

KINGS CHEM GUIDE: SECOND EDITION

KINGS CHEMISTRY GUIDE™

A book designed to teach beginner, intermediate, and advanced high school students and first year college chemistry students, hobbyists, enthusiasts, and amateurs about the basic fundamentals of general chemistry

By Jared Ledgard

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Chapter 1: Introduction to Chemistry: A quick lesson in general chemistry

1a. Introduction to chemistry

Introduction to chemistry and the elements

What is chemistry? The answer is rather simple and may surprise you. Chemistry is the art of dealing with, altering, and changing molecules from one form to the next. However, this process of change tends to confuse people at first, as this confusion is primarily due to the fact that the acts of altering or changing molecules from one form to the next have many rules, and utilize unfamiliar formulas, numbers, mathematics and equations, letters and abbreviations, and chemical apparatus and processes. Despite all the rules involved in chemistry and such, chemistry is much easier to learn than most would think. As previously mentioned, chemistry is the art of altering or changing molecules from one form to the next. This art is not confined strictly to the laboratory, and the art of chemistry has been mastered by the natural world for billions of years. As you live your life, you come into contact with chemistry every second of every day. Atoms and molecules and virtually thousands of chemical reactions, chemicals, and processes stare you in the face at any given moment. Unfortunately, few people ever sit back and ponder on this world that exists all around them. For example, as you read this text, you're staring at chemical compounds. The chemicals that make up this paper, the chemicals that make up the ink of the text you're reading, and even the chemicals that make up the air you breathe, you're engaged in an arsenal of chemical compounds and chemical reactions. In another example, your body alone is a huge biochemical factory where millions of chemical reactions take place every single day. In other examples, the geology of the earth dates back 4 billion years and has witnessed trillions of chemical reactions from volcanic eruptions, erosion, oceans and lakes, the atmosphere, to advanced ecosystems. This entire world is chemistry based and is composed of elements, chemical compounds, and chemical reactions of all kinds.

So what can we say about all this? Chemistry involves every corner stone of life, and its foundations are small particles called atoms, otherwise known as elements. These elements make up all the various chemical compounds that in turn, make up the entire earth, solar system, and universe. If you could just ponder for two minutes on the chemistry that exists all around you, you would be astonished, and that's just the room your body mass occupies at this exact moment. Now, before I get carried away and start talking about totally confusing and highly advanced subjects, let's begin to understand the basics of chemistry first.

First things first, let's take a look at the periodic table of elements. What is the periodic table of the elements? Well, this famous chart outlines the basic particles of all matter as known everywhere, meaning that the elements listed in the chart not only exist on earth, but make up the entire universe. All the elements on this chart have been arranged perfectly and exist as their individual physical properties dictate.

Periodic Table of the Elements

The periodic table displays elements in a grid. Each element's cell contains its atomic number (top left), symbol (center), and name (bottom). Elements are grouped into columns (groups) and rows (periods). The table includes elements from Hydrogen (1) to Oganesson (118), plus additional elements like Actinides and Lanthanides shown in separate rows at the bottom.

The periodic table of the elements.

Notice on the above image of the periodic table how the elements are represented by single letters or double letters. Don't get confused about the elements that are represented by single letters and those represented by double letters. There is nothing special about the elements represented by single letters or double letters and it's solely arbitrary. Notice how each element is arranged in columns and rows. This does have significant meaning, as each column or row of elements have similarities to them, meaning the elements in each column or row for example, have similar physical properties. These similar physical properties can be vast in definition, so they will not be discussed here.

In many cases, the letter representing the corresponding element matches the first letter or so of the name of the element. For example, the element oxygen is represented by the letter "O". Also, notice how letters "Al" represent the element Aluminum; however, this representation is not true for all elements. For example, the element Tungsten is represented by the letter "W", and the element Tin is represented by the letters "Sn".

Familiarization with atoms

As you have noticed, on the periodic table, the elements are listed in rows and columns, and these rows and columns are placed as such because atoms share things in common, and hence, they fit into a particular row or column. So what exactly are atoms. An atom is one of the smallest units of mass known to man, and makes-up all matter on earth, the solar system, and the universe. Each atom is composed of a nucleus, and an outer sphere. In the nucleus of an atom, there exists two defined particles of mass. One particle is called a "proton", and the other particle is called a "neutron". Protons and neutrons have nearly identical weights, but the proton is positively charged, and the neutron is neutral with no charge. The sphere, or atmosphere of the nucleus of an atom is occupied by electrons. Electrons have negative charges, and hence orbit the nucleus of an atom because of the positively charged protons. The size of electrons are very tiny, even despite their strong negative charges, making them the smallest units of mass known to man. In atoms, the electrons spin and orbit around the nucleus of an atom like satellites orbiting the earth. However, free flowing electrons make-up the electromagnetic spectrum, and concentrated flows of electrons can be seen as electric arcs such as a bolt of lightning. Electricity flowing through a metal wire, is composed of electrons moving from atom to atom like a flying airplane. When we glance at the periodic table again. The atomic number of each element tells you how many protons it has. For example, the element Boron has an atomic number of 5, meaning it contains 5 protons. Each atom has the same number of protons as neutrons, so if Boron has 5 protons, it has five neutrons as well. You can also simply divide the atomic mass of any given element in half, and that gives you the number of neutrons and protons. The atomic number also represents how many electrons an atom has. For example, the element Aluminum has an atomic number of 13, and hence contains 13 electrons per nucleus. To take this a bit further, there exist atoms called "isotopes". Isotopes are atoms that contain unbalanced numbers of protons and neutrons. For example, Tritium is an isotope of

hydrogen, and contains two neutrons but only one proton and one electron. Some isotopes occur in nature, but most are made synthetically utilizing nuclear reactions.

Introduction to the four main categories of chemistry

Now that your familiar with the periodic table and the elements, its time to understand the four main categories of chemistry. Over the last few centuries, scientists have discovered that the chemistry of this amazing universe fits into four main categories. These categories include a) Inorganic chemistry, b) Organic chemistry, c) Biochemistry, and d) Nuclear chemistry. Inorganic chemistry is the chemistry of simple and basic compounds, most of which take up about 98% of the earth by weight, and about 99.8% of the entire universe. Inorganic compounds are simple non-carbon based compounds ranging from common rocks, to salts such as rock salt, Epsom salt, and simple acids such as nitric acid or sulfuric acid. Organic chemistry deals with all compounds that contain centralized carbon, and all other carbon based compounds. Biochemistry deals with the special chemical reactions seen in life, such as DNA/RNA, peptides, and amino acid sequences, and nuclear chemistry deals with the chemistry of atoms on an atomic level, rather than on a chemical one. Currently, there are about 180,000 inorganic compounds known to man, and about 26 million organic compounds. The number of biochemical compounds is not known, but may be estimated at around 1 million. Note: Biochemistry deals primarily with bioorganic processes and chemical reactions, many of which utilize both inorganic and organic compounds. Nuclear chemistry deals mainly with radioactive atoms and their corresponding compounds.

A few examples of atoms (elements) would be the following (note: the chemistry of the elements fits into the category of inorganic chemistry):

1. Sulfur
2. Tin
3. Lead
4. Chlorine
5. Potassium
6. Chromium
7. Iron and Steel
8. Zinc
9. Copper
10. Gold
11. Silver
12. Platinum
13. Arsenic
14. Beryllium
15. Boron
16. Magnesium
17. Cobalt
18. Carbon

A few examples of inorganic compounds and materials would be the following:

1. Rock salt (sodium chloride)
2. Epsom salt (magnesium sulfate heptahydrate)
3. Calcite mineral
4. Sulfuric acid
5. Bleach
6. Baking soda (sodium hydrogen carbonate)
7. Potassium chloride
8. Boric acid
9. Potassium nitrate
10. Sedimentary rock
11. Metamorphic rock
12. Pyrite (Iron sulfide)
13. Galena (Lead sulfide)
14. Cinnabar (Mercury sulfide)
15. Chlorine dioxide
16. Nitrogen dioxide
17. Sulfur dioxide
18. Hydrogen peroxide
19. Ammonia

A few examples of organic compounds and materials would be the following:

1. Benzene
2. Aspirin
3. TNT
4. Nitroglycerine
5. Vanillin
6. Vitamin B12
7. PVC
8. Cellulose
9. Nerve gas
10. Oil
11. Black pepper
12. Capsaicin (hot sauce)
13. Beef
14. Styrofoam
15. Methamphetamine
16. Paper
17. Wood
18. Citric acid
19. Coal
20. Charcoal
21. Chloroform
22. Xanax
23. Cocaine
24. Malathion
25. Nicotine
26. Vodka (solution of ethyl alcohol in water)
27. Sugar
28. Solvents

A few examples of bioorganic compounds and materials include the following:

1. DNA
2. RNA
3. Peptides
4. Proteins
5. Lipoproteins
6. G-proteins
7. Poly peptides
8. Amino acids
9. Viruses
10. Enzymes
11. Bacteria
12. Skin cells
13. Tissue
14. Finger nails
15. Hair
16. Multifunctional cytokines
17. Ribonucleoproteins
18. Hormones
19. Insulin

A few examples of nuclear materials would be the following:

1. Uranium
2. Plutonium
3. Radon
4. Deuterium
5. Tritium
6. Iodine¹²⁴
7. Lead²⁰³

8. Gold¹⁷⁷
9. Phosphorus³⁰
10. Thorium
11. Polonium

Basic physical properties of elements, inorganic compounds and materials, organic compounds and materials, bioorganic compounds and materials, and nuclear materials:

1. **Water/solvent solubility:** All elements known to man are insoluble in water and most known solvents; however, some elements such as oxygen, chlorine, bromine, and iodine are very slightly soluble in water, but the halogens show moderate solubility in certain organic solvents, and sulfur is soluble in carbon disulfide. Most inorganic compounds are insoluble in water and most solvents, but some are soluble in water forming solutions, otherwise known as electrolytes. Most organic compounds are insoluble in water, but a select few are somewhat soluble in water and only a few are miscible with water forming solutions. Many bioorganic compounds and materials are soluble in water and many solvents. Practically all nuclear (radioactive) elements are insoluble in water and all known solvents; however, some radioactive compounds and materials are somewhat soluble in water, but relatively insoluble in solvents.
2. **Melting and boiling points:** Many elements have relatively high melting points and boiling points, but a select few have low melting points and boiling points, and the elements of mercury and gallium have very low melting points and low boiling points. The gaseous elements have extremely low melting points and relatively low boiling points. Many inorganic compounds have relatively high melting points and boiling points. Some inorganic compounds have low melting points, but high boiling points. Many organic compounds have relatively low melting points and boiling points. Most bioorganic compounds and materials decompose when heated so their melting and boiling points cannot be determined. Most radioactive elements as with non-radioactive elements have high melting points and high boiling points, but some radioactive compounds have moderate melting points with high boiling points.
3. **Flammability/combustibility:** Under normal conditions all elements with the exception of sulfur are non-flammable; however most powdered elements will burn in air when ignited with a flame. Almost all inorganic compounds have no flammability or combustibility; however, a select few inorganic compounds such as the nitrates, chlorates, perchlorates, dichromates, and permanganates will burn when mixed with a combustible material and ignited using a flame, i.e., potassium nitrate mixed with sulfur. Many organic compounds are flammable or have some degree of combustibility, and some organic compounds and materials are highly flammable. Most bioorganic compounds and materials are non-flammable, and decompose when heated. All radioactive elements under normal conditions are non-flammable, but some radioactive elements when in powdered form will burn in air when ignited with a flame. Some radioactive compounds such as the nitrates, chlorates, perchlorates, dichromates, and permanganates will burn when mixed with a combustible substance, i.e., Uranyl nitrate mixed with charcoal and/or sulfur and ignited using a flame.

1b. The nature of Chemical reactions: Chemical bonding

Now that you have an idea about elements, and the four major categories of chemistry and a few of their compounds, its time to discuss the foundation to chemistry, and that foundation is the nature of “chemical reactions”. What is a chemical reaction, and why does a chemical reaction take place? Chemical reactions take place because of many different reasons; however, a few of those reasons will be discussed here. As you have seen, the four main categories of chemistry are inorganic chemistry, organic chemistry, biochemistry, and nuclear chemistry; however, we will only discuss the nature of inorganic and organic reactions that take place.

In the nature of chemical reactions we will only deal with inorganic and organic reactions and we will not discuss the reactions of bioorganic compounds or nuclear reactions as these are too complex and complicated for this book. Inorganic compounds and organic compounds undergo chemical reactions based on two types of “bonding”. This bonding utilizes two major types of chemical bonding, 1) ionic bonding, and 2) covalent bonding. Remember these two types of bonding, as they are very very important in understanding the nature of chemical reactions.

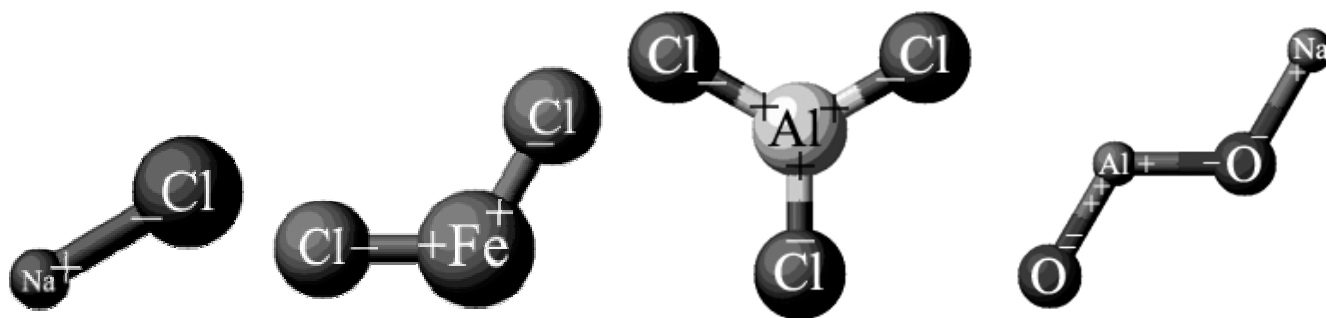
Ionic bonding

Ionic bonding is the simplest form of chemical interaction. In an ionic compound, the atoms within the molecule are held together by positive and negative charges. These charges are what form ionic compounds. If we look at sodium chloride for example, the ionic bond is between the positive sodium atom, and the negative chlorine atom. So why is the sodium positive and the chlorine negative? This is answered by discussing electro negativities of the various atoms, meaning each atom or element, possesses its own unique electro negativity. The element sodium has a really low electro negativity, whereby the element chlorine has a rather high electro negativity; therefore, the element sodium possesses positive energy, and the chlorine atom possesses negative energy. In essence, because the chlorine possesses negative energies, and the sodium possesses positive energies, when the two atoms, or elements come in contact with one another, they chemical react, and form an ionic

bond, i.e., the chlorine swipes one of the sodium's electrons to fulfill its electrical demands. When an atom loses an electron from its nucleus, it becomes positively charged. At the same time, on the other end, when an atom gains an electron from a weaker atom, it in-itself becomes negatively charged. Lets look at the following table:

Atom (element)	Electro negativity
Lithium	1.00
Sodium	1.00
Magnesium	1.20
Calcium	1.0
Titanium	1.30
Iron	1.60
Copper	1.80
Aluminum	1.50
Lead	1.60
Nitrogen	3.10
Oxygen	3.50
Sulfur	2.40
Fluorine	4.10
Chlorine	2.8
Iodine	2.2

You will notice by looking at the table that the element sodium possesses an electro negativity of 1.0, and the element chlorine posses an electro negativity of 2.8. Because the difference between the electro negativities is greater then 1.5, the compound sodium chloride is ionic. Lets look at a few examples of ionic compounds.

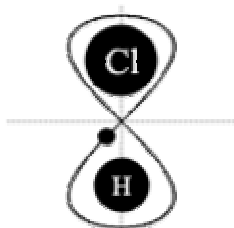


Far left molecule: sodium chloride; second from the left molecule: Ferrous chloride; second from the right molecule: Aluminum chloride; and far right molecule: sodium aluminate. Notice how the number of positives equals the number of negatives.

As previously mentioned, when sodium and chlorine react with each other, the chlorine swipes one of the sodium's electrons, thus making sodium positive and chlorine negative. The reason this happens is because the element sodium has a weak electrical charge. Because sodium has such a weak charge, and the chlorine as a more powerful electrical charge, the sodium must give up an electron to the chlorine. If this sounds confusing, think about two magnets, and how two magnets stick together when exposed to opposite poles. When two magnets are placed side-by-side with identical poles, they repel each other. Ionic compounds work in the same manner. Obviously identical charges would repel each other, therefore, when ionic compounds are formed, their positive and negative charges attract each other, but in order for this to happen, each atom must either gain or give-up electrons. As briefly mentioned before, when an atom gives-up electrons, it becomes positive, and when an atom gains electrons, it becomes negative. Therefore, in every single case, when one atom gives-up an electron, another atom gains it simultaneously. As noticed in the above illustration atoms can give-up and gain more then one electron. Notice the second from the right molecule, i.e., aluminum chloride. Aluminum is capable of giving up three electrons, and hence becoming a positively charged atom with a positive charge of 3. At the same time, you need three negative charges to balance things out, and hence, the aluminum bonds with three chlorine atoms, each chlorine atom has a negative charge. In this case, each chlorine atom only swipes one of the aluminums three available electrons. So why does the chlorine atom only swipe one electron, and why does aluminum give-up three electrons. This is a good question and will be discussed later.

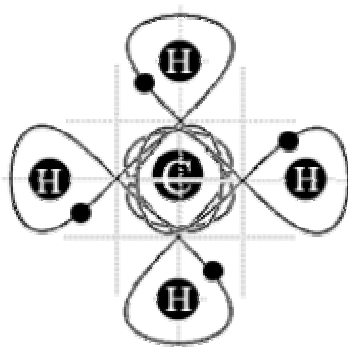
Covalent bonding

As you have learned, ionic compounds form their bonds by utilizing differences in electro negativities. Covalent compounds on the other hand, form their bonds by sharing electrons rather than taking them, or giving them up. So what exactly does this mean? Well, in sharing, the electrons orbit around each atom of the molecule that makes up the bond. If this sounds confusing, lets study the following illustration.



Covalent bonding structure of hydrogen chloride. As with covalent bonding the electrons form figure eight patterns around each other. This is how the atoms in the molecule share their electrons.

As seen in the above illustration, the electrons in the molecule orbit around both atoms using a figure eight type orbital pattern. This is the foundation to covalent bonding. Now lets look at a more difficult covalently bonded system in the following illustration.

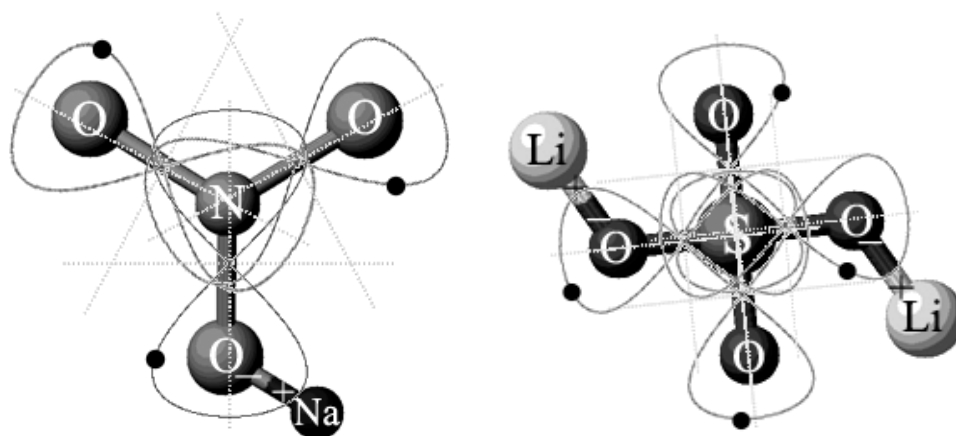


In this illustration we see the molecule methane, where four hydrogen atoms are covalently bonded to a central carbon atom whereby the electrons use figure eight orbital patterns. This is another great example of the sharing of electrons that takes place in covalent bonding.

In covalent bonding, the difference between electro negativities of the atoms usually does not exceed 1.50. What this means is that when atoms with differences in electro negativities are less 1.50, the bonding is said to be covalent. For example, when we see the above structure of hydrogen chloride, the hydrogen has an electro negativity of 2.2, and the chlorine atom has an electro negativity of 2.8, making the difference between the two electro negativities 0.60. Because this difference of 0.60 is less then 1.50, the molecule is covalent. The bonding in the majority of organic compounds is covalent in nature.

Dual bonding

In some cases, molecules will be composed of both ionic, and covalent bonding. Not to confuse you, but remember that if the difference between electro negativities between two atoms is less then 1.50, the bonding is covalent, and if the difference is greater then 1.50, the bonding is predominantly ionic. Therefore, molecules will often have dual bonding, composed of both ionic and covalent bonds. For a better example, lets look at the following illustrations:

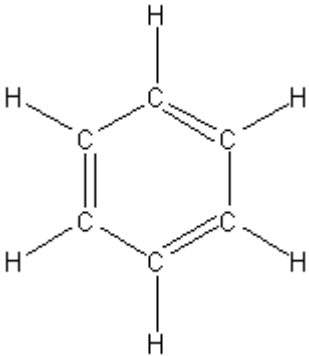
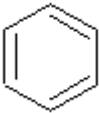
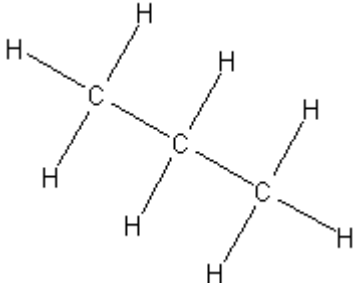
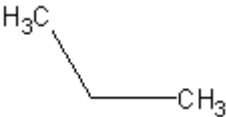
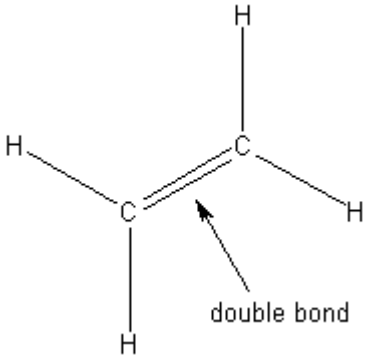
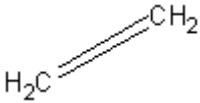


In the left illustration the molecule sodium nitrate contains both an ionic bond between the oxygen atom and the sodium atom, and three covalent bonds between the central nitrogen atom and the three adjacent oxygen atoms. In the right illustration we see the molecule lithium sulfate, with two ionic bonds, each from the lithium atom to a single oxygen, and we see four covalent bonds between the central sulfur atom, and the four adjacent oxygen atoms.

1c. Understanding chemical bonding: Chemical structures and formulas

Before we discuss the oxidation states of the various elements, we need to take a quick lesson into chemical structures and formulas. In chemistry, we use chemical structures and formulas to explain what is taking place during a chemical reaction, and to explain to the reader what chemical is being talked about. When writing structures and formulas, we use letters to represent the atoms involved, and we use lines to represent the bonding between the various atoms involved. Therefore, let's get familiarized with the chemical structures and their formulas by looking at the following table:

Expanded chemical structure	Condensed chemical structure	Condensed structural formula
<p>4 hydrogen atoms</p> <p>single carbon atom</p>		CH ₄
<p><i>The expanded structure of methane. Notice how four hydrogen atoms are covalently bonded to a central carbon atom.</i></p>	<p><i>The condensed chemical structure of methane.</i></p>	<p><i>Condensed structural formula</i></p>
<p>3 chlorine atoms</p> <p>single phosphorus atom</p>		PCl ₃
<p><i>The expanded structure of phosphorus trichloride.</i></p>	<p><i>The condensed chemical structure of phosphorus trichloride.</i></p>	<p><i>Condensed structural formula</i></p>

 <p>6 carbon ring, containing 6 carbon atoms 6 hydrogen atoms, each one bonded to a single carbon atom</p>		C_6H_6
<i>The expanded structure of benzene. Notice how every other carbon is double bonded to the next one.</i>	<i>Condensed chemical structure of benzene</i>	<i>Condensed structural formula</i>
 <p>3 carbon chain, 8 hydrogen atoms</p>		C_3H_8
<i>The expanded structure of propane. Each hydrogen bonded to a single carbon atom.</i>	<i>Condensed chemical structure of propane</i>	<i>Condensed structural formula</i>
 <p>2 carbon chain; 6 hydrogen atoms</p>		C_2H_4
<i>The expanded structure of Ethene. Notice the double bond between the two carbon atoms. Each hydrogen is bonded to a single carbon atom.</i>	<i>Condensed chemical structure of Ethene</i>	<i>Condensed structural formula</i>

1d. Understanding chemical bonding: Oxidation states and the number of bonds atoms form


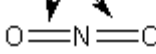
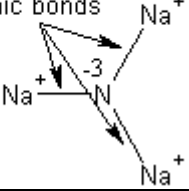
Now that you have a rough-cut idea about atoms, chemical bonding, and structure, its time to understand why chemical reactions take place in the first place. Each atom has what are called oxidation states. Oxidation states are closely related to the number of bonds an individual atom can form. These oxidation states dictate the atoms within a molecule, and defines the what, when, how, and why they chemically react with and why the reactions take place between atoms and molecules in the first place. As briefly mentioned in section 1b under ionic bonding, aluminum likes to give up three electrons. So why is this the case? The answer is oxidation states. Aluminum has an oxidation state of +3, meaning when it reacts with other elements or

when it reacts with molecules, it always involves three electrons in the reaction. In other words, three electrons will always be given up if the aluminum is involved in a chemical reaction involving ionic compounds, or it will always share three electrons if involved in a chemical reaction involving covalent compounds. The oxidation state of aluminum will always be +3 and nothing will change that. Therefore, let's get familiarized with the oxidation states of the most common elements. Note: Some elements have only one oxidation state, and some have more than one oxidation state.

Element	Oxidation State(s)	Number of bonds formed
Hydrogen (H)	+1, -1	1
Sodium (Na)	+1	1
Magnesium (Mg)	+2	2
Calcium (Ca)	+2	2
Iron (Fe)	+2, +3	2 and 3
Copper (Cu)	+1, +2	1 and 2
Aluminum (Al)	+3	3
Carbon (C)	+2, -4, +4	2, and 4
Nitrogen (N)	+1, -1, +2, -2, +3, -3, +4, +5	1, 2, 3, 4, and 5
Oxygen (O)	-2	2
Phosphorus (P)	+3, -3, +5	3 and 5
Sulfur (S)	-2, +4, +6	2, 4, and 6
Chlorine (Cl)	+1, -1, +5, +7	1, 5, and 7
Iodine (I)	+1, -1, +5, +7	1, 5, and 7

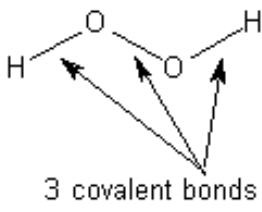
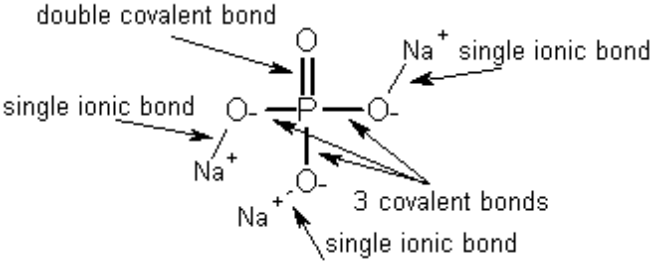
Now, understanding the oxidation states of the various elements can be confusing, but what it really comes down to is simple memorization. However, we could go into much greater detail to why the elements form their oxidation states, but it would require some pretty advanced information, which will not be discussed in this book. In the meantime however, let's focus on several elements, such as Nitrogen, Oxygen, and Chlorine. When you zoom in on these elements, you will notice the various oxidation states that they have, and the corresponding number of bonds they can form. Remember that the number of oxidation states in each element is closely related to the number of bonds the element can form; therefore, let's focus on the following tables for examples:

The Element Nitrogen

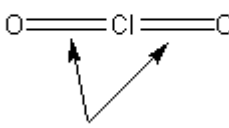
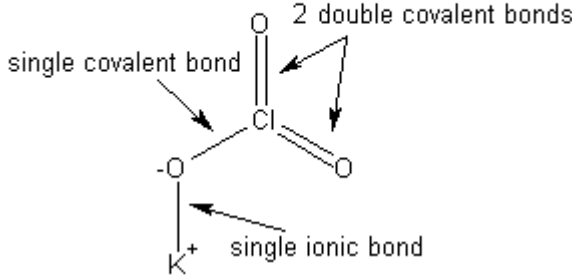
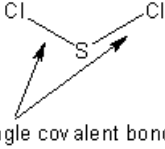
Structure example	Structure example	Structure example
<p>NITRIC OXIDE</p> <p>1 double covalent bond</p> 	<p>NITROGEN DIOXIDE</p> <p>2 double covalent bonds</p> 	<p>SODIUM NITRIDE</p> <p>3 single ionic bonds</p> 
<p>1. Nitrogen is in the +2 oxidation state</p> <p>2. Oxygen is in the -2 oxidation state</p>	<p>1. Nitrogen is in the +4 oxidation state</p> <p>2. Oxygen is in the -2 oxidation state</p>	<p>1. Nitrogen is in the -3 oxidation state</p> <p>2. Sodium is in +1 oxidation state</p>
<p>Because the nitrogen is in the +2 oxidation state, and its electro negativity is only slightly smaller than the oxygen, it forms a double covalent bond with the oxygen, which is in the -2 oxidation state. In other words, nitrogen and oxygen both form 2 bonds. Note how the positive and negative oxidation charges are in relation to the number of total bonds formed, i.e., +2 and -2. Don't confuse these charges with ionic bonding</p>	<p>Because the nitrogen is in the +4 oxidation state, and its electro negativity is only slightly smaller than the oxygen, it forms two double covalent bonds with the oxygens, each of which have -2 oxidation state. In other words, the nitrogen forms 4 bonds, and the two oxygen atoms both form 2 bonds. Note how the positive and negative oxidation charges are in relation to the number of total bonds formed, i.e., +4 and -4. Don't confuse</p>	<p>In this case, the nitrogen is in the -3 oxidation state because its electro negativity is greater than 1.5 over the sodium. Therefore, its electrical charges are in exact relation to one another, i.e., +3 and -3. Each sodium atom is bonded to the negatively charged nitrogen atom using ionic bonding.</p>

	these charges with ionic bonding.	
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The element oxygen

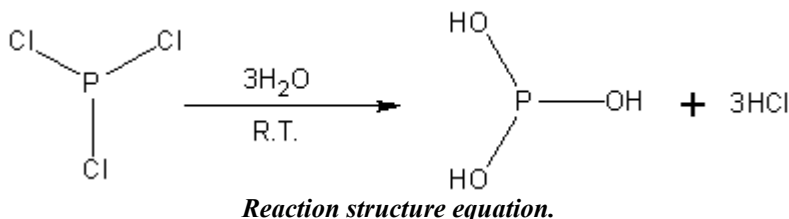
Structure example	Structure example
<p>HYDROGEN PEROXIDE</p>  <p>3 covalent bonds</p>	<p>TRISODIUM PHOSPHATE</p>  <p>double covalent bond single ionic bond Na⁺ single ionic bond 3 covalent bonds single ionic bond Na⁺</p>
<p>1. Oxygen is in the -2 oxidation state 2. Hydrogen is in $+1$ oxidation state</p>	<p>1. Oxygen is in the -2 oxidation state 2. Phosphorus is in the $+5$ oxidation state 3. Sodium is in $+1$ oxidation state</p>
<p>In this example, the oxygen has a -2 oxidation state, and the hydrogens have a $+1$ oxidation state each. Because the difference in electro negativities is less than 1.5, the bonds are covalent. In this example, the two oxygen atoms form 2 bonds each, and the hydrogen forms 1 bond each. Notice how the two oxygen atoms share a single covalent bond.</p>	<p>In this example, we have three different atoms bonded in the same molecule. We see a central phosphorus atom, which is in a $+5$ oxidation state. Because its electro negativity is less than 1.5 as compared to the oxygen atoms, it forms 5 bonds. To do so, it forms a double covalent bond with a single oxygen and three single covalent bonds between three other oxygen atoms. On the other end of these three oxygen atoms, we have three single ionic bonds formed to three sodium atoms as the result of the sodium atoms difference in electro negativity, being more than 1.5.</p>

The element chlorine

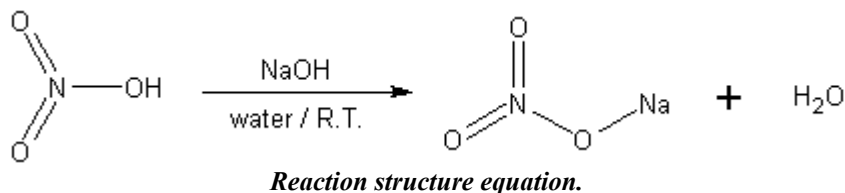
Structure example	Structure example	Structure example
<p>CHLORINE DIOXIDE</p>  <p>2 double covalent bonds</p>	<p>POTASSIUM CHLORATE</p>  <p>2 double covalent bonds single covalent bond single ionic bond K⁺</p>	<p>SULFUR CHLORIDE</p>  <p>2 single covalent bonds</p>
<p>1. Chlorine is in $+4$ oxidation state 2. Oxygen is in the -2 oxidation state</p>	<p>1. Chlorine is in the $+5$ oxidation state 2. Oxygen is in the -2 oxidation state 3. Potassium is in the $+1$ oxidation state</p>	<p>1. Chlorine is in the -1 oxidation state 2. Sulfur is in the $+2$ oxidation state</p>
<p>In this example, the chlorine is in the $+4$ oxidation state therefore it forms 4 bonds. In doing so, it forms 2 double covalent bonds with two oxygen atoms, both of which have a -2 oxidation state, and hence, forming 2 bonds each. Notice how the number of bonds balances out, i.e., 4 bonds to 4 bonds total.</p>	<p>In this example, the chlorine is in a $+5$ oxidation state, therefore, it forms 5 bonds. In doing so, it forms two double covalent bonds with two oxygen atoms, and a single covalent bond with a third oxygen atom. This third oxygen in turn, satisfies its 2 bond needs by forming an ionic bond between a potassium atom because its difference in electro negativity is greater than 1.5.</p>	<p>In this simple example, two chlorine atoms are forming 1 bond each, so in doing so, they are bonded to a single sulfur atom, which requires 2 bonds because of its $+2$ oxidation state.</p>

1e. Understanding chemical bonding: Chemical reactions

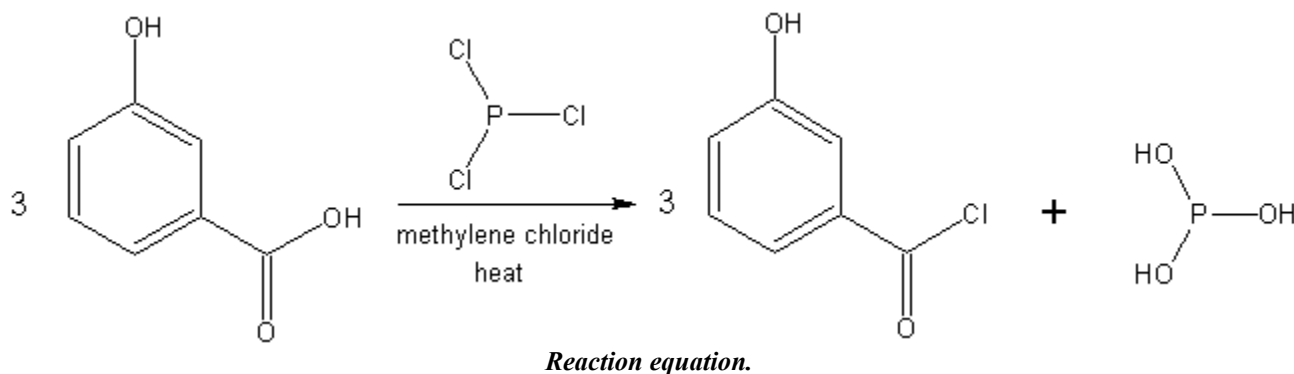
Understanding chemical reactions is very important in the field of chemistry. To better understand chemical reactions you must apply what you have learned so far, i.e., chemical structures and formulas, and oxidation states. Therefore, to better understand chemical reactions, let's look at the following examples.



In the above example, we see phosphorus trichloride written on the left, and then we see an arrow pointing towards the right. Above the arrow is water, and below the arrow is R.T., which represents reaction temperature. It should be noted that the reactant(s) are always placed on the left hand side of the arrow, and an arrow is then written pointing to the right. The reaction products are always written on right hand side of the arrow, and as we see above, we have the products phosphorous acid, and hydrogen chloride. Reaction conditions, and reagents are always written above and below the arrow in the reaction equation. It should be also noted, that the reaction equation must be balanced in order for it to make sense. In the above reaction equation, on the left hand side, and the reactant water we have 1 phosphorus atom, 3 chlorine atoms, 6 hydrogen atoms, and 3 oxygen atoms ($3\text{H}_2\text{O} = 3 \times 2$ hydrogen atoms + 3×1 oxygen atoms); therefore, to balance out the equation on the right-hand side, we have 1 phosphorus atom, 6 hydrogen atoms, 3 oxygen atoms, and 3 chlorine atoms, identical to the left hand side and the above arrow reactant. Now let's look at another example:



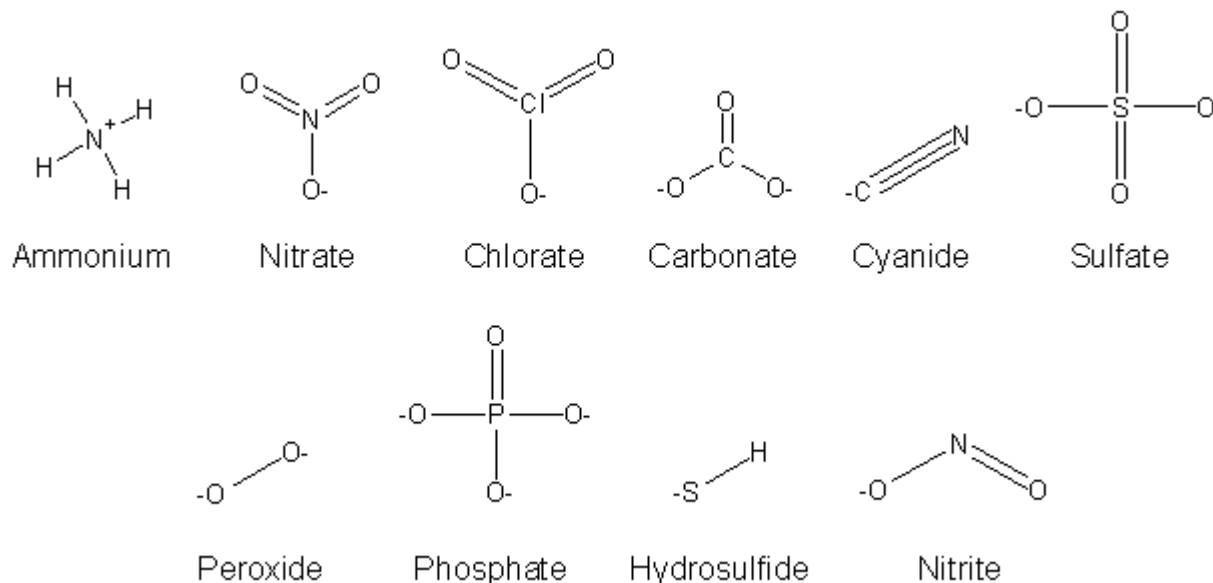
In the above reaction equation, we see nitric acid on left hand side, and then we see the reactant sodium hydroxide (NaOH) above the center arrow. Below the arrow, we see water as a non-reactant, and R.T., for room temperature. On the right-hand side of the reaction equation, we see the products of sodium nitrate, and water as a by-product. As you will notice, the reaction equation is balanced, i.e., 1 nitrogen atom, 4 oxygen atoms, 2 hydrogen atoms, and 1 sodium atom. Now, let's focus on a more complicated reaction.



In the above reaction equation, we see the compound 3-hydroxybenzoic acid on the left hand side, and we see the reactant phosphorus trichloride above the central arrow. Below the arrow, we see the reaction conditions stating the solvent methylene chloride is used and the reaction mixture is heated to a general heat. On the right hand side of the equation we see the products 3-hydroxybenzoyl chloride and the by-product phosphorous acid.

If. Understanding chemical bonding and reactions: Functional groups

Now that you have an understanding of general chemical structure, oxidation states and the like, its time to explore what are called functional groups. Understanding functional groups in chemistry will better help you understand why certain chemical reactions take place. So what are functional groups? Functional groups are classes or groups of chemical compounds that have similarities in physical properties and chemical structure. Lets look at the following table for general functional groups in Inorganic chemistry:



In this illustration we see some inorganic functional groups.

An ion, is a functional group of atoms and molecules that posses either a positive or negative charge. In the above illustration, we see the Ammonium ion (NH_4^+), we see the nitrate ion (NO_3^-), we see the chlorate ion (ClO_3^-), we see the carbonate ion (CO_3^{2-}) Note: carbonates contain carbon, but are not classified as organic compounds because of their degree of ionic bonding; however, organic carbonates do exist as well, we now see the cyanide ion (CN^-), we see the sulfate ion (SO_4^{2-}), we see the peroxide ion (O_2^{2-}), we see the classic phosphate ion (PO_4^{3-}), we see the hydrosulfide ion (HS^-), and finally we see the nitrite ion (NO_2^-).

Now lets look at some classic inorganic compounds and their condensed structures. Notice their functional groups and how they have similarities in chemical structure.

Ammonium ion NH_4^+	Nitrate ion NO_3^-	Chlorate ion ClO_3^-	Cyanide ion CN^-	Sulfate ion SO_4^{2-}	Phosphate ion PO_4^{3-}
Ammonium chloride (NH_4Cl)	Potassium nitrate (KNO_3)	Sodium chlorate (NaClO_3)	Lithium cyanide (LiCN)	Sodium sulfate (Na_2SO_4)	Trisodium phosphate (Na_3PO_4)
Ammonium carbonate ($[(\text{NH}_4)_2\text{CO}_3]$)	Magnesium nitrate ($[\text{Mg}(\text{NO}_3)_2]$)	Potassium chlorate (KClO_3)	Potassium cyanide (KCN)	Magnesium sulfate (MgSO_4)	Calcium phosphate tribasic ($[\text{Ca}_3(\text{PO}_4)_2]$)
Ammonium Iodide (NH_4I)	Copper nitrate ($[\text{Cu}(\text{NO}_3)_2]$)	Ferric chlorate ($[\text{Fe}(\text{ClO}_3)_3]$)	Calcium cyanide ($[\text{Ca}(\text{CN})_2]$)	Zirconium sulfate ($[\text{Zr}(\text{SO}_4)_2]$)	Aluminum phosphate tribasic (AlPO_4)
Ammonium nitrate (NH_4NO_3)	Aluminum nitrate ($[\text{Al}(\text{NO}_3)_3]$)	Lead chlorate ($[\text{Pb}(\text{ClO}_3)_2]$)	Zinc cyanide ($[\text{Zn}(\text{CN})_2]$)	Lead sulfate (PbSO_4)	Nickel phosphate tribasic ($[\text{Ni}_3(\text{PO}_4)_2]$)

Now let's look at some examples of functional groups in organic chemistry. Unlike inorganic chemistry however, there are many many functional groups in organic chemistry, so we will only discuss the most common ones here.

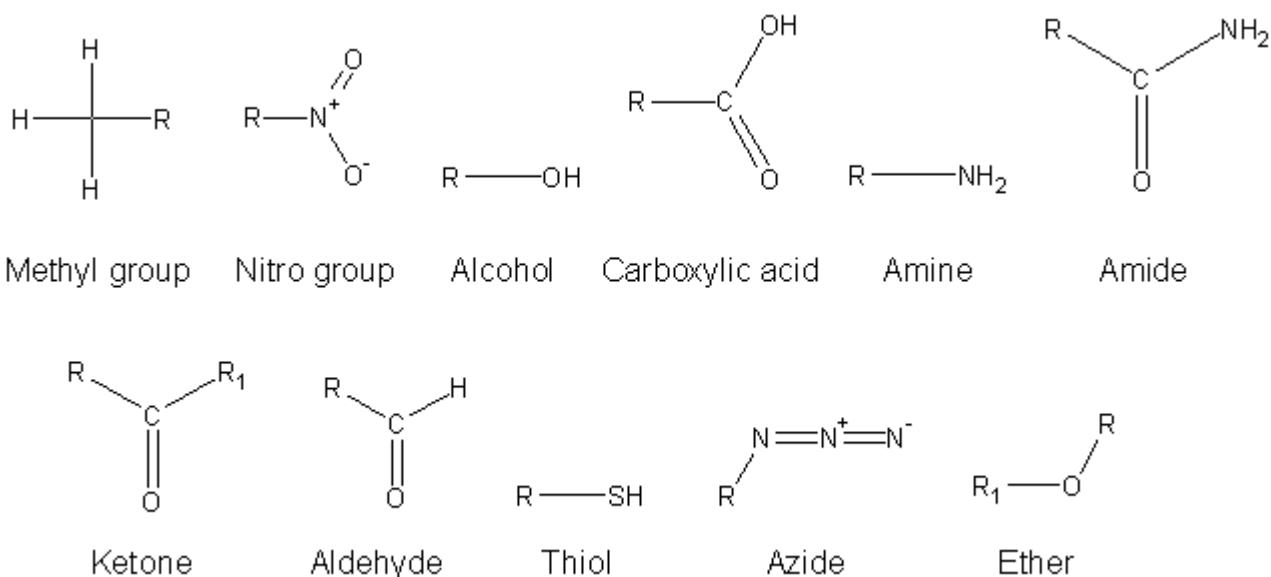


Illustration of some common functional groups in organic chemistry.

The following tables will now emphasize some common organic compounds and their specific functional groups. Notice the patterns involved in their names and structure:

Table 1:

Nitro functional group	Alcohol functional group	Carboxylic acid functional group	Amine functional group
<p>Nitropropane</p>	<p>Ethyl alcohol</p>	<p>Acetic acid</p>	<p>Isopropyl amine</p>
<p>Nitrobenzene</p>	<p>Propylene glycol</p>	<p>Propionic acid</p>	<p>Diethylamine</p>
<p>TNT (Trinitrotoluene)</p>	<p>Phenol</p>	<p>Benzoic acid</p>	<p>Aniline</p>

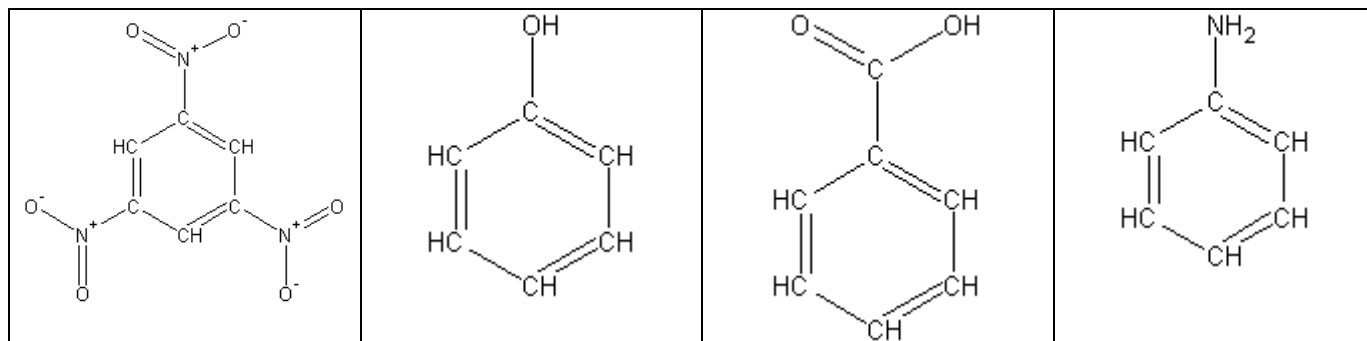


Table 2:

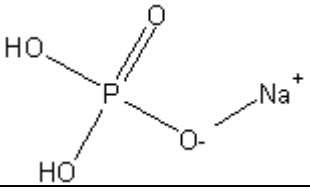
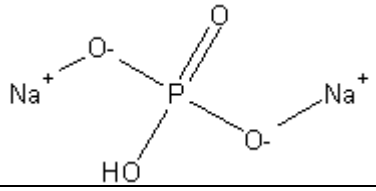
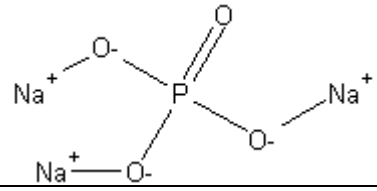
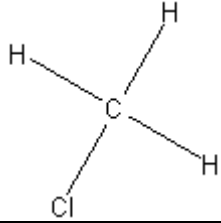
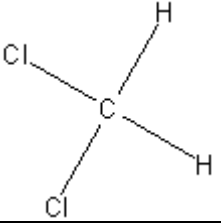
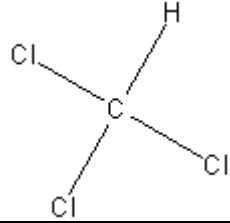
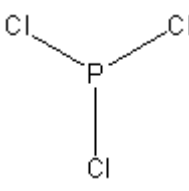
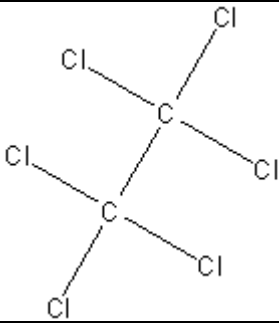
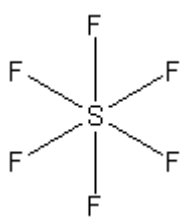
Ketone functional group	Aldehyde functional group	Ether functional group
<p>Acetone</p>	<p>Formaldehyde</p>	<p>Methyl ether</p>
<p>Methyl ethyl ketone</p>	<p>Acetaldehyde</p>	<p>Ethyl ether</p>
<p>Acetophenone</p>	<p>Benzaldehyde</p>	<p>Tetrahydrofuran</p>

1g. Understanding the language of chemistry

Chemistry has a unique language all its own. This language of chemistry was developed by a foundation called the IUPAC system, which stands for International Union of Pure and Applied Chemistry. This organization is an independent agency, which exists sole for the naming of chemical compounds, and providing the language of chemistry. The language of chemistry can be quit complicated, so we will stick to the rater simple attributes, rather then engage into anything to confusing. First, lets get familiar with the IUPAC system of numbering. In the following table you will see the numbers 1 through 10

1	2	3	4	5	6	7	8	9	10
mono	di	tri	tetra	penta	hexa	hepta	octa	nona	deca

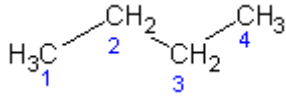
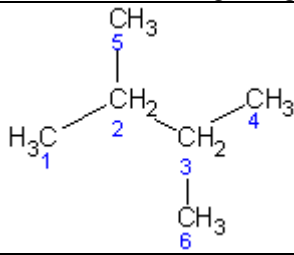
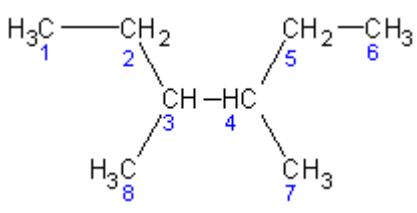
Now, when we apply these numbers to various compounds we get the following examples:

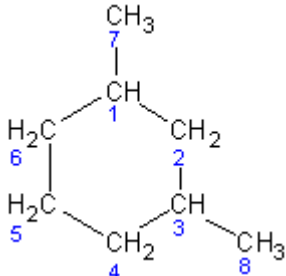
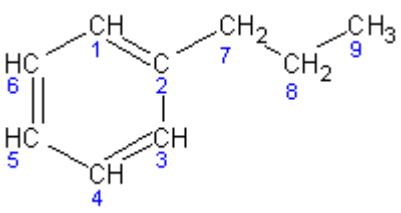
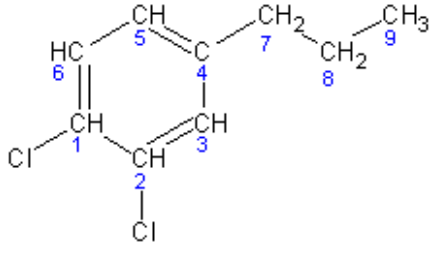
		
"Mono"sodium phosphate	"Di"sodium phosphate	"Tri"sodium phosphate
		
"Mono"chloromethane	"Di"chloromethane	"Tri"chloromethane
		
Phosphorus "tri"chloride	"Hexa"chloroethane	Sulfur "hexa"fluoride

In the naming of organic compounds, we start by numbering the compounds organic chain. All organic compounds have carbon chains, and the number of carbons in the chain represents the particular name for that compound. For example, when we look at simple carbon chains we see the following:

Methyl	Ethyl	Propyl	Butyl	Pentyl
CH ₃	CH ₃ CH ₃	CH ₃ CH ₂ CH ₃	CH ₃ CH ₂ CH ₂ CH ₃	CH ₃ (CH ₂) ₃ CH ₃
Methane	Ethane	Propane	Butane	Pentane
Hexyl	Heptyl	Octyl	Nonyl	Decyl
CH ₃ (CH ₂) ₄ CH ₃	CH ₃ (CH ₂) ₅ CH ₃	CH ₃ (CH ₂) ₆ CH ₃	CH ₃ (CH ₂) ₇ CH ₃	CH ₃ (CH ₂) ₈ CH ₃
Hexane	Heptane	Octane	Nonane	Decane

Now lets see some examples of how numbering is used in the naming of organic compounds.

		
Butane	2,3-dimethyl butane	3,4-dimethyl hexane

		
1,3-dimethyl cyclohexane	2-propyl benzene	1,2-dichloro-4-propylbenzene

1h. Conversion factors

Understanding conversion factors is not important for this book, as each metric unit is given in English units; however, it never hurts to explore the differences in the metric and English measurements and units. The following table will help emphasize how to convert English to metric, and vice versa.

To convert	Into	Multiply By	To convert	Into	Multiply By
Atmospheres	Cm of mercury	76	Liters	Gallons	0.2642
Atmospheres	Mm of mercury	760	Liters	Ounces (fluid)	33.814
Atmospheres	Torrs	760	Meters	Feet	3.281
Atmospheres	In of mercury	29.92	Meters	Inches	39.37
Atmospheres	psi	14.7	Milligrams	Ounces	3.527×10^{-5}
Celsius	Fahrenheit	$1.8 + 32$	Milligrams	Pounds	2.2046×10^{-6}
Centimeters	Inches	0.3937	Milliliters	Gallons	2.642×10^{-4}
Centimeters	Meters	0.01	Milliliters	Ounces (fluid)	0.0338
Centimeters of mercury	Atmospheres	0.01316	Millimeters	Feet	3.281×10^{-3}
Centimeters of mercury	psi	0.1934	Millimeters	Inches	0.03937
Fahrenheit	Celsius	$0.556 - 17.8$	Ounces	Grams	28.349527
Feet	Meters	0.3048	Ounces	Kilograms	0.0283
Feet	Millimeters	304.8	Ounces	Milligrams	28,349.5
Gallons	Liters	3.785	Pints (liquid)	Liters	0.4732
Gallons	Milliliters	3,785	Pints (liquid)	Milliliters	473.2
Grams	Ounces	0.03527	Pounds	Grams	453.5924
Inches	Centimeters	2.540	Pounds	Kilograms	0.4536
Inches	Millimeters	25.40	psi	Atmospheres	0.06804
Inches of mercury	Atmospheres	0.03342	Quarts (liquid)	Liters	0.9464
Inches of mercury	psi	0.4912	Quarts (liquid)	Milliliters	946.4
Kilograms	Ounces	35.274	Torr	Mm of mercury	1.0
Kilograms	Pounds	2.205	Torr	Atmospheres	1.316×10^{-3}

Chapter 2:

Familiarization with

Laboratory Techniques

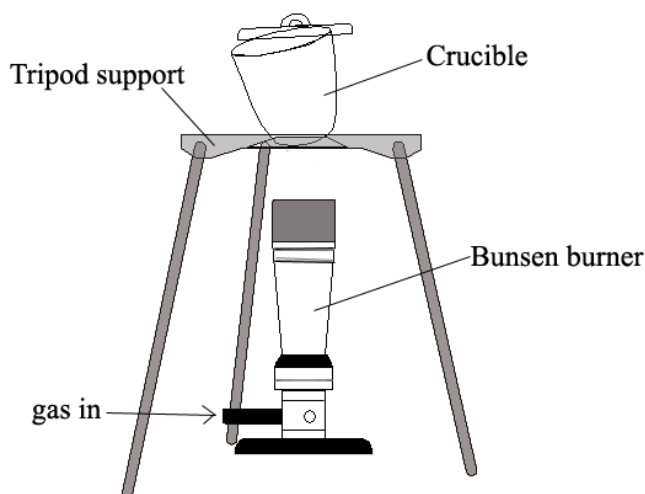
2a: General Laboratory Techniques: Methods of heating

Heating is one of the most widely utilized tools in the general lab. Heat is used to stimulate a chemical reaction, boil a reaction mixture or solution, or to drive-off liquids as seen in distillation. The most important forms of heating in the general lab are discussed in the following:

1) Free flame

Free flames refer to Bunsen burners or alcohol lamps. Both utilize an open flame and can be used for general heating purposes. The pros of using free flames are that solutions and chemicals can be heated rapidly; however, the drawbacks to free flames are flammable liquids and their vapors. Heating combustible liquids with a free flame can be dangerous, as looming vapors can ignite. Other drawbacks to using free flames are that they don't often heat mixtures or chemicals evenly, and the free flames can over-heat certain chemicals leading to dangerous situations such as dangerous decompositions resulting in toxic fumes, or just plain decomposition of the desired product.

In using free flame devices such as Bunsen burners or alcohol lamps, the fuel is relatively cheap, owing to another pro for using free flames. When using Bunsen burners, simple natural gas or propane or even butane can be used. In the lab, natural gas outlets are readily available, and for home and school use, simple store bought propane tanks or butane tanks can be used as the fuel source.

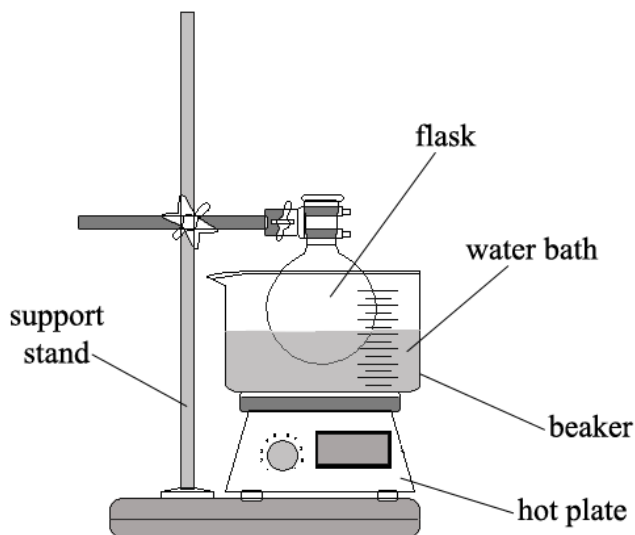


Common laboratory Bunsen burner with support stand.

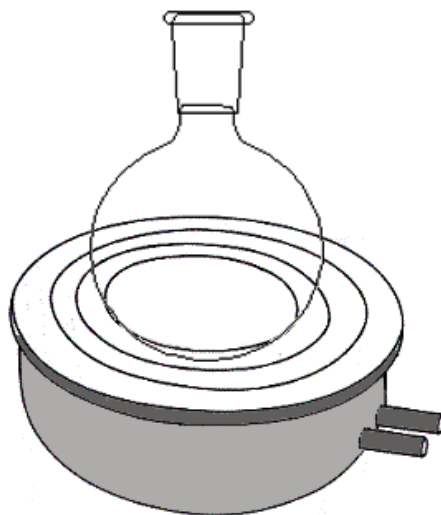
2) Steam bath, or water bath

Steam baths, or water baths are used primarily to heat reaction vessels such as flasks to temperatures ranging from 50 to 125 Celsius. The beneficial aspect of steam baths are that they provide even heating without the dangers of forming blind spots. Blind spots occur when there is uneven heating. Blind spots can cause decomposition of a chemical compound, or can lead to

unwanted chemical reactions due to poor and uneven heating. Another advantage steam baths or water baths have, is they eliminate the risk of igniting flammable fumes or the like. Steam baths, or water baths can be made using beakers or other heatable containers. The only draw back to steam baths or water baths is that they cannot heat to temperatures higher than 125 Celsius.



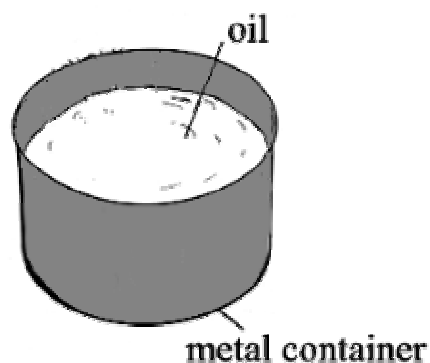
A classic setup for water bath heating. The hot plate is used to heat the water. Note: Notice how the flask is suspended in the water bath, and is not touching the bottom of the beaker.



A common steam bath. A steam bath is composed of a water container, and a heating element (not illustrated). The heating element boils the water, and the steam, is what actually heats the vessel. The vessel rests upon a system of rings that can be removed to accommodate larger vessels.

3) Oil bath

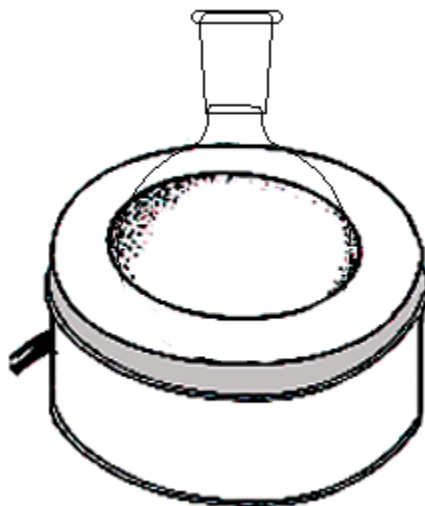
Oil baths are used in an identical manner as water baths, with the water simply being replaced by a non-volatile oil, such as a cooking oil, or crude oil compound. Oil baths are very useful for heating liquids and reaction mixtures to temperatures ranging from 125 to 350 Celsius. The only drawbacks to oil baths are they are slow to heat and cool, and they leave a greasy film on the flask being used, making it an annoyance for clean up.



A typical container for heating a flask with oil. The flask should be immersed only 1/3 of the way into the oil. Note: glass containers for holding the oil should be avoided.

4) Electric Heating Mantles

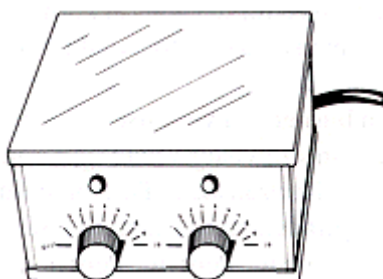
Heating mantles are very common for the use of heating flasks and other apparatus. Heating mantles are just like hot plates, except their shaped circular to accommodate round bottom flasks, which they are predominantly designed for. Heating mantles are very simple to operate, and come with variable temperature controllers. Heating mantles can be used to heat round bottom flasks and apparatus ranging from 50 milliliters up to 12 liters in capacity.



A typical heating mantle.

5) Hot Plates

Hot plates are by far the most common tool for heating in the general lab, and they are used extensively in heating flasks and beakers, and other flat bottom glassware or containers. Hot plates work in the same manner as heating mantles, except hot plates contain a flat smooth heating surface, usually composed of aluminum. Hot plates come in many different sizes, and some come with magnetic stirring devices built in.



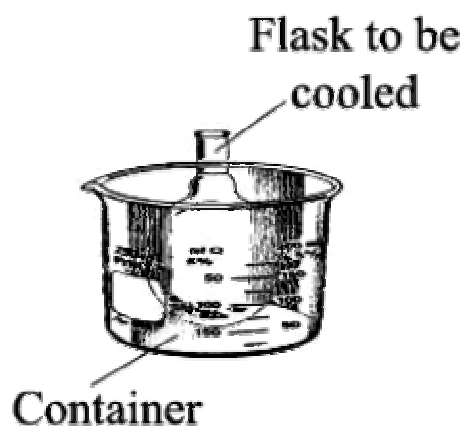
A typical hot plate. Most hot plates come doubled as hot plates and magnetic stirrers.

2b. Methods of Cooling

As you will find in many chemical reactions, heat is often evolved; therefore, methods of cooling must be applied in order to maintain proper reaction conditions. Excessive heat build up during chemical reactions can lead to dangerous conditions including violent decomposition of a chemical or mixture, the evolution of harmful fumes or vapors, or the decomposition or loss of your desired product

When forming a method of cooling, one utilizes a large outer container, from which the cooling agent is then placed there into. For example, ice is the most common cooling agent, so when its used to form an ice bath, the bath, or container is usually three to four times the volume of your flask or reaction apparatus. If for example, your using a 1-liter flask to contain your chemicals or your reaction mixture, a 3-liter to 4-liter container should be used to house this 1-liter flask. Note: before adding the ice or cooling agent, make sure the flask or apparatus to be cooled is situated and seated inside the cooling vessel/container, as adding the ice first, will make it difficult to place the flask there into properly. Then when filling the ice bath with ice or other cooling agent, make sure to fill the ice bath all the way up. In short, if your using a 1-liter flask, it should be perfectly situated in a 3 to 4 liter cooling bath, and the ice or cooling agent, when placed there into, should submerge your 1-liter flask by at least 2/3 of the way up. In other words, 80% of the total height of your 1-liter flask should be totally covered or submerged in ice.

Note: In some cases your desired flask to be cooled may actually displace your cooling media (cold water, or ice water) before hand, or as the cooling media such as ice melts causing the flask to float and possible tip over. To alleviate this potential, Lead rings which fit around the necks of the flasks can be used to weight them down and keep them from tipping over.

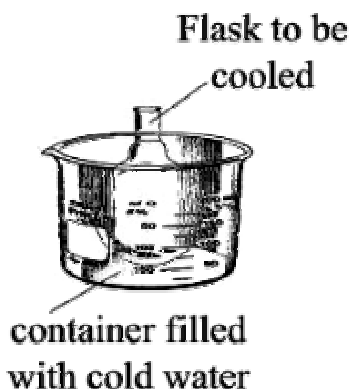


A typical setup for cooling a flask or reaction mixture. The container is filled with cooling agent.

Now, the five most common methods of cooling a flask or reaction apparatus will be discussed in the following:

1) Cold water bath

In some cases, cooling a flask can be done by the use of simple old-fashioned cold water. Cold-water baths are used to cool and maintain the flasks temperature from 15 to 60 Celsius. Note in some cases, during the chemical reaction or during the cooling process, the water will have to be quickly removed, and then replaced with fresh cold water, in order to maintain the proper temperature.



2) Ice water bath

Ice water baths are used in the same circumstances as plain old cold-water baths; however, they are generally used when the flask temperature is to be kept below 50 Celsius. In forming ice water baths, the water to ice by volume should be about 50/50. Ice water baths are generally used to cool the flask to 15 to 40 Celsius.

3) Standard ice bath

Ice baths are the most common method for cooling flasks during reactions and other operations. Ice baths are generally used when cooling the flask to 0 to 25 Celsius is desired. For best results, the ice should be pulverized to allow for maximum surface area; unfortunately, a drawback to doing this can result in faster melting of the ice; therefore, small cubes of ice about half inch by half inch should be used to maximize surface area and allow the ice to adhere to the walls of the flask. Always remember to place your flask into the cooling container before adding the ice. During the cooling process, the ice will have to be replaced periodically, and this can be a pain. Note: for long term cooling and storage of a flask not involved in a chemical reaction, you can place the ice bath into a freezer for prolonged periods.

4) Salt/ice bath

The salt/ice bath is a modified version of the standard ice bath, and utilizes both ice and salt. In doing so, a cooling temperature of -10 to 5 Celsius can be maintained for short to moderate periods of time. Salt/ice baths can be easily prepared by simply placing the ice into your cooling container, after your desired flask has been placed, and then filling the container with crushed ice in the usual manner as for preparing an ice bath; thereafter, finely grained salt is added to the ice by sprinkling it over the ice. The ratio of ice to salt by weight should be about 90:10, meaning 90% ice, and 10% salt. As with standard ice baths, the cooling media may have to be replaced periodically, and this can be a pain. The most common salt used in salt/ice baths is common table salt (sodium chloride), but other salts can be used; for example, the salts of potassium chloride, magnesium chloride, and calcium chloride can be used as well. These salts generally produce cooling temperatures from -30 to 5 Celsius for short to moderate cooling periods.

5) Dry ice/acetone bath

Dry ice baths are very common in the modern lab, and can be used to achieve cooling temperatures of -70 to -10 Celsius. Because dry ice is so volatile, meaning it evaporates readily when exposed to air, the dry ice is often slurried with a solvent such as acetone, which preserves its cooling power for longer durations of time. As dry ice is readily available and inexpensive, it makes for a perfect cooling media. The drawbacks, however, are the fact that it may over cool your flask, causing the contents therein to solidify or freeze. Therefore, dry ice/acetone slurries are commonly used where low temperatures in general are required, such as maintaining a very low boiling liquid in its liquid state. Acetone isn't the only solvent that can be used, and other liquids, such as ethanol, ethyl acetate, or ether can be used. To create a dry ice/acetone bath, firmly secure your flask into the cooling container as with previous methods, and then pour in acetone (or other solvent) to the point where the cooling container is filled about half way. Thereafter, add in pulverized chunks of dry ice to completely surround the flask to be cooled and maximize surface area. The ratio of solvent to dry ice ranges from 50:50 (acetone to dry ice) for moderate to long cooling durations, to 70:30 (acetone to dry ice), for short term cooling operations. The advantage of using dry ice/solvent slurries is that the dry ice is readily replaced once it evaporates during the operation.

Cooling tricks of the trade

In some cases you will be able to cool your flask by the use of a refrigerator and/or freezer. For long periods of time, a refrigerator/freezer can be used with great success. However, it is not wise to use a refrigerator for cooling if a chemical reaction is taking place in your flask, or if there are gases being evolved from your flask.

2c. Extraction

The extraction process is a very crucial step in purifying and collecting a specific product. During the extraction process a special liquid, or solvent is chosen and used, and is mixed with the substance to be extracted. The substance to be extracted can be a reaction mixture, liquid, oil, plant, seed, or any desired substance or mixture that contains the desired product to be purified. To carry out an extraction, the chosen solvent is broken down into several portions, and then each portion is shaken vigorously with the substance to be extracted for several minutes. During the shaking process, what takes place is that the desired product to be purified moves from the substance to be extracted and dissolves in the solvent portion. Once the extraction process is complete, the solvent portions, also called extracts are combined, and then evaporated to leave behind the extracted product. The chosen solvent is based on its insolubility in the substance to be extracted, meaning the chosen solvent

must not be soluble in the substance to be extracted; however, the product to be purified should have a relatively good solubility in the chosen solvent. Now we will look at the general steps for extracting a reaction mixture or liquid.

1) The right funnel and size must be chosen first

The size of the separatory funnel is crucial, and must be chosen carefully before performing an extraction. A separatory funnel is a piece of laboratory glassware that is shaped like a pear. The following illustration shows what a common separatory funnel looks like. Now, during the extraction process, there must be enough room in the separatory funnel in order to allow the contents to shake during the extraction process. In essence, the separatory funnel should not be filled more than $\frac{2}{3}$ of the total volume. In a specific example, if we use a 250-milliliter separatory funnel for extracting 100 milliliters of a reaction mixture, we would use a 50 milliliter portion of chosen solvent. If you have to extract a large volume of reaction mixture or liquid, and you don't have a proper sized separatory funnel, you can simply divide the total volume of the reaction mixture or liquid into smaller portions, and then do the same for the chosen solvent to be used.



A standard laboratory separatory funnel.

2a) Performing the extraction of a liquid mixture

The first thing to do in an extraction process is to choose the solvent. Once the solvent has been chosen, it should be divided into three portions of equal volume. Thereafter, pour the mixture to be extracted into the separatory funnel, and then add in one of the three solvent portions. After the solvent portion has been added to the separatory funnel, a two-phase mixture will result. In some cases the solvent portion will be the upper layer, and in some cases it will be the lower layer. If the density of the chosen solvent is less than the mixture to be extracted, then it will be the upper layer, and if its density is greater than the mixture to be extracted, it will be the lower layer.

Now, shake the separatory funnel vigorously for about 1 to 2 minutes, and then allow the separatory funnel to stand for several moments to allow the two-phase mixture to fully separate. Thereafter, drain-off the lower layer and save it. If the lower layer is the chosen solvent portion, then set it aside for the moment. If however, the chosen solvent is less dense than the mixture to be extracted it will be the upper layer so you will have to drain off the bottom layer first, and then pour it back into the same separatory funnel after you have recovered the upper solvent portion. In any sense, when the solvent portion has been collected, repeat the process with the remaining two solvent portions. Once the extraction process has been fully completed, the three solvent portions, also called extracts are combined, dried, and then evaporated to recover the dissolved desired product. This latter process will be discussed in greater detail under drying solvent mixtures, recrystallization and distillation. Note: Sometimes, when performing an extraction process, such as a water solution of sodium hydroxide and chloroform for example, an emulsion forms making it very difficult to properly carry out the extraction process. Emulsification cannot always be anticipated, so the solvent must be chosen wisely.

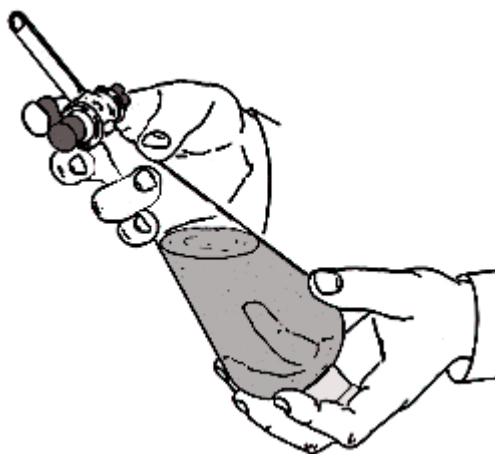
2b) Extracting a solid substance such as plant, root, seed, ect.,

Extracting a solid substance such as a plant for example, takes on a different approach. To extract a solid substance, you should use a typical blender. Place the solid substance to be extracted into the blender, and then add in the entire chosen solvent. Thereafter, blend the entire mixture on low speed for a good 10 to 15 minutes. Thereafter, filter the entire mixture to collect the

liquid portion. Once the liquid portion has been collected, the mixture is dried, and then evaporated to recover the desired product. This latter process will be discussed in greater detail under drying solvent mixtures, recrystallization and distillation.

3) How to properly vent the separatory funnel after the shaking

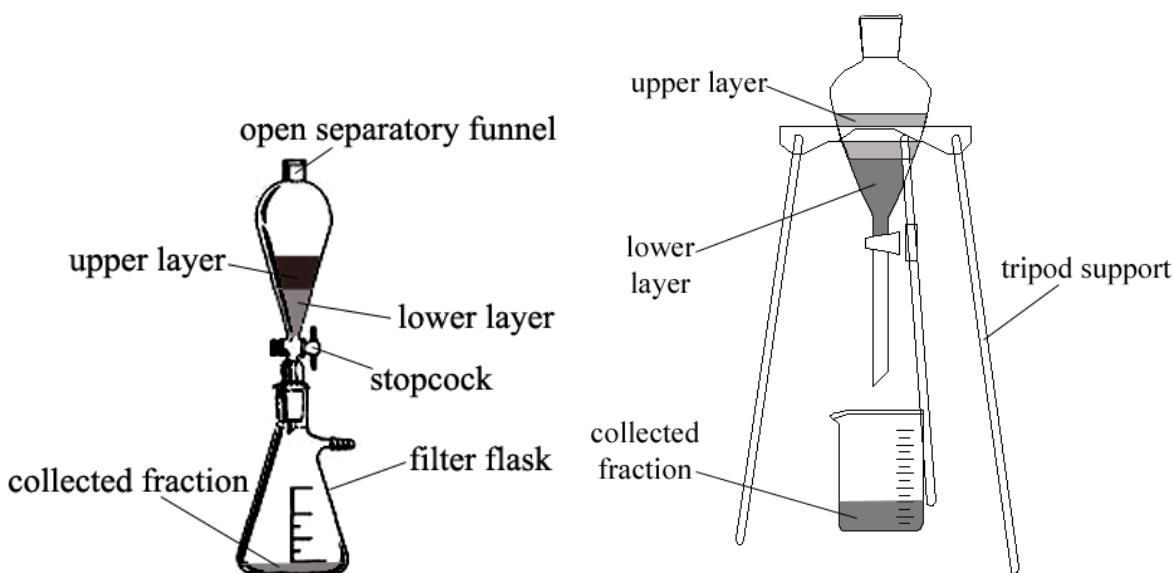
While shaking the funnel, vapors from the reaction mixture or solvent can increase pressures inside the separatory funnel. Therefore, proper venting of the separatory funnel is necessary in order to prevent pressure from pushing the contents out of the funnel. In order to properly vent a separatory funnel, rest the funnel in one hand while grasping the glass stopper. Thereafter, tilt the separatory funnel so that the stopcock is pointed up and away from you and others. Thereafter, rotate the stopcock until it is parallel to the separatory funnel, and in the open position. Note: make sure the level of the liquid is below the opening of the separatory funnel so no liquid is pushed out while opening.



Proper method of venting the separatory funnel.

4) How to properly drain the funnel

After vigorously shaking the funnel, the two-phase mixture must be separated. In order to properly do this, place the separatory funnel onto a single neck flask, or use a ring support stand, and then remove the separatory funnels stopper. Then open the stopcock very slowly, so only a slow flow of liquid can be seen. Note: if you open stopcock before removing the separatory funnels stopper, it will become very difficult to proceed, so make sure to remove the separatory funnels stopper beforehand. While draining the bottom layer of the two-phase mixture, bring the flow of the draining liquid to a drip as the boundary of the two-phase liquid approaches the stopcock. This needs to be done in order to prevent parts of the upper layer from draining out.



Classic setups for draining the separatory funnel after the shaking period.

Working example for extracting a liquid chemical reaction mixture:

1. Choose your separatory funnel and make sure it is the right size
2. Choose your solvent wisely, and then divide it into three equal portions by volume
3. Pour the reaction mixture to be extracted into the separatory funnel (make sure the funnel's stopcock is closed) making sure it doesn't occupy more than 2/3 of the total volume of the separatory funnel.
4. Pour in the first of the chosen solvent portions into the separatory funnel, and then stopper the funnel to seal it water tight.
5. Vigorously shake the funnel using two hands for about 1 to 2 minutes.
6. Vent the separatory funnel as described earlier.
7. Hold the funnel upright for a few moments to allow the two-phase mixture to fully separate.
8. Drain-off the bottom solvent portion and then save it. If however, the solvent portion is the upper layer, drain-off the bottom reaction mixture layer, save it for the moment, and then drain-off the solvent portion and set it aside for the moment. Then pour the previously drained-off reaction mixture layer back into the same separatory funnel.
9. Repeat steps 4 through 7 two more times using a fresh solvent portion each time.
10. After the third extraction process has been complete, combine all three solvent portions. What to do next will be discussed later. In the meantime, at this point you have carried out a successful extraction process.

Note: In some extraction process, the liquid mixture to be extracted will be dark in nature, making it difficult to see where the two-phase boundary is. If this happens simply hold the separatory funnel up to a bright light, and then mark the phase boundary with a piece of tape.

Working example on extracting a solid substance such as a plant, seed, etc:

1. Place the solid substance into a mortar and pestle, or any desired means and pulverize or ground the solid substance to increase its surface area.
2. Setup a standard kitchen blender, and then add in the pulverized solid substance.
3. Then choose a proper solvent, and then add all of the solvent into the blender.
4. Turn on the blender and blend the mixture on low speed for about 15 to 20 minutes at room temperature.
5. Thereafter, turn off the blender, and then filter the entire mixture to recover the liquid portion and to separate out the solid insoluble material.

Specialty extracting process: "Salting Out"

What is salting out? Salting out is a unique process whereby a salt is added to a liquid mixture in order to force a dissolved product to separate out. In most salting out processes, the desired product to be separated is usually a liquid, and the mixture from which it is to be separated from is usually a mixture containing water, otherwise known as aqueous solutions. To carry out a salting out process, an excess of a salt, usually sodium chloride, sodium sulfate, or magnesium sulfate is added to the aqueous mixture, and the resulting mixture is then vigorously shaken or stirred for about 5 minutes. Upon standing of the resulting mixture, a two-phase mixture will result, which can then be separated using a separatory funnel, or drained off if a separatory funnel was already in use. Now, let's use an example to better familiarize you with the salting out process.

1. Into a common separatory funnel or beaker, place the aqueous mixture to be salted out. In this case, we use rubbing alcohol, which is an aqueous solution of isopropyl alcohol and water.
2. Add in an excess of sodium chloride, say about 1 part salt per 15 parts of aqueous solution.
3. Then shake the mixture vigorously for about 5 minutes if using a separatory funnel, or vigorously stir the mixture for about 5 minutes if you're using a beaker.
4. Then allow the mixture to stand for about 5 minutes to allow a two-phase mixture to form.
5. Then pour the mixture into a separatory funnel, if you used a beaker, and then drain-off the bottom aqueous layer. The upper layer of the two-phase mixture will be composed of relatively straight isopropyl alcohol.

2d. Filtration

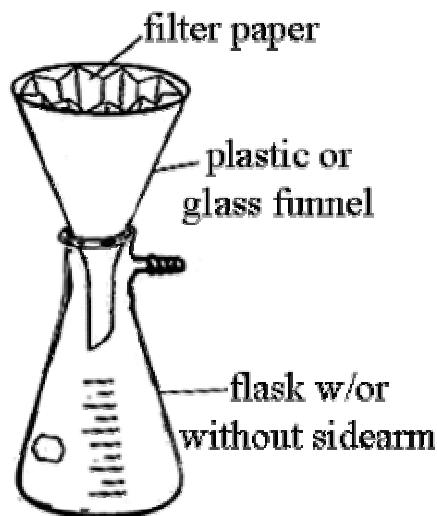
Introduction

Filtration is a rather simple process and involves two different forms, gravity filtration and vacuum filtration. In either case, filtration is a necessary tool used in chemistry and many other applications to recover insoluble or precipitated matter or products. Filtration utilizes a filter media, or also called filter paper, where the insoluble or precipitated matter or products collect on. Filter paper is specially designed with micro small holes so that insoluble matter or products collect, but liquids and solutions are allowed to pass through. Note: Many people use filtration in their daily lives, such as when you brew up a cup

of coffee, you place the coffee into a filter, and then hot water passes there through; in essence, the act of preparing a cup of coffee is an extraction process, and a filtration process in one.

1) Gravity filtration

Gravity filtration is the oldest form of filtration and requires a filter, which utilizes gravity to pull the liquid through the micro small holes of the filter paper. Gravity filtration is the most common form of filtration in general labs, but its use should be limited due to the severe slowness of the filtration, especially when trying to filter very fine precipitated matter. When filtering very fine precipitated matter, or also called particles, the gravity filtration process can take hours even days. To carryout a gravity filtration process, the liquid to be filtered is poured into the plastic or glass funnel, and then you have to wait for all the liquid to drain through the filter, leaving the insoluble material collected on the filter paper. Once the insoluble material has been collected, and the liquid has thoroughly passed through the filter, you can collect the insoluble mass from the filter paper. Note: the insoluble mass on the filter paper will often be moist with the liquid that you filtered, and this can be a pain to deal with, so you will need to wash the collected material, see section 2f to learn about this.



Classic setup for gravity filtration.

Instead of using gravity filtration to recover a precipitated product, it is sometimes used to purify liquids and mixtures by the use of a filter aid. A filter aid is an insoluble solid material that is placed inside the filter paper prior to use. To use, the desired mixture or solution to be purified is simply poured into the filter and allowed to drain and collect into the flask. The filter aid is designed to absorb impurities from the mixture or solution, or in most cases a reaction mixture. The filter aid ranges from granulated charcoal, activated carbon, silicon dioxide, silica gel, sand, zeolites, diatomaceous earth, or even aluminum oxide.

Fluting Filter Paper for use in gravity filtration

When carrying out gravity filtration, glass or plastic cones or funnels are often used. In this regards, filter paper, which is often sold in circular form must be fluted and formed to fit properly in the glass or plastic cone or funnel. Before you flute your filter paper, the size of the circular filter paper to be used should be at least 2 times the diameter of the top of the cone or funnel your using for the filtration. Note: In gravity filtrations, fluted filter paper is often superior to other forms of filtering paper because the “flutedness” of the filter paper allows for better airflow between the glass or plastic cone or funnel wall, and the fluted filter paper. To flute standard circular filter paper:

1. Take three circular sheets of filter paper, lay them on top of each other, and then fold them in half.



2. Next, take this half, and fold it into quarters, i.e, fold this portion into half.



3. Fold this portion into eighths, i.e, fold this halved portion into half's.



4. Open the halved filter paper up, to the original half-folded position.



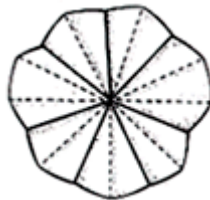
5. Take the opened filter paper, and fold to the eighth line.



6. Then fold to the quarter line.

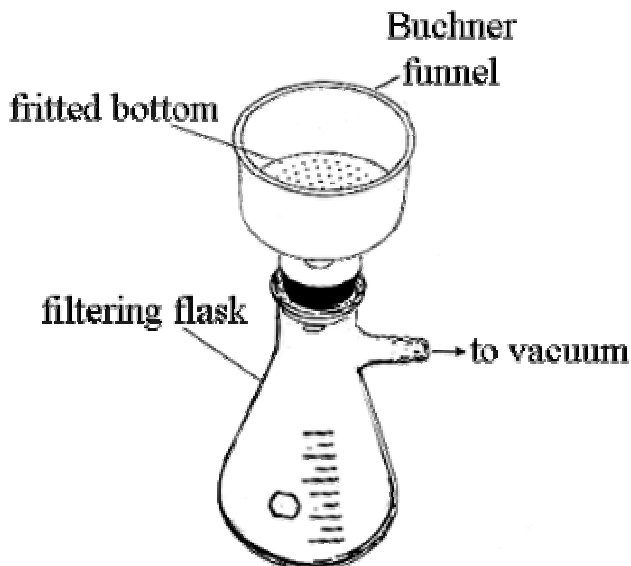


7. Finally, this is what the paper will look like when it is fully opened.



2) Vacuum Filtration

Vacuum filtration is by far the most widely used and most important form of filtration, and its use in the modern lab is practically a must have and indispensable tool. Vacuum filtration is a process where a vacuum is applied to the filtering flask in order to pull the solution being filtered through the filter at a much faster rate. Unlike gravity filtration, vacuum filtration is very quick and virtually spontaneous in its rate of filtration, as the suction of the vacuum quickly pulls the liquid through the filter. The most common apparatus for use in vacuum filtration is a piece of equipment called a “Buchner” funnel, named after a famous chemist, and a vacuum source. A Buchner funnel is composed of a specially designed porcelain, plastic, or glass funnel, that contains a fritted flat bottom filled with tiny holes, as illustrated in the following illustration. The vacuum source can be either a standard vacuum pump, which are readily available, or a hand operated mechanical pump. Note: Buchner funnels often come in two pieces, the upper piece is the funnel and fritted bottom, and the bottom piece is usually the stem, and rubber stopper. To carryout a vacuum filtration process, simply pour the liquid you wish to filter into the Buchner funnel, and allow the vacuum to suck all the liquid through the filter. Once the filtration process is complete, you can simply vacuum dry your filtered-off mass, see section 2f for vacuum drying techniques.



Classic setup for vacuum filtration utilizing a Buchner funnel and a filtering flask. The Buchner funnel can either be plastic, porcelain, or glass.

Unlike gravity filtration, the filter paper used in vacuum filtration has a diameter only slightly smaller than the diameter of the Buchner funnel. This filter paper is placed on the inside of the Buchner funnel above the fritted bottom.

2e. Recrystallization, and solid product recovery

Recrystallization

Recrystallization is the process of separating a dissolved solid product from a solvent solution. A solvent solution could be a previous extract from a reaction mixture, plant, etc., or a direct reaction mixture itself, or any type of solution containing a dissolved solid there into. A solution is what you get when a solid is dissolved into a liquid. For example, if we dissolved table salt into a glass of tap water, we would get a saline solution of sodium chloride in water. Now, in order to carry out a recrystallization we use three major methods of recovering the dissolved solid product, and these will be discussed below:

1) General recrystallization utilizing heat only

General recrystallization is a crucial tool used in chemistry to purify a non-heat sensitive solid product. Recrystallization sometimes follows the extraction process, which was briefly explained earlier. Unlike the extraction process however, recrystallization involves removing the chosen solvent to recover the dissolved product there into. In recrystallization, the idea is to remove the chosen solvent to recover a dry solid product. To do this, heat is applied to the solution in order to boil-off the chosen solvent. As the solvent is evaporated/boiled-off and removed, the solution containing the dissolved product becomes more and more concentrated. The more concentrated the solution gets, the closer you get to recrystallization. In recrystallization the goal is to remove enough solvent from the solution so as to form a "super saturated" solution. A super saturated solution is what happens when the dissolved product is more soluble in hot solution rather than in cold. What this means is, when we take a solution containing a dissolved solid, and we heat this solution to the point where its solvent begins to boil and evaporate-off, the solution becomes more and more concentrated. As such, the dissolved product becomes more soluble in the hot solution; however, when the heat is removed, and the solution is allowed to cool, the excess crystals in the super saturated solution have no solvent to go to as it was removed by evaporation/boiling, so they separate out as fine crystals. Once a few crystals form, a whole mess of crystals then grow off of them; thus, a recrystallization process has been successful. The precipitated crystals are then removed by filtration, and the remaining liquid solution is then heated to a boil again to restart the concentration process over again. This process is continued until the bulk of the dissolved solid has recrystallized and precipitated out, and the bulk of the solvent has been removed. Note: super saturated solutions are often called "mother liquors".

Working example of recrystallization using heat only

To use a working example of recrystallization using heat only, we will use a compound called ammonium chloride as the desired product, and water as the solvent.

1. Into a suitable beaker or flask, add and dissolve 100 grams of ammonium chloride into 400 milliliters of tap water.
2. Then place your flask or beaker onto a hot plate, and then heat the solution to 100 Celsius.
3. When the solution begins to boil, continue to boil the solution until you see a few small crystals floating on the surface of the solution.
4. When you see a few crystals of ammonium chloride floating on the surface of the boiling solution, remove the heat source and allow the solution to cool to room temperature. Note: you will begin to notice crystals separating out long before the solution is cooled to room temperature.
5. Once the solution has pretty much cooled to room temperature, or a few degrees above room temperature, filter-off all the precipitated crystals. At this point, you will notice that the total volume of the original solution has drastically changed.
6. Now, place the filtered solution back into your flask or beaker, and then heat the solution to 100 Celsius once again.
7. Allow the solution to boil, until you see a few tiny crystals floating on the surface of the solution.
8. When you see a few tiny crystals floating on the surface of the solution, remove the heat source, and allow the mixture to cool to room temperature as before. Note: you will begin to notice crystals separating out long before the solution is cooled to room temperature.
9. Once the solution has cooled to room temperature or near it, filter-off the precipitated crystals, using the same filter as for the first crop of precipitated crystals, and then place the filtered solution, which will have a reduced volume again, back into your flask or beaker.
10. At this point, your solution will have been reduced by at least 2/3rds in total volume. Continue the recrystallization process by heating the solution for a third time to 100 Celsius to boil-off the water once again.
11. Now, for a third time, when you see tiny crystals floating on the surface of the solution, remove the heat source and allow the solution to cool to room temperature for a third time. Note: you will begin to notice crystals separating out long before the solution is cooled to room temperature as before.
12. Finally, filter-off these crystals using the same filter as the previous two. By this point the bulk of the ammonium chloride should have been recovered, and the bulk of the solution will have been removed.

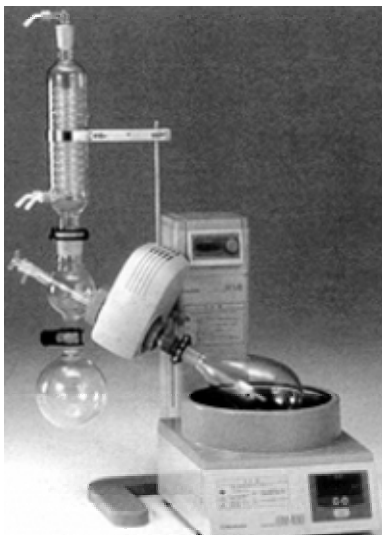
Note: in many cases, water will not be the solvent employed, and hence, you will want to use a distillation apparatus to boil off the solvent, and at the same time, recover the solvent. When using water, you can simply allow it to boil-off and dissipate in the air, but when using solvents such as methylene chloride or ether, you will probably want to collect the solvent by using distillation instead of just allowing the vapors to boil away.

2) Recrystallization using seed crystals

Seed crystals are often used to induce a crystallization of a dissolved solid. To carryout a seed crystal process, take your solution containing the dissolved solid, and place it in the open, and allow the solvent to evaporate naturally. As the solvent evaporates the solution will become more and more concentrated. However, because a super saturated solution cannot form unless the mixtures boiled, as previously discussed, the solution will come to a point where its maximum solubility is achieved. When this point is reached, the crystals will only separate out slowly as the solvent evaporates from then on. In order to speed up the formation of crystals, we would employ a seed crystal. To do so, we simply take a couple of small dried crystals of the desired product, that we would get from a chemical supplier, and we place these seed crystals into the solution. In doing so, the crystals will begin to grow and will get larger and larger as the evaporation of the solvent continues. In most cases, seed crystals are primarily used to induce recrystallization from aqueous solutions and not organic solvents such as ethers, methylene chloride or others.

3) Recovering the product through low heat and vacuum

In most modern labs, the recrystallization process by heat or seed crystal is primarily reserved for aqueous solutions, and not organic mixtures. Organic mixtures on the other hand, such as solutions containing the solvents ether, methylene chloride, ethyl acetate, or other common organic solvents are placed into devices called rotary evaporators to remove the solvent and recover the dissolved product. A rotary evaporator, as illustrated below, is a special device, which utilizes low heat and high vacuum in order to remove the solvent. When a vacuum is applied to a system containing a mixture, the boiling point of the solvent is drastically reduced. With lower boiling points comes lower heating temperature needed to drive off the solvent. In doing so, low heat can be used to drive-off the solvent, and collect the dissolved solid product from the solution. By using rotary evaporators, the solvents can be driven-off at faster rates drastically speeding up the process of separation. Also, with lower heats being used, heat and other sensitive products can be recovered without danger of decomposition. Note: liquids have decreased boiling points with decreased pressures, and higher boiling points with increasing pressures (water boils at 100 Celsius at sea level, but boils at 93 Celsius above 15,000 feet, and so on). Note: Processes involving rotary evaporators will not be discussed in this book.



A classic rotary evaporator. The solution to be evaporated is placed into a diagonal flask, which is partially submerged in a water bath. A vacuum is applied, and the water bath is gently heated to the desired temperature. As the solvent is removed, it collects in the receiver flask located on the bottom left of the illustration.

2f. Washing and drying liquids and solids

Washing liquids

Liquids are not always needed to be washed, but every so often, they will need a quick washing or scrubbing. To wash a liquid, simply place the liquid into a separatory funnel, and then add in water or another solvent. Note: the water or liquid being used for the washing should not dissolve the liquid to be washed, and when added to the separatory funnel should form a two-phase mixture. Thereafter, shake the separatory funnel vigorously for a few moments, and then allow the separatory funnel to stand to allow the two-phase mixture to form—similar to an extraction process. Now, if the liquid you're using to wash is the upper layer, you will need to drain-off the bottom layer first, before draining off the upper layer, and then you will have to place the first drained-off portion back into the same separatory funnel after draining off the upper layer. If the liquid you're using to wash is the lower layer, then simply drain it off, leaving the upper layer in the separatory funnel. Repeat this process three or four more times to thoroughly wash your liquid.

Washing solids using non-vacuum techniques

The method of washing solids via non-vacuum techniques is accomplished by placing the solid product to be washed into a beaker or other suitable container, and then adding in an excess of water or solvent. Note: the solid to be washed should be insoluble in the water or solvent used. The ratio of solid product to liquid should be about 1 part solid product to 10 parts liquid. Thereafter, stir the entire mixture for a few moments, and then allow the mixture to stand for several minutes. During this standing time, the insoluble solid product will slowly settle to the bottom. After the solid product has settled to the bottom, most of the water or solvent will be above the settled solid product. Now is the tricky part, the excess liquid should be carefully drained-off by titling the beaker or container at an angle so that the liquid slowly drains out without the insoluble solid being removed. Thereafter, more water or solid is then added and the process is repeated again. This repeating can be as many times as possible but usually requires about 6 or 7 repeats before the solid is totally washed.

Washing solids using vacuum techniques

Solids are easily washed by vacuum filtration by placing the solids to be washed on a piece of filter paper placed above the fritted bottom of a Buchner funnel. Thereafter, a clean liquid, such as water or any desired pure solvent is then poured in, in moderate size portions, over the solids in the Buchner funnel. After each pouring, the liquid is sucked through the filter paper, thereby cleaning the solids in the Buchner funnel. This process of pouring can be repeated a many times as desired. Note: the liquid you're using to clean and wash the solids in the Buchner funnel should not be able to dissolve the solids being washed. If washing a product after it has been filtered from a liquid or mixture, simply pour the filtered liquid used during the vacuum filtration process, back into the Buchner funnel. This pouring of the filtered liquid back into the Buchner funnel can be repeated as many times as necessary. Each time, the liquid will be sucked through the filter via the vacuum, and will act to wash the insoluble material. In most cases, the filtered liquid is only poured back into the Buchner funnel a few times, before a fresh liquid is used. The fresh liquid is usually water when dealing with inorganic and organic substances. Note: of coarse water

cannot be used to wash water reactive materials. When using a fresh liquid such as water, the liquid is poured in portions, and allowed to be sucked through the filter via the vacuum thereby washing the insoluble mass.

Drying solids

Solids can be dried using four main techniques. These four techniques include using an oven to dry the solids, using a vacuum to dry the solids, using airflow to dry the solids, or using a hot plate to heat and dry the solids. When using an oven, the solids are merely placed into an oven, and heated to a temperature that will dry the solids. Ovens are usually used to dry solids that are moist with water, and solids moist with flammable solvents should not be dried using ovens.

Vacuum drying is the most common method of drying many types of solids and works in the same manner as vacuum filtration. In vacuum drying, the solids to be dried are placed onto a piece of filter paper inside a Buchner funnel, and a strong vacuum is applied. As the suction of the vacuum sucks air through the filter paper, the airflow will dry the solids as a result. Vacuum drying is the most common form of drying solids in the lab.

One method of drying solids is to place the solids onto a shallow pan or tray, and allowing them to air dry. To speed up the air drying process, a small fan can be used to blow air over the solids, thereby drying them at a moderate rate. This form of drying is useful in drying large quantities of heat sensitive solids.

The final form of drying solids is to use direct heat from a hot plate. To dry solids in this manner, the solids are simply placed into a beaker or similar container, and then heated directly using a hot plate. This form of drying can be used to dry solids quickly, but is limited to solids that are not heat sensitive. Heat sensitive solids cannot be dried in this manner.

Drying liquids to remove water

Drying liquids containing traces of water can be accomplished by mixing the liquid to be dried with a drying agent. This form of drying is commonly used after an extraction process, whereby the combined extract portions are freed from traces of water by using a drying agent. Reaction mixtures and other liquids can also be dried and freed from traces of water by using drying agents. To dry a combined extract portion, reaction mixture or any other liquid, simply place a small amount of a drying agent into the mixture, and then stir the mixture for a few minutes. During the stirring process, the drying agent will absorb any traces of water that may be present. Thereafter, the drying agent, which will be insoluble in the mixture, can be removed by filtration.

The following are the most commonly used drying agents used in the modern lab:

1) Anhydrous sodium sulfate

Anhydrous sodium sulfate is one of the most widely used drying agents used in the lab to dry organic liquids to remove traces of water. It is very inexpensive, relatively inert, can be recycled over and over again, and it has a very high absorption of water as it can form a decahydrate. The sodium sulfate can be recycled after its use by placing it into a crucible and heating it to about 200 Celsius using a Bunsen burner.

2) Anhydrous magnesium sulfate

Anhydrous magnesium sulfate is another very common and inexpensive drying agent used to dry organic liquids to remove traces of water. Anhydrous magnesium sulfate has very similar properties and next to sodium sulfate, is the second most common drying agent used in the lab. One advantage that anhydrous magnesium sulfate has over sodium sulfate, is that it has a higher affinity for water than sodium sulfate.

3) Calcium chloride

Calcium chloride is the third most common drying agent used to dry organic liquids to remove traces of water. Calcium chloride has a very high affinity for water, and its affinity for water is greater than that of sodium or magnesium sulfates. Calcium chloride is very effective, and it removes traces of water readily and rapidly; however, the draw back to calcium chloride is its reactivity towards substances and organic liquids. Calcium chloride cannot be used to dry organic liquids containing ammonia, amines, alcohols, acids, and tetrahydrofurans.

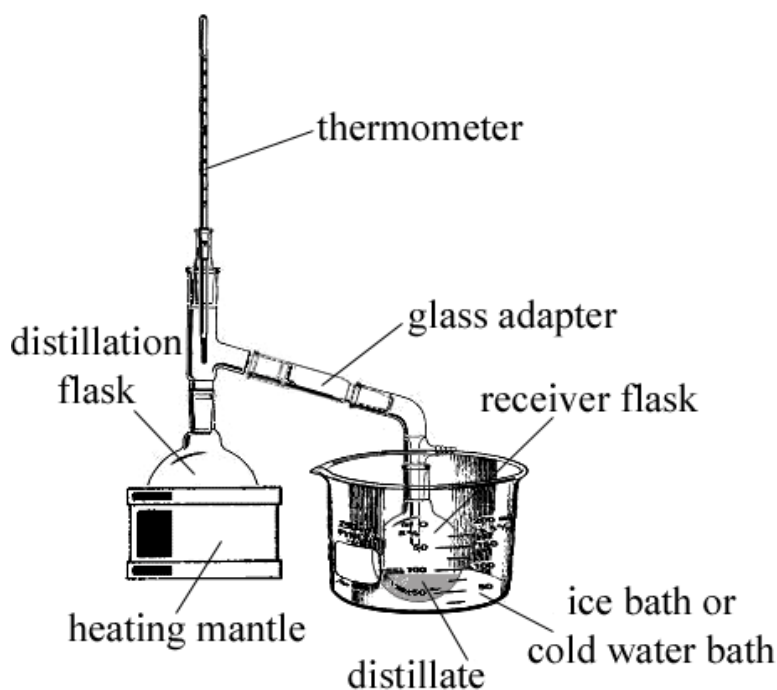
2g. Distillation

Distillation is a very common process used in the lab to remove solvents from extracts or reaction mixtures, or to separate and purify liquids from mixtures. Distillation can be broken down into five main categories, 1) short path atmospheric distillation, 2) fractional atmospheric distillation, 3) Standard atmospheric distillation, 4) vacuum distillation, and 5) steam distillation.

Each one will be discussed in detail below. So what exactly is distillation? Distillation is the process of heating a liquid to its boiling point, and then collecting the vapors as they pass over. In basic, every liquid has a boiling point, and when a liquid is heated to its boiling point it boils into a vapor or gas. This gas or vapor then dissipates into the air when the liquid is heated in the open, such as when your boiling water on a stove; however, in distillation, instead of simply boiling a liquid and allowing its vapors to dissipate, we use a close system, or distillation apparatus to isolate and condense the vapors back into a liquid. In this case, we can separate liquids from mixtures by boiling them in a closed apparatus, and then collecting the vapors as they pass over.

1) Short path atmospheric distillation

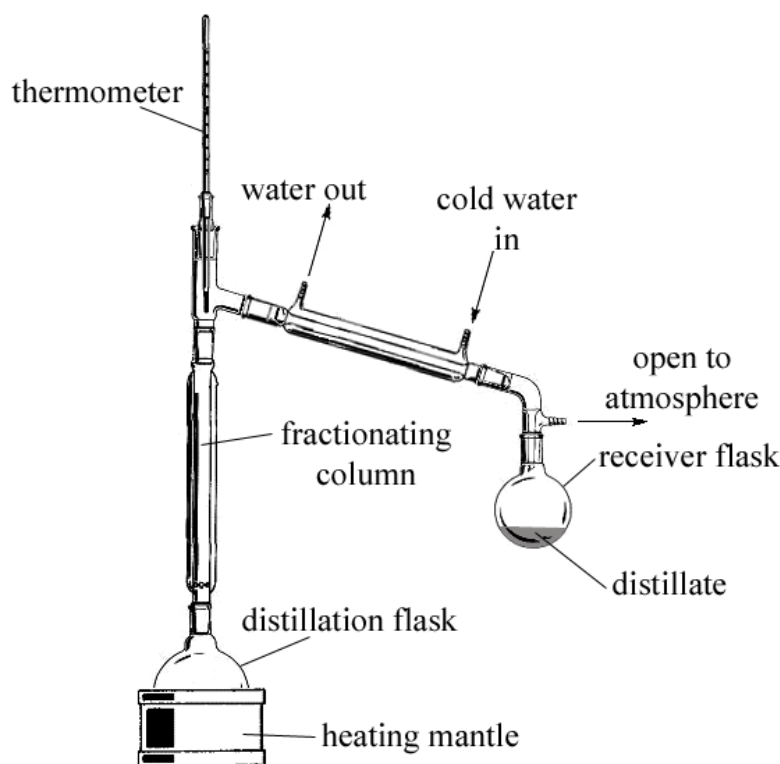
Short path atmospheric distillation involves a distillation apparatus utilizing an ice or cold-water bath to cool the vapors. Short path distillation is very useful in quickly removing water from a reaction mixture or other mixture, or for removing non-volatile liquids from reaction mixtures or other mixtures. Short path atmospheric distillation means that the apparatus is open to the atmosphere.



Common setup for short path atmospheric distillation.

2) Fractional atmospheric distillation

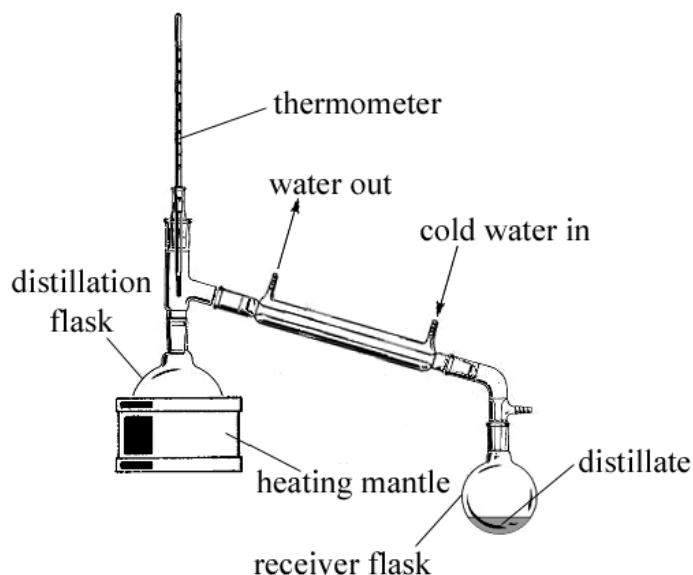
Fractional atmospheric distillation is distillation that utilizes a fractionating column in order to distill mixtures that have two or more liquids with similar boiling points. In essence, fractional distillation is used to fractionally remove liquids with similar physical properties from mixtures. In fractional atmospheric distillation, the apparatus is open to the air as in short path distillation.



Classic setup for fractional distillation.

3) Standard atmospheric distillation

Standard atmospheric distillation is the most common form of distillation in the modern lab. This form of distillation can be used for many types of processes such as removing solvents from mixtures, removing liquids from reaction mixtures, and for purifying liquid mixtures. As with the previous two forms of distillation, the standard atmospheric distillation apparatus is open to the air.

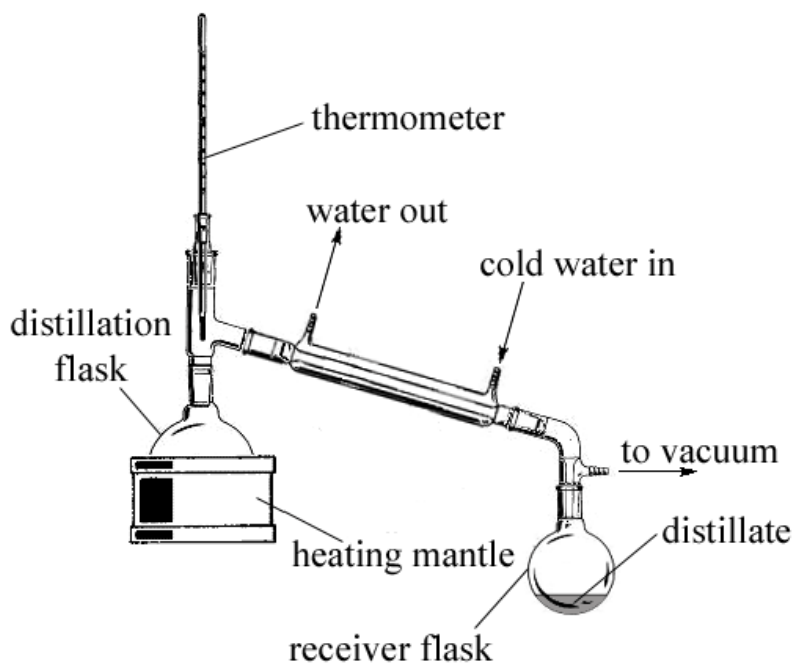


Classic setup for standard atmospheric distillation.

4) Vacuum distillation

Vacuum distillation is a special form of distillation that utilizes reduced pressures during the distillation. As previously mentioned earlier, liquids have reduced boiling points under reduced pressures, and higher boiling points with increased pressures. Therefore, in vacuum distillation, we use reduced pressure in order to reduce the boiling point of the liquid to be

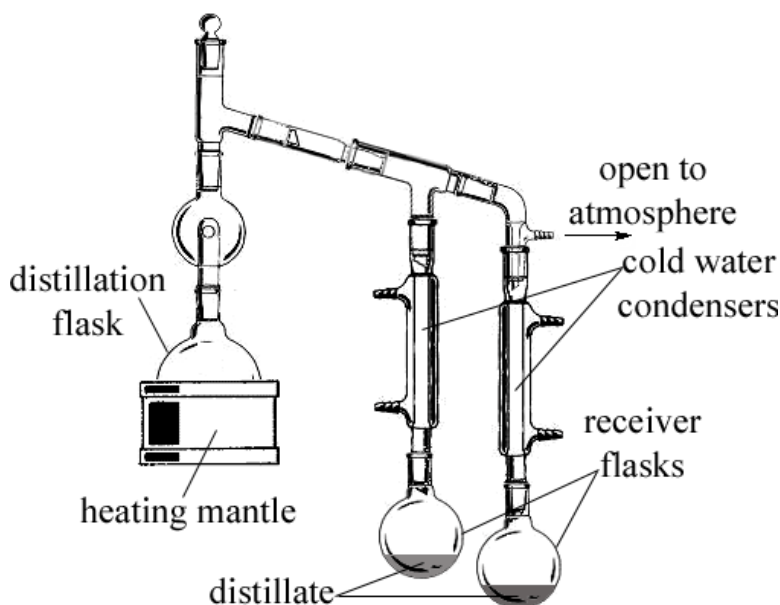
boiled. Vacuum distillation is also used to speed up the distillation process, as with reduced pressure, comes increased volatility of the liquid to be distilled; and hence, the distillation is more rapid as the more volatile the liquid to be distilled is, the faster its vapor carries over.



A classic setup for vacuum distillation. The apparatus is identical to the one used in standard distillation, except the apparatus is connected to a vacuum to reduce the pressure inside.

5) Steam distillation

Steam distillation is a special and unique form of distillation that involves using large excess amounts of water. In steam distillation, steam is used to provoke volatilization of certain solids and liquids. In essence, some mixtures and solids contain compounds that are volatile in the presence of steam, and hence can be recovered and removed by steam distillation. In other words, during the distillation process, which is carried out like any other, steam causes certain solids or liquids to volatilize. This volatilization causes the solids or liquids to be carried over with the steam simultaneously. They can then be collected from the distillate.



Common steam distillation apparatus, excess water is placed in the distillation flask, and then the desired solid mixture or liquid mixture to be steam distilled is placed there into. Thereafter, the distillation flask is then heated to 100 Celsius.

During the steam distillation process water and the volatile solids or liquids are carried over to the two receiver flasks. In this illustration, two receiver flasks are used to separate volatile solids and liquids with different densities, i.e, the products with the lower density will collect in the left receiver flask, and the products with the higher density will collect in the right receiver flask.

2h. Laboratory and chemical safety

Now that you have a brief understanding of basic chemistry and general laboratory techniques, now is the time to familiarize yourself with laboratory and chemical safety. Laboratory and chemical safety is crucial in order to maintain proper organization and discipline when dealing with laboratory procedures, chemicals, and general techniques. When dealing with laboratory procedures and chemicals, the best form of safety is to avoid and prevent accidents before they happen. **Under all circumstances, eye protection and gloves should be used at all times when dealing with chemicals. Eye protection includes eye goggles that form a complete seal around the eyes and face, and eye glasses alone should not be used for eye protection. Most gloves worn in the lab are called “Nitrile” gloves, which are chemically resistant to almost all compounds.** When dealing with chemicals, you need to familiarize yourself with chemical warning hazard signs as illustrated below:



Common laboratory chemical warning signs.

For the general dealing with and handling of chemicals, the following checkpoints should be observed:

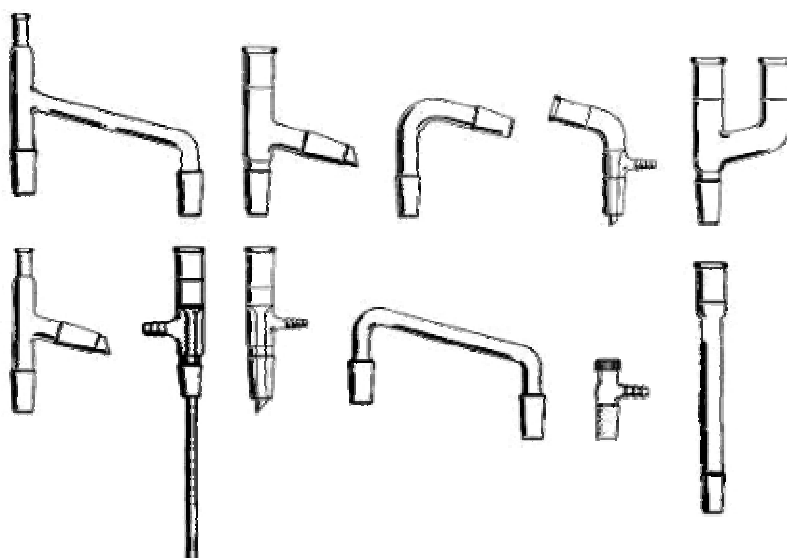
- 1) Always wear safety goggles at all times. Clothing, laboratory apparatus, and equipment can be replaced, but your eyes cannot. Do not use contact lenses or eyeglasses alone as a substitute for safety goggles. Always remember, if you get chemicals into your eyes under any circumstance, immediately flush your eyes with large excessive volumes of fresh cold tap water.
- 2) If you spill a chemical on your body, immediately remove any clothing that comes into contact, and flush any exposed skin with large amounts of cold tap water. It should be noted that most chemicals are only dangerous if they are allowed to linger on the clothing or skin, so take action immediately. Note: even concentrated sulfuric acid spilled on your clothing or skin is harmless as long as you take action immediately. Waiting around and the acid will dissolve through your clothing and begin to burn your skin.
- 3) In the case of fire or possible explosion, always remember to save yourself first. Trying to be a hero by battling the blaze or trying to prevent an explosion can get you hurt. You can keep fire extinguishers in arms reach, and you can keep an adequate water source within reach, but do not attempt to take on the accident by yourself.

- 4) For simple accidents such as acid spills, keep baking soda in arms reach. Baking soda can be spread upon acid spills to neutralize them.
- 5) When working with flammable solvents and liquids, make sure that all sources of ignition are suppressed. In the event of a fire you should carefully yet calmly move away from the burning area. If the fire is small and appears to be fightable, go ahead and try to extinguish the flames; if however, the fire is fast and progressive, evacuate the area. As before, do not attempt to be a hero.
- 6) It is of great importance that you do not eat or drink anything while handling chemicals or carrying out laboratory processes. Food and beverages can become contaminated without your knowledge, and by accident.
- 7) By all means, never attempt to taste chemicals or attempt to place chemicals on your body in anyway.
- 8) Never smell chemicals directly by sticking your nose up to beakers, flasks, or any other containers. Chemicals in open containers can be sampled by wafting the vapors with your hand to your nose, while keeping a safe distance from the open container.
- 9) Make sure not to rub your eyes or scratch your body while wearing gloves and handling chemicals. Traces of chemicals on your gloves or bare hands can be transferred to other parts of your body.
- 10) It should be noted that breathing and/or skin contact with small concentrations or small amounts of noxious liquids, mixtures, or any other substances does not pose an immediate danger, but you should avoid contact with any potentially noxious chemical under all circumstances. Toxic chemicals should only be handled by personnel who know what they are doing. In any case, toxic substances should be handled with great care and attention. Monitor for any potential leaks or spills.
- 11) Always handle volatile solvents and liquids in well-ventilated areas, and avoid breathing fumes. Flammable liquids like ether are highly volatile, and their fumes can fill rooms and travel great distances in short periods of time. Also, try to avoid breathing the vapors of flammable solvents as they can get you buzzed and make you feel dizzy, decreasing your bodies ability to focus and concentrate on what your doing.
- 12) When working with chemicals, always try to wear inexpensive clothing such as work clothing since the possibility of an accident is always present. Inexpensive clothing can be damaged during the accident, but replaceable. When working with chemicals, laboratory coats and/or aprons are readily available and can be worn over existing clothing.
- 13) Do not wear loose clothing, sandals, or casual clothing while working with chemicals. Obviously, shorts, skirts, blouses, or any other clothing that leaves large areas of skin unprotected should not be worn when handling chemicals.
- 14) Never experiment with unfamiliar chemicals by heating them, mixing them, or treating them with any other chemicals, or any other sort of interaction. Never heat unfamiliar chemicals as decomposition forming toxic fumes may result.
- 15) When carrying out chemical reactions, always make sure you know what you are doing, or have knowledge about what you are doing before you do it. Playing around with chemicals can lead to the generation of toxic fumes, explosions, or fire hazards.

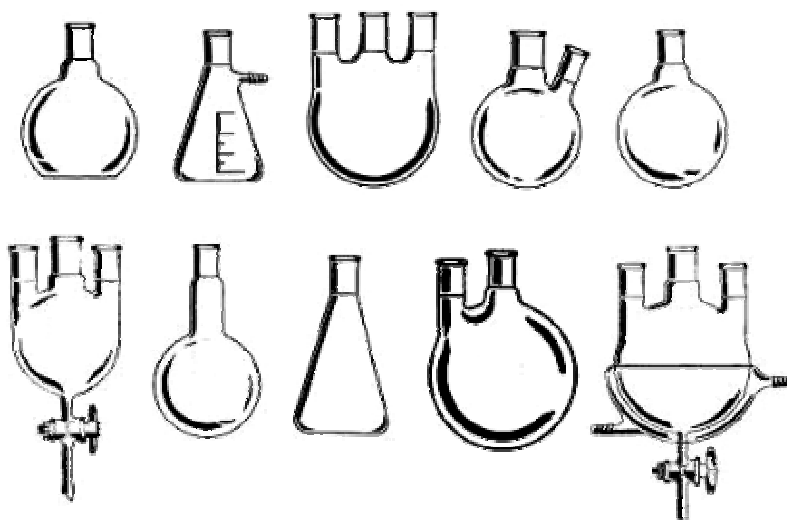
2i. Familiarization with laboratory glassware and equipment

Laboratory glassware

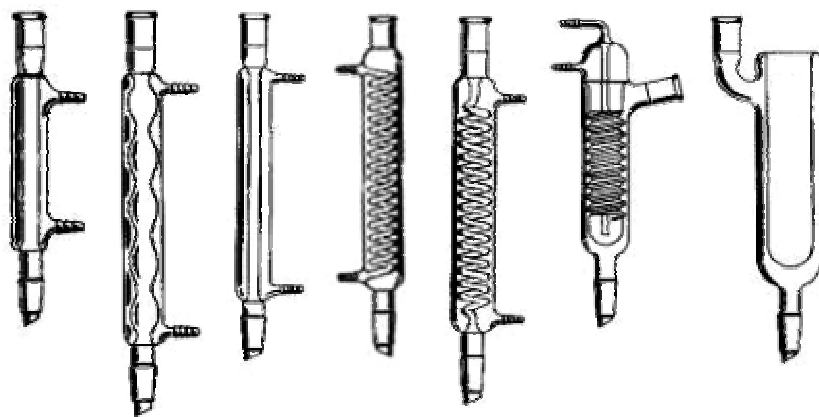
Laboratory glassware are essential when carrying out extractions, chemical reactions, or chemical processes. Laboratory glassware comes in many different sizes and forms, all of which are made of heat resistant glass called “Pyrex”, or “Kimax”. This heat resistant glassware can be heated to temperatures of up to 500 Celsius without breakage, cracking, or scarring. Because laboratory glassware varies in size and type, the glassware ranges in price, and some glassware can be quit expensive. In any sense, its time you get familiar with general laboratory glassware. Laboratory glassware is divided into five main categories, and these categories include 1) adapters, 2) flasks, 3) condensers, 4) general lab glass, and 5) specialty apparatus.



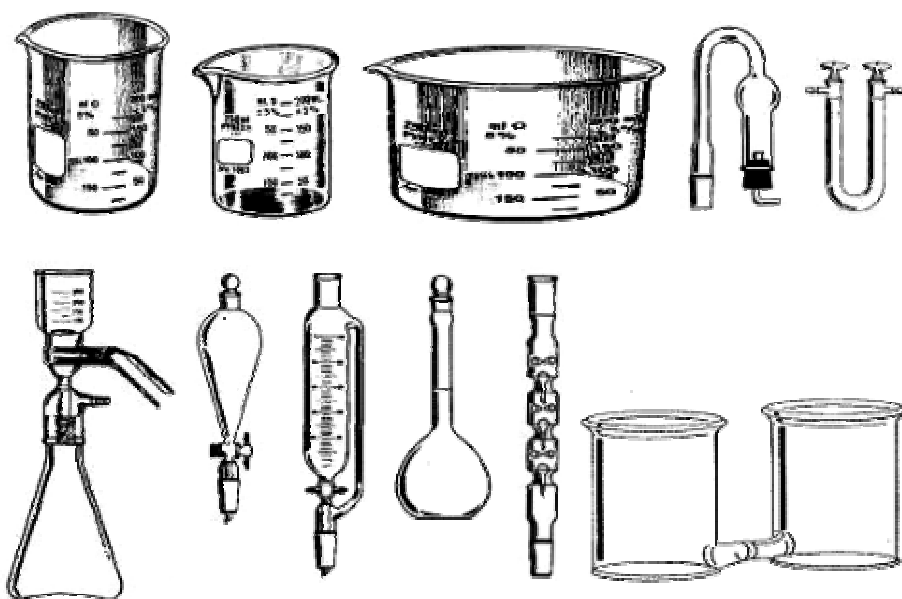
Adapters. Laboratory glassware adapters come in many different sizes, and are composed of male and female glass joints. The joints range in size from 10/30, to 19/22, to 24/40. The adapters have male and female joints that are composed of etched glass surfaces, which form an airtight seal when placed male to female. Adapters range in price from \$25.00 to \$150.00.



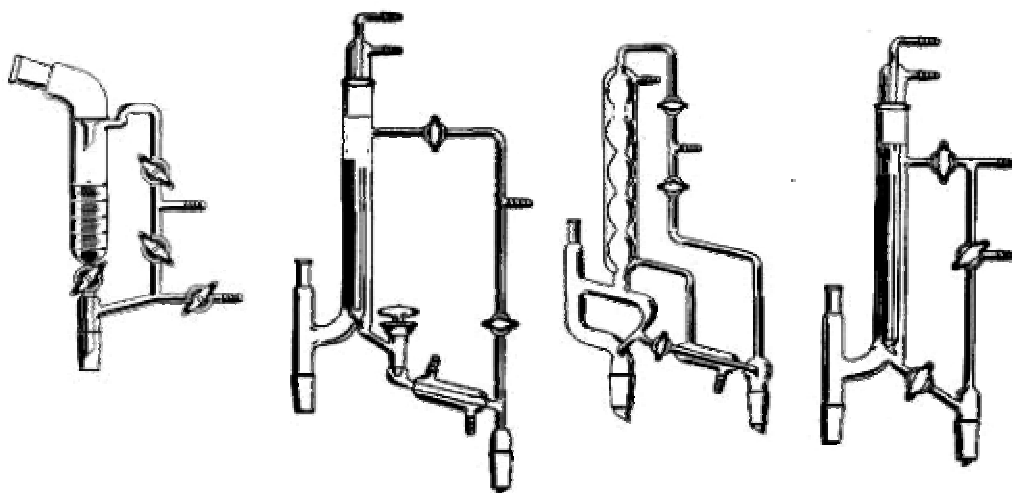
Glass flasks. As with adapters, many flasks in the chemical laboratory are composed of etched joints ranging from 10/30 to 24/40. Flasks range in price from \$15.00 to \$300.



Laboratory glassware condensers. Like adapters and flasks, most condensers in the modern lab have etched glass joints. Condensers range in price from \$45.00 to \$500.



Common laboratory glassware. Other forms of common laboratory glassware are test tubes, which are not shown in the illustration. Some common laboratory glassware contains etched glass joints as seen in adapters, flasks, and condensers. Common laboratory glassware ranges from \$1.00, to \$250.



Advanced laboratory glassware. These types of glassware can be very expensive and range in price from \$500 to \$1000.

Even though laboratory glassware can be heated to temperatures ranging from 100 Celsius to 500 Celsius. Laboratory glassware should not be subjected to cool temperatures after heating, as crackage can occur.


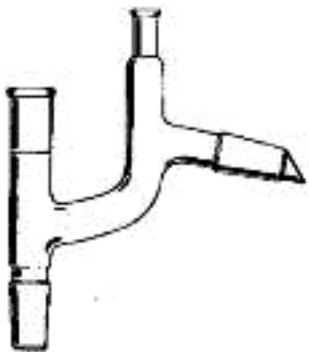






Laboratory equipment









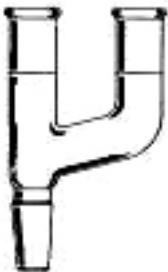



General laboratory equipment ranges from hot plates, Bunsen burners, to ovens. Other types of laboratory equipment include vacuum pumps, oil baths, and rotary evaporators. Laboratory equipment ranges from \$30 to \$6000. Rotary evaporators can cost as much as \$6000. Advanced laboratory equipment includes HPGC, and NMR systems, HPGC stands for high performance gas chromatography, and NMR stands for Nuclear Magnetic Resonance. High performance gas chromatography can range from \$6000 to \$15,000 and are used to identify chemical compounds in parts per million. High performance gas chromatography is widely used in modern labs, and is practically indispensable. High Performance gas chromatography is widely used by research scientists and analytical scientists to identify chemical compounds.

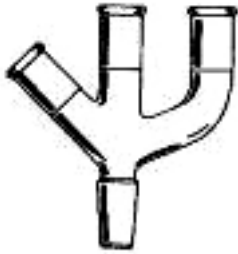
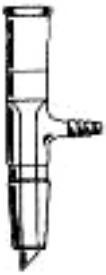







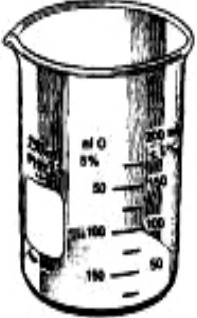

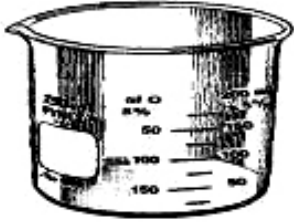
Chapter 3: Laboratory Apparatus




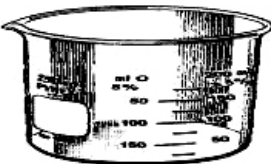




Familiarization with laboratory glassware






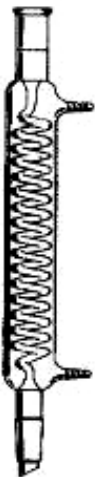
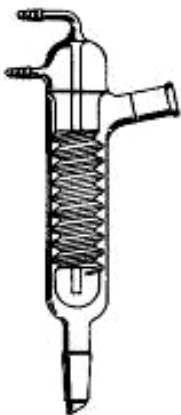

Most laboratory glassware, as previously discussed are composed of all glass, one-piece construction with etched male and female joints. The most common joints are 19/22 and 24/40, meaning millimeters in width by milliliters in length; therefore, 24/40 means 24mm width by 40mm length. The following pictures illustrate common laboratory glassware with joints of 19/22 or 24/40 on average.

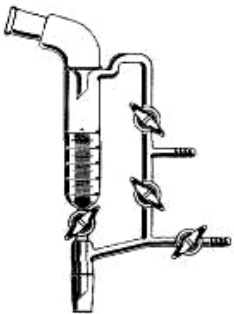
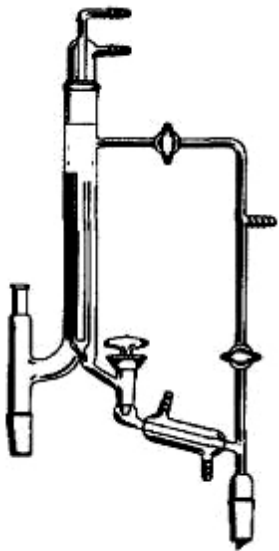

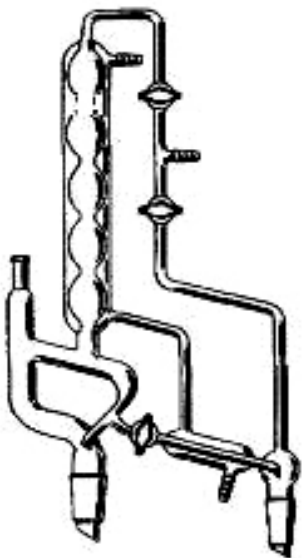
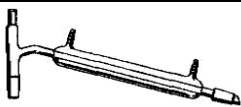

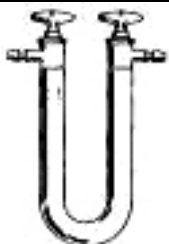
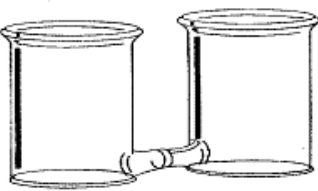
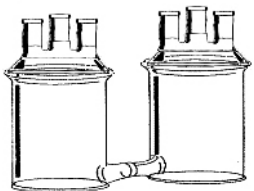



			
Straight vacuum take-off adapter with stopcock; Average price: \$25.00 to \$35.00	3-way Claisen adapter with thermometer joint; Average price: \$45.00 to \$85.00	Curved Vacuum take-off adapter with stopcock; Average price: \$27.00 to \$40.00	Distilling adapter with thermometer joint; Average price: \$45.00 to \$95.00
			
3-way distilling head; Average price: \$35.00 to \$55.00	Curved 2-way male to male adapter; Average price: \$25.00 to \$35.00	3-way inverted distilling adapter; Average price: \$37.00 to \$55.00	2-way curved female to male adapter; Average price: \$25.00 to \$35.00




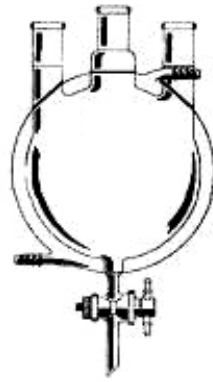




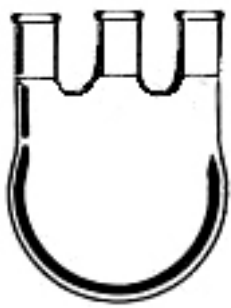
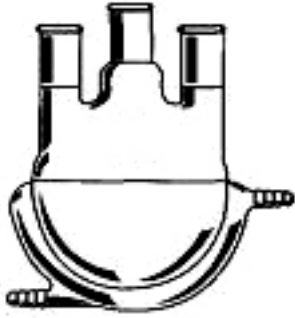

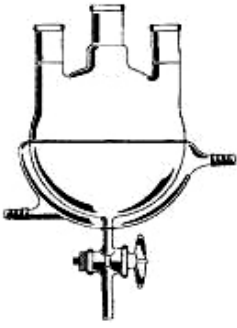
			
Curved drip-tip adapter; Average price: \$45.00 to \$60.00	Vacuum drip-tip adapter; Average price: \$35.00 to \$50.00	Female to male straight adapter; Average price: \$20.00 to \$30.00	Female to female straight adapter; Average price: \$20.00 to \$30.00
			
Thermometer adapter; Average price: \$15.00 to \$35.00	Distilling head with trap and stop cock outlet; Average price: \$45.00 to \$65.00	Curved tapered adapter; Average price: \$27.00 to \$40.00	Vacuum adapter with hose barb; Average price: \$15.00 to \$25.00
			
Claisen adapter; Average price: \$45.00 to \$75.00	3-way distilling adapter; Average price: \$35.00 to \$45.00	2-way distilling adapter with thermometer joint; Average price:	Vacuum adapter with extended drip-tip; Average price: \$37.00 to \$55.00

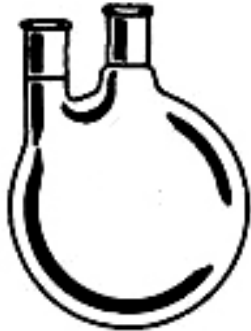


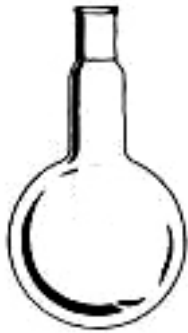








		\$35.00 to \$55.00	
			
Modified Claisen 4-way adapter; Average price: \$55.00 to \$75.00	Straight vacuum distilling adapter; Average price: \$29.00 to \$47.00	Anti-splash bulb adapter; Average price: \$55.00 to \$67.00	Distilling adapter male to male; Average price: \$29.00 to \$34.00
			
Advanced 3-way distilling head with thermometer joint and stopcock; Average price: \$89.00 to \$125.00	Tube or thermometer adapter with hose barb; Average price: \$15.00 to \$20.00	Straight distilling column; Average price: \$34.00 to \$45.00	Tube or thermometer adapter; Average price: \$12.00 to \$15.00
			
Male to male straight adapter; Average price: \$25.00 to \$34.00	Standard beaker, tall form; Average price: \$1.75 to \$65.00	Standard beaker low form; Average price: \$7.95 to	Large low form beaker; Average price: \$55.00 to \$105.00


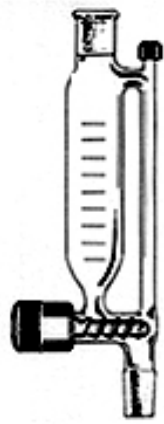


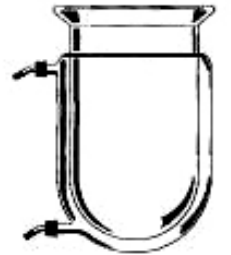

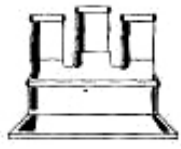

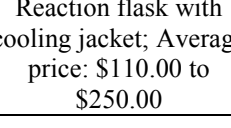
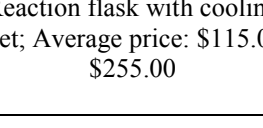
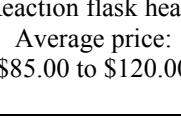
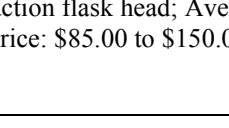
		\$80.00	
			
Bleed tube; Average price: \$2.75	Thermometer adapter with bleed tube; Average price: \$15.00 to \$25.00	Oil bubbler; Average price: \$14.95	Large pan like low form beaker; Average price: \$65.00 to \$120.00
			
Double oiler bubbler; Average price: \$ 20.00	Burret; Average price: \$45.00 to \$55.00	Vacuum distilling column, with vacuum jacket, long; Average price: \$95.00 to \$200.00	Vacuum distilling column, with vacuum jacket, short; Average price: \$75.00 to \$110.00


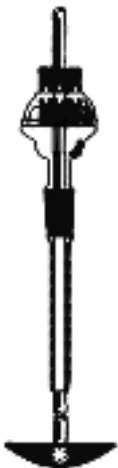

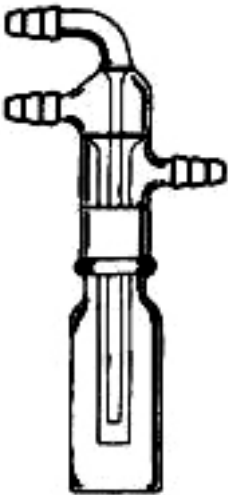


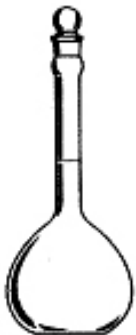
			
Snyder distilling column; Average price: \$45.00 to \$75.00	Standard Liebig condenser, short; Average price: \$35.00 to \$75.00	Allihn condenser; Average price: \$40.00 to \$65.00	Cold finger adapter with cooling inlet tubes; Average price: \$65.00 to \$75.00
			
Standard Liebig condenser, medium; Average price: \$35.00 to \$55.00	Coil distilling condenser; Average price: \$55.00 to \$80.00	Fredrichs distilling condenser; Average price: \$95.00 to \$150.00	Dry-ice cold trap condenser; Average price: \$50.00 to \$100.00

			
Advanced vacuum take-off pressure distilling head with stopcocks; Average price: \$150.00 to \$200.00	Advanced cold-trap distilling take-off adapter with short Liebig condenser and vacuum take-off with stopcocks and thermometer joint; Average price: \$195.00 to \$260.00	Calcium chloride drying tube; Average price: \$15.00	Advanced Allihn vacuum take-off adapter with stop cocks and short path Liebig condenser with thermometer joint; Average price: \$190 to \$230.00
			
Liebig distilling head with thermometer joint; Average price: \$65.00 to \$100.00	Standard drying tube; Average price: \$15.00	U-tube drying tube with side arms; Average price: \$21.00	Electrolytic cell with porous membrane separator; Average price: \$55.00
			
Electrolytic cell with reaction heads and porous membrane separator; Average price: \$95.00	Buchner funnel with support clamp; Average price: \$35.00	Buchner funnel with support clamp and flask with vacuum arm; Average price: \$65.00	Standard all glass Buchner funnel with vacuum side arm; Average price: \$42.00

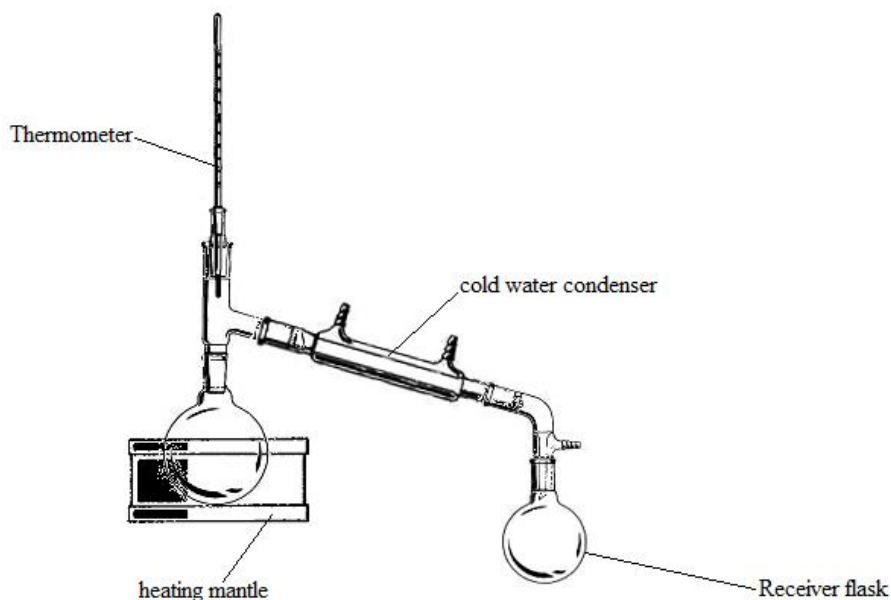
			
Erlenmeyer flask with 2-way adapter and side arm stopcock; Average price: \$65.00 to \$95.00	Dewer flask with side arm and stop cock; Average price: \$75.00 to \$98.00	Flat bottom boiling flask with joint; Average price: \$10.00 to \$95.00	3-neck round bottom flask with cooling jacket and drainage stop cock; Average price: \$110.00 to \$190.00
			
Pear shaped flask with joint; Average price: \$15.00 to \$98.00	Erlenmeyer filter flask with joint and side arm; Average price: \$45.00 to \$85.00	Long neck pear shaped flask with vacuum side arm; Average price: \$55.0 to \$78.00	3-neck flask with angled joints; Average price: \$55.00 to \$110.00
			
Standard 3-neck round bottom flask; Average price: \$65.00 to \$120.00	3-neck round bottom flask with cooling jacket; Average price: \$85.00 to \$150.00	3-neck flask with drainage outlet and stopcock; Average price: \$95.00 to \$145.00	3-neck flask with drainage outlet and stopcock with cooling jacket; Average price: \$95.00 to \$150.00

			
2-neck round bottom flask; Average price: \$35.00 to \$65.00	2-neck round bottom flask with angled neck; Average price: \$35.00 to \$70.00	Standard single neck round bottom flask; Average price: \$25.00 to \$120.00	Long neck round bottom flask; Average price: \$25.00 to \$110.00
			
Erlenmeyer flask with joint; Average price: \$15.00 to \$65.00	Funnel with joint; Average price: \$8.00	Separatory funnel with stop cock; Average price: \$45.00 to \$100.00	Addition funnel with pressure equalization and stop cock; Average price: \$65.00 to \$110.00
			

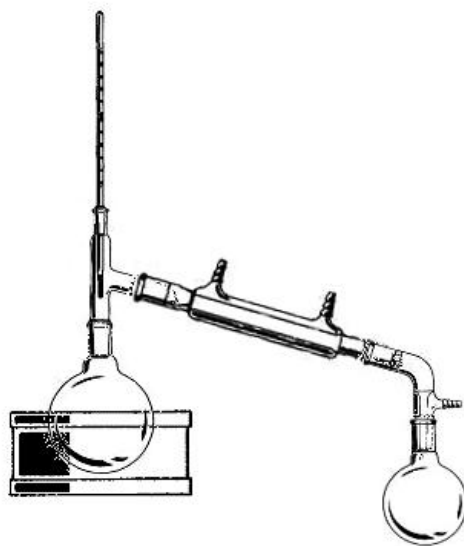
Addition funnel with pressure equalization with stopcocks; Average price: \$75.00 to \$145.00	Addition funnel with stopcock and cooling jacket; Average price: \$90.00 to \$150.00	Inlet gas addition tube; Average price: \$25.00 to \$45.00	Inlet gas addition adapter with female to male joints; Average price: \$30.00 to \$60.00
			
Heating mantle; Average price: \$85.00 to \$250.00	Powder addition funnel; Average price: \$75.00	Electric probe thermometer with 10/30 joint; Average price: \$65.00	Electric probe thermometer with joint adapter; Average price: \$75.00
			
Reaction flask with cooling jacket; Average price: \$110.00 to \$250.00	Reaction flask with cooling jacket; Average price: \$115.00 to \$255.00	Reaction flask head; Average price: \$85.00 to \$120.00	Reaction flask head; Average price: \$85.00 to \$150.00
			

			
Electric stirrer with Teflon stir blade and joint; Average price: \$95.00 to \$120.00	Electric stirring motor with Teflon stir blade and joint; Average price: \$110.00 to \$135.00	Assorted glass stoppers; Average price: \$0.95 to \$5.00	All glass sublimation apparatus with vacuum take-offs; Average price: \$78.00
			
Thermometer with 10/30 joint; Average price: \$15.00	Thermometer with joint adapter; Average price: \$25.00 to \$35.00	Volumetric flask; Average price: \$8.95 to \$75.00	

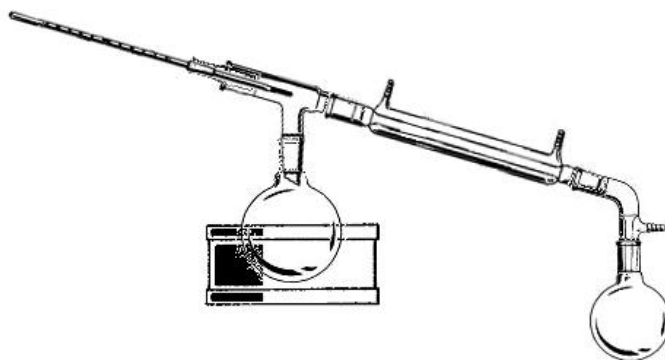
Standard distillation apparatus



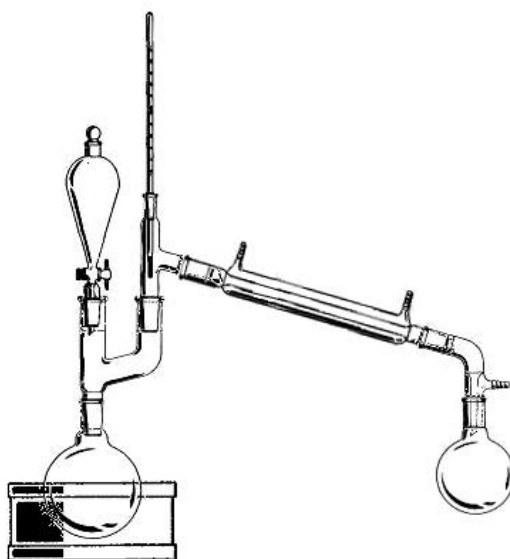
A standard laboratory distillation apparatus is composed of six pieces of labware, including a thermometer with joint, a three-way adapter, condenser, and two single neck round bottom flasks. This apparatus is designed for general distillation at atmospheric pressure



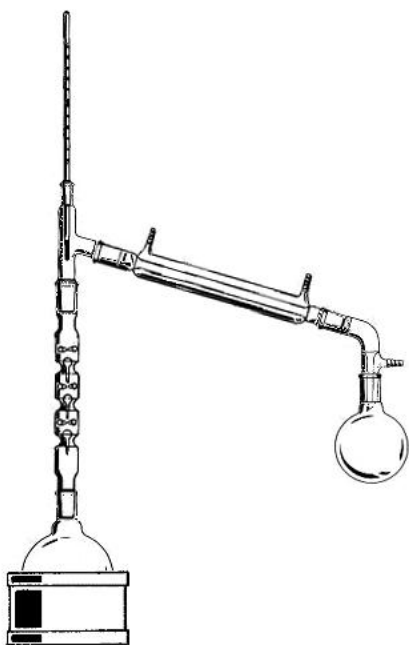
A standard laboratory distillation apparatus is composed of six pieces of labware, including a thermometer with 10/30 joint, a three-way adapter, condenser, and two single neck round bottom flasks. This apparatus is designed for general distillation at atmospheric pressure



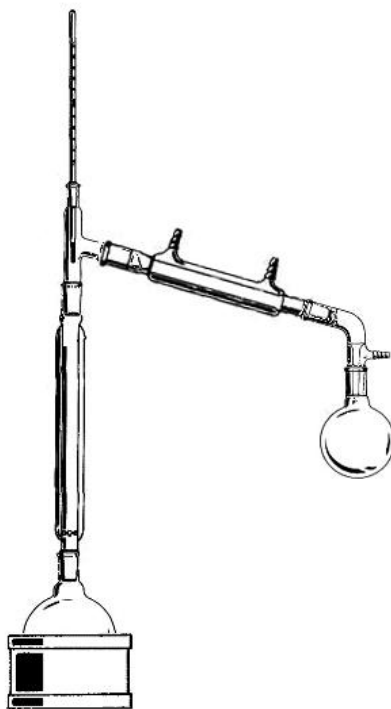
A standard "short path" laboratory distillation apparatus is composed of seven pieces of labware, including a thermometer with 10/30 joint, a three-way adapter, condenser, and two single neck round bottom flasks. This apparatus is designed for general distillation at atmospheric pressure



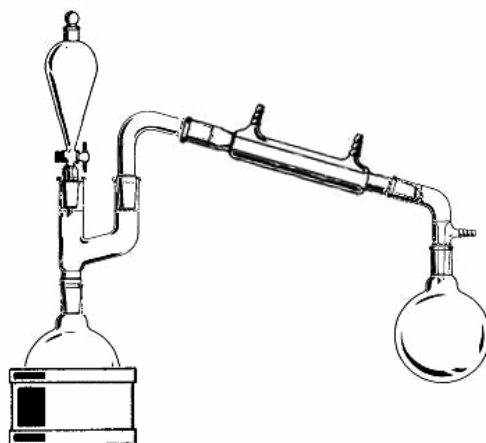
A standard distillation apparatus with addition funnel. This apparatus is composed of 8 pieces including a Claisen adapter. This apparatus is designed for distillation at atmospheric pressure.



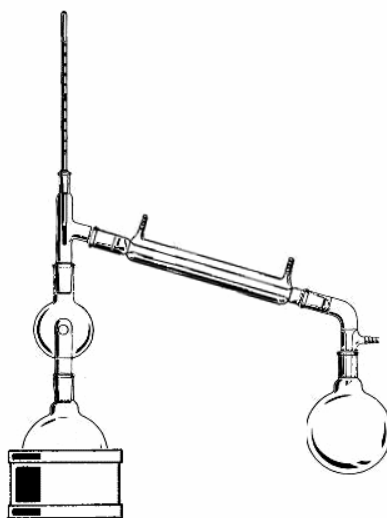
This is a standard distillation apparatus with Snyder column. This setup is used for distillation at atmospheric pressure or under vacuum.



Fractional distillation apparatus. This apparatus is used to distill liquid mixtures that contain liquids that have close boiling points. A vacuum jacked distilling column is used in this setup, and at atmospheric pressure or under vacuum.

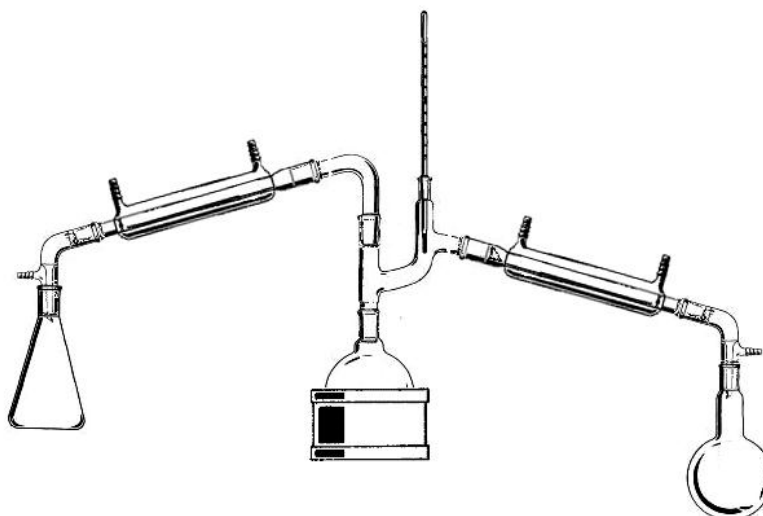


Standard steam distillation apparatus. The steam is generated internally from boiling water. The addition funnel contains additional water, which is added when necessary.

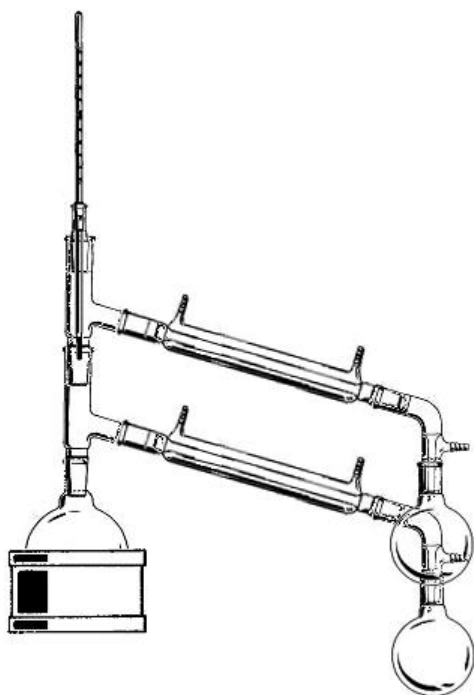


Standard distillation apparatus with anti-splash bulb. This apparatus is used to distill mixtures that bump and foam.

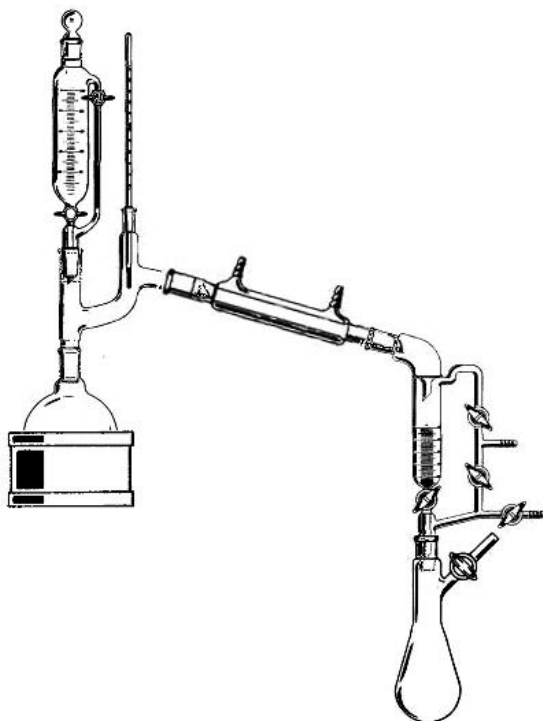
Advanced Distillation apparatus



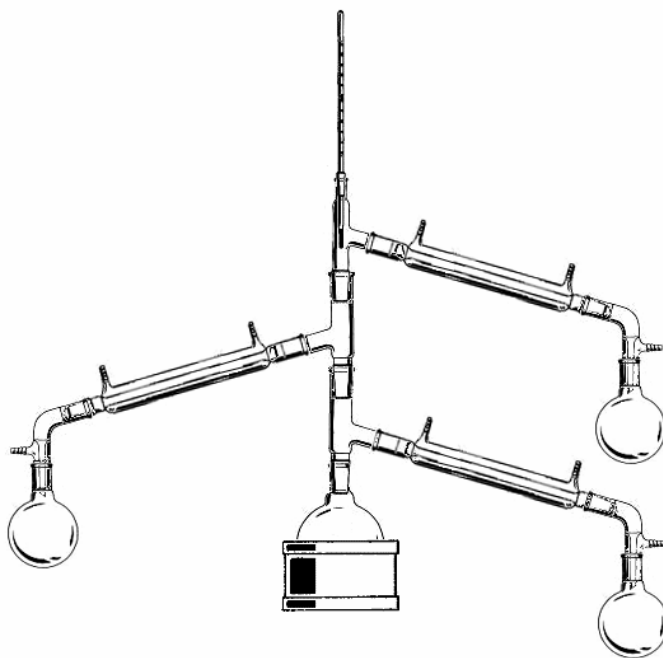
2-Path distillation apparatus designed to distill two liquids with different densities at atmospheric pressure. The heavier density liquid distills over in the right flask.



2-Path distillation apparatus designed to distill two liquids with different densities at atmospheric pressure. The heavier density liquid distills over in the lower right flask.

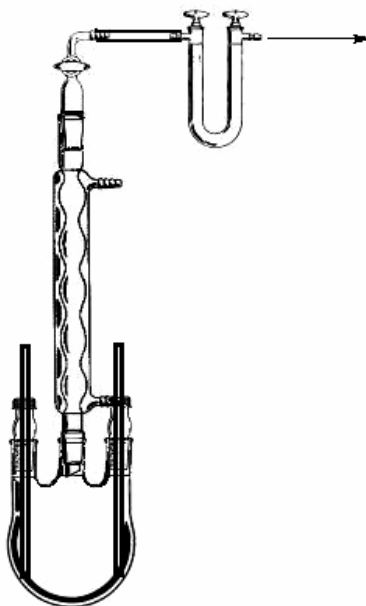


Advanced vacuum distillation apparatus with vacuum manifold and take-off valves, with addition funnel.

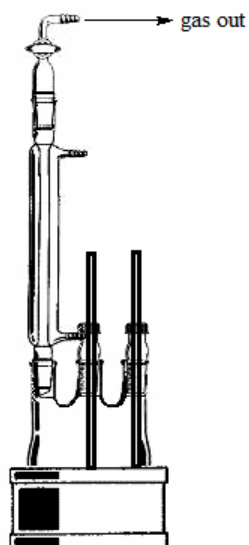


Triple path distillation apparatus under atmospheric pressure or under vacuum. This apparatus is used to distill liquids with different boiling points and densities.

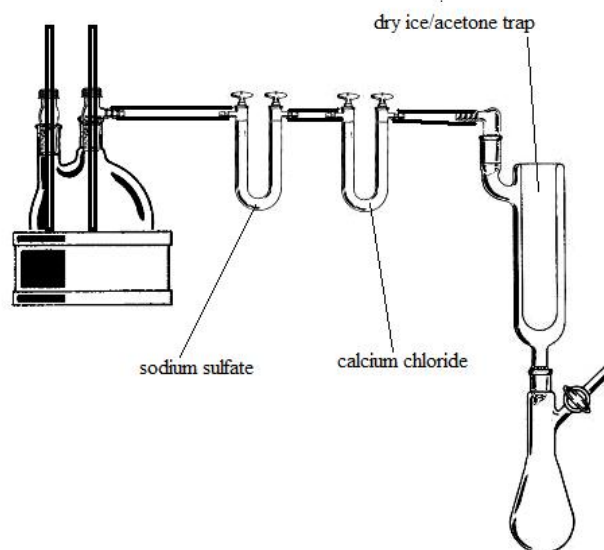
Electrolysis apparatus



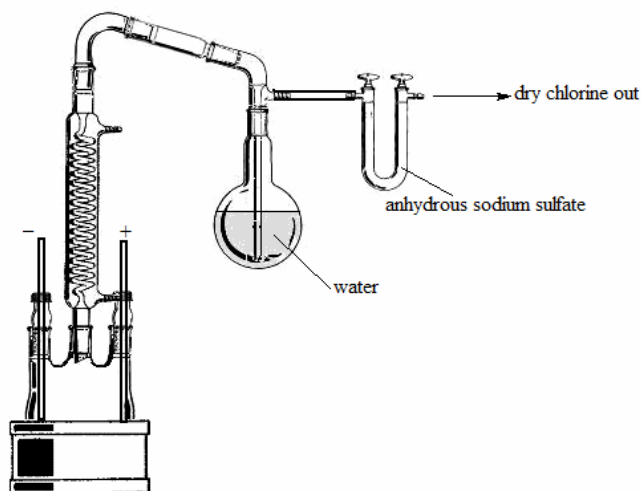
Classic electrolysis apparatus utilizing a 3-neck flask. This apparatus is used to generate hydrogen and oxygen by electrolysis of alkaline salts such as table salt or magnesium sulfate using inert electrodes of graphite or lead. If metal electrodes such as copper or aluminum are used, the corresponding metal hydroxides will be formed, and precipitate. In the above apparatus a bulb condenser is used to control temperature, and a calcium chloride drying tube is attached to dry the gas mixture.



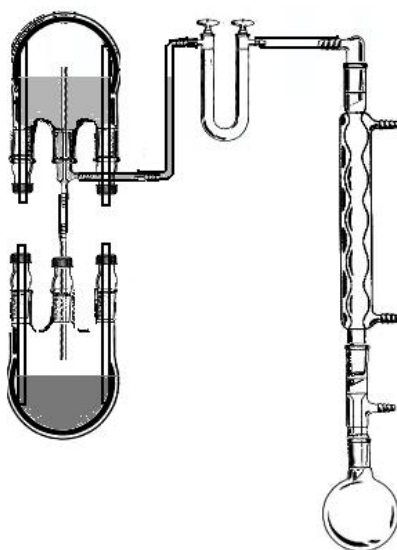
Electrolysis apparatus for the oxidation of neutral salts. In this apparatus uses a heating mantle to control temperature, and the electrodes are made of platinum coated steel. The salts used are sodium chloride and magnesium sulfate. The sodium chloride is converted into sodium chlorate, and the magnesium sulfate is converted into magnesium peroxydisulfate.



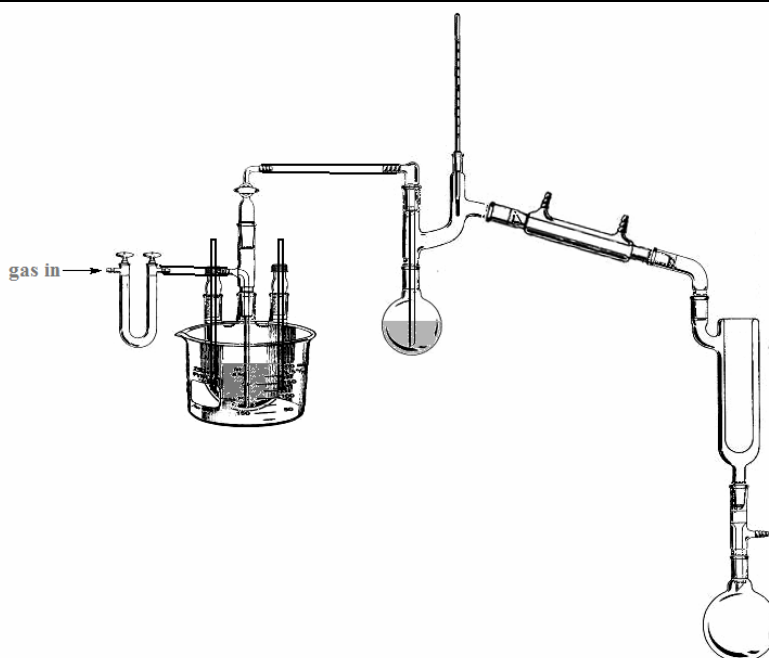
This apparatus is for generating chlorine gas by electrolysis of hydrochloric acid. The chlorine gas is dried by passing it through drying tubes, and is then liquefied by use of a dry ice cooling condenser.



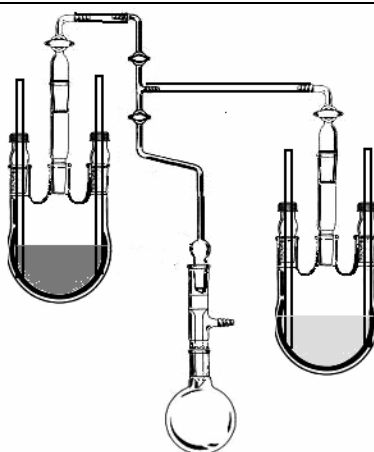
Conventional apparatus for the electrolysis of hydrochloric acid to form chlorine gas. The chlorine gas is first bubbled through water to remove acid, and then dried by use of sodium sulfate.



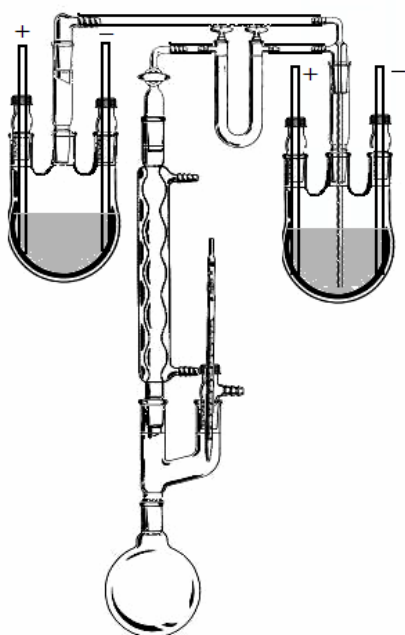
Advanced electrochemical apparatus for the combining of gases from two separate cells. The lower cell generates a gas that combines with the gas generated from the upper cell. The gas mixture is then dried using a drying tube, and then cooled by means of the condenser. The electrodes are made of platinum plated metal, graphite, or nickel.



Electro apparatus for the generation of gas. A reaction gas is introduced below the surface of the electrolyte. The gas generated is then liquefied by means of a dry ice trap.

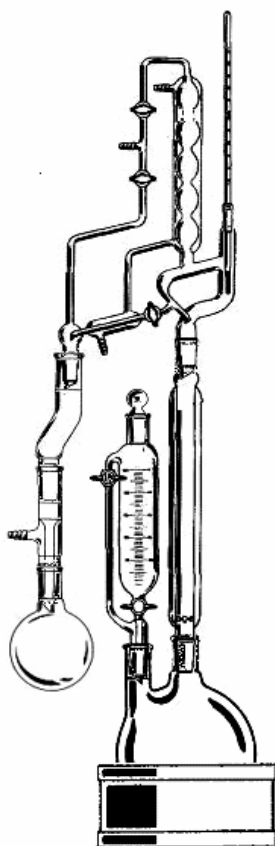


Double electrolysis apparatus for generating more than one gas. The gas mixture is then used as a reactant for bubbling into a reaction mixture (not illustrated).

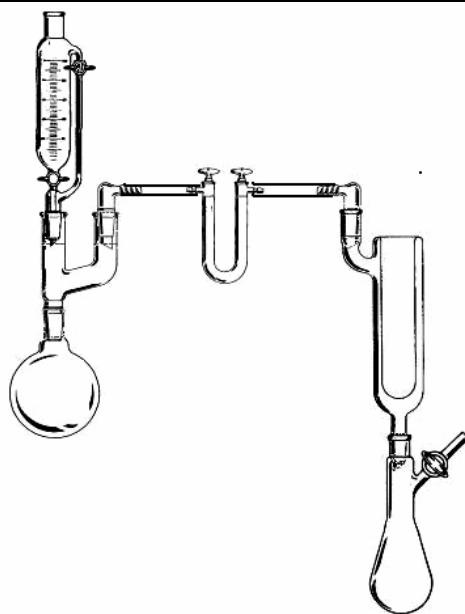


Electrochemical setup for reacting electrolysis products with a reaction mixture contained in the lower flask. The gases from the two electrolysis apparatus react with the vapors of a particular compound(s) generated from the lower flask.

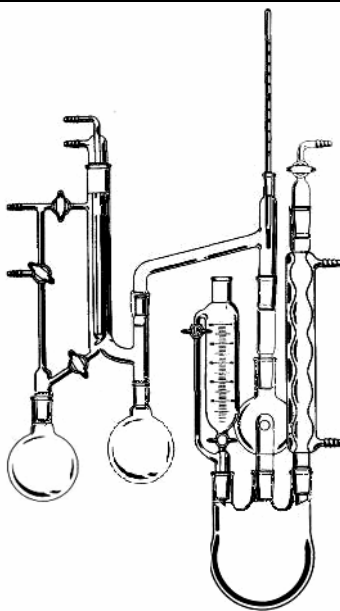
Advanced apparatus for various laboratory procedures



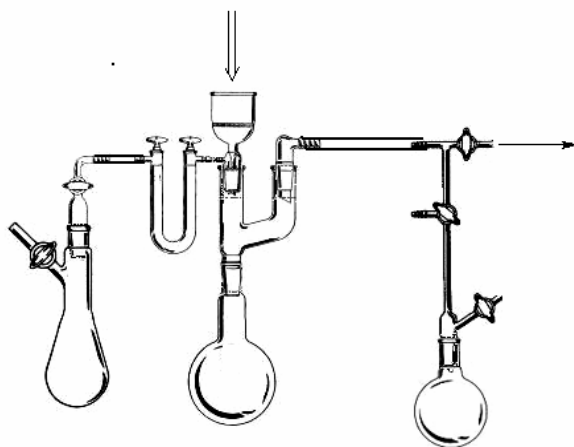
Advanced reaction apparatus with addition funnel with vacuum manifold and two vacuum ports. This apparatus is used for various laboratory procedures involving volatile materials.



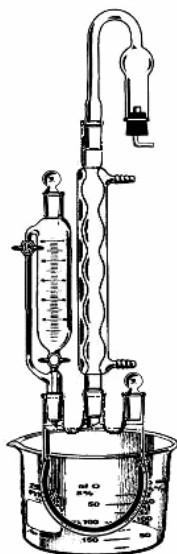
Typical apparatus for the generation of gas. Such gasses include phosphine and sulfur dioxide. To generate phosphine, sodium phosphide is placed in the reaction flask, and then concentrated sulfuric acid is dripped onto it. To generate sulfur dioxide, sodium hydrogen sulfite is placed in the reaction flask, and then concentrated hydrochloric acid is dripped onto it. The gases are then liquefied by use of a dry ice condenser.



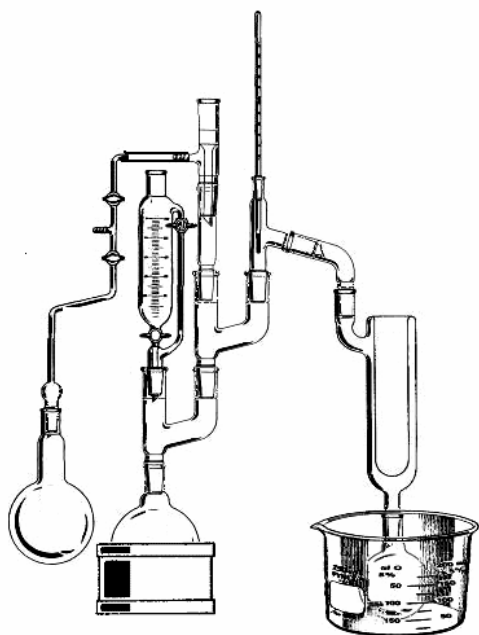
Advanced apparatus for reacting ketones with strong oxidizing agents to form volatile liquids and gas. Apparatus is equipped with vacuum manifold.



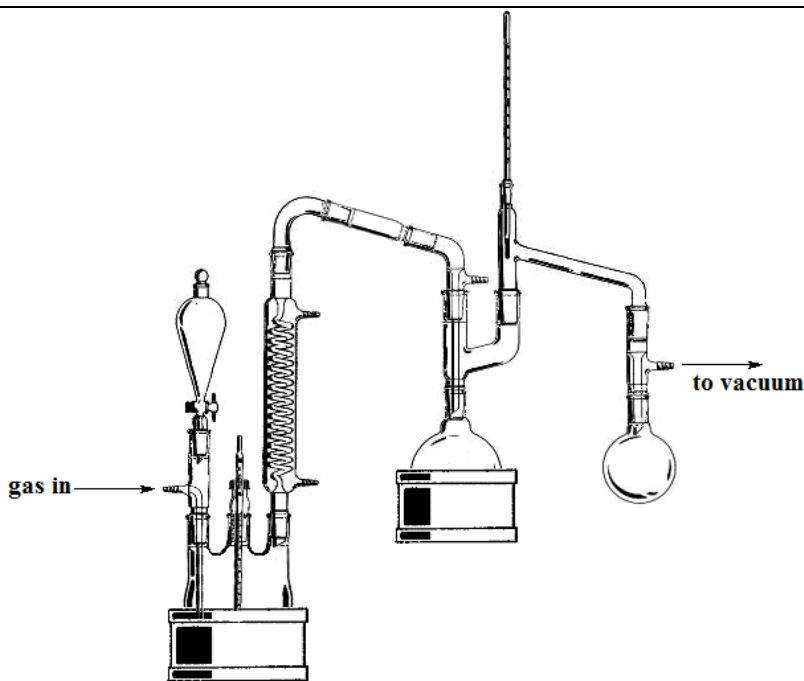
Reaction of filtrate with substrate utilizing a vacuum manifold.



Standard reaction apparatus for the preparation of a Grignard Reagent. A calcium chloride drying tube is used to keep moisture out of the apparatus, and a cold water bath is used to keep the reaction mixture cool.



Advanced reaction assembly for the liquefaction of reaction gas produced by reacting a substrate with an intermediate. This apparatus comes with a vacuum manifold to reduce the pressure inside the reaction apparatus. A dry ice cold condenser is used to liquefy the product gases.



Advanced reaction assembly for the reaction of volatile liquid with a reaction mixture. The volatile liquid is generated in the reaction flask to the left, which is then passed into reaction flask at the right. A reaction gas is bubbled into the reaction flask to the left.

Chapter 4: Chemistry

Theory and Calculations

Chemical theory and calculations are very diverse and include numerous operations; however, most chemistry calculations are easier to understand than one might expect. Unlike overpriced and overrated chemistry college textbooks, this guide will better help you understand the basics of chemistry theory and calculations without all the hoop-law, fancy pants operations, and difficult to know equations. First of all, we should start with the very basics of chemistry theory and calculations.

1. Let's start with determining the molecular weight of Aluminum chloride (AlCl_3).
 - a) Answer: Let's look at the periodic table, and find the atomic weights of aluminum and chlorine
 - b) Aluminum has a atomic weight of 26.98, and chlorine has an atomic number of 35.45. Now, since we have three chlorine atoms in aluminum chloride we write the simple equation $35.45 \times 3 + 26.98$
 - c) Therefore the molecular weight of aluminum chloride is 133.33
2. Let's try another example. Determine the molecular weight of acetic acid (CH_3COOH)
 - a) Answer, here we have 2 carbon atoms, 4 hydrogen atoms, and 2 oxygen atoms; therefore we look at the periodic table and see that carbon = 12.011, Hydrogen = 1.008, and oxygen = 15.999. Therefore we have $12.011 \times 2 + 1.008 \times 4 + 15.999 \times 2 = 60.052$. The molecular weight of acetic acid is 60.052
3. Let's look at some of the elements from the periodic table, and determine the atomic weights of a) Hydrogen; b) Lithium; c) silver; and d) Calcium
 - a) Answer, look the periodic table and you will find that the atomic weight of Hydrogen is 1.008; Lithium is 6.94; Silver is 107.87; and Calcium is 40.08.
4. Now let's determine the molecular weight of some gasses. Let's look at hydrogen, oxygen, nitrogen, and Chlorine.
 - a) Because these gases exist in the free state as H_2 , O_2 , N_2 , and Cl_2 , we use the following simple equation to determine their molecular weights.
 - b) Hydrogen = 1.008×2 ; Oxygen = 15.999×2 ; Nitrogen = 14.007×2 ; and Chlorine = 35.45×2
 - c) Therefore we have $1.008 \times 2 = 2.016$; $15.999 \times 2 = 31.998$; $14.007 \times 2 = 28.014$; and $35.45 \times 2 = 70.9$
 - d) So the molecular weight of hydrogen is 2.016; the molecular weight of oxygen is 31.998; the molecular weight of nitrogen is 28.014; and the molecular weight of chlorine is 70.9
5. Now let's discuss what a mole is? A mole in chemistry is not the small furry creature that lives in the ground, no a mole in chemistry is the number of atoms in a given sample of matter; for example, 1 mole of hydrogen (H_2), which is 2.016, has the same number of atoms as 1 mole of silver, which is 107.87. This means that 2.016 grams of hydrogen (1.008×2) contain the same number of atoms as 107.87 grams of silver. A mole is the Avogadro's number of atoms, which is 6.02×10^{23} .
6. Now let's determine the number of moles in 230 grams of chlorine (Cl_2).
 - a) First we locate chlorine on the periodic table and then we get the atomic weight of chlorine, which is 35.45.
 - b) Second, because chlorine in the free state has the structure Cl_2 we use this to get $35.45 \times 2 = 70.9$
 - c) Second we simply divide 230 grams by 70.9, and we get 3.244; therefore 230 grams of chlorine equals 3.244 moles. Note: We use Cl_2 because that is what chlorine is in its free state. Second note: don't get confused and use chlorine atom in the equation, as mono Cl does not exist only Cl_2 .
6. Now let's determine the number of moles in 126 grams of pure distilled water.
 - a) First we simply determine the molecular weight of water which has the structure H_2O ; therefore we have hydrogen, $1.008 \times 2 = 2.016 + 15.999$ (a single oxygen atom) = 18.015 (molecular weight of water and 1 mole, meaning that 18.015 grams is one mole of pure water.
 - b) Now, we simply divide 126 grams by 18.015 = 6.994 moles of water in 126 grams of water.
7. Now let's determine how many moles are in 356 grams of $\text{K}_2\text{Cr}_2\text{O}_7$ (potassium dichromate).
 - a) As before, we must determine the molecular weight of potassium dichromate; therefore we have potassium = $39.1 \times 2 = 78.2$; Chromium = $52 \times 2 = 104$; and Oxygen = $15.999 \times 7 = 111.993$
 - b) Therefore we have $78.2 + 104 + 111.993 = 294.193$ (molecular weight of potassium dichromate)
 - c) Now we simply divide 356 grams by 294.193, which = 1.21 moles of potassium dichromate.
8. Let's calculate how many grams of sample there are in 1.67 moles of Potassium hydroxide.
 - a) First we calculate the molecular weight of potassium hydroxide; therefore, we have potassium = 39.10 + oxygen = 15.999 + hydrogen = 1.008, so we have 56.107 as the molecular weight of potassium hydroxide.

- b) Now to determine the mass in grams of 1.67 moles of potassium hydroxide we simply multiply 1.67 by molecular weight of potassium hydroxide, which is $56.107 = 93.698$ grams of potassium hydroxide.
9. Now lets get familiarized with balancing chemical reactions. Balance the following equation $\text{Al} + \text{O}_2 = \text{Al}_2\text{O}_3$
- a) Since aluminum is reacting with oxygen it forms aluminum oxide, because we have 2 aluminum atoms, and three oxygen atoms in the formula we need to balance the equation; therefore we would say $4\text{Al} + 3\text{O}_2 = 2\text{Al}_2\text{O}_3$. Notice how we have a total of 4 aluminum atoms, and 6 oxygen atoms on both sides of the equation.
10. Now lets balance another equation. $\text{NH}_3 + \text{O}_2 + \text{catalyst} = \text{NO}_2 + \text{H}_2\text{O}$
- a) In this equation we need to balance out the ammonia and oxygen so we have the same number of atoms on each side of the equation especially because we have water as a by-product in the reaction.
- b) Therefore we would say $4\text{NH}_3 + 7\text{O}_2 + \text{catalyst (platinum)} = 4\text{NO}_2 + 6\text{H}_2\text{O}$. Notice how there is the same number of atoms on both sides of the equation. Note: the catalyst does not need to be balanced as it is unchanged during the process and remains the same.
11. Finally, lets balance out this equation. $\text{MnO}_2 + \text{HCl} = \text{MnCl}_2 + \text{Cl}_2 + \text{H}_2\text{O}$
- a) In this equation we do like the previous two, we simply need to balance the equation so it makes sense; therefore we would say $\text{MnO}_2 + 4\text{HCl} = \text{MnCl}_2 + \text{Cl}_2 + 2\text{H}_2\text{O}$
12. Determine how many grams of sodium hydroxide are in 1.45 moles of sodium hydroxide.
- a) First we must determine the molecular weight of sodium hydroxide; therefore, we have sodium = 22.99; oxygen = 15.999; and hydrogen = 1.008; therefore we have $22.99 + 15.999 + 1.008 = 39.997$
- b) Now, we simply multiply the molecular weight of sodium hydroxide with 1.45 moles so we get $39.997 \times 1.45 = 57.995$ grams of sodium hydroxide in 1.45 moles of sodium hydroxide.
13. Preparing solutions. Lets say we want to make 1000 grams of a 25% by weight solution of potassium permanganate in water. How many grams of potassium permanganate do we need to add to how much water?
- a) Since water has a density of 1.0, one milliliter of water weighs approximately 1 gram; therefore, to make 1000 grams of a 25% potassium permanganate solution all we need to do is multiply 0.25 (the decimal percentage of 25%) by 1000 (in grams) = 250 grams. Finally we simply subtract 1000 by 250 = 750 milliliters of water. So, to make 1000 grams of a 25% potassium permanganate solution we dissolve 250 grams of potassium permanganate into 750 milliliters of water.
14. Preparing solutions. Lets say we want to make 560 grams of a 5% by weight sodium carbonate solution in water.
- a) Since water has a density of 1.0, one milliliter of water weighs approximately 1 gram; therefore we simply multiply 0.05 (the decimal percentage of 5%) by 560 = 28 grams of sodium carbonate. Now, we simply subtract 560 from 28 = 532 milliliters of water. So to make 560 grams of a 5% sodium carbonate solution, we simply dissolve 28 grams of sodium carbonate into 532 milliliters of water.
15. Preparing solutions. Lets say we want to make a total of 870 grams of aqueous solution containing 15% by weight sodium chlorate, and 10% by weight sodium sulfate. Since water has a density of 1.0, one milliliter of water weighs approximately 1 gram.
- a) Step 1: Determine the number of grams of sodium chlorate so we multiply 0.15 (the decimal percentage of 15) by 870 = 130.5 grams
- b) Step 2: Determine the number grams of sodium sulfate so we multiply 0.10 (the decimal percentage of 10%) by 870 = 87 grams
- c) Step 3: All we need to do now is add up 130.5 grams sodium chlorate with the 87 grams of sodium sulfate = 217.5 grams, then we subtract this number from 870 = 652.5 milliliters of water. Therefore to prepare 870 grams of this solution all we need to do is dissolve 130.5 grams of sodium chlorate, and 87 grams of sodium sulfate into 652.5 milliliters of water.
16. Preparing solutions. How many grams of sodium chloride would be in 50 grams of a 30% by weight sodium chloride solution in water?
- a) First, since water has a density of 1.00 at stp, all we need to do is multiply 0.30 (representing the decimal percentage of 30%) by 50 = 15 grams of sodium chloride in 50 grams of a 30% solution.
17. Calculate Molarity of solution. What is the Molarity of a solution that has a sample weight of 100, the sample has a molecular weight of 123, and the solvent used has a volume of 450 milliliters.
- a) $(100)/(123)/(450 \times 10^{-3}) = 1.80066$
- b) Note: type in the above equation exactly as written, a graphing calculator works best.
18. Calculate a chemical reaction. We have 45 grams of sulfur, and we want to know how many grams of oxygen it will take to react with the sulfur to form sulfur dioxide.
- a) First we must balance the equation; therefore we have $\text{S} + \text{O}_2 = \text{SO}_2$
- b) Second we determine the molecular weights of sulfur and oxygen, so we have sulfur = 32.06, and we have oxygen = $15.999 \times 2 = 31.998$
- c) Now, take the mass of sulfur which is 45 grams = x1; then we have the molecular weight of known sample, which is 32.06 = x2; then we have moles of known sample which is $1 = x3$; then we have the molecular weight of the unknown oxygen sample is 31.998 = x4; finally we have moles of unknown oxygen sample, which is $1 = x5$.

- d) Now we set up the equation: $(x1)x(1)/(x2)x(x5)/(x3)x(x4)/(1) = 44.91$ grams of oxygen. Note: enter the equation exactly as written using preferably a graphing calculator.
19. Calculate a chemical reaction. We have 24 grams of aluminum, and we want to know how many grams of dry hydrogen chloride gas it will take to react with the aluminum to form aluminum chloride and hydrogen gas.
- First we must balance the equation; therefore we have $2Al + 6HCl = 2AlCl_3 + 3H_2$
 - Second we determine the molecular weights of aluminum and hydrogen chloride, so we have aluminum = 26.982, and we have hydrogen chloride is chlorine = 35.45 + hydrogen = 1.008, so we have 35.45 + 1.008 = 36.458.
 - Now, take the mass of aluminum which is 24 grams = x1; then we have the molecular weight of known sample, which is 26.982 = x2; then we have moles of known sample which is 2 (Al) = x3; then we have the molecular weight of the unknown hydrogen chloride gas sample is 36.458 = x4; finally we have moles of unknown hydrogen chloride gas sample, which is 6 (HCl) = x5.
 - Now we set up the equation: $(x1)x(1)/(x2)x(x5)/(x3)x(x4)/(1) = 97.286$ grams of hydrogen chloride. Note: enter the equation exactly as written using preferably a graphing calculator.
20. Calculate a chemical reaction. We have 123 grams of magnesium sulfate, and we want to know how many grams of sodium hydroxide it will take to react with the magnesium sulfate to form magnesium hydroxide.
- First we must balance the equation; therefore we have $MgSO_4 + 2NaOH = Mg(OH)_2 + Na_2SO_4$
 - Second we determine the molecular weights of magnesium sulfate and sodium hydroxide, so we have magnesium sulfate = magnesium, 24.305 + sulfur, 32.06 + oxygen, 15.999 x 4 = 120.361, and we have sodium hydroxide = sodium, 22.99 + oxygen, 15.999 + hydrogen, 1.008 = 39.997; therefore the molecular weight of magnesium sulfate is 120.361, and that of sodium hydroxide is 39.997.
 - Now, take the mass of magnesium sulfate which is 123 grams = x1; then we have the molecular weight of known magnesium sulfate sample, which is 120.361 = x2; then we have moles of known magnesium sulfate sample which is 1 ($MgSO_4$) = x3; then we have the molecular weight of the unknown sodium hydroxide sample is 39.997 = x4; finally we have moles of unknown sodium hydroxide sample, which is 2 (NaOH) = x5.
 - Now we set up the equation: $(x1)x(1)/(x2)x(x5)/(x3)x(x4)/(1) = 81.747$ grams of sodium hydroxide. Note: enter the equation exactly as written using preferably a graphing calculator.
21. Determine the amount in grams of sodium in 129.6 grams of sodium chloride, NaCl.
- First we calculate the molecular weight of sodium chloride which 22.99 (sodium) + 35.45 (chlorine) = 58.44
 - Second we multiply the atomic weight of sodium, which is 22.99 by the weight of sample sodium chloride, which is 129.6 grams, divide by the molecular weight of sample sodium chloride, which is 58.44; therefore we have $(22.99) \times (129.6)/58.44 = 50.98$ grams of sodium in 129.6 grams of sodium chloride.
22. Determine the amount in grams of potassium and oxygen in 345 grams of potassium dichromate, $K_2Cr_2O_7$.
- First we calculate the molecular weight of potassium dichromate in the usual manner; therefore we have potassium, which is $39.1 \times 2 = 78.2$; then we have chromium, which is $52 \times 2 = 104$; and then we have oxygen, which is $15.999 \times 7 = 111.993$; therefore we have a molecular weight of $78.2 + 104 + 111.993 = 294.193$.
 - Second we multiply the atomic weight of potassium, which is $39.1 \times 2 = 78.2$, by the weight of sample of potassium dichromate, which is 345 grams, then divide this by the molecular weight of potassium dichromate, which is 294.193; therefore we have $(39.1) \times (2) \times (345)/294.193 = 91.7$ grams of potassium in 345 grams of potassium dichromate.
 - Third, we multiply the atomic weight of oxygen, which is $15.999 \times 7 = 111.993$ by the weight of sample of potassium dichromate, which is 345 grams, then divide this by the molecular weight of potassium dichromate, which is 294.193; therefore we have $(15.999) \times (7) \times (345)/294.193 = 131.33$ grams of oxygen in 345 grams of potassium dichromate.
23. At Standard Temperature and Pressure, pure water has a density of 1.00; therefore, one milliliter of water weighs approximately 1 gram. Going on standard pressure and temperature what would be the weight of 150 milliliters of pure methylene chloride?
- First we must look up the density of methylene chloride using the Merck index, or any other desired source, and we see that the density of methylene chloride is 1.3
 - Therefore, we simply multiply 1.3 by 150 = 195 grams.
24. At standard Temperature and pressure, what would be the weight of 230 milliliters of pure isopropyl alcohol?
- First we must look up the density of Isopropyl alcohol using the Merck index, or any other desired source, and we see that the density of isopropyl alcohol is 0.78.
 - Therefore we simply multiply 0.78 by 230 = 179.4 grams
25. At standard temperature and pressure we have 156 grams of pure acetone. What would be the volume in milliliters?
- First we find the density of acetone at STP, which is 0.788; therefore, we simply divide 156 grams by 0.788 = 197.96 milliliters of acetone at STP.
26. Calculate each weight in grams, of 220 milliliters of a mixture of 50% acetone, and 50% water by volume.

- a) First we gather the densities of both liquids; therefore we have water = 1.00, and acetone = 0.788.
 - b) Second we calculate the weight in grams of acetone so we multiply 0.50 (the percentage in decimal) by 220 = 110. Then we multiply 0.788 by 110 = 86.68 grams of acetone in 220 milliliters of 50% acetone, and 50% water solution.
 - c) Thirdly, we simply subtract 220 by 86.68 grams = 133.32 grams of water.
27. Calculate each weight in grams, of 309 milliliters of a mixture of 23% ethanol, and 77% methylene chloride by volume.
- a) First we gather the densities of both liquids at STP, so we get ethanol = 0.789, and that of methylene chloride = 1.3
 - b) Second, we calculate the weight in grams of ethanol so we multiply 0.23 (the percentage in decimal) by 309 = 71.07. Then we multiply 71.07 by 0.789 = 56.07 grams ethanol in 309 milliliters of ethanol/methylene chloride mixture.
 - c) Thirdly, we subtract 309 by 56.07 grams = 252.92 grams of methylene chloride.
28. Calculate chemical reaction using mole-to-mole theory. Calculate the reaction of 123 grams of Barium carbonate (BaCO_3) with an unknown amount of concentrated sulfuric acid.
- a) First we setup the equation and balance it out. Thus we have $1\text{BaCO}_3 + 1\text{H}_2\text{SO}_4 = 1\text{BaSO}_4 + 1\text{CO}_2 + 1\text{H}_2\text{O}$ (notice how the number of atoms is identical on both sides of the equation).
 - b) Now, we calculate how many moles are in 123 grams of barium carbonate, so we must first determine the molecular weight of barium carbonate, which is Barium = 137.33; Carbon = 12.011; and Oxygen is $15.999 \times 3 = 47.997$; therefore we have $137.33 + 12.011 + 47.997 = 197.338$ molecular weight of barium carbonate.
 - c) Now, we take the mass of barium carbonate of sample, which is 123 and divide it by the molecular weight of barium carbonate, which is 197.338; thus we have, $123 \text{ divide by } 197.338 = 0.623$ moles.
 - d) Now, since our original equation is already balanced, meaning 1 mole of barium carbonate with 1 mole of sulfuric acid, we must calculate the weight in grams of 0.623 moles of concentrated sulfuric acid; therefore we tally the molecular weight of sulfuric acid, which is sulfur = 32.06; hydrogen = $1.008 \times 2 = 2.016$; and oxygen = $15.999 \times 4 = 63.996$; therefore the molecular weight of sulfuric acid is $32.06 + 2.016 + 63.996 = 98.072$
 - e) Now to determine the weight of 0.623 moles of sulfuric acid, we simply multiply 0.632 by the molecular weight of sulfuric acid, so we have $0.623 \times 98.072 = 61.098$ grams of sulfuric acid to react with 123 grams of barium carbonate.
29. Calculate chemical reaction using mole-to-mole theory. Calculate the reaction of 203 grams sodium chloride (NaCl) with an unknown amount of concentrated sulfuric acid.
- a) First we setup the equation and balance it out. Thus we have $2\text{NaCl} + 1\text{H}_2\text{SO}_4 = 2\text{HCl} + 1\text{Na}_2\text{SO}_4$ (notice how the number of atoms is identical on both sides of the equation).
 - b) Now, we calculate how many moles are in 203 grams of sodium chloride, so we must first determine the molecular weight of sodium chloride, which is Sodium = 22.99; and Chlorine = 35.45; therefore we have $22.99 + 35.45 = 58.44$ molecular weight of sodium chloride.
 - c) Now, we take the mass of sodium chloride of sample, which is 203 grams and divide it by the molecular weight of sodium chloride, which is 58.44; thus we have, $203 \text{ divide by } 58.44 = 3.47$ moles.
 - d) Now comes the confusing part; even though 203 grams of sodium chloride is 3.47 moles, we have a 2 next to the NaCl in the equation. This means we must divide 3.47 moles by 2 = 1.735 moles
 - e) Now, since our original equation is already balanced, meaning 2 moles of sodium chloride with 1 mole of sulfuric acid, we must calculate the weight in grams of the new mole, which is 1.735 moles of concentrated sulfuric acid; therefore we tally the molecular weight of sulfuric acid, which is sulfur = 32.06; hydrogen = $1.008 \times 2 = 2.016$; and oxygen = $15.999 \times 4 = 63.996$; therefore the molecular weight of sulfuric acid is $32.06 + 2.016 + 63.996 = 98.072$
 - f) Now to determine the weight of 1.735 moles of sulfuric acid, we simply multiply 1.735 moles by the molecular weight of sulfuric acid, so we have $1.735 \times 98.072 = 170.15$ grams of sulfuric acid to react with 203 grams of sodium chloride.
30. Calculate chemical reaction using mole-to-mole theory. Calculate the reaction of 45 grams of aluminum with an unknown amount of hydrogen chloride.
- a) First we setup the equation and balance it out. Thus we have $2\text{Al} + 6\text{HCl} = 2\text{AlCl}_3 + 3\text{H}_2$ (notice how the number of atoms is identical on both sides of the equation).
 - b) Now, we calculate how many moles are in 45 grams of aluminum, so in this case, we must first determine the atomic weight of aluminum, which is 26.982.
 - c) Now, we take the mass of aluminum of sample, which is 45 grams and divide it by the atomic weight of aluminum, thus we have $45 \text{ divide by } 26.982 = 1.6677$ moles of aluminum.
 - d) Now comes the confusing part; even though 45 grams of aluminum is 1.6677 moles, we have a 2 next to the Al (2Al) in the equation. This means we must divide 1.6677 moles by 2 = 0.83385.

- e) Now comes the second confusing yet crucial command. Since our original equation is already balanced, meaning 2 moles of aluminum with 6 moles of hydrogen chloride, we must multiply 0.83385 (from step d) by 6 (6HCl) = 5.0031.
- f) Now we tally the molecular weight of hydrogen chloride, which is hydrogen = 1.008; and chlorine = 35.45; therefore the molecular weight of hydrogen chloride is $1.008 + 35.45 = 36.458$
- g) Now we determine the weight of hydrogen chloride by multiplying 5.0031 (from step e) by the molecular weight of hydrogen chloride, thus we have $5.0031 \times 36.458 = 182.4$ grams of hydrogen chloride to react with 45 grams of aluminum.

Chapter 5: Chemical mixtures

Chemical mixtures are substances containing a mixture of ingredients, rather than a single entity. Chemical mixtures are widely found not only in the synthetic world, but in the natural world as well. Trees, plants, paints, foodstuffs, grasses, seeds, roots, ect., all contain simple to complex mixtures of various individual compounds. In many cases, these individual compounds can be extracted and isolated if need be. In essence, chemical mixtures contain a mixture of chemical compounds (either natural or manmade) that coexist in a uniform manner forming either a solid, liquid, or semi-solid phase. All chemical mixtures are composed of various chemical compounds, all of which can be separated and isolated from said mixture. Also, chemical mixtures contain chemical compounds that do not react with one another.

I. Commercial and Industrial Application Compositions

001. Commercial cosmetic lotion composition for face cleansing:

Into a suitable empty ball mill, place **25 grams of potassium nitrate**, followed by **75 grams of tincture of benzoin**. Thereafter, tumble the mixture at 75 RPM for about 30 minutes. Thereafter, into a separate empty ball mill, place **100 grams of cucumber juice**, followed by **50 grams of cologne spirits**, and then followed by **275 grams of elderflower water**. Thereafter, tumble the mixture at 100 RPM for about 1 hour. Finally, place both tumbled mixtures into a suitable mixing vessel equipped with motorized stirrer, and then blend the combined mixture for about 45 minutes to form a uniform mix. Thereafter, place the combined mixture into a suitable container for storage, sealed airtight.

Percentage: 52.38% elderflower water, 19.04% cucumber juice, 14.28% tincture of benzoin, 9.52% cologne spirits, 4.76% potassium nitrate, 0.02% balance

002: Commercial cosmetic cream composition for face cleansing:

Into a standard mixing bowl, equipped with motorized stirrer utilizing a plastic stir blade, place **136 grams of white beeswax**, followed by **37 grams of spermaceti compound**, followed by **240 milliliters of distilled water**, followed by **136 grams of sweet whole milk**, followed by **544 grams of sweet almond oil**, followed by **1 gram of borax**, and then finally followed by **6 grams of flower perfume**, and then blend the mixture on high speed for about 1 hour to form a uniform blend. After the blending operation, the mixture should be stored in a suitable airtight container for storage.

Percentage: 49.45% sweet almond oil, 21.81% distilled water, 12.36% white bees wax, 12.36% sweet whole milk, 3.36% spermaceti compound, 0.54% flower perfume, 0.09% borax, 0.03% residual balance

003A: Commercial cosmetic vanishing cream composition for facial use:

Into a standard mixing bowl, equipped with motorized stirrer utilizing a plastic stir blade, place **2600 grams of stearic acid**, followed by **100 grams of potassium hydroxide**, and then followed by **300 grams of coconut oil**. Thereafter, blend the mixture for about 30 minutes to form a uniform mixture. Thereafter, add in **12 liters of warm water**, and then continue to blend the mixture for about 30 minutes. After the blending operation, the mixture should be stored in a suitable airtight container for storage.

Percentage: 80% water, 17.33% stearic acid, 2% coconut oil, 0.66% potassium hydroxide, 0.01% mixed residual balance

003B: Commercial cosmetic vanishing cream composition for facial use:

Into a standard mixing bowl, equipped with motorized stirrer utilizing a plastic stir blade, place **1200 grams of stearic acid**, followed by **900 grams of sodium carbonate decahydrate**, and then followed by **500 grams of butyl stearate**. Thereafter, blend the mixture for about 30 minutes to form a uniform mixture. Thereafter, add in **3600 milliliters of warm water**, followed by **60 grams of borax**, and then continue to blend the mixture for about 30 minutes. After the blending operation, the mixture should be stored in a suitable airtight container for storage.

Percentage: 57.5% water, 19.16% stearic acid, 14.37% sodium carbonate decahydrate, 7.98% butyl stearate, 0.95% borax, 0.04% mixed residual balance

004A: Commercial cosmetic composition for facial use “smooth ointment”:

Into a suitable heat resistant container, such as a glass beaker, place **250 grams of white petrolatum**, and then gently heat the mixture using a Bunsen burner or electric hot plate until the wax melts. Once the wax has melted, add in **750 grams of a sulfurated hydrogenated castor oil** having a value of 3 and containing 10% sulfur trioxide, and then blend the total mixture on high speed for about 30 minutes while continuing to heat the mixture. After 30 minutes, remove the heat source, and allow the mixture to cool. During this cool down period, continue to blend the mixture. Once the mixture has cooled, it is ready to be used.

Percentage: 75% sulfurated hydrogenated castor oil, 25% petrolatum wax compound

004B: Commercial cosmetic composition for facial use “Belladonna ointment”:

Into a suitable heat resistant container, such as a glass beaker, place **250 grams of white petrolatum**, and then gently heat the mixture using a Bunsen burner or electric hot plate until the wax melts. Once the wax has melted, add in **250 grams of a sulfurated hydrogenated castor oil** having a value of 3 and containing 10% sulfur trioxide, and then blend the mixture for about 30 minutes. Thereafter, or in the meantime, into a suitable container, equipped with a motorized stirrer, place **400 milliliters of deionized water**, followed by **100 grams of Pilular extract of Belladonna**, and then blend the mixture for about 10 minutes at room temperature. After blending the petrolatum wax mixture for about 30 minutes, slowly add in, in small portions at a time, the water and Belladonna extract mixture, while continuing to heat and blending the total mixture for about 45 minutes. After blending the combined mixture for about 45 minutes, remove the heat source, and allow the mixture to cool. During this cool down period, continue to blend the mixture. Once the mixture has cooled, it is ready to be used.

Percentage: 40% deionized water, 25% sulfurated hydrogenated castor oil, 25% petrolatum wax compound, 10% Belladonna extract

005: Commercial cosmetic cream composition for facial use:

Into a suitable heat resistant container, such as glass beaker, place **150 grams of white wax**, and then gently heat the mixture using a Bunsen burner or electric hot plate until the wax melts. Once the wax has melted, add in **500 grams of liquid petrolatum**, and then blend the mixture for about 30 minutes. At the same time, into a separate clean container equipped with motorized stirrer, place **300 milliliters of hot deionized water**, and then followed by **10 grams of a sulfurated hydrogenated castor oil** having a value of 3 and containing 10% sulfur trioxide, and then blend the mixture for about 30 minutes. After blending the wax mixture for about 30 minutes, slowly add in, in small portions at a time, the water and castor oil mixture, while continuing to heat and blending the total mixture for about 45 minutes. After blending the combined mixture for about 45 minutes, remove the heat source, and allow the mixture to cool. During this cool down period, continue to blend the mixture. Once the mixture has cooled, it is ready to be used.

Percentage: 52.08% liquid petrolatum, 31.25% deionized water, 15.625% white wax, 1.04% sulfurated hydrogenated castor oil, 0.005% residual balance

006: Commercial cosmetic cream composition for facial use “liquefying cleansing cream”:

Into a suitable heat resistant container, such as glass beaker, place **10 grams of hydroxystearic acid**, and then gently heat the mixture using a Bunsen burner or electric hot plate until the material melts. Once the material has melted, add in **150 grams of spermaceti**, and then blend the mixture for about 30 minutes. Thereafter, add in **300 grams of peach kernel oil**, followed by **50 grams of oleyl alcohol**, and then continue to heat and blend the mixture for about 15 minutes. Thereafter into a separate clean container equipped with motorized stirrer, place **400 milliliters of hot deionized water**, and then add **100 grams of a sulfurated hydrogenated castor oil** having a value of 3 and containing 10% sulfur trioxide, and then blend the mixture for about 30 minutes. After blending the hydroxystearic acid/spermaceti mixture for about 30 minutes, slowly add in, in small portions at a time, the water and castor oil mixture, while continuing to heat and blending the total mixture for about 45 minutes. After blending the combined mixture for about 45 minutes, remove the heat source, and allow the mixture to cool. During this cool down period, continue to blend the mixture. Once the mixture has cooled, it is ready to be used.

Percentage: 39.6% deionized water, 29.7% peach kernel oil, 14.85% spermaceti, 9.9% sulfurated hydrogenated castor oil, 4.95% oleyl alcohol, 0.99% hydroxystearic acid, 0.01% residual balance

007: Commercial cosmetic vanishing cream composition for facial use:

Into a suitable heat resistant container, such as glass beaker, place **150 grams of glyceryl monostearate**, followed by **150 grams of stearic acid**. Thereafter, gently heat the mixture using a Bunsen burner or electric hot plate until both materials melt. In the meantime, prepare a solution by adding and mixing **100 grams of a sulfurated hydrogenated castor oil** having a value of 3 and containing 10% sulfur trioxide, and **10 grams of tri-isopropanolamine**, into **600 milliliters of hot water**. Thereafter, blend this water mixture on moderate speed for about 10 minutes. Then, to the melted mixture first prepared, slowly add in, in small portions at a time, the water and castor oil mixture, while blending the melted mixture on moderate speed until all the water mixture has been added. After all the water mixture has been added to the melted mixture, blend the combined mixture for about 45 minutes while continuing to heat the combined mixture. After blending the combined mixture for 45 minutes, remove the heat source, and allow the mixture to cool. During this cool down period, continue to blend the mixture. Once the mixture has cooled, it is ready to be used.

Percentage: 59.4% water, 14.85% glyceryl monostearate, 14.85% stearic acid, 9.9% sulfurated hydrogenated castor oil, 0.99% tri-isopropanolamine, 0.01% mixed balance

008: Commercial cosmetic sunburn cream composition for facial use:

Into a suitable heat resistant container, such as glass beaker, place **100 grams of glyceryl monostearate**, followed by **200 grams of petrolatum**. Thereafter, gently heat the mixture using a Bunsen burner or electric hot plate until both materials melt. In the meantime, prepare a solution by adding and mixing **200 grams of a sulfurated hydrogenated castor oil** having a value of 3 and containing 10% sulfur trioxide, into **400 milliliters of hot water**. Thereafter, blend this water mixture on moderate speed for about 10 minutes. Then, to the melted mixture first prepared, slowly add in, in small portions at a time, the water and castor oil mixture, while blending the melted mixture on moderate speed until all the water mixture has been added. After all the water mixture has been added to the melted mixture, blend the combined mixture for about 45 minutes while continuing to heat the combined mixture. After blending the combined mixture for 45 minutes, remove the heat source, and allow the mixture to cool. During this cool down period, continue to blend the mixture. Once the mixture has cooled, it is ready to be used.

Percentage: 44.44% water, 22.22% sulfurated hydrogenated castor oil, 22.22% petrolatum, 11.11% glyceryl monostearate, 0.01% mixed balance

009: Commercial cosmetic liquid vanishing cream:

Into a suitable mixing container, equipped with motorized stirrer, place **345 milliliters of deionized water**, followed by **5 grams of perfume**, of any desired odor, followed by **50 grams of triethanolamine stearate**. Thereafter, blend the mixture for about 30 minutes to form a uniform mixture. Thereafter, add in **50 grams of 2-hexyldecanol**, and then followed by **50 grams of paraffin oil**. Thereafter, blend the mixture on moderate speed for about 45 minutes to form a uniform mass. Thereafter, the mixture is ready for use.

Percentage: 69% deionized water, 10% triethanolamine stearate, 10% paraffin oil, 10% 2-hexyldecanol, 1% perfume

010: Commercial cosmetic skin oil:

Into a suitable mixing container, equipped with motorized stirrer, place **100 grams of paraffin oil**, followed by **200 grams of almond oil**, followed by **690 grams of 2-butyloctadecanol**, and then followed by **10 grams of perfume**, of any desired odor. Thereafter, blend the mixture for about 30 minutes to form a uniform mixture. Thereafter, the mixture is ready for use.

Percentage: 69% 2-butyloctadecanol, 20% almond oil, 10% paraffin oil, 1% perfume

011: Commercial cosmetic skin powder:

Into a suitable empty ball mill, filled with 500 grams of steel shot of any desired diameter, place **250 grams of talcum powder**, followed by **200 grams of kaolin**, followed by **25 grams of magnesium stearate**, followed by **5 grams of zinc oxide**, and then followed by **20 grams of 2-decyldodecanol**. Thereafter, tumble the mixture at 250 RPM for about 4 hours. Thereafter, remove the pulverized powdered mixture from the steel shot by straining it through a metal screen. Once the powder has been screened and collected it should be stored in airtight containers. Thereafter, the mixture is ready for use.

Percentage: 50% talcum, 40% kaolin 5% magnesium stearate, 4% 2-decyldodecanol, 1% zinc oxide

012: Commercial cosmetic hair oil:

Into a suitable empty ball mill, filled with 500 grams of steel shot of any desired diameter, place **500 grams of 2-hexyldecanol**, and then followed by **5 grams of perfume**, of any desired odor. Thereafter, tumble the mixture at 50 RPM for about 2 hours. Thereafter, remove the pulverized mixture from the steel shot by straining it through a metal screen. Once the mixture has been screened and collected, place it into any desired mixing container equipped with motorized stirrer, and then add in **300 grams of paraffin oil**, and then followed by **200 grams of isopropyl alcohol**. Thereafter, blend the mixture on moderate speed for about 45 minutes. After blending for about 45 minutes, the mixture is ready for use.

Percentage: 49.75% 2-hexyldecanol, 29.85% paraffin oil, 19.9% isopropyl alcohol, 0.49% perfume, 0.01% mixed balance

013: Commercial cosmetic eye makeup removing lotion:

Into a suitable empty ball mill, filled with 500 grams of steel shot of any desired diameter, place **10 grams of dry sarsaparilla extract**, followed by **2 grams of potassium dihydrogen phosphate**, followed by **8 grams of potassium hydrogen phosphate tri hydrate**, followed by **600 milligrams of sodium benzoate**, and then followed by **1.4 grams of monochloroacetamide**.

Thereafter, tumble the mixture at 150 RPM for about 2 hours. Thereafter, remove the pulverized mixture from the steel shot by straining it through a metal screen. Once the mixture has been screened and collected, place it into any desired mixing container equipped with motorized stirrer, and then add in **20 grams of hexylene glycol**, followed by **1.6 grams of chlorohexidine dihydrochloride**, and then followed by **2 liters of deionized water**. Thereafter, blend the mixture on moderate speed for about 45 minutes. After blending for about 45 minutes, the mixture is ready for use.

Percentage: 97.86% deionized water, 0.97% hexylene glycol, 0.48% sarsaparilla extract, 0.39% potassium hydrogen phosphate, 0.097% potassium dihydrogen phosphate, 0.07% chlorohexidine dihydrochloride, 0.068% monochloroacetamide, 0.036% residual balance, 0.029% sodium benzoate

014: Commercial cosmetic hair treatment composition:

Into a suitable empty ball mill, filled with 50 grams of steel shot of any desired diameter, place *120 grams of Epsom salt*, followed by *140 grams of sodium bicarbonate*, followed by *70 grams of pulverized sea kelp*, and then followed by *70 grams of hask placenta*. Thereafter, tumble the mixture at 250 RPM for about 1 hour. Thereafter, remove the pulverized mixture from the steel shot by straining it through a metal screen. Once the mixture has been screened and collected, place it into any desired mixing container equipped with motorized stirrer, and then add in *125 grams of apple cider vinegar (5% acidity)*, and then followed by *650 milliliters of deionized water*. Thereafter, blend the mixture on moderate speed for about 45 minutes. After blending for about 45 minutes, the mixture is ready for use.

Percentage: *55.31% deionized water, 11.91% sodium bicarbonate, 10.63% apple cider vinegar, 10.21% Epsom salt, 5.95% pulverized sea kelp, 5.95% hask placenta, 0.04% mixed residual balance*

015: Commercial nasal spray composition:

Into a suitable mixing vessel, equipped with motorized stirrer, place *12 liters of purified water*, and then add in *5 grams of oxymetazoline hydrochloride*, followed by *200 milligrams of phenylmercuric acetate*, followed by *2 grams of benzalkonium chloride*, followed by *37 grams of glycine*, followed by *570 grams of a 70% sorbitol solution*, and then followed by *35 grams of cherry flavor*, and then blend the entire mixture for about 45 minutes. Thereafter, the mixture is ready for use.

Percentage: *94.86% purified water, 4.5% sorbitol solution, 0.29% glycine, 0.27% cherry flavor, 0.039% oxymetazoline hydrochloride, 0.0245% mixed residual balance, 0.015% benzalkonium chloride, 0.0015% phenylmercuric acetate*

016: Commercial body rubbing composition to relieve aches and pains:

Into a suitable mixing vessel, equipped with motorized stirrer, place *300 grams of methyl salicylate*, and then add in *200 grams of olive oil*, and then blend the mixture at room temperature for about 15 minutes to form a uniform mass. Thereafter, add in, in small portions at a time, *200 grams of oil of eucalyptus* while rapidly blending the mixture during the addition. Once all the eucalyptus oil has been added, add in *300 grams of isopropyl alcohol* all at once. Thereafter, blend the total mixture on moderate speed for about 1 hour to form a fine consistency. Thereafter, the mixture is ready for use.

Percentage: *30% methyl salicylate, 30% isopropyl alcohol, 20% oil of eucalyptus, 20% olive oil*

017: Commercial shower gel composition:

Into a suitable mixing vessel, equipped with motorized stirrer, place *400 grams of sodium lauryl ether sulphate*, followed by *100 grams of sodium coco amido propyl betaine*, followed by *500 grams of alkylpolyglucoside*, followed by *37 grams of sorbic acid*, and then blend the mixture on moderate speed for about 15 minutes. Thereafter, add in *49 grams of trisodium citrate dihydrate*, followed by *90 grams of cross-linked polyacrylate*, and then followed by *25 grams of sodium hydroxide*, and then continue to blend the mixture on moderate speed for about 1 hour. Finally, add in *1000 milliliters of purified water*, and then continue to blend the mixture for about 45 minutes to form a uniform mass. Thereafter, the mixture is ready for use.

Percentage: *45.43% purified water, 22.71% alkylpolyglucoside, 18.17% sodium lauryl ether sulphate, 4.54% sodium coco amido propyl betaine, 4.08% cross linked polyacrylate, 2.22% trisodium citrate dihydrate, 1.68% sorbic acid, 1.13% sodium hydroxide, 0.04% mixed residual balance*

018: Commercial facial cleansing and moisturizing composition:

Into a suitable mixing bowl, equipped with motorized stirrer, place *234 grams of MEA sulphosuccinate*, followed by *2000 milliliters of deionized water*. Thereafter blend the mixture on high speed for about 15 minutes. Thereafter, add in *6 grams of a cross-linked polycarylate*, followed by *170 grams of sodium cocoyl isethionate*, and then followed by *316 grams of coco amidopropyl betaine*. Thereafter, continue to blend the mixture for about 1 hour to form a uniform mass. After the blending operation, the mixture is ready for use.

Percentage: *73.36% deionized water, 11.59% coco amidopropyl betaine, 8.58% MEA sulphosuccinate, 6.23% sodium cocoyl isethionate, 0.22% cross-linked polycarylate, 0.02% mixed residual balance*

II. Foodstuffs Compositions

001: Commercial edible composition:

Into a suitable clean and sterilized mixing bowl, equipped with motorized stirrer of the same, place *500 milliliters of purified boiled water*, and then add in *150 grams of confectioners sugar*, and then stir the mixture on moderate speed to dissolve all the sugar. Thereafter, add in *1.5 grams of gellan gum*, followed by *1 gram of locust bean gum*, followed by *1.6 grams of sodium citrate*, and then blend the mixture for about 45 minutes. Thereafter, add in *500 milligrams of calcium lactate*, followed by *1.5 grams of citric acid*, and then followed by *50 grams of maltodextrin*. Thereafter, blend the mixture on moderate speed for about 45 minutes. After the blending operation, the mixture is ready for use. To use, the mixture should be pressed into molds of any desired shape, and then cured in an oven for a suitable period of time. Thereafter, the mixture is ready for consumption.

Percentage: *70.81% purified water, 21.24% confectioners sugar, 7.08% maltodextrin, 0.22% sodium citrate, 0.21% gellan gum, 0.21% citric acid, 0.14% locust bean gum, 0.07% calcium lactate, 0.02% mixed residual balance*

002: Commercial edible composition:

Into a suitable clean and sterilized mixing bowl, equipped with motorized stirrer of the same, place **1000 milliliters of purified boiled water**, and then add in **350 grams of cane sugar**, and then stir the mixture on moderate speed to dissolve all the sugar. Thereafter, add in **2.5 grams of gellan gum**, followed by **2 grams of locust bean gum**, followed by **2 grams of sodium citrate**, and then blend the mixture for about 45 minutes. Thereafter, add in **500 milligrams of calcium chloride**, and then followed by **2.5 grams of Carrageenan**. Thereafter, blend the mixture on moderate speed for about 45 minutes. After the blending operation, the mixture is ready for use. To use, the mixture should be pressed into molds of any desired shape, and then cured in an oven for a suitable period of time. Thereafter, the mixture is ready for consumption.

Percentage: 73.55% purified water, 25.74% sugar, 0.18% gellan gum, 0.18% Carrageenan, 0.14% locust bean gum, 0.14% sodium citrate, 0.036% calcium chloride, 0.034% mixed residual balance

003: Commercial edible dairy dessert composition:

Into a suitable clean and sterilized mixing bowl, equipped with motorized stirrer of the same, place **300 grams of Gelatine**, and then add in **3000 grams of cane sugar**, and then stir the mixture on moderate speed for about 45 minutes. Thereafter, add in **2000 grams of fresh Cream of 35%**, followed by **2000 grams of skim milk powder**, and then continue to blend the mixture on moderate speed for about 30 minutes. Thereafter, add in **2000 grams of regular skim milk**, and then blend the mixture for another 30 minutes. Thereafter, add in **15 grams of calcium lactate**, followed by **30 grams of sodium hexametaphosphate**, and then followed by **30 grams of Gellan gum**. Thereafter, blend the mixture on moderate speed for about 45 minutes. After the blending operation, the mixture is ready for use. To use, the mixture should be pressed into molds of any desired shape, and then cured in an oven for a suitable period of time. Thereafter, the mixture is ready for consumption.

Percentage: 32% cane sugar, 21.33% skim milk powder, 21.33% regular skim milk, 21.33% fresh cream, 3.2% gelatine, 0.32% Gellan gum, 0.32% sodium hexametaphosphate, 0.16% calcium lactate, 0.01% mixed balance

004: Commercial edible “two part” dairy dessert milk chocolate composition:

Into a suitable clean and sterilized mixing bowl, equipped with motorized stirrer of the same, place **44 grams of fresh cream**, and then add in **58 grams of skim milk powder**, followed by **600 grams of skim milk**, and then blend the entire mixture for about 30 minutes to form a uniform mixture. Thereafter, add in **120 grams of sugar**, followed by **60 grams of deep chocolate product**, and then blend the mixture on moderate speed for about 15 minutes. Thereafter, add in **15 grams of cocoa powder**, followed by **2 grams of Gellan gum**, followed by **30 grams of starch**, and then followed by **1 gram of calcium lactate**, and then followed by **2 grams of sodium hexametaphosphate**, and then continue to blend the entire mixture on moderate speed for about 50 minutes. Thereafter, the mixture is ready for use.

Percentage: 64.37% skim milk, 12.87% sugar, 6.43% chocolate, 6.22% skim milk powder, 4.72% fresh cream, 3.21% starch, 1.6% cocoa powder, 0.21% Gellan gum, 0.21% sodium hexametaphosphate, 0.10% calcium lactate, 0.06% mixed residual balance

005: Commercial edible “cookie confection” composition:

Into a suitable clean and sterilized mixing bowl, equipped with motorized stirrer of the same, place **1180 grams of flour**, followed by **462 grams of sugar**, followed by **31 grams of invert sugar**, and then followed by **385 grams of regular shortening product**, and then blend the mixture on high speed at room temperature for about 15 minutes. Thereafter, add in **7 grams of ammonium bicarbonate**, followed by **1.8 grams of baking soda**, followed by **280 milligrams of pure vanilla**, followed by **100 milliliters of water**, and then blend the mixture on high speed for about 45 minutes at room temperature or slightly above room temperature. Thereafter, add in **450 milligrams of yellow food dye**, followed by **6.8 grams of salt**, followed by **13.6 grams of egg powder**, followed by **1.1 grams of baking powder**, and finally followed by **450 milligrams of lecithin**, and then continue to blend the mixture on high speed for about 45 minutes at slightly above room temperature. Thereafter, the mixture is ready for use. To use, the material should be pressed or extruded into any desired shape and then cured in an oven at a suitable temperature.

Percentage: 53.89% flour, 21.1% sugar, 17.58% shortening, 4.56% water, 1.41% invert sugar, 0.61% egg powder, 0.31% ammonium bicarbonate, 0.30% salt, 0.081% baking soda, 0.059% mixed balance, 0.05% baking powder, 0.02% yellow dye, 0.02% lecithin, 0.01% pure vanilla extract

006: Commercial edible “oatmeal” composition:

Into a suitable clean and sterilized mixing bowl, equipped with motorized stirrer of the same, place **739 milliliters of hot water**, and then add in **80 grams of fresh cut oats**, and then allow the mixture to stand for about 30 minutes at room temperature. Thereafter, add in **80 grams of regular honey**, followed by **7 grams of vanilla extract**, followed by **4 grams of ground cinnamon powder**, and then followed by **2.4 grams of salt**. Thereafter, blend the mixture on high speed for about 45 minutes at a temperature slightly above room temperature. Thereafter, add in **80 grams of rolled oats**, and then followed by **7.6 grams of oat bran**, and then continue to blend the mixture on moderate speed for about 45 minutes. Thereafter, the mixture is ready for use. To use, the material should be pressed or extruded into any desired shape and then cured in an oven at a suitable temperature.

Percentage: 73.9% water, 8% cut oats, 8% honey, 8% rolled oats, 0.76% oat bran, 0.70% vanilla extract, 0.40% ground cinnamon, 0.24% salt

007: Commercial edible “oatmeal” composition:

Into a suitable clean and sterilized mixing bowl, equipped with motorized stirrer of the same, place **386 milliliters of hot water**, and then add in **40 grams of fresh cut oats**, and then allow the mixture to stand for about 30 minutes at room temperature. Thereafter, add in **25 grams of regular honey**, followed by **3 grams of vanilla extract**, and then followed by **800 milligrams of salt**. Thereafter, blend the mixture on high speed for about 45 minutes at a temperature slightly above room temperature. Thereafter, add in **1.25 grams of oat bran**, followed by **40 grams of no. 3 rolled oats**, and then followed by **1.25 grams of cinnamon**, and then continue to blend the mixture on moderate speed for about 45 minutes. Thereafter, the mixture is ready for use. To use, the material should be pressed or extruded into any desired shape and then cured in an oven at a suitable temperature.

Percentage: 77.61% water, 8% cut oats, 8% rolled oats, 5% honey, 0.6% vanilla extract, 0.25% cinnamon, 0.25% oat bran, 0.16% salt, 0.13% mixed residual balance

008: Commercial edible nutritional bar composition:

Into a suitable ball mill, filled with 150 grams of steel shot, place **330 grams of oat bran**, followed by **360 grams of rolled oats**, followed by **140 grams of soy polysaccharides**, followed by **5500 grams of fructose**, followed by **2350 grams of invert sugar**, and then followed by **100 grams of cinnamon**. Thereafter, tumble the mixture at 150 RPM for about 1 hour. Thereafter, remove the mixture from the steel shot by passing it through a screen in the usual manner. Thereafter, into a separate clean container, equipped with motorized stirrer, place **140 grams of currant raisons**, followed by **140 grams of crushed almonds**, followed by **880 grams of honey**, followed by **2190 grams of sugar**, followed by **400 grams of starch**, followed by **1480 grams of cholestyramine**, and then followed by **80 grams of vanilla**, and then blend the mixture on moderate speed for about 45 minutes. Thereafter, add in the tumbled mixture prepared previously, and then finally add in **850 grams of hot water**, and then blend the entire mass for about 1 hour at a temperature just above room temperature. Thereafter, the mixture is ready for use. To use, the mixture should be pressed into any desired shape under high pressure, and then cured in an oven at moderate temperature for several hours.

Percentage: 36.96% fructose, 15.79% invert sugar, 14.71% sugar, 9.94% cholestyramine, 5.91% honey, 5.71% water, 2.68% starch, 2.41% rolled oats, 2.21% oat bran, 0.94% currant raisons, 0.94% almonds, 0.94% soy polysaccharides, 0.67% cinnamon, 0.53% vanilla, 0.19% mixed residual balance

009: Commercial edible binder composition:

Into a suitable mixing bowl or similar container, equipped with large capacity stirring blade and mechanism, place **225 grams of corn syrup**, and then add in **110 grams of honey**, followed by **225 grams of brown sugar**, followed by **150 grams of standard edible cooking fat**, and then blend the mixture on moderate speed for about 45 minutes. After 45 minutes, add in **5 grams of lecithin**, followed by **175 grams of water**, followed by **11 grams of salt**, and then finally followed by **37 grams of glycerin**. Thereafter, blend the mixture on moderate speed for about 50 minutes to form a uniform mass. Thereafter, the material is ready for use.

Percentage: 23.98% corn syrup, 23.98% brown sugar, 18.65% water, 15.99% edible cooking fat, 11.72% honey, 3.94% glycerin, 1.17% salt, 0.53% lecithin, 0.04% mixed balance

010: Commercial edible nutritional “trail mix” composition:

Into a suitable tumbling machine such as an empty ball mill, place **443 grams of raisons**, followed by **590 grams of peanuts**, followed by **443 grams of granola**, followed by **443 grams of dried pulverized apricots**, followed by **593 grams of almonds**, followed by **150 grams of dehydrated apples**, and then followed by **590 grams of crushed walnuts**, and then tumble or rotate the mixture at 500 RPM for about 2 hours to form a uniform mass. Thereafter, the mixture is ready for consumption.

Percentage: 18.23% almonds, 18.14% peanuts, 18.14% walnuts, 13.62% raisins, 13.62% apricots, 13.62% granola, 4.6% apples, 0.03% mixed residual balance

011: Commercial orange gelatin desert composition:

Into a suitable empty ball mill, place **131 grams of sucrose**, followed by **14.7 grams of gelatin**, followed by **2.6 grams of adipic acid**, followed by **2 grams of disodium phosphate**, followed by **1.8 grams of fumaric acid**, and then followed by **300 milligrams of orange flavor** compound. Thereafter, tumble the mixture at 150 RPM for about 1 hour to form a uniform mixture. Thereafter, the mixture is ready for use. To use, the mixture simply needs to be mixed with hot water, and then allowed to cool.

Percentage: 85.95% sucrose, 9.64% gelatin, 1.7% adipic acid, 1.31% disodium phosphate, 1.18% fumaric acid, 0.19% orange flavor, 0.03% mixed residual balance

012: Commercial cherry drink mix composition:

Into a suitable empty ball mill, place *94 grams of sucrose*, followed by *2.1 grams of citric acid*, followed by *100 milligrams of ascorbic acid*, followed by *2.3 grams of maltodextrin*, and then followed by *310 milligrams of cherry flavor compound*. Thereafter, tumble the mixture at 150 RPM for about 1 hour to form a uniform mixture. Thereafter, the mixture is ready for use. To use, the mixture simply needs to be mixed with cold water.

Percentage: *95.13% sucrose, 2.32% maltodextrin, 2.12% citric acid, 0.31% cherry flavor, 0.10% ascorbic acid, 0.02% mixed balance*

013: Commercial brownie mix composition:

Into a suitable empty ball mill, place *955 grams of invert sugar*, and then add in *345 grams of shortening*, followed by *215 grams of cake flour*, followed by *310 grams of all purpose flour*, and then followed by *370 grams of maltodextrin*.

Thereafter, tumble the mixture at 150 RPM for about 1 hour. Thereafter, add in *155 grams of cocoa*, followed by *30 grams of starch*, followed by *15 grams of dried egg whites*, followed by *12.5 grams of salt*, followed by *8.5 grams of vanilla flavor*, and then followed by *650 milligrams of baking soda*. Thereafter, continue to tumble the mixture at 150 RPM for about 45 minutes. Thereafter, the mixture is ready for use.

Percentage: *39.51% invert sugar, 15.31% maltodextrin, 14.27% shortening, 12.82% all purpose flour, 8.89% cake flour, 6.41% cocoa, 1.24% starch, 0.62% dried egg whites, 0.51% salt, 0.35% vanilla flavor, 0.044% mixed residual balance, 0.026% baking soda*

014: Commercial instant dry vanilla pudding composition:

Into a suitable empty ball mill, place *370 grams of sugar*, and then add in *2.5 grams of Durem emulsifier*, followed by *2.5 grams of vegetable oil*, followed by *3 grams of disodium phosphate*, and then followed by *5 grams of tetrasodium phosphate*.

Thereafter, tumble the mixture at 150 RPM for about 1 hour. Thereafter, add in *2.5 grams of salt*, followed by *1 gram of yellow food coloring*, followed by *7.5 grams of vanilla flavor*, and then followed by *100 grams of starch*. Thereafter, continue to tumble the mixture at 150 RPM for about 50 minutes. Thereafter, the mixture is ready for use.

Percentage: *74.89% sugar, 20.24% starch, 1.51% vanilla flavor, 1.01% tetrasodium phosphate, 0.60% disodium phosphate, 0.5% salt, 0.5% vegetable oil, 0.5% Durem emulsifier, 0.2% yellow food coloring, 0.05% mixed residual balance*

015: Commercial dry “trail mix” composition:

Into a suitable dry mixing container, equipped with heavy duty stir blade, place *907 grams of cocoa powder*, and then add in *226 grams of freshly thawed frozen corn material*, followed by *283 grams of regular store bought oats*, followed by *283 grams of regular plain white rice*, and then followed by *113 grams of regular finely ground wheat*. Thereafter, add in *150 grams of sugar*, followed by *50 grams of almonds*, and then followed by *50 grams of fine ground chestnuts*. Thereafter, blend the mixture thoroughly on high speed for about 1 hour to form a uniform mass. Thereafter, the mixture is ready for use.

Percentage: *43.98% cocoa powder, 13.72% oats, 13.72% white rice, 10.96% corn, 7.27% sugar, 5.48% wheat, 2.42% almonds, 2.42% chestnuts, 0.03% residue*

016A: Commercial vegetable “raspberry” jelly composition:

Into a suitable empty ball mill, place *300 grams of sugar*, and then add in *1.5 grams of raspberry flavoring agent*. Thereafter, tumble the mixture at 150 RPM for about 45 minutes to form a uniform mixture. Thereafter, place this tumbled mixture into a suitable mixing bowl, equipped with large capacity stirring blade, and then add in *20 grams of agar agar gel*, and then blend the mixture on high speed for about 1 hour. Thereafter, add in *20 grams of karaya compound*, and then continue to blend the mixture on high speed for about 1 hour. Thereafter, add in *750 milligrams of red food coloring*, and then followed by *3.2 grams of tartaric acid*. Thereafter, blend the mixture on moderate speed for about 45 minutes. Thereafter, the mixture is ready for use.

Percentage: *86.84% sugar, 5.78% agar agar, 5.78% karaya, 0.92% tartaric acid, 0.43% raspberry flavor agent, 0.21% red food coloring, 0.04% mixed residual balance*

016B: Commercial vegetable “cherry” jelly composition:

Into a suitable empty ball mill, place *300 grams of sugar*, and then add in *500 milligrams of cherry flavoring agent*.

Thereafter, tumble the mixture at 150 RPM for about 45 minutes to form a uniform mixture. Thereafter, place this tumbled mixture into a suitable mixing bowl, equipped with large capacity stirring blade, and then add in *20 grams of agar agar gel*, and then blend the mixture on high speed for about 1 hour. Thereafter, add in *15 grams of karaya compound*, and then continue to blend the mixture on high speed for about 1 hour. Thereafter, add in *750 milligrams of red food coloring*, and then followed by *16 grams of tartaric acid*. Thereafter, blend the mixture on moderate speed for about 45 minutes. Thereafter, the mixture is ready for use.

Percentage: *85.16% sugar, 5.67% agar agar, 4.54% tartaric acid, 4.25% karaya, 0.21% red food coloring agent, 0.14% cherry flavoring agent, 0.03% mixed balance*

III. Pyrotechnic Compositions (courtesy of The Preparatory Manual of Black Powder and Pyrotechnics)

001: “Ammonium perchlorate” moderate performance “rubber-like” propellant utilizing polysulfide binder:

Into a standard mixing bowl, equipped with motorized stirrer utilizing a plastic stir blade, place **128.5 grams of a polysulfide binder compound** sold as Thiokol LP-2, followed by **9.5 grams of lead dioxide**, followed by **2 grams of stearic acid**, and then blend the mixture on high speed for a bout 5 minutes. After 5 minutes, add in **320 grams of ammonium perchlorate**, followed by **40 grams of furfuryl alcohol**, and then continue to blend the mixture on high speed for about 5 to 10 minutes to form a uniform mix. After blending, the fluidized mixture is then ready for use. To use, it should be poured and vibrated directly into your rocket motor, and then cured at room temperature for about 2 days.

Burn rate: Average at 1000 psi

Water resistance: Very good

Stability: Can be stored for many years.

Flammability (1 to 10): 9

Ease of ignition (1 to 10): 9

Tendency to cake: None

Explosive ability: Can be detonated but only severe conditions—requires significant TNT, RDX, or HMX booster.

Percentage: **64% ammonium perchlorate, 25.7% polysulfide binder, 8% furfuryl alcohol plasticizer, 1.9% lead dioxide vulcanizing agent, 0.4% stearic acid catalyst**

Classification: Deflagrating explosive (classified as propellant)

Use: Can be used to propel a wide variety of low velocity missiles especially of the wire-guided type.

002: “Ammonium perchlorate” high Performance Military grade rocket propellant with PVC:

Into a suitable beaker or container, add 600 milliliters of tetrahydrofuran solvent. Thereafter, add in **80 grams of PVC** and then stir the mixture to dissolve the PVC. Note: PVC pipes from the hardware store can be used. Once the PVC has dissolved, filter the solution, to remove any insoluble materials, and then pass the filtered solvent mixture (three times) through a layer of aluminum oxide contained in a silica gel column. Thereafter, add to your filtered solvent mixture containing the dissolved PVC, **4 grams of Epon 815 epoxy agent**, followed by **4 grams of magnesium sulfide**, and then followed by **320 grams of finely divided ammonium perchlorate**. Thereafter, rapidly stir the entire mixture for about 15 to 30 minutes at a temperature around 40 Celsius. Afterwards, remove the heat source, and then quickly add in, before the mixture cools, 600 milliliters of warm water. Thereafter, allow the entire mixture to cool to room temperature, and rapidly stir it during this cool down period. Once the mixture has cooled, continue to rapidly stir the mixture at room temperature for about 30 minutes. After 30 minutes, filter the mixture to collect the insoluble propellant mixture. You can use gravity filtration or vacuum filtration. Gravity filtration takes the longest amount time, so vacuum filtration is recommended. If using, vacuum filtration, suck the propellant mixture dry. If you used gravity filtration, once your propellant mixture has been removed, recover the pasty material from the filter paper, and place it onto a shallow pan and spread it out, and allow it to air-dry for several days or until completely dry. Either way, once your propellant is completely dry, you can move onto the packing process. Now, place your propellant into a suitable beaker or container, and then add in 50 milliliters of 99% isopropyl alcohol or 95% ethyl alcohol, and then vigorously stir the entire mixture to form a paste of similar mixture. Thereafter, press this mixture into your rocket motor or rocket mold, which you should already have designed and built. Once your rocket motor or mold has been filled, gently but firmly shake (for several minutes) your rocket motor or rocket mold to remove any air bubbles that might be trapped. Note: removing air bubbles is essential to prevent possible rocket failure (explosions). Finally, place your rocket motor or mold into a warm dry place and allow it to cure for up to 7 days.

Burn rate: Fast

Water resistance: Good.

Stability: Can be stored for many years.

Flammability (1 to 10): 10

Ease of ignition (1 to 10): 8 ½

Tendency to cake: None

Explosive ability: Can be detonated but only severe conditions—requires significant TNT, RDX, or HMX booster.

Percentage: **78.43% ammonium perchlorate, 19.6% PVC polymer, 0.98% Epon 815 epoxy resin, 0.98% magnesium sulfide catalyst, 0.01% impurities**

Classification: Deflagrating explosive (classified as propellant)

Use: Standard military rocket fuel for rockets, missiles, and the like. Has excellent thrust capability, and is used for propelling high explosive warheads.

003: “Ammonium perchlorate” high Performance military rocket propellant (reduced flash):

Into a suitable blender equipped with plastic stirring blade, place **344 grams of polyethylene glycol**, followed by **416 grams of nitroguanidine**, and then thoroughly blend this mixture for 15 to 30 minutes at room temperature on high. Thereafter, add in **200 grams of aluminum powder**, and then thoroughly blend for 15 to 30 minutes at room temperature on high. Finally, add in **510 grams of ammonium perchlorate**, and **100 grams of ammonium nitrate**, and then thoroughly blend the mixture for 2 hours at room temperature on high. After the blending, the propellant mixture needs to be pressed into your rocket motor or mold under high pressure, and thereafter the rocket motor should be cured for several days at 30 Celsius.

Burn rate: 0.23 to 0.26 inches per second at 500 psi

Water resistance: Good.

Stability: Can be stored for many years.

Flammability (1 to 10): 9

Ease of ignition (1 to 10): 9

Tendency to cake: None

Explosive ability: Can be detonated but only severe conditions—requires significant TNT, RDX, or HMX booster.

Percentage: **32% ammonium perchlorate, 26.4% nitroguanidine, 21.9% polyethylene glycol binder, 12.7% aluminum, 6.3% ammonium nitrate, 0.70% impurities**

Classification: Deflagrating explosive (classified as propellant)

Use: Specialty military rocket fuel for missiles.

004: “Ammonium perchlorate” high Performance polyurethane rocket propellant:

Into a suitable mixing bowl, mixer, blender, or similar container, equipped with heating source, place **75 grams of aluminum powder of standard mesh**, followed by **19.5 grams of dioctylsebacate plasticizer**, followed by **35.8 grams of polybutadiene diol** (of 6000 molecular weight, available from Phillips Petroleum Company), followed by **1.5 grams of polypropylene oxide triol** (of 700 molecular weight), followed by **250 milligrams of dihydroxypropyl bis(cyanoethylamine)**, followed by **2 grams of 2,6-toluene diisocyanate**, and then followed by **100 milligrams of ferric acetylacetonate**, and then blend the mixture on moderate speed for about 10 minutes. Thereafter, add in **365 grams of standard ammonium perchlorate** of average mesh, and then continue to blend the mixture on moderate speed for about 15 to 20 minutes to form a uniform mix. Thereafter, add in **750 milligrams of finely divided nickel-II-hydroxide**, and then continue to blend the mixture on moderate speed for about 5 minutes. Finally, the mixture is ready to cure. To do so, it should be pressed and vibrated into any desirable rocket motor, engine, mold, ect., and then cured in an oven at 57 Celsius for 10 days. The excessive amount of curing time is to allow proper polymerization. Requires proper ignition composition.

Burn rate: 0.3 to 0.4 inches per second at 1000 psi

Water resistance: Good.

Stability: Can be stored for many years.

Flammability (1 to 10): 9

Ease of ignition (1 to 10): 8 ¾

Tendency to cake: None

Explosive ability: Very low.

Percentage: **73% ammonium perchlorate, 15% aluminum, 7.1% polybutadiene diol binder, 3.9% dioctylsebacate plasticizing agent, 0.4% 2,6-toluene diisocyanate, 0.3% polypropylene oxide triol binding agent, 0.15% nickel-II-hydroxide curing agent, 0.08% residue, 0.05% dihydroxypropyl bis(cyanoethylamine curing catalyst, 0.02% ferric acetylacetonate,**

Classification: Deflagrating explosive (classified as propellant).

Use: Can be used in high performance rockets for military and commercial use.

005: “Ammonium perchlorate” thermally and impact resistant “blue” signaling flare producing no ash or sparks:

Into a suitable mixing bowl or blender, equipped with motorized stirrer and plastic stir blade, place **18 grams of paraffin**, followed by **55.5 grams of standard copper dust**, followed by **55.5 grams of stearic acid**, and then moderately blend the mixture for 1 hour at room temperature. After 1 hour, add in **371 grams of ammonium perchlorate**, and then continue to blend the mixture on moderate speed for about 30 minutes. Thereafter, the mixture is ready to be used. To use, it needs to be pressed into any desirable container, tube, candle, ect., under a pressure of about 8000 psi. Requires igniter composition for proper ignition.

Burn rate: 0.20 inches per second at ¾ inch diameter flare “candle”

Ignition temperature: 250 Celsius

Day-light visibility: 1500 yards

Water resistance: Very good.

Stability: Can be stored for many years.

Flammability (1 to 10): 6

Ease of ignition (1 to 10): 6 ½

Tendency to cake: None.

Explosive ability: None.

Percentage: 74.2% ammonium perchlorate, 11.1% copper, 11.1% stearic acid, 3.6% paraffin

Classification: Deflagrating explosive (classified as pyrotechnic mixture)

Use: Used in signaling on the battlefield for military operations.

006: “Potassium chlorate” high “heaving” action gun propellant suitable for artillery guns:

Into a suitable beaker or similar container, place 340 milliliters of water, followed by **170 grams of glycerin**, and then followed by **198 grams of sugar**. Thereafter, boil the mixture at 100 Celsius and stir until all the sugar dissolves. Once the sugar dissolves, remove the heat source, and allow the mixture to cool to room temperature. However, during the cool down period, when the temperature of mixture reaches about 50 Celsius, add in **226 gram of potassium chlorate** while rapidly stirring the mixture. After the addition of the potassium chlorate, continue to rapidly blend the mixture for about 2 hours. After 2 hours, pour the entire mixture onto a shallow pan, and allow it to thoroughly air-dry. Once it has, place the dried mass into mixing bowl, or blender equipped with plastic stir blade, followed by 150 milliliters of hexane, and then blend the mixture on moderate speed until 95% of the hexane evaporates. When this point is reached, stop the blending and then place the loose granules onto a shallow pan, and allow them to thoroughly air-dry. Once they have, the granules are ready for used. Note: not all the granules will be the same size, so they should be separated using the appropriate sieves.

Burn rate: Rapid.

Water resistance: Moderate.

Stability: Can be stored for many years.

Flammability (1 to 10): 9

Ease of ignition (1 to 10): 9

Tendency to cake: None.

Explosive ability: Can be detonated but only severe conditions—requires significant TNT, RDX, or HMX booster.

Percentage: 38% potassium chlorate, 33.3% sugar, 28.6% glycerin, 0.10% residue

Classification: Deflagrating explosive (classified as propellant)

Use: Can be used in field cannons and artillery guns.

007: “Potassium chlorate” standard green smoke composition for use in fireworks:

Into a suitable mixing bowl, or similar container, equipped with motorized stirrer, place **390 grams of standard commercially available indigo**, followed by **225 grams of synthetic commercially available yellow auramine compound**, followed thereafter by **525 grams of potassium chlorate**, and then followed by **390 grams of lactose**. Thereafter, add in 250 milliliters of acetone, and then blend the mixture until the bulk of the acetone evaporates. Thereafter, place the semi-dry mass onto a shallow pan or tray, and allow to thoroughly air-dry. Once it has, place the dried mass into a suitable ball mill, filled with steel shot of the usual diameter, and then tumble the mass at 250 RPM for about 30 to 40 minutes to form a uniform powder. Thereafter, the mixture is ready for use. To use, the mixture simply needs to be pressed under high pressure into any desirable smoke ball, container, mold, ect., under a pressure of about 10,000 psi. The mixture should be ignited using any standard ignition composition.

Burn rate: Slow.

Water resistance: Very good.

Stability: Can be stored for many years.

Flammability (1 to 10): 4+

Ease of ignition (1 to 10): 5

Tendency to cake: None.

Explosive ability: None.

Percentage: 34.31% potassium chlorate, 25.49% lactose, 25.49% indigo, 14.7% auramine yellow, 0.01% impurities

Classification: Deflagrating explosive (classified as consumer fireworks composition).

Use: Very common composition for the production of green smoke for various purposes.

008: “Potassium chlorate” classic blue Bengal light composition for use in blue lights:

Into a suitable mixing bowl or similar container, equipped with motorized stirrer in the usual means, place **75 grams of sulfur**, followed by **75 grams of potassium sulfate**, followed by **75 grams of copper-II-ammonium sulfate**, Thereafter, add in 150 milliliters of hexane, and then blend the mixture for about 15 to 20 minutes. Thereafter, add in **135 grams of potassium nitrate**, followed by **140 grams of potassium chlorate**, and then continue to blend the mixture for about 15 to 30 minutes to form a uniform paste. Thereafter, the pasty mass is ready for use. To use, the solvent wet mass simply needs to be pressed into any desirable tube, mold, ect. and then allowed to dry, or coated on any suitable rod for making sparklers in the usual manner, and allowed to dry.

Burn rate: Average.

Water resistance: Very good.

Stability: Can be stored for many years.

Flammability (1 to 10): 5 ¼

Ease of ignition (1 to 10): 6

Tendency to cake: None.

Explosive ability: None.

Percentage: *28% potassium chlorate, 27% potassium nitrate, 15% sulfur, 15% potassium sulfate, 15% copper-II-ammonium sulfate*

Classification: Deflagrating explosive (classified as consumer fireworks composition)

Use: Can be used in Bengal lights, sparklers and for making colored fires for multiple uses.

009: “Potassium chlorate” classic Bengal light composition for use in yellow fires:

Into a ball mill, filled with 250 grams of steel shot of small diameter, but average weight, place *180 grams of potassium chlorate*, followed by *60 grams of sodium oxalate*, followed by *60 grams of flours of sulfur*, and then followed by *30 grams of shellac*. Thereafter, tumble the mixture at 150 RPM for about 30 minutes at room temperature to form a uniform mixture. Thereafter, the mixture is ready for use. To use, simply moisten the mixture with a small amount of alcohol to form a paste. Thereafter, press the mixture into any cone, fountain, mold, ect., in the usual manner, and then allow the munitions to cure until thoroughly dry. Can be ignited using any standard means.

Burn rate: Average.

Water resistance: Very good.

Stability: Can be stored for many years.

Flammability (1 to 10): 6

Ease of ignition (1 to 10): 6

Tendency to cake: None.

Explosive ability: None.

Percentage: *54% potassium chlorate, 18% sodium oxalate, 18% sulfur, 9% shellac, 1% residue*

Classification: Deflagrating explosive (classified as consumer fireworks composition)

Use: Can be used in display fires for multiple uses.

010: “Potassium chlorate” standard bursting charge for small aerial shells:

Into a suitable mixing drum, container, ect., equipped with motorized stirrer, place *375 grams of potassium chlorate*, followed by *115 grams of finely powdered hemp coal*, and then followed by *10 grams of glutinous rice starch*. Thereafter, add in 125 milliliters of acetone or ether, and then blend the mixture until the bulk of the acetone or ether evaporates. When this point has been achieved, place the semi-dried mass onto a shallow pan or tray, and then allow it to thoroughly air-dry. Once it has, the dried mass needs to be ball milled using small diameter shot under mild RPM (avoid any significant grinding or bumping) to form a uniform powder. Thereafter, wet the dried powder with a little alcohol to form a paste. This paste can then be rolled into balls, or wrapped into any paper containers, or material in the usual manner, and then allowed to dry. Instead of forming a paste, the dry tumbled powder can be used in loose form if desired, but should be confined in the usual manner, i.e., as in the center of the aerial shell. Readily ignites, but should be primed in the usual manner.

Burn rate: Burns good.

Water resistance: Very good.

Stability: Can be stored for many years.

Flammability (1 to 10): 9

Ease of ignition (1 to 10): 9

Tendency to cake: None.

Explosive ability: Ignites rapidly, producing a bursting effect.

Percentage: *75% potassium chlorate, 23% hemp coal, 2% rich starch*

Classification: Deflagrating explosive (classified as consumer fireworks composition)

Use: Used in small aerial shells, 2 to 3 inches in diameter to burst open the shell. Can also be used in Hollywood style effects to burst apart and ignite gasoline mixtures.

011: “Potassium chlorate” classic whistle mixture for use in bottle rockets and ground devices:

Into a suitable mixing bowl, or blender, equipped with motorized stirrer in the usual manner, place *375 grams of potassium chlorate*, and then followed by *125 grams of gallic acid*. Thereafter, add in 150 milliliters of methylene chloride, and then blend the mixture for about 30 minutes. Thereafter, the mixture is ready for use. To use, simply press the mixture under high pressure into any cone, tube, container, mold, or bottle rocket in the usual manner, and then allow the devices to cure for several days at room temperature.

Burn rate: Typical.

Water resistance: Very good.

Stability: Can be stored for many years.

Flammability (1 to 10): 5 to 6

Ease of ignition (1 to 10): 8

Tendency to cake: None.

Explosive ability: None.

Percentage: *75% potassium chlorate, 25% gallic acid*

Classification: Deflagrating explosive (classified as consumer fireworks composition)

Use: Used in fireworks for generating loud whistling effects.

012: “Potassium perchlorate”: moderate performance “rubber-like” propellant utilizing potassium perchlorate and polysulfide binder:

Into a standard mixing bowl, equipped with motorized stirrer utilizing a plastic stir blade, place **97.5 grams of a polysulfide binder compound sold as Thiokol LP-2**, followed by **7.5 grams of lead dioxide**, followed by **7.5 grams of dibutyl phthalate**, followed by **1.75 grams of stearic acid**, and then blend the mixture on high speed for about 5 minutes. After 5 minutes, add in **300 grams of potassium perchlorate**, followed by **50 grams of ammonium perchlorate**, followed by **36 grams of furfuryl alcohol**, and then continue to blend the mixture on high speed for about 5 to 10 minutes to form a uniform mix. After blending, the fluidized mixture is then ready for use. To use, it should be poured and vibrated directly into your rocket motor, and then cured at room temperature for about 2 days.

Burn rate: Average at 1000 psi

Water resistance: Very good

Stability: Can be stored for many years.

Flammability (1 to 10): 9

Ease of ignition (1 to 10): 9

Tendency to cake: None

Explosive ability: None.

Percentage: **59.9% potassium perchlorate, 19.4% polysulfide binder, 9.9% ammonium perchlorate, 7.1% furfuryl alcohol plasticizer, 1.4% lead dioxide vulcanizing agent, 1.4% dibutyl phthalate dispersion medium, 0.56% mixed residues, 0.34% stearic acid catalyst,**

Classification: Deflagrating explosive (classified as propellant)

Use: Can be used to propel a wide variety of low velocity missiles especially of the wire-guided type.

013: “Potassium perchlorate” moderate performance “rubber-like” propellant utilizing potassium perchlorate and polysulfide binder:

Into a standard mixing bowl, equipped with motorized stirrer utilizing a plastic stir blade, place **115 grams of a polysulfide binder compound sold as Thiokol LP-2**, followed by **2.5 grams of paraquinone dioxime**, followed by **3.4 grams of stearic acid**, followed by **6.5 grams of zinc oxide**, and then blend the mixture on high speed for about 5 minutes. After 5 minutes, add in **250 grams of potassium perchlorate**, followed by **85 grams of ammonium perchlorate**, followed by **37.5 grams of furfuryl alcohol**, and then continue to blend the mixture on high speed for about 5 to 10 minutes to form a uniform mix. After blending, the fluidized mixture is then ready for use. To use, it should be poured and vibrated directly into your rocket motor, and then cured in an oven at 65 Celsius for several hours.

Burn rate: Average at 1000 psi

Water resistance: Very good

Stability: Can be stored for many years.

Flammability (1 to 10): 9

Ease of ignition (1 to 10): 9

Tendency to cake: None

Explosive ability: None.

Percentage: **50% potassium perchlorate, 23% polysulfide binder, 17% ammonium perchlorate, 7.5% furfuryl alcohol plasticizer, 1.3% zinc oxide, 0.68% stearic acid catalyst, 0.50% paraquinone dioxime, 0.02% mixed residues**

Classification: Deflagrating explosive (classified as propellant).

Use: Can be used to propel a wide variety of low velocity missiles especially of the wire-guided type.

014: “Potassium perchlorate” high Performance potassium perchlorate rocket propellant:

Into a suitable mixing bowl, blender, or similar container, equipped with motorized stirrer utilizing plastic stir blades, place **190 grams of potassium perchlorate**, followed by **50 grams of finely powdered aluminum** of the usual mesh, and then followed by **10 grams of copper chromite**. Immediately thereafter, add in 150 milliliters of diethyl ether, and then blend the mixture on high speed to form a dough. Thereafter, place the dough onto a shallow tray or pan, and allow it to thoroughly air-dry. Afterwards, place the dried mass into a ball mill, filled with steel shot of the usual weight and diameter, and then tumble the mixture at 100 RPM for about 30 minutes to form a uniform powder. Now, into a clean mixing bowl, blender, ect., equipped with motorized stirrer in the usual means, place the uniform tumbled mixture, followed by **62.5 grams of a unhardened polyurethane compound** (commercially available), followed by **172.5 grams of potassium perchlorate**, followed by **10 grams of finely powdered aluminum**, and then blend the mixture on high speed for about 15 to 20 minutes to form a uniform fluidized mass. Thereafter, the mixture is ready for use. To use, the fluidized mass needs to be poured and pressed into any desirable rocket motor, engine, mold, ect., under the usual conditions, and the resulting engine or mold should be cured at room temperature, or at temperatures raising from 50 to 80 Celsius for several hours or for several days. Should be ignited using a black powder charge or equivalent ignition composition.

Burn rate: 0.30 to 0.35 inches per second at 500 psi

Water resistance: Good.

Stability: Can be stored for many years.

Flammability (1 to 10): 8 ½

Ease of ignition (1 to 10): 8 ½

Tendency to cake: None

Explosive ability: May explode under severe conditions only.

Percentage: 73.2% *potassium perchlorate*, 12.6% *polyurethane compound binder*, 12.1% *aluminum*, 2% *copper chromite burn rate catalyst*, 0.10% *balanced*

Classification: Deflagrating explosive (classified as propellant).

Use: Can be used in military and commercial rockets and missiles for multiple uses.

015: “Potassium perchlorate” high Performance potassium perchlorate rocket propellant:

Into a suitable mixing bowl, blender, or similar container, equipped with motorized stirrer utilizing plastic stir blades, place **190 grams of potassium perchlorate**, followed by **50 grams of finely powdered aluminum** of the usual mesh, and then followed by **10 grams of copper chromite**. Immediately thereafter, add in 150 milliliters of diethyl ether, and then blend the mixture on high speed to form a dough. Thereafter, place the dough onto a shallow tray or pan, and allow it to thoroughly air-dry.

Afterwards, place the dried mass into a ball mill, filled with steel shot of the usual weight and diameter, and then tumble the mixture at 100 RPM or so for about 30 minutes to form a uniform powder. Now, into a clean mixing bowl, blender, ect., equipped with motorized stirrer in the usual means, place the uniform tumbled mixture, followed by **62.5 grams of a unhardened polyurethane compound** (commercially available), followed by **172.5 grams of ammonium perchlorate**, followed by **10 grams of finely powdered aluminum**, and then blend the mixture on high speed for about 15 to 20 minutes to form a uniform fluidized mass. Thereafter, the mixture is ready for use. To use, the fluidized mass needs to be poured and pressed into any desirable rocket motor, engine, mold, ect., under the usual conditions, and the resulting engine or mold should be cured at room temperature, or at temperatures raising from 50 to 80 Celsius for several hours or for several days. Should be ignited using a black powder charge or equivalent ignition composition.

Burn rate: 8 millimeters per second at 500 psi

Water resistance: Good.

Stability: Can be stored for many years.

Flammability (1 to 10): 8 ½

Ease of ignition (1 to 10): 8 ½

Tendency to cake: None

Explosive ability: May explode under sever conditions only.

Percentage: 73.2% *potassium perchlorate*, 12.6% *polyurethane compound binder*, 12.1% *aluminum*, 2% *copper chromite burn rate catalyst*, 0.10% *balanced*

Classification: Deflagrating explosive (classified as propellant).

Use: Can be used in military and commercial rockets and missiles for multiple uses.

016: “Potassium perchlorate” high performance gun propellant:

Into a suitable mixing bowl, blender, or similar container, equipped with motorized stirrer utilizing a plastic stir blade, place **380 grams of potassium perchlorate**, followed by **47.5 grams of finely powdered aluminum**, followed by **72.5 grams of Armstrong epoxy resin E-301-14**, and then thoroughly blend the mixture for about 10 to 15 minutes to form a uniform fluidized pasty mass. After the mixing process, the mixture is ready for pressing. To do so, it needs to placed into a beaker of suitable container and gently heated to 180 Celsius. When the mixtures temperature reaches 180 Celsius, the hot mixture needs to be immediately placed into an extruding machine fitted with die cast holes ranging from 1.5 to 5 millimeters in diameter, depending on desired grain size, and extruded under a hydraulic pressure of 15,000 psi to form rods, or “spaghetti-like” strands ranging from 200 to 300 millimeters in length. Thereafter, the spaghetti strands should then immediately be cut into even pieces of 1.5 to 5 millimeters lengths, depending on desired grain size. Afterwards, the grains should be cured on a shallow pan for a day or so. Thereafter, the grains are ready for loading into any desirable shell casing utilizing the normal techniques.

Burn rate: Rapid.

Water resistance: Very good.

Stability: Can be stored for many years.

Flammability (1 to 10): 9

Ease of ignition (1 to 10): 9

Tendency to cake: None.

Explosive ability: Cannot be detonated under normal conditions.

Percentage: 76% *potassium perchlorate*, 14.5% *epoxy resin*, 9.5% *aluminum*

Classification: Deflagrating explosive (classified as propellant)

Use: Can be used as general propellant for field guns, cannons, and ship guns. Can also be used as a solid rocket propellant.

017: “Potassium perchlorate” urea-formaldehyde composition for fountains/cones:

Into a suitable mixing bowl, blender, or suitable container, equipped with motorized stirrer utilizing a plastic stir blade, place **30 grams of finely divided aluminum**, followed by **10 grams of strontium oxalate**, followed by **180 grams of potassium perchlorate**, and then gently dry blend the mixture for about 10 minutes. Thereafter, add in **90 grams of a urea-formaldehyde resin** (commercially available), and then continue to gently blend the mixture for about 15 minutes. Thereafter, press the mixture into any desirable cone, fountain, ect, utilizing the normal techniques and then cure the cones, fountains, ect., in an oven at 80 Celsius for about 3 to 4 minutes. Can be ignited using any standard means.

Burn rate: Average.

Water resistance: Very good.

Stability: Can be stored for many years.

Flammability (1 to 10): 6 ½

Ease of ignition (1 to 10): 6 ¾

Tendency to cake: None.

Explosive ability: None.

Percentage: **58% potassium perchlorate, 29% urea-formaldehyde resin, 9.6% aluminum, 3.2% strontium oxalate, 0.2% residue**

Classification: Deflagrating explosive (classified as consumer fireworks composition)

Use: Used for making fountains and cones.

018: “Ammonium nitrate”: high Performance military rocket propellant (JPL X350 type):

Into a standard ball mill, place **196 grams of ammonium nitrate** of 100 mesh, followed by **84 grams of ammonium nitrate** of 30 microns, followed by **40 grams of dioctyl azelate**, followed by **4 grams of copper chromite**, followed by **200 milligrams of ferric acetylacetonate**, and then 100 grams of steel shot of 4 to 5 millimeters in diameter. Thereafter, thoroughly tumble the mixture for 10 to 15 minutes at room temperature. At the same time, into a standard blender or mixing bowl, equipped with plastic stirring blades rather than sharp steel ones, place **5.4 grams of poly propylene glycol**, followed by **24 grams of hexane triol**. Thereafter, thoroughly blend this mixture for 15 to 30 minutes at room temperature on high speed. After preparing these two mixtures, place both of them into a specially deigned heated blender with vacuum pump and gauge. Thereafter, thoroughly blend the two mixtures on high speed at 30 Celsius under a vacuum of 10 millimeters of mercury for about 35 minutes.

Immediately after 35 minutes, equalize the reduced pressure, open the machine, and toss in **6 grams of 2,4-toluene diisocyanate**, and then continue to heat and vacuum blend the mixture on high speed and under a vacuum of about 20 millimeters of mercury at 23 to 25 Celsius for about 10 minutes. Afterwards, the propellant mixture is ready to cast. To do so, simply pour and press the propellant into any desired rocket motor or mold, and then vibrate the motor or mold vigorously to remove any air-bubbles or the like. Finally, let your motor or mold cure for about 1 to 2 weeks at room temperature.

Burn rate: 0.2 to 0.3 inches per second at nominal pressure

Water resistance: Moderate.

Stability: Can be stored for many years.

Flammability (1 to 10): 9

Ease of ignition (1 to 10): 8 ½

Tendency to cake: None

Explosive ability: Can be detonated but only severe conditions—requires significant TNT, RDX, or HMX booster.

Percentage: **77.8% ammonium nitrate, 11% dioctyl azelate, 6.6% hexane triol plasticizer, 1.6% 2,4-toluene diisocyanate, 1.5% poly propylene glycol plasticizer, 1.1% copper chromite burn rate accelerator, 0.05% ferric acetylacetonate curing catalyst, 0.35% impurities**

Classification: Deflagrating explosive (classified as propellant)

Use: Standard military rocket fuel for rockets and missiles.

019: “Ammonium nitrate” based military rocket propellant with TNT power additive:

Into a suitable mixing bowl, blender, or similar container, place **66.5 grams of a thermoplastic phenoxy resin** (commercially available as Bakelite PKDA-8500 resin), followed by **20 grams of TNT**, followed by **7.5 grams of sodium barbiturate**, followed by **15 grams of finely divided standard carbon black**, followed by **1 gram of N-phenylmorpholine**, followed by **2 grams of ammonium oxalate**, and then finally followed by **350 grams of ammonium nitrate**, and then blend the mixture for about 15 minutes. After 15 minutes, add in **33.3 grams of acetyl triethylcitrate**, and then immediately thereafter add in **4 grams of 2,4-toluene diamine**, and then continue to blend the mixture for about 5 to 10 minutes. Immediately thereafter, cast the semi-slurry mixture into any desirable rocket motor, mold, ect., under mild pressure, and then vibrate said munitions to remove air bubbles (if any), and then allow the rocket motor to cure for a day or so.

Burn rate: 10 seconds per inch at 1000 psi at 70 Celsius

Water resistance: Very good

Stability: Can be stored for many years.

Flammability (1 to 10): 9

Ease of ignition (1 to 10): 9

Tendency to cake: None

Explosive ability: Can be detonated but only severe conditions—requires significant TNT, RDX, or HMX booster.

Percentage: *70% Ammonium nitrate, 13.3% thermoplastic phenoxy resin, 6.6% acetyl triethylcitrate catalyst, 4% TNT, 3% carbon black filler, 1.5% sodium barbiturate stabilizer, 0.8% 2,4-toluene diamine additive, 0.4% ammonium oxalate burn rate catalyst, 0.2% N-phenylmorpholine inert filler, 0.20% residue*

Classification: Deflagrating explosive (classified as propellant)

Use: Can be used for general military and commercial use.

020: “Ammonium nitrate” high Performance JPL X360 rocket propellant:

Into a suitable horizontal ball mill, filled with steel shot of the usual diameter and size, place *105 grams of ground ammonium nitrate*, followed by *245 grams of ungrounded ammonium nitrate*, followed by *10 grams of copper chromite*, and then tumble the mixture for about 30 minutes at 150 RPM to form a uniform mixture. Afterwards, into a suitable mixing container, equipped with vacuum gauge, motorized stirrer, and heating source, place the dry tumbled mixture previously prepared, followed by *119.5 grams of polypropylene glycol 2025*, and then blend the mixture on moderate speed for about 30 minutes under a vacuum of 10 millimeters of mercury at a temperature of 21 Celsius. Thereafter, prepare a curing mixture by adding and mixing with, *500 milligrams of ferric acetyl acetate* to *24 grams of toluene diisocyanate*, and then quickly remove the vacuum, open the mixing container, and then throw in the curing mixture, and then reseal the container, apply a vacuum of 10 millimeters of mercury, and then continue to blend the mixture on moderate speed for about 10 minutes at 21 Celsius. Thereafter, open the container once again, and quickly add in *6 grams of 1,2,6-hexanetriol*, and then re-seal the container, apply the same vacuum as before, and then continue to blend the mixture on moderate speed for about 10 additional minutes. Thereafter, the propellant mixture is ready to be casted. To do so, simply pour it into any desirable rocket motor, engine, mold, ect., and then briefly vibrate the munitions to remove air bubbles, and then cure the munition(s) in an oven at 60 Celsius for about 16 hours. Requires standard rocket propellant ignition composition.

Burn rate: 0.30 to 0.40 inches per second at 1000 psi

Water resistance: Good.

Stability: Can be stored for many years.

Flammability (1 to 10): 9

Ease of ignition (1 to 10): 9

Tendency to cake: None

Explosive ability: Very low.

Percentage: *68.6% ammonium nitrate, 23.4% polypropylene glycol 2025 plasticizer, 4.7% toluene diisocyanate, 1.9% copper chromite burn rate catalyst, 1.1% 1,2,6-hexanetriol, 0.21% mixed impurities, 0.09% ferric acetyl acetate*

Classification: Deflagrating explosive (classified as propellant).

Use: Widely used high performance rocket propellant with multiple uses.

021: “Ammonium nitrate” high Performance rocket propellant:

Into a suitable mixing blender, such as a “Baker-Perkins mixer” equipped with vacuum technology, or equivalent, place *450 grams of polybutadiene*, followed by *50 grams of 2-methyl-5-vinylpyridine*, and then blend the mixture for about 5 minutes at room temperature. Thereafter, add in *50 grams of carbon black*, and then continue to blend the mixture for another 5 minutes. After 5 minutes, add in *8.7 grams of finely powdered sulfur*, followed by *15 grams of zinc oxide*, followed by *5 grams of “Aerosol OT”*, which is a dioctyl ester of sodium sulfosuccinic acid, followed by *15 grams of “Felxamine”*, a commercially available binder composed of 65% complex diarylamineketone, and 35% N,N'-diphenyl-p-phenylenediamine, and continue to blend the mixture for about 5 minutes. After 5 minutes, add in *40 grams of benzophenone*, followed by *40 grams of Pentaryl A”*, which is a amylbiphenyl, and then continue to blend the mixture for about 60 minutes. After 60 minutes, apply a vacuum of 749 millimeters of mercury for 5 minutes. After 5 minutes, remove the mild vacuum, and then add in *36.6 grams of epichlorohydrin*, and then continue to blend the mixture without vacuum, for about 10 minutes. After 10 minutes, slowly add in, in small portions at a time, *4083 grams of ammonium nitrate of 40 microns on average*, and blend the mixture on moderate speed during the addition. After the addition of the nitrate, continue to blend the mixture for about 60 minutes to form a uniform mixture. After 60 minutes, add in *99 grams of milori blue pigment agent*, and then blend the mixture for about 5 minutes. After 5 minutes, add in *12.2 grams of “Butyl-Eight”*, which is a dithiocarbamate rubber accelerator, and then blend the mixture on high speed for about 15 minutes. At this point, the temperature of the propellant mixture may have increased above room temperature. Either way, if the temperature of the propellant mixture is below 30 Celsius, heat the propellant mixture to 67 Celsius, and then blend the propellant mixture at this temperature for about 10 to 15 minutes. Thereafter, press the mixture into any desirable rocket motor, engine, ect, under standard pressure, and the cure the rockets in an oven at 60 to 80 Celsius for 12 to 18 hours.

Burn rate: 0.15 to 0.21 inches per second at 500 psi

Water resistance: Good.

Stability: Can be stored for many years.

Flammability (1 to 10): 9

Ease of ignition (1 to 10): 8+

Tendency to cake: None

Explosive ability: Very low.

Percentage: 83.2% ammonium nitrate, 9.1% polybutadiene polymer binder, 2% milori blue pigment curing agent, 1% 2-methyl-5-vinylpyridine copolymers, 1% carbon black catalyst filler, 0.81% benzophenone burn rate catalyst, 0.81% Pentary A copolymer catalyst, 0.74% epichlorohydrin polymerization catalyst, 0.30% zinc oxide filler, 0.30% flexamine binder additive, 0.24% Butyl-eight rubber accelerator, 0.23% mixed residues, 0.17% sulfur burn modifier, 0.1% Aerosol OT catalyst,

Classification: Deflagrating explosive (classified as propellant).

Use: Can be used for high performance rocket propellants with multiple uses.

022: "Ammonium nitrate" high Performance smokeless polymer rocket propellant:

Into a suitable mixing bowl, blender, or similar container equipped with motorized stirrer, place 375 milligrams of finely powdered sulfur, followed by 1.5 grams of zinc oxide, followed by 10 grams of carbon black, followed by 9 grams of Milori blue agent, followed by 376.5 grams of ammonium nitrate, followed by 500 milligrams of Aerosol-OT wetting agent (dioctyl ester of sodium sulfosuccinic acid), and then followed by 1.5 grams of Flexamine antioxidant (25% diarylamine-ketone complex, and complex mixture of amines and phenols, commercially available), and then blend the mixture for about 10 to 15 minutes. Thereafter, add in 50 grams of a copolymer composed of butadiene of 2-methyl-5-vinylpyridine, followed by 10 grams of dibutoxyethoxyethyl formal plasticizer, followed by 500 milligrams of SA-113, which is N,N-dimethyl-tertiary butyl sulfenyl dithiocarbamate vulcanization accelerator, and then continue to blend the mixture for about 15 minutes. After 15 minutes, the mixture is ready to be casted. To do so, pour, press, and vibrate the mixture into any desirable rocket engine, motor, mold, ect, under the usual techniques, and then cure the munitions in an oven at 76 Celsius for 7 to 14 days. Can be ignited using any standard high performance rocket ignition composition.

Burn rate: 0.15 to 0.2 inches per second at 1000 psi

Water resistance: Good.

Stability: Can be stored for many years.

Flammability (1 to 10): 9

Ease of ignition (1 to 10): 8+

Tendency to cake: None

Explosive ability: Very low.

Percentage: 81.8% ammonium nitrate, 10.8% Butadiene copolymer, 2.1% dibutoxyethoxyethyl formal plasticizer, 2.1% carbon black filler, 1.9% milori blue burning catalyst, 0.38% mixed residues, 0.32% zinc oxide catalyst, 0.32% flexamine antioxidant agent, 0.10% aerosol-OT wetting agent, 0.10% SA-113 vulcanization accelerator, 0.08% sulfur

Classification: Deflagrating explosive (classified as propellant).

Use: Can be used for high performance rockets, missiles, and take-off-assisted rocket packs.

023: High Performance rocket propellant:

Into an empty heated ball mill, place 39 grams of dinitrotoluene, followed by 39 grams of 2,4-dinitrodiphenyl ether, and then gradually heat the nitro compounds to 120 Celsius. When the nitro compounds reach a temperature of about 120 Celsius, add in 26 grams of finely divided PVC, and then tumble the mixture at 150 RPM at 120 Celsius for about 5 minutes. Thereafter, add in 5 grams of Prussian blue pigment, followed by 10 grams of ammonium dichromate, and then followed by 380 grams of ammonium nitrate of average microns, and then continue to tumble the mixture at 150 RPM for about 1 hour at 120 Celsius to form a uniform mass. Thereafter, the propellant mixture is ready to be poured. To do so, pour, press, and vibrate the mixture, before it cools, into any desirable rocket motor, engine, mold, ect., and then allow the munitions to cool and cure for several days.

Burn rate: 0.20 to 0.25 inches per second at 1000 psi

Water resistance: Good.

Stability: Can be stored for many years.

Flammability (1 to 10): 9

Ease of ignition (1 to 10): 8 ½

Tendency to cake: None

Explosive ability: Very low.

Percentage: 76% ammonium nitrate, 7.8% dinitrotoluene plasticizer, 7.8% 2,4-dintrodiphenyl ether plasticizer, 5.2% PVC binder, 2% ammonium dichromate catalyst, 1% Prussian blue burn rate catalyst, 0.20% mixed residues

Classification: Deflagrating explosive (classified as propellant).

Use: Used as a propellant in anti-ship missiles, and long-range rockets and missiles.

024: "NAPALM" military incendiary agent for use in non-bursting Napalm bombs (tail ejection bombs):

Into a suitable mixing drum, equipped with motorized stirrer, place 70 grams of aluminum salts of coconut oil (aluminum soap of coconut), followed by 20 grams of finely ground lampblack, and then followed by 870 grams of regular super unleaded gasoline. Thereafter, blend the mixture for about 5 minutes, and then add in 40 grams of a mixture containing 40%

oleic acid, and 60% linoleic acid (Neo-Fat 3R), and then continue to blend the mixture for about 15 minutes to form a uniform gel. Thereafter, the gel is ready for packing. For packing, the gel simply needs to be lightly pressed into any desirable bomb casing in the usual loading means. Not recommend for “bursting” type munitions.

Burn rate: 100 c.c. sample will burn for 3 minutes (estimated)

Heat output: N/A

Water resistance: Very good.

Stability: Can be stored for many years, but should be used within 18 months if applicable.

Flammability (1 to 10): N/A

Ease of ignition (1 to 10): 9 (based on ignition of flammable liquids).

Tendency to cake: None

Explosive ability: Stable.

Percentage: *87% gasoline, 7% aluminum soap of coconut, 4% oleic acid/linoleic acid mixture, 2% lampblack*

Classification: Highly flammable semi-liquid.

Use: Widely used in military napalm bombs for various operations.

025: “NAPALM” military incendiary agent for use in bursting Napalm bombs (modified):

Into a suitable mixing drum, equipped with motorized stirrer, place *90 grams of aluminum salts of coconut oil* (aluminum soap of coconut), followed by *20 grams of finely ground lampblack*, and then followed by *840 grams of regular super unleaded gasoline*. Thereafter, blend the mixture for about 5 minutes, and then add in *50 grams of a mixture containing 40% oleic acid, and 60% linoleic acid (Neo-Fat 3R)*, and then continue to blend the mixture for about 15 minutes to form a uniform gel. Thereafter, the gel is ready for packing. For packing, the gel simply needs to be lightly pressed into any desirable bomb casing in the usual loading means. This composition is preferred for bursting type bombs.

Burn rate: 100 c.c. sample will burn for 3 minutes (estimated)

Heat output: N/A

Water resistance: Very good.

Stability: Can be stored for many years, but should be used within 18 months if applicable.

Flammability (1 to 10): N/A

Ease of ignition (1 to 10): 9 (based on ignition of flammable liquids).

Tendency to cake: None

Explosive ability: Stable.

Percentage: *84% gasoline, 9% aluminum soap of coconut, 5% oleic acid/linoleic acid mixture, 2% lampblack*

Classification: Highly flammable semi-liquid.

Use: Widely used in military napalm bombs for various operations.

026: “NAPALM” military incendiary agent for non bursting Napalm munitions:

Into a suitable mixing drum, equipped with motorized stirrer, place *900 grams of super unleaded gasoline*, and then add in *50 grams of aluminum naphthenate*. Thereafter, blend the mixture on moderate speed for about 15 minutes. Thereafter, add in *50 grams of aluminum soap of coconut*, and then continue to blend the mixture for about 20 minutes. Thereafter, the gel is ready for packing. For packing, the gel simply needs to be lightly pressed into any desirable bomb casing in the usual loading means. This composition is not preferred for bursting type bombs.

Burn rate: 100 c.c. sample will burn for 2 to 3 minutes (estimated)

Heat output: N/A

Water resistance: Very good.

Stability: Can be stored for many years, but should be used within 18 months if applicable.

Flammability (1 to 10): N/A

Ease of ignition (1 to 10): 9 (based on ignition of flammable liquids).

Tendency to cake: None

Explosive ability: Stable.

Percentage: *90% gasoline, 5% aluminum soap of coconut, 5% aluminum naphthenate*

Classification: Highly flammable semi-liquid.

Use: Widely used in military napalm bombs for various operations.

027: “NAPALM” military incendiary agent (modified with lampblack):

Into a suitable mixing drum, equipped with motorized stirrer, place *915 grams of super unleaded gasoline*, and then add in *30 grams of aluminum naphthenate*. Thereafter, blend the mixture on moderate speed for about 15 minutes. Thereafter, add in *50 grams of aluminum soap of coconut*, and then followed by *5 grams of finely powdered lampblack*, and then continue to blend the mixture for about 20 minutes. Thereafter, the gel is ready for packing. For packing, the gel simply needs to be lightly pressed into any desirable bomb casing in the usual loading means. This composition can be used in bursting type bombs.

Burn rate: 100 c.c. sample will burn for 2 to 3 minutes (estimated)

Heat output: N/A

Water resistance: Very good.

Stability: Can be stored for many years, but should be used within 18 months if applicable.

Flammability (1 to 10): N/A

Ease of ignition (1 to 10): 9 (based on ignition of flammable liquids).

Tendency to cake: None

Explosive ability: Stable.

Percentage: *91.5% gasoline, 5% aluminum soap of coconut, 3% aluminum naphthenate, 0.5% lampblack*

Classification: Highly flammable semi-liquid.

Use: Widely used in military napalm bombs for various operations.

Chapter 6: Extraction

Procedures and processes

Extraction is a major process used on many levels including industrial, and laboratory. Extraction is used to isolate a single material from a mixture of compounds. Earlier, you were familiarized with making chemical mixtures, such as commercial edible and pyrotechnics mixtures from individual compounds. Now is the time to learn about extraction, and how to isolate an individual compound from a predetermined mixture. Extraction is carried out on two primary levels; 1) the industrial level, where extraction is used primarily to remove impurities from a mixture, or to isolate an individual compound in order to purify it; and 2) on a laboratory scale, which is used primarily to isolate an individual compound in order to purify it. Also, extraction is often used to prepare “essential” oils or perfumes from a natural plant material. The following procedures will better explain how extraction is used.

Procedure 001: Extraction process 1: Separation of ammonium perchlorate from a pyrotechnic mixture:

Method 1: Extraction of ammonium perchlorate from a pre-prepared pyrotechnic composition

Materials:

1. 75 grams (2.6 oz.) of sulfur	4. 375 grams (13.2 oz.) of ammonium perchlorate
2. 50 grams (1.7 oz.) of charcoal	5. 250 milliliters (8.4 fluid oz.) of 95% ethyl alcohol
3. 10 grams (0.35 oz) of aluminum powder	6. 1500 milliliters (50.7 fluid oz.) of tap water

Hazards: 95% ethyl alcohol is flammable so extinguish all flames before using.

Procedure:

Step 1: Preparation of a pyrotechnic composition containing ammonium perchlorate

Into a suitable ball mill, filled with 500 grams (1.1 pounds) of steel shot, place 75 grams (2.6 oz.) of sulfur, followed by 50 grams (1.7 oz.) of charcoal, followed by 10 grams (0.35 oz) of aluminum powder, and then tumble the mixture for about 45 minutes at 150 RPM. Thereafter, separate this mixture from the steel shot, by screening it through a metal screen in the usual manner. Thereafter, place the mixture into a suitable mixing drum or vessel, equipped with motorized stirrer, and then add in 375 grams (13.2 oz.) of ammonium perchlorate, followed by 250 milliliters (8.4 fluid oz.) of 95% ethyl alcohol. Thereafter, blend the mixture on moderate speed for about 45 minutes to form a uniform mass. Thereafter, the mixture is ready for use. To use, the mixture can be pressed into any desired rocket motor, and then allowed to cure in an oven under mild heat. However, for use in the following step, place the freshly prepared mixture onto a shallow pan or tray, and allow it to thoroughly air-dry. Once the mixture has thoroughly air-dried, it is ready for step 2.

Step 2: Separation of ammonium perchlorate from a pyrotechnic mixture.

Into a suitable beaker or similar container, place 500 grams (1.1 pounds) of the ammonium perchlorate pyrotechnic mixture prepared in step 1, and then add in 1500 milliliters (50.7 fluid oz.) of tap water, and then bring the mixture to a gentle boil at around 100 Celsius (212 Fahrenheit). Allow the mixture to boil for about 15 to 20 minutes with occasional stirring. After which, remove the heat source, and allow the mixture to cool to about 50 to 60 Celsius. When the mixture cools to 50 to 60 Celsius, quickly filter the mixture using gravity filtration, or preferably, vacuum filtration to remove the insoluble matter (the charcoal, aluminum powder, and sulfur). After the filtration process, place the filtered mixture back into a large beaker, and then bring the mixture to a gentle boil. Once the mixture has reached a gentle boil, continue to boil the mixture until tiny

crystals begin to form on the surface of the liquid. When this happens, quickly remove the heat source, and allow the mixture to cool to room temperature fully. Note: during the cool down period, crystals of the perchlorate will form. Once the mixture has cooled to room temperature, filter-off the precipitated crystals, using a clean filter setup, see filtration for more information. Once the crystals have been filtered-off, place the filtered liquid back into the same beaker, and bring it to a gentle boil once again. Continue to boil the mixture until tiny crystals form on the surface of the liquid as before. When this point is reached, remove the heat source, and allow the mixture to cool to room temperature once again. Note: like before, during this cool down period, crystals of the perchlorate will separate out. Once the mixture has cooled to room temperature, filter-off the precipitated crystals, using the same filter as before. Thereafter, place the filtered liquid back into the same beaker, or a smaller cleaner one, and repeat the whole process about two more times to collect as much of the ammonium perchlorate as possible, this process is what recrystallization is, see vide supra.

Procedure 002: Extraction process 2: Separation of potassium nitrate from black powder:

Method 1: Extraction of potassium nitrate from black powder

Materials:

1. 1500 milliliters (50.7 fluid oz.) of hot tap water	2. 500 grams (1.1 pounds) of finely divided black powder
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Hazards: Black powder is highly flammable so extinguish all flames before using.

Procedure: Into a clean large beaker or similar container, place 1500 milliliters (50.7 fluid oz.) of hot tap water, and then add in 500 grams (1.1 pounds) of finely divided black powder (note: commercial black powder will have to be pulverized in a crucible or ball mill in order to turn it into a fine powder). Thereafter, boil the mixture at 100 Celsius (212 Fahrenheit) for about 45 minutes. During which time, the potassium nitrate will be dissolved into the water. Thereafter, remove the heat source and allow the mixture to cool. When the mixture's temperature reaches that of about 40 to 50 Celsius, filter the hot liquid to remove the insoluble material (sulfur and charcoal). Thereafter, pour the filtered liquid into a clean beaker or similar heat resistant container. Now, perform a recrystallization process by first, boiling the mixture to concentrate it. When the mixture reaches saturation point, tiny crystals will form on the surface of the liquid. When this point is reached, remove the heat source, and allow the mixture to cool to room temperature. During the cool down period, crystals will crystallize and precipitate out of solution. When the mixture has cooled, it should be filtered to recover the precipitated crystals of the nitrate. The filtered mixture should then be placed back into the same beaker or a clean one, and brought to a boil once again, this process should be repeated about two or three more times to recover the bulk of the potassium nitrate, see recrystallization for more information.

Procedure 003: Extraction process 3: Preparation of essential oil of caraway by use of steam distillation

Constitution of oil of caraway: Carvone, and d-Limonene

Method 1: Separation of oil of caraway from the spice

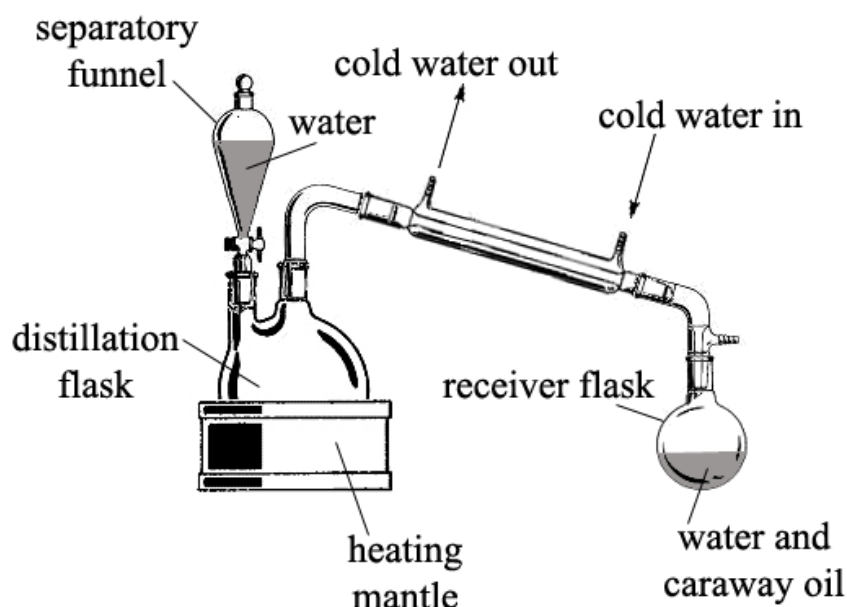
Materials:

1. 150 grams (5.2 oz.) of finely powdered caraway	2. 1000 milliliters (16.9 fluid oz.) of tap water
3. 225 milliliters (7.6 fluid oz.) of methylene chloride	4. 25 grams (0.88 oz.) of anhydrous magnesium sulfate

Hazards: Avoid inhalation of methylene chloride fumes.

Procedure: Assemble the apparatus as illustrated below, and then place 150 grams (5.2 oz.) of finely powdered caraway into the distillation flask along with 500 milliliters (16.9 fluid oz.) of tap water. Thereafter, bring the contents in the distillation flask to a boil, and then continue to boil the mixture for about 4 hours. Note: during the 4 hours, drain some water from the separatory funnel into the distillation flask to maintain the water level at around 500 milliliters. If the water level in the distillation flask get to low, the steam distillation process will not function properly. After 4 hours, remove the heat source, and allow the apparatus to cool to room temperature. Thereafter, pour the contents of the receiver flask into a clean separatory funnel, and then extract the aqueous mixture with three 75-milliliter portions (three 2.5 fluid oz. portions) of methylene chloride, see extraction for more information on this process. After the extraction process, combine all extract portions if not already done so, and then dry the combined extracts by adding in 25 grams (0.88 oz.) of anhydrous magnesium sulfate. Thereafter, blend the mixture for a few minutes to allow the magnesium sulfate to absorb any water present, and then filter-off

the magnesium sulfate. Thereafter, place the dried methylene chloride mixture into a distillation apparatus, and distill-off the methylene chloride by heating the mixture to about 40 to 50 Celsius. Continue the distillation until no more methylene chloride distills over. When this point has been reached, the operation is complete, so remove the heat source, and allow the remaining oil in the distillation flask to cool to room temperature. Thereafter, remove the oil of caraway from the distillation flask and store it in any suitable airtight container.



Apparatus for the steam distillation of caraway spice.

Procedure 004: Extraction process 4: Extraction of DNA from food stuffs

Method 1: Extraction of DNA from Onions

Materials:

1. 500 grams (1.1 pounds) of pre-chopped regular store bought onions	4. 90 milliliters (3 fluid oz.) of regular store bought shampoo or 90 milliliters (3 fluid oz.) of regular store bought liquid detergent
2. 5 grams (0.17 oz.) of regular store bought table salt	5. 1 gram (1 good sized pinch) of regular store bought meat tenderizer
3. 1500 milliliters (50.7 fluid oz.) of warm tap water	6. 1500 milliliters (50.7 fluid oz.) of 70% rubbing alcohol (non-colored)

Hazards: Rubbing alcohol is flammable.

Procedure: Into a suitable standard kitchen blender, place 500 grams (1.1 pounds) of pre-shopped regular store bought onions, and then add in 5 grams (0.17 oz.) of regular store bought table salt (non iodized), and then followed by 1500 milliliters (50.7 fluid oz.) of warm tap water. Thereafter, blend the mixture three times, blending for 15 seconds each time, for a total of about 45 seconds. Now, pour the total blended mixture through a regular strainer into a separate container, to separate out any large non-water soluble materials. After the straining process, add to the strained liquid, about 90 milliliters (3 fluid oz.) of regular store bought shampoo or 90 milliliters (3 fluid oz.) of regular store bought liquid detergent, and then gently blend the total mixture to evenly distribute the shampoo or detergent. Thereafter, allow the total mixture to stand at room temperature for about 10 minutes. Thereafter, divide the mixture up into 20 different 75-milliliter fractions (2.5 fluid oz. fractions), and then pour each 75-milliliter fraction into a respective 150-milliliter sized test tube or any suitable vessel that is long and slender. Now, into each 150-milliliter test tube, add in 1 gram (1 good sized pinch) of regular store bought meat tenderizer (which contains a mixture of special enzymes), and then very gently blend the mixture in each test tube for about 15 to 20 seconds to evenly distribute the meat tenderizer. Now, into each 150-milliliter test tube, gradually and gently pour in 75 milliliters (2.5 fluid oz.) of 70% rubbing alcohol (non-colored, available in most stores). Note: add the alcohol by tilting each 150-milliliter test tube and then slowly adding in the alcohol; during the slow addition of the alcohol, a second upper layer will form. Note: do not add the alcohol too fast, or do not shake or blend the mixture as the alcohol will dissolve into the water and hence, the

DNA will not precipitate properly. The best way to add the alcohol is to allow it run down the inner side of the test tube. The rubbing alcohol will comprise the top layer, and is where the DNA will end up. After the addition of the alcohol, allow each test tube to stand at room temperature for about a good 2 hours, at which time the DNA will form a stringy insoluble mass, that can be collected by “hooking it” with a needle or small metal hook, or simply “grabbed” by a glass stir rod or slender spatula and removed. The collected DNA should then be air-dried.

Method 2: Extraction of DNA from wheat germ

Materials:

1. 3 grams (1/10 th an oz.) of raw wheat germ	3. 10 milliliters (0.33 fluid oz.) of regular store bought laundry detergent
2. 60 milliliters (2 fluid oz.) of hot tap water	4. 42 milliliters (1.4 fluid oz.) of 95% ethyl alcohol

Hazards: Alcohol is flammable.

Procedure: Into a 150-milliliter test tube, or equivalent, place 3 grams (1/10th an oz.) of raw wheat germ, (toasted wheat germ will not work). Thereafter, add in 60 milliliters (2 fluid oz.) of hot tap water, and then thoroughly blend the mixture using a glass stir rod for about 10 minutes. Thereafter, add in 10 milliliters (0.33 fluid oz.) of regular store bought laundry detergent, and then continue to blend the mixture for about 10 minutes to form a uniform mixture. Note: while blending, try not to blend so hard that a foam forms. After mixing for about 10 minutes, allow the mixture to stand, and then slowly add in 42 milliliters (1.4 fluid oz.) of 95% ethyl alcohol. Note: to add the alcohol, tilt the test tube, and then slowly add in, the alcohol drop by drop. If the alcohol is added to fast, or the mixture is shaken or stirred, the alcohol will dissolve in the water, and the DNA cannot precipitate. The best way to add the alcohol is to allow it run down the inner side of the test tube. Note: the alcohol will form an upper layer, which is where the DNA will form. The DNA precipitates between the boundaries of the bottom and upper layers. After the addition of the alcohol, allow the test tube to stand at room temperature for about a good 20 to 30 minutes, at which time the DNA will form a stringy insoluble mass, that can be collected by “hooking it” with a needle or small metal hook, or simply “grabbed” by a glass stir rod or slender spatula and removed. The collected DNA should then be air-dried

Method 3: Extraction of DNA from Kiwi

Materials:

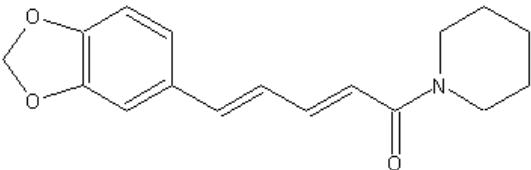
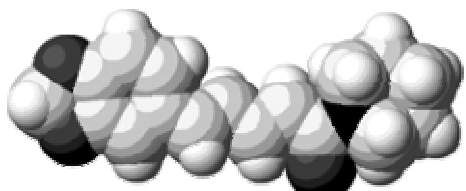
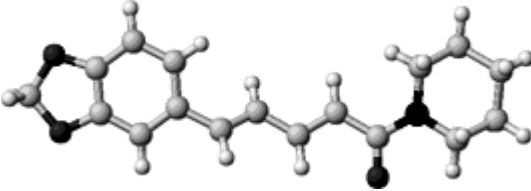
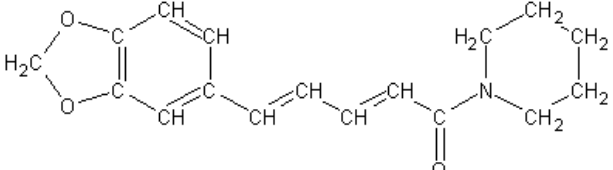
1. Three whole kiwi fruits	4. 200 milliliters (6.7 fluid oz.) of distilled water
2. 30 milliliters (1 fluid oz) of any standard store bought liquid detergent or shampoo	5. 200 milliliters (6.7 fluid oz.) of 70% rubbing alcohol
3. 4.5 grams (0.99 oz.) of regular store bought table salt	

Hazards: Alcohol is flammable.

Procedure: Obtain three whole kiwi fruits, and then peel the fruits. Thereafter, cut the fruit up into moderate sized pieces, and then crush these pieces into a mush, by mashing the pieces together. Thereafter, prepare a solution by adding 30 milliliters (1 fluid oz) of any standard store bought liquid detergent or shampoo, and 4.5 grams (0.99 oz.) of regular store bought table salt (non-iodized salt), into 200 milliliters (6.7 fluid oz.) of distilled water. Thereafter, blend the mixture to dissolve the salt. Note: try not to stir so hard so as a foam forms. If a foam forms, allow the solution to stand until the bulk of the foam disappears. Now, add in the mashed kiwi, and then gently stir the mixture to form a uniform mass. Note: do not blend or stir the mixture to hard so as a foam forms. Thereafter, filter the mixture using a single coffee filter. Note: this filtration process can take up to 24 hours, so set the mixture aside to allow it to filter overnight. The next day, most of the liquid should have been collected. Thereafter, pour this liquid into five, 100-milliliter test tubes filling each test tube about half way up with the filtered kiwi mixture. Thereafter, into each test tube, gradually and gently pour in 50 milliliters (1.6 fluid oz.) of 70% rubbing alcohol (non-colored, available in most stores). Note: add the alcohol by tilting each test tube and then slowly adding in the alcohol; during the slow addition of the alcohol, a second upper layer will form. Note: do not add the alcohol too fast, or do not shake or blend the mixture as the alcohol will dissolve into the water and hence, the DNA will not precipitate properly. The best way to add the alcohol is to allow it run down the inner side of the test tube. The rubbing alcohol will comprise the top layer, and is where the DNA will end up. After the addition of the alcohol, allow each test tube to stand at room temperature for about a good 20 to 30 minutes, at which time the DNA will form a stringy insoluble mass, that can be collected by “hooking it” with a needle or small metal hook, or simply “grabbed” by a glass stir rod or slender spatula and removed. The collected DNA should then be air-dried.

Procedure 005: Extraction process 5: Extraction of Piperine from black pepper

Chemical name of Piperine:

	 3D Structure 01  3D Structure 02
Chemical structure of Piperine	3D Structure(s)
	$C_{17}H_{19}NO_3$
Structure make-up	Condensed chemical structure

Piperine

Piperine forms monoclinic crystals or prisms when recrystallized from alcohol. The crystals have a melting point of 130 Celsius. The crystals are at first tasteless, but then rapidly impart a burning taste when ingested. Piperine is insoluble in water, slightly soluble in alcohol, and soluble in chloroform, benzene, and acetic acid. Piperine is readily extracted from black pepper, and is one of the chief compounds responsible for the characteristic taste of black pepper.

Method 1: Extraction of piperine from black pepper (courtesy of A Laboratory History of Narcotics vol. 1, by Jared Ledgard)

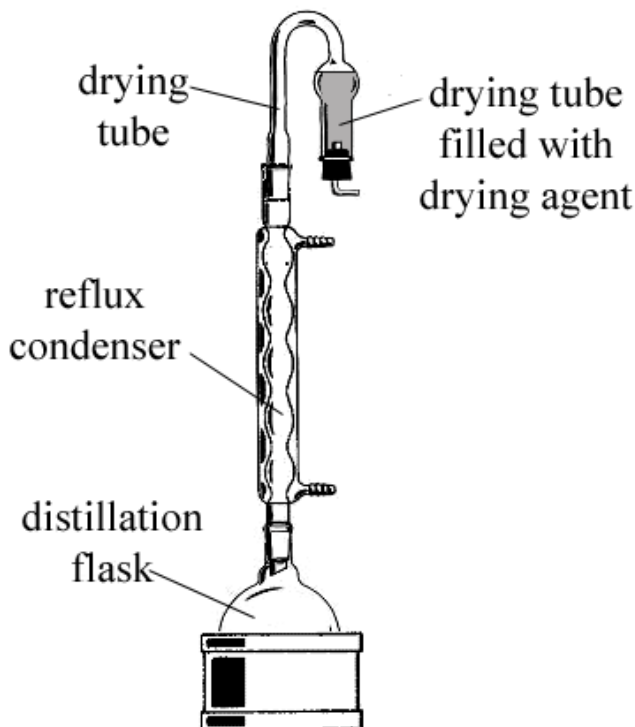
Materials:

1. 75 grams (2.6 oz.) of powdered or finely ground black pepper	4. 65 milliliters (2.2 fluid oz.) of warm water
2. 750 milliliters (25.3 fluid oz.) of 95% ethyl alcohol	5. 65 milliliters (2.2 fluid oz.) of more water
3. 50 milliliters (1.7 fluid oz.) of a 10% potassium hydroxide solution in 95% ethyl alcohol	6. 100 milliliters (3.4 fluid oz.) of acetone

Hazards: Wear gloves when handling potassium hydroxide, which is very corrosive. Extinguish all flames before using ethyl alcohol, and acetone, both of which are flammable.

Procedure: Into a standard reflux apparatus, place 75 grams (2.6 oz.) of powdered or finely ground black pepper (if using fresh black pepper corns or granules, the corns or granules should be finely ground before using). Note: 75 grams of black pepper is about 2/3 of a normal bottle sold in the grocery store. After adding the black pepper to the reflux apparatus, add in 750 milliliters (25.3 fluid oz.) of 95% ethyl alcohol. Thereafter, reflux the mixture at 78 Celsius for about 4 or 5 hours. After the reflux extraction process, remove the heat source, and allow the alcohol mixture to cool to room temperature. Thereafter, filter the alcohol extract to remove insoluble materials, and then place this filtered alcohol extract into a distillation apparatus, and distill-off the ethyl alcohol at 78 Celsius until the total remaining volume is about 75 milliliters (2.5 fluid oz.). When most

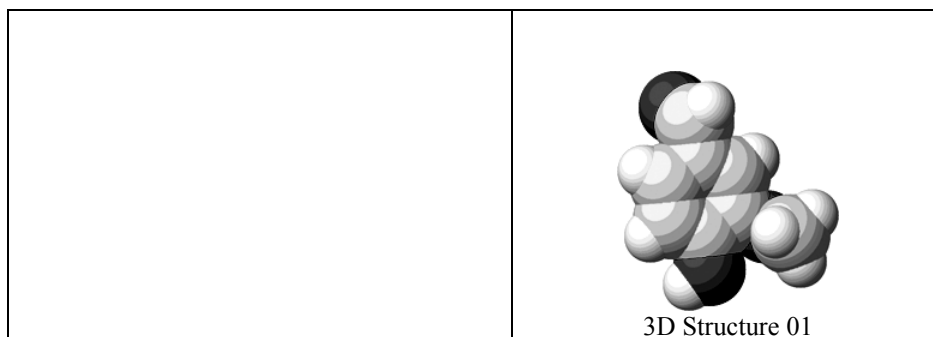
of the ethyl alcohol has been removed, and the left over remaining alcohol concentrate is around 75 milliliters (2.5 fluid oz.) in volume, stop the distillation process, and collect the left over remaining alcohol concentrate (after it has cooled), and place it into a clean beaker. Then, into a second clean beaker, add in 50 milliliters (1.7 fluid oz.) of a 10% potassium hydroxide solution in 95% ethyl alcohol. Thereafter, to the potassium hydroxide/alcohol solution, add in the concentrated alcohol extract, and thereafter, heat the total mixture at about 60 to 70 Celsius. When the temperature of this mixture reaches 60 to 70 Celsius, slowly add drop wise, 65 milliliters (2.2 fluid oz.) of warm water. Note: during the addition of the water, the desired piperine compound will gradually precipitate. When precipitation begins, remove the heat source, and allow the alcohol mixture to cool to room temperature, and during this cooling period continue to add the water, slowly and drop-wise. When the mixture has cooled to room temperature, add in 65 milliliters (2.2 fluid oz.) of more water (cold water this time), and then stir the entire mixture for about 30 minutes at room temperature, and then allow the entire mixture to stand (no stirring) for several hours at room temperature. Afterwards, filter-off the precipitated solid, and then vacuum dry or air-dry it. Finally, recrystallize this dry solid from 100 milliliters (3.4 fluid oz.) of acetone, and after the recrystallization process, vacuum dry or air-dry the filtered-off crystals. The result will be about 3 grams (0.1 oz.) of the desired piperine compound with a melting point of 128 Celsius.

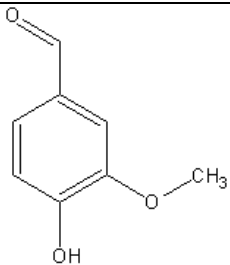
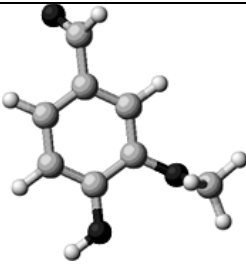
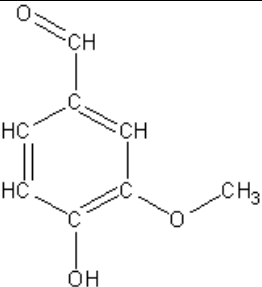


Reflux apparatus equipped with drying tube for the extraction of piperine from black pepper. Cold water should be circulated through the reflux condenser jacket.

Procedure 006: Extraction process 6: Extraction of vanillin from vanilla extract

Chemical name of vanillin: 4-hydroxy-3-methoxybenzaldehyde



	 3D Structure 02
Chemical structure of vanillin	3D Structure(s)
	$C_8H_8O_3$
Structure make-up	Condensed chemical structure

Vanillin (4-hydroxy-3-methoxybenzaldehyde)

Vanillin forms white to slightly yellow needle like crystals, which have a very pleasant taste and odor. The crystals are slowly oxidized on exposure to air and light, and should be stored in airtight amber glass bottles. The crystals have a melting point of 80 to 81 Celsius, and a boiling point of 285 Celsius with some possible decomposition. The crystals are not very soluble in water, but are freely soluble in alcohol, chloroform, and most common solvents. Vanillin is one of the major compounds responsible for the characteristic taste of vanilla.

Method 1: Extraction of vanillin from store bought vanilla extract (courtesy of A Laboratory History of Narcotics vol. 1, by Jared Ledgard)

Materials:

1. 75 milliliters to 118 milliliters (2.5 to 4 fluid oz.), of grocery store brand vanilla extract	3. Three 50-milliliter portions (three 1.6 fluid oz. portions) of diethyl ether
2. 50 milliliters (1.6 fluid oz.) of warm water	4. 10 grams (0.35 oz.) of anhydrous magnesium sulfate

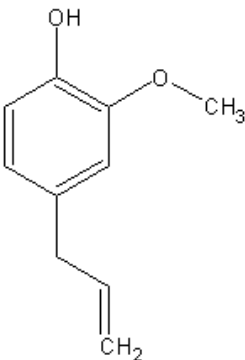
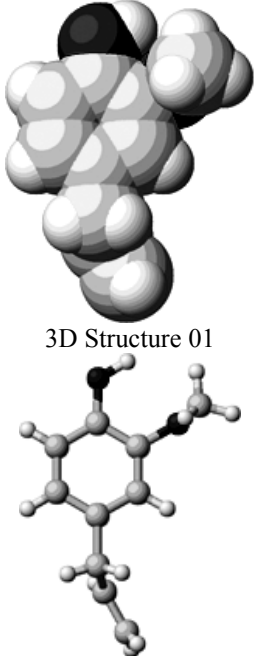
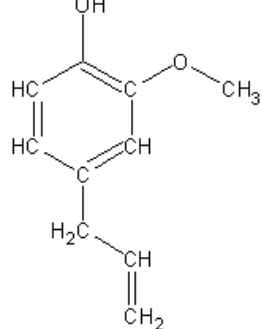
Hazards: Extinguish all flames before using diethyl ether, which is highly flammable and capable of forming explosive mixtures with air.

Procedure: Pour a large bottle (75 milliliters to 118 milliliters, 2.5 to 4 fluid oz.) of grocery store brand vanilla extract into a suitable beaker, and then add in 50 milliliters (1.6 fluid oz.) of warm water. Then extract this entire mixture with three 50-milliliter portions (three 1.6 fluid oz. portions) of diethyl ether, and after the extraction process, combine all ether portions (if not already done so), and then dry this combined ether portion by adding to it, 10 grams (0.35 oz.) of anhydrous magnesium sulfate. Then stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Then place this filtered ether mixture into a distillation apparatus, and distill-off the ether at 40 Celsius. When no more ether passes over or is collected, stop the heating process, and recover the left over remaining residue (after it has cooled to room temperature), and then vacuum dry or air-dry this collected residue. Thereafter, set this dry residue aside just for a moment. Now, depending on how much residue you have (based on what quantity of grocery store vanilla extract you purchased), add your collected left over residue into heated water contained in suitable sized beaker. In other words, place 20 milliliters (0.67 fluid oz.) of water per 1 gram (0.04 oz.) of your residue into a breaker, and heat to 80 Celsius—thereby, add in your residue. After you add in the residue, continue to heat the water mixture at 80 Celsius with moderate stirring for about 15 minutes, and then quickly filter this water mixture (before it cools), and then place the filtered water mixture into a clean beaker, and allow it to cool to room temperature—whereby crystals of vanillin will form. After the water mixture has cooled to room temperature, place it into an ice bath (or use a freezer), and allow the mixture to stand at 0 Celsius for 1 hour. Then filter-off the precipitated crystals of

vanillin, and then vacuum dry or air-dry the crystals. Note: the crystals should be stored in airtight bottles in a cool place to prevent oxidation. Note: there are numerous modifications to this extraction process.

Procedure 007: Extraction process 7: Extraction of Eugenol from cloves

Chemical name of Eugenol: 4-allyl-2-methoxyphenol

	 <p>3D Structure 01</p> <p>3D Structure 02</p>
Chemical structure of Eugenol	3D Structure(s)
	$C_{10}H_{12}O_2$
Structure make-up	Condensed chemical structure

Eugenol (4-allyl-2-methoxyphenol)

Eugenol forms a colorless to pale yellowish liquid with a boiling point of 255 Celsius. Eugenol slowly turns dark on exposure to air, so it should be stored in airtight bottles in a cool place. Eugenol has a powerful odor of cloves, from which it is readily extracted from ordinary spice cloves. Eugenol has a melting point of -9 Celsius, so the oil may crystallize on standing under cold temperatures. Eugenol is miscible with alcohol, methylene chloride, and ether, but insoluble in water. Eugenol is a major starting point for the preparation of psychedelic amphetamines.

Method 1: Extraction of eugenol from store bought cloves (courtesy of A Laboratory History of Narcotics vol. 1, by Jared Ledgard)

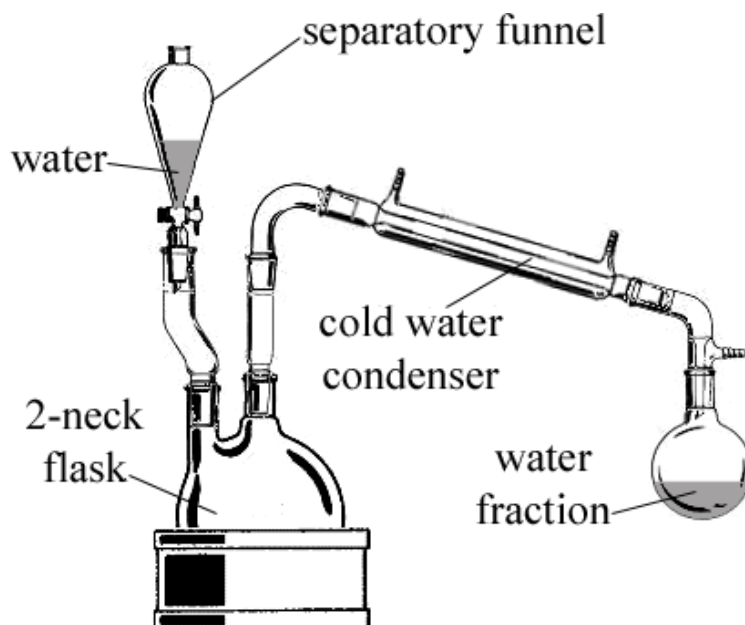
Materials:

1. 100 grams (3.5 oz.) of cloves (regular store bought cloves)	7. 250 to 300 milliliters (8.5 to 10.1 fluid oz.) of a 5% hydrochloric acid solution
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2. 500 milliliters (17 fluid oz.) of water	8. Four 40-milliliter portions (four 1.4 fluid oz. portions) of methylene chloride
3. 250 milliliters (8.4 fluid oz.) of water	9. 50-milliliter portion (1.7 fluid oz.) of water
4. three 50-milliliter portions (three 1.7 fluid oz. portions) of methylene chloride	10. 50 milliliter portion (1.7 fluid oz.) of a 23% sodium chloride solution
5. six 50-milliliter portions (six 1.7 fluid oz. portions) of a 5% potassium hydroxide solution	11. 15 grams (0.52 oz.) of anhydrous sodium sulfate
6. 50 milliliters (1.7 fluid oz.) of methylene chloride	

Hazards: Wear gloves when handling potassium hydroxide and hydrochloric acid, both of which are capable of causing skin burns.

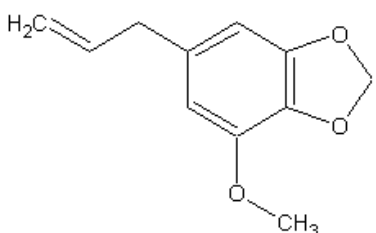
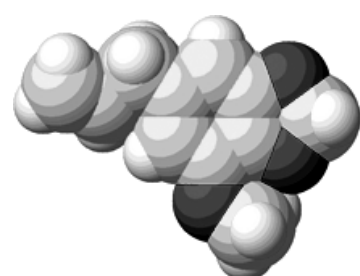
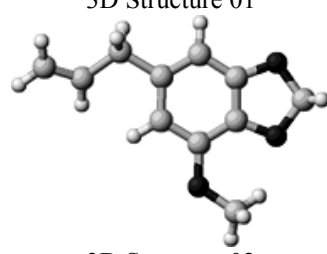
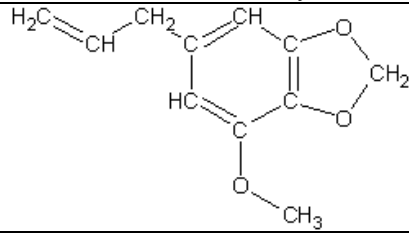
Procedure: Into a suitable steam distillation apparatus (fitted with a 250 milliliter addition funnel, or better), place 100 grams (3.5 oz.) of cloves (regular store bought cloves). Thereafter, add in 500 milliliters (17 fluid oz.) of water, and then add 250 milliliters (8.4 fluid oz.) of water to the addition funnel. This 250-milliliter addition funnel should contain about 200 milliliters of water at all times, and the water therein should be added to the cloves and water mixture periodically to keep the flask's water volume at around 500 milliliters all throughout the steam distillation process. Then heat the cloves and water mixture to 105 to 110 Celsius, and allow the mixture to be steam distilled. The process should take about 150 minutes, and thereafter, stop the steam distillation process, and then recover the oily distillate in the receiver flask. Then extract this oily distillate with three 50-milliliter portions (three 1.7 fluid oz. portions) of methylene chloride, and after the extraction, combine all methylene chloride portions (if not already done so). Note: after each extraction, the methylene chloride will be the bottom layer each time. After the extraction, the upper water layer can be discarded. Now, extract the combined methylene chloride portion with six 50-milliliter portions (six 1.7 fluid oz. portions) of a 5% potassium hydroxide solution. After the extraction, combine all aqueous alkaline portions (if not already done so), and then briefly wash this combined aqueous alkaline portion with one portion of 50 milliliters (1.7 fluid oz.) of methylene chloride. Note: after the extraction and washing, the aqueous alkaline portion will be the upper layer each time. After the extraction and washing, the methylene chloride can be recycled if desired. Then place this combined aqueous alkaline portion into a large beaker, and then carefully add in, slowly, 250 to 300 milliliters (8.5 to 10.1 fluid oz.) of a 5% hydrochloric acid solution. Note: more or less acid may or may not be needed, and the acid is added solely to bring the pH of the aqueous mixture (in the beaker) to about 1—add as much acid as needed to reach a pH of about 1. After adding the acid, moderately stir the entire acidic mixture for about 30 minutes. Then, extract this entire acidic mixture with four 40-milliliter portions (four 1.4 fluid oz. portions) of methylene chloride. After the extraction process, combine all methylene chloride portions (if not already done so), and then wash this combined methylene chloride portion with one 50-milliliter portion (1.7 fluid oz.) of water, followed by one 50 milliliter portion (1.7 fluid oz.) of a 23% sodium chloride solution. Note: after the extraction and washings, the methylene chloride will be the lower layer each time. After the extraction and washing portions, dry the washed methylene chloride portion by adding to it, 15 grams (0.52 oz.) of anhydrous sodium sulfate, and then stir the entire mixture for about 10 minutes—thereafter, filter-off the sodium sulfate. Finally, place this filtered dried methylene chloride portion into a distillation apparatus, and distill-off the methylene chloride at 40 Celsius. When no more methylene chloride passes over or is collected, remove the left over remaining pale yellow oil (after it has cooled), and then store it in an amber glass bottle in a refrigerator until use. Note: the eugenol at this point will have a purity of about 98%.



Standard steam distillation apparatus. The addition funnel should be filled with water at all times.

Procedure 008: Extraction process 8: Extraction of Myristicin from nutmeg or nutmeg butter

Chemical name of Myristicin: 6-allyl-4-methoxy-1,3-benzodioxole

	 <p>3D Structure 01</p>  <p>3D Structure 02</p>
<p>Chemical structure of Myristicin</p>	<p>3D Structure(s)</p>
	<p>$C_{11}H_{12}O_3$</p>
<p>Structure make-up</p>	<p>Condensed chemical structure</p>

Myristicin (6-allyl-4-methoxy-1,3-benzodioxole)

Myristicin forms a colorless to yellowish oil (depending on purity), with a boiling point (at 40 milliliters of mercury) of 173 Celsius. Myristicin exists naturally in nutmeg, carrots, and parsley, from which it can be extracted—especially from the corresponding oils.

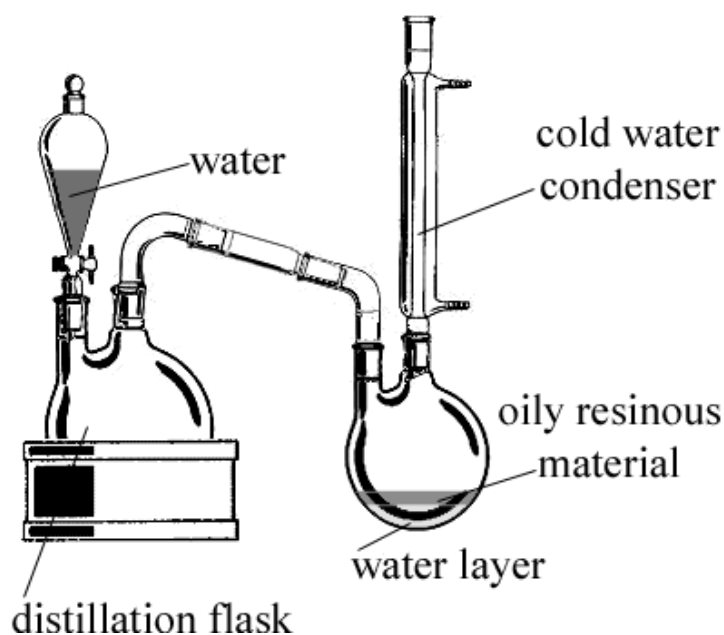
Method 1: Extraction of myristicin from store bought nutmeg (courtesy of A Laboratory History of Narcotics vol. 1, by Jared Ledgard)

Materials:

1. 100 grams (3.5 oz.) of powdered nutmeg (regular store bought nutmeg)	4. 10 grams (0.35 oz.) of anhydrous magnesium sulfate
2. 750 milliliters (25.3 fluid oz.) of water	5. 200 milliliters (6.8 fluid oz.) of boiling 95% ethyl alcohol
3. Three 75-milliliter portions (three 2.5 fluid oz. portions) of pre-heated methylene chloride	6. 5 grams (0.17 oz.) of anhydrous sodium sulfate

Hazards: Ethyl alcohol is flammable, so extinguish all flames before using.

Procedure: Into the steam distillation apparatus as illustrated in the following drawing, place 100 grams (3.5 oz.) of powdered nutmeg (regular store bought nutmeg), followed by 750 milliliters (25.3 fluid oz.) of water. Thereafter, steam distill this mixture at 100 Celsius for about 4 to 6 hours. Note: the exact steam distillation process may vary, and should be continued until no more oily resinous material is seen collecting in the receiver flask. When no more oily resinous material is seen collecting in the receiver flask, stop the steam distillation process, and then recover the entire oily resinous aqueous mixture from the receiver flask, and then place this mixture into a beaker, and then gently heat to about 50 Celsius for about 10 minutes. Then, before the oily water mixture cools to below 50 Celsius, place it into a separatory funnel, and then collect the upper oil layer. In some cases, the oil layer will be the bottom layer. Thereafter, extract this collected oil layer (before it cools to below 40 Celsius), with three 75-milliliter portions (three 2.5 fluid oz. portions) of pre-heated methylene chloride (pre-heated to about 40 Celsius), and after the extraction process, combine all warm methylene chloride portions (if not already done so), and then dry this combined warm methylene chloride portion by adding to it, 10 grams (0.35 oz.) of anhydrous magnesium sulfate. Note: after each extraction, the warm methylene chloride portion can be simply decanted-off rather than recovered by using a separatory funnel. After adding in the anhydrous magnesium sulfate, stir the entire combined warm methylene chloride portion for about 10 minutes, and then filter-off the magnesium sulfate. Note: if during the stirring process (with the magnesium sulfate), the combined methylene chloride portion cools to below 30 Celsius, gently warm the entire mixture to 40 Celsius. Then place this warm methylene chloride portion into a distillation apparatus, and distill-off the methylene chloride at 40 Celsius. When no more methylene chloride passes over or is collected, stop the distillation process, and then recover the left over remaining oil (before it cools to below 40 Celsius). Immediately thereafter, dissolve this recovered warm oil into 200 milliliters (6.8 fluid oz.) of boiling 95% ethyl alcohol (pre-heated to about 78 Celsius), and then quickly stir the entire alcohol mixture for about 5 minutes, and then filter-off any insoluble impurities (if any). Note: filter the alcohol mixture while its still boiling hot. After the filtration process, allow the alcohol mixture to slightly cool to about 60 Celsius, and then place it into an ice bath, and chill it to about 0 Celsius for about 2 hours. Note: a freezer can be used by itself or in combination with the ice bath. After chilling the alcohol mixture for about 2 hours, filter-off the crystallized myristicin, and then quickly vacuum dry this myristicin product (before it warms to above 5 Celsius). Note: air drying will not work, and if desired, the myristicin can be dried by gently heating the crystals of the myristicin to induce liquification, and then adding in 5 grams (0.17 oz.) of anhydrous sodium sulfate (to absorb any moisture). After adding in the sodium sulfate, stir the entire mixture for about 10 minutes, and then filter-off the sodium sulfate. The oil should then be stored in an amber glass bottle until use. Note: there are numerous modifications to this process, and those with experience should attempt any modifications they see fit.



Setup for the steam distillation of nutmeg.

Method 2: Extraction of myristicin from nutmeg butter (courtesy of A Laboratory History of Narcotics vol. 1, by Jared Ledgard)

Materials:

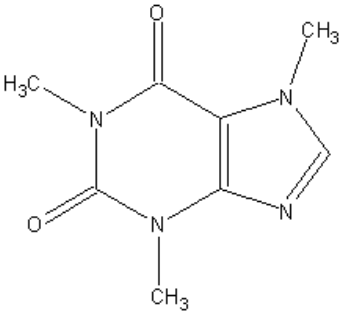
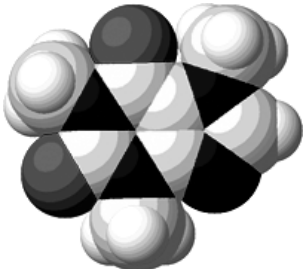
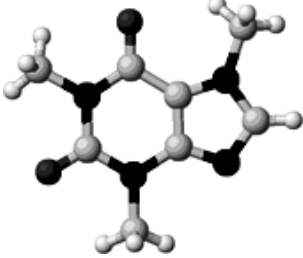
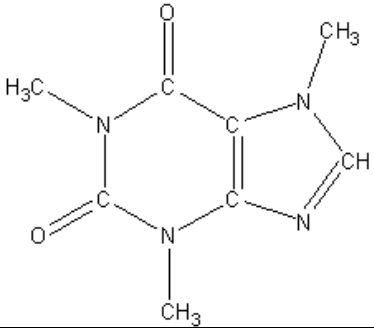
1. 50 grams (1.8 oz.) of commercially available nutmeg butter	3. 100 milliliters (3.4 fluid oz.) of pre-heated diethyl ether
2. 500 milliliters (17 fluid oz.) of boiling 95% ethyl alcohol	4. 5 grams (0.17 oz.) of anhydrous sodium sulfate

Hazards: Use care when handling diethyl ether, which is highly flammable, and capable of forming explosives mixtures with air—use proper ventilation and extinguish all flames before using.

Procedure: Nutmeg butter is a product that is obtained by pressing nutmeg between heated plates in the presence of a small amount of steam. Nutmeg butter is composed primarily of myristicin, glycerides of myristic acid and other fats, and residue. The myristicin portion can be obtained by treating the nutmeg butter with ether or alcohol. To isolate myristicin from nutmeg butter, thoroughly mix 50 grams (1.8 oz.) of commercially available nutmeg butter with 500 milliliters (17 fluid oz.) of boiling 95% ethyl alcohol. Note: make sure the 95% ethyl is boiling at 78 to 79 Celsius before adding in the nutmeg butter. While adding in the nutmeg butter, rapidly stir the boiling alcohol mixture, and after the addition of the nutmeg butter, place the entire alcohol mixture (including any and all insoluble solids) into a reflux apparatus (before the alcohol cools), and then reflux the entire mixture at about 79 Celsius for 2 hours. After 2 hours, quickly remove the reflux condenser, and replace it with a conventional condenser fitted with a receiver flask, and then distil-off the 95% ethyl alcohol until about 50% of the total volume remains (distill-off about 250 milliliters of the ethyl alcohol). When the alcohol mixture has been reduced to a total volume of about 50%, allow the alcohol concentrate to cool to about 60 Celsius, and then filter the entire alcohol mixture to remove any insoluble impurities. Note: this filtration process should be carried out before the alcohol mixture cools to below 60 Celsius. After the filtration process, place the entire filtered alcohol concentrate (even if two or more layers exist) into an ice bath, and chill it to about 0 Celsius. Note: a freezer can be used by itself or in combination with the ice bath. Then allow the alcohol concentrate to chill at 0 Celsius for about 2 hours. After 2 hours, filter-off the precipitated crystals of the myristicin (before the alcohol concentrate warms to above 5 Celsius), and then place these filtered-off crystals (before they have a chance to warm to above 10 Celsius) into a suitable beaker, and then add in 100 milliliters (3.4 fluid oz.) of pre-heated diethyl ether (pre-heated to about 40 Celsius). Thereafter, stir the entire warm ether mixture for about 30 minutes, and then filter-off any insoluble impurities (if any). Then, place this warm ether mixture into a distillation apparatus, and distil-off the ether only until about 25% of the total volume has been reduced (distill-off only about 25 milliliters of ether). When 75% of the total ether volume remains, stop the distillation process, and then place the ether concentrate into an ice bath (before it cools), and then chill it to about 0 Celsius for about 1 hour. Note: a freezer can be used instead of an ice bath or in combination with. After chilling this ether concentrate to about 0 Celsius for 1 hour, filter the ether mixture to recover the crystallized myristicin (before it warms to above 5 Celsius), and then vacuum dry these filtered-off crystals of the myristicin (before they warm to above 5 Celsius). Note: air drying will not work, and if desired, the myristicin can be dried by gently heating the crystals of the

myristicin to induce liquefaction, and then adding in 5 grams (0.17 oz.) of anhydrous sodium sulfate (to absorb any moisture). After adding in the sodium sulfate, stir the entire mixture for about 10 minutes, and then filter-off the sodium sulfate. The oil should then be stored in an amber glass bottle until use. Note: there are numerous modifications to this process, and those with experience should attempt any modifications they see fit.

Procedure 009: Extraction process 9: Extraction of Caffeine from tealeaves

	 3D Structure 01  3D Structure 02
Chemical structure of Caffeine	3D Structure(s)
	$\text{C}_8\text{H}_{10}\text{N}_4\text{O}_2$
Structure make-up	Condensed chemical structure

Caffeine

Caffeine forms white hexagonal crystals by sublimation. Caffeine has a melting point of 238 Celsius, but the crystals begin to sublime when heated to 178 Celsius. Caffeine is only moderately soluble in water, but more soluble in hot water. The crystals are also moderately soluble in alcohol, acetone, but are much more soluble in methylene chloride, chloroform, and practically insoluble in ether. Caffeine is capable of forming a hydrate, which loses its water of hydration when heated to 80 Celsius. Caffeine is a widely used stimulant, ingested by millions in the form of coffee, tea, etc.,

Method 1: Extraction of caffeine from tea leaves

Materials:

1. 825 milliliters (28 fluid oz.) of water	5. Four 90-milliliter portions (four 3 fluid oz. portions) of methylene chloride
2. 60 grams (2.1 oz.) of sodium carbonate	6. 15 grams (0.52 oz.) of anhydrous sodium sulfate
3. 30 to 40 tea bags (any brand of tea can be used)	7. 21 milliliters (0.71 fluid oz.) of toluene

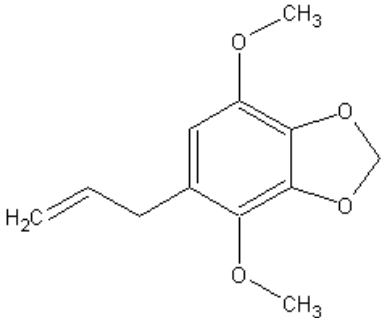
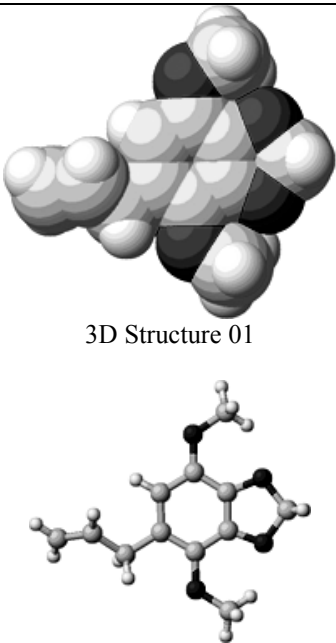
4. 90 milliliters (3 fluid oz.) of methylene chloride

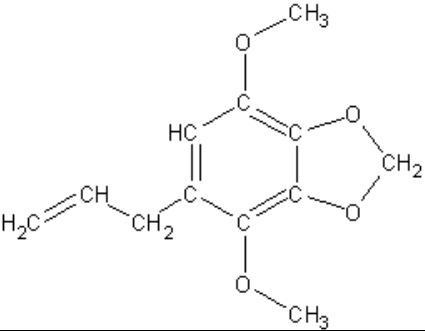
8. 30 milliliters (1 fluid oz.) of hexane

Hazards: Use proper ventilation when using toluene and hexane, and avoid inhalation of the fumes.

Procedure: Into a suitable beaker or flask, place 825 milliliters (28 fluid oz.) of water, and then add and dissolve 60 grams (2.1 oz.) of sodium carbonate. Thereafter, boil the mixture, and once the water begins to boil, add in 30 to 40 tea bags (any brand of tea can be used). Thereafter, boil the mixture and allow the tea bags to soak for 15 minutes in the usual manner. After 15 minutes, remove the heat source, and allow the tea mixture to cool to about 50 Celsius. Thereafter, remove the tea bags, and then allow the tea mixture to cool to room temperature. Thereafter, add in 90 milliliters (3 fluid oz.) of methylene chloride, and then stir the mixture gently for about 30 to 40 minutes. Note: do not shake the mixture vigorously as an emulsion will form. After stirring the mixture for about 30 to 40 minutes, gently pour the mixture into a separatory funnel, and then remove the lower organic solvent layer. Thereafter, place this lower organic layer aside temporarily, and then repeat the extraction process with four 90-milliliter portions (four 3 fluid oz. portions) of methylene chloride upon the upper water layer. After extracting the upper water layer four more times, combine all lower methylene chloride portions, if not already done so, and then dry the combined methylene chloride portions by adding in 15 grams (0.52 oz.) of anhydrous sodium sulfate. Then stir the mixture briefly, and then filter-off the sodium sulfate. Now, place the dried methylene chloride portion into a distillation apparatus, and distill-off the methylene chloride until a dry residue remains. When this point is achieved, remove the heat source, and then collect the dry residue. Finally, recrystallize this dry residue from a toluene/hexane solvent mixture prepared by adding and dissolving 21 milliliters (0.71 fluid oz.) of toluene to 30 milliliters (1 fluid oz.) of hexane, and after the recrystallization process, vacuum dry or air dry the collected caffeine crystals. These crystals can be sublimed using a standard sublimation setup (see iodine) to afford highly pure crystals of 99% purity.

Procedure 010: Extraction process 10: Extraction of Apiole from parsley (advanced process)

 <p>The chemical structure of Apiole is shown. It consists of a benzene ring with two methoxy groups (-OCH₃) at the 1 and 3 positions. At the 4 position, there is a propenyl group (-CH=CH-CH₂-). At the 2 position, there is a five-membered furan ring fused to the benzene ring.</p>	 <p>3D Structure 01</p> <p>3D Structure 02</p>
Chemical structure of Apiole	3D Structure(s)
	C ₁₂ H ₁₄ O ₄

	
Structure make-up	Condensed chemical structure

Apiole

Apiole forms crystals with a melting point of 30 Celsius. Fresh apiole may be a semi-solid liquid. The compound can be distilled at 294 Celsius. Apiole is soluble in alcohol, benzene and chloroform, but insoluble in water. Apiole is a major constituent of parsley, and is responsible for the aroma and taste of parsley.

Method 1: Extraction of Apiole from parsley seeds (courtesy of A Laboratory History of Narcotics vol. 1, by Jared Ledgard)

Materials:

1. 1 kilogram (2.2 pounds) of parsley seeds	5. 50 milliliters (2 fluid oz.) of warm water
2. 500 milliliters (17 fluid oz.) of 95% ethyl alcohol	6. 10 grams (0.35 oz.) of lead-II-oxide
3. Three 50-milliliter portions (three 2 fluid oz. portions) of diethyl ether	7. 50 milliliters (2 fluid oz.) of additional warm water
4. 10 grams (0.35 oz.) of anhydrous sodium sulfate	8. 10 grams (0.35 oz.) of anhydrous sodium sulfate

Hazards: Extinguish all flames before using diethyl ether, which is highly flammable, and can form explosive mixtures with air—use caution.

Procedure: Grind up 1 kilogram (2.2 pounds) of parsley seeds until the seeds are of a finely ground nature. Then place the finely ground seeds into a large reflux apparatus (equipped with motorized stirrer or other stirring means), and then add in 500 milliliters (17 fluid oz.) of 95% ethyl alcohol. Thereafter, reflux the entire mixture at 78 Celsius for about 6 to 8 hours while moderately stirring the ethyl alcohol mixture. After refluxing for about 6 to 8 hours, remove the heat source, and then allow the entire alcohol mixture to cool to room temperature. Thereafter, filter the entire alcohol mixture to remove any insoluble materials, and then place this ethyl alcohol mixture into a distillation apparatus, and distill-off the ethyl alcohol until about 50% of its total volume has been reduced (about 250 milliliters of ethyl alcohol removed). Note: the recovered ethyl alcohol can be recycled if desired. When about 50% of the ethyl alcohol mixture has been removed, stop the distillation process, and then place the ethyl alcohol mixture into a suitable sized beaker (before it cools), and then allow it to cool to room temperature. Then, quickly filter this alcohol concentrated mixture to remove any potential insoluble impurities (if any). Now, extract this entire alcohol mixture with three 50-milliliter portions (three 2 fluid oz. portions) of diethyl ether, and after the extraction process, combine all ether portions (if not already done so), and then dry this combined ether portion by adding to it, 10 grams (0.35 oz.) of anhydrous sodium sulfate. Note: after each extraction, the ether will be the upper layer each time. After adding in the sodium sulfate, stir the entire ether mixture for about 10 minutes, and then filter-off the sodium sulfate. Then, place this filtered ether mixture into a distillation apparatus, and distill-off the ether at 40 Celsius. When no more ether passes over or is collected, stop the distillation process, and then recover the left over remaining oily residue (when it cools to about 40 Celsius). Then place this warm collected left over oily residue into a clean beaker, and then add in 50 milliliters (2 fluid oz.) of warm water, followed by 50 grams (1.8 oz.) of sodium carbonate, and then followed by 10 grams (0.35 oz.) of lead-II-oxide. Thereafter, rapidly blend this entire mixture for about 1 hour at a temperature of about 40 Celsius—a hot plate will be needed in order to keep the temperature of the mixture at about 40 Celsius. After rapidly stirring for about 1 hour, add in 50 milliliters (2 fluid oz.) of additional warm water, and then continue to stir the entire mixture at about 40 Celsius for an additional hour. Thereafter, filter the entire mixture through a layer of charcoal (place a bed of charcoal over the filter paper), before the mixtures temperature drops below 40 Celsius. After the filtration process, place the entire filtered mixture into a clean beaker. Now, extract this entire mixture with three 50-milliliter portions of diethyl ether, and after the extraction process, combine all ether portions (if not already done so), and then dry this combined ether portion, by adding to it, 10 grams (0.35 oz.) of anhydrous sodium sulfate. Note: after each ether extraction, the ether will be the upper layer each time. After adding in the anhydrous sodium sulfate, stir the entire ether mixture for about 10 minutes, and then filter-off the sodium sulfate. Finally,

place this entire ether mixture into a distillation apparatus, and distill-off the ether at 40 Celsius. When no more ether passes over or is collected, stop the distillation process, and then recover the left over remaining oily residue (before it cools to below 40 Celsius). Then place this warm collected left over residue (composed primarily of the desired apiole) into an amber glass bottle, and then store it in a refrigerator until use. Note: There are numerous modifications to this process, and those who are willing, should carryout any modifications that would seem fit.

Method 2: Extraction of Apiole from oil of parsley (courtesy of A Laboratory History of Narcotics vol. 1, by Jared Ledgard)

Materials:

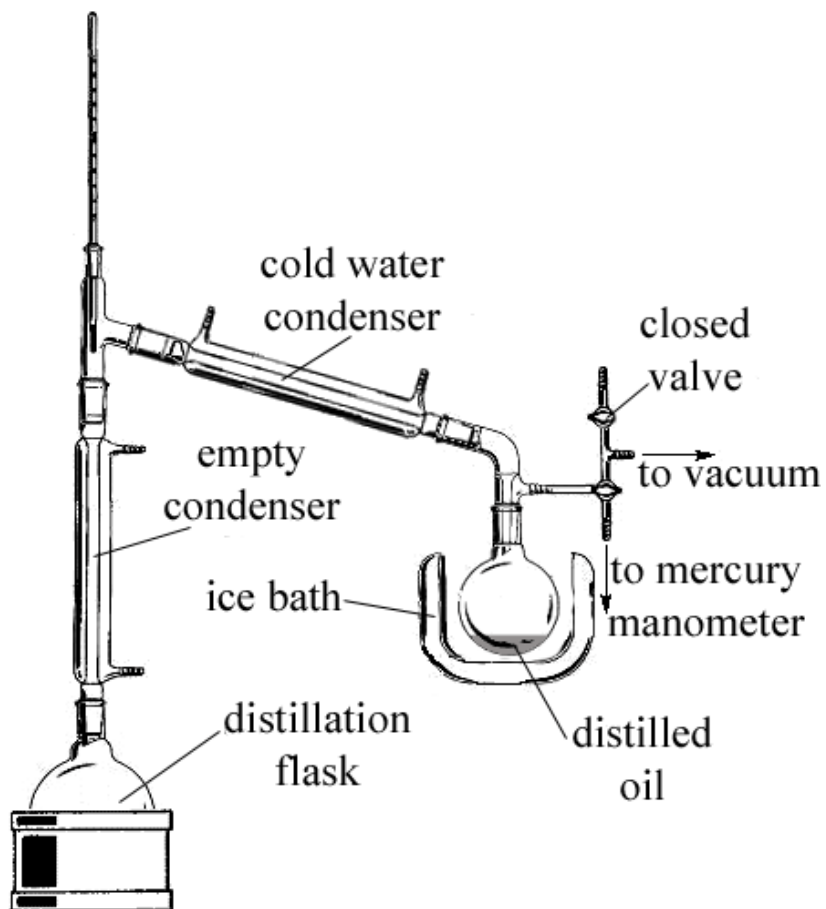
1. 150 grams (5.3 oz.) of commercially available “Oil of Parsley	2. 150 milliliters (5.1 fluid oz.) of ether
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Hazards: Extinguish all flames before using diethyl ether, which is highly flammable, and can form explosive mixtures with air—use caution.

Procedure:

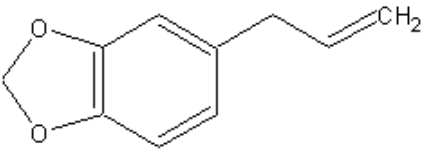
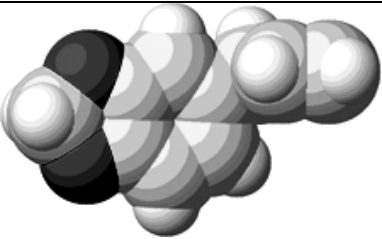
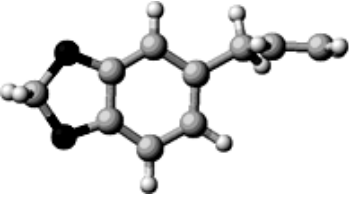
This procedure is an advance procedure for the extraction of apiole from oil of parsley. This process utilizes vacuum distillation.

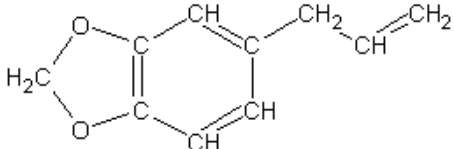
Set-up the vacuum fractional distillation apparatus as illustrated below, and then fractionally distil 150 grams (5.3 oz.) of commercially available “Oil of Parsley”, at 167 Celsius under a vacuum of 27 millimeters of mercury. After the vacuum distillation process, remove the ice trap from the receiver flask, and then allow the receiver flask to warm to room temperature. Thereafter, gently warm the receiver flask using a small Bunsen flame or other means, and allow the crystallized apiole to liquefy into an oil. Then pour this oil into an amber glass bottle, and then store it in a refrigerator until use. Note: in some cases, the apiole can be obtained by allowing the commercial oil of parsley to stand in an ice bath for several hours (a freezer can be used as well). During the chilling process, crystals of apiole will slowly form. These crystals can then be filtered-off, and then dissolved into 150 milliliters (5.1 fluid oz.) of ether. The ether mixture should then be briefly stirred for about 30 minutes, and then filtered to remove any potential insoluble impurities (if any). Then place this filtered ether mixture into a distillation apparatus, and distill-off the ether at 40 Celsius. When all the ether has been removed, place the left over remaining oily residue of the apiole (after it has cooled) into a amber glass bottle and store in a refrigerator until use. Note: There are numerous modifications to this process, and those who are willing, should carryout any modifications they would seem fit.



Advanced setup for vacuum fractional distillation for the isolation of apiole from commercial oil of parsley.

Procedure 011: Extraction process 11: Extraction of Safrole (advanced process 2)

	 3D Structure 01  3D Structure 02
Chemical structure of Safrole	3D Structure(s)
	C ₁₀ H ₁₀ O ₂

	
Structure make-up	Condensed chemical structure

Safrole

Safrole forms a colorless to slightly yellow liquid with the odor of sassafras. The oil is insoluble in water, but very soluble in alcohol, and miscible with chloroform and ether. The oil has a boiling point of 232 Celsius, but can be distilled under high vacuum at 100 Celsius under 11 millimeters of mercury. Safrole is the main component of sassafras oil, from which it makes up 70 to 75% of the oil by weight. Safrole also exists in *Ocotea cymbarum* oil (Brazilian oil of sassafras), which it exists up to 90% by weight. The oil of massoria bark and *Cinnamomum massoia* contains about 14% safrole. Safrole can be extracted from sassafras oil by the means described later, and it can be extracted from Massoria bark oil and *Cinnamomum massoia* by washing the corresponding oil with sodium hydroxide solution to remove the phenols, and then vacuum distilling to obtain the safrole boiling at about 100 Celsius under a vacuum of 11 millimeters of mercury, or by carefully fractionally distilling (two path distillation) the phenol free oil at 228 to 235 Celsius. Safrole can also be made synthetically from rather inexpensive reagents (see safrole). Sassafras oil can be obtained by steam distilling the ground up roots of the sassafras tree, which grows in the mid western United States. Other sassafras species of trees elsewhere in the world can also be used to obtain the safrole by steam distillation from the root. To identify a sassafras tree, consult a book that discusses the various types of trees and plants. The dried root bark of the sassafras tree contains about 10% safrole by weight, and the remainder of the root contains only about 1%. The dried root bark can be obtained from numerous sources, including herb stores, health food suppliers, and botanical suppliers. Sassafras oil can also be obtained from these aforementioned sources; if however your local suppliers do not offer the sale of sassafras oil, request them to order some for you, which they should have no problem doing. Safrole is also used in perfumes, so check out the types of perfumes, and their ingredients. Note: check out your local aromatherapy suppliers, as they are major consumers of oils, one of which may be sassafras oil. Sassafras oil may be used in adulterants in massage oils for use in aromatherapy. *Ocotea cymbarum* oil is obtained by steam distillation of the wood of the *Ocotea pretiosa* tree, which grows in South America. The wood contains about 1% oil by weight, which is easily collected by steam distillation of the wood chips, and the resulting steam distilled product contains about 90% safrole by weight. Distributors of perfume and flavoring compounds may contain this *Ocotea cymbarum* oil. Check the OPD directory for essential oils and botanical companies; also check out small herb shops nationwide.

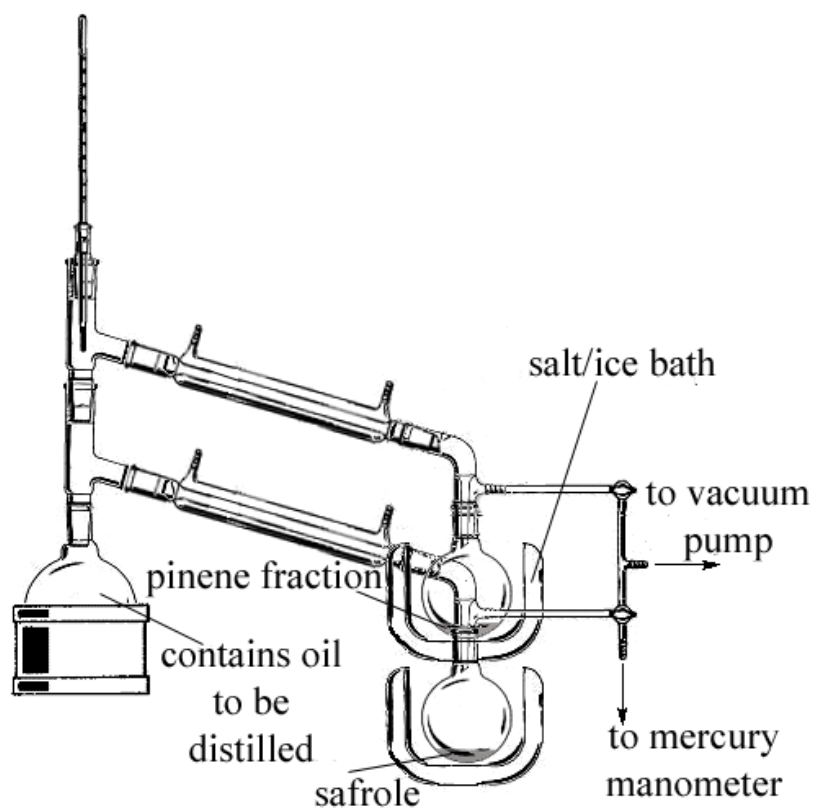
Method 1: Extraction of Safrole from sassafras oil (courtesy of A Laboratory History of Narcotics vol. 1, by Jared Ledgard)

Materials:

1. Sassafras oil or any safrole containing oil, plant, root, ect.	
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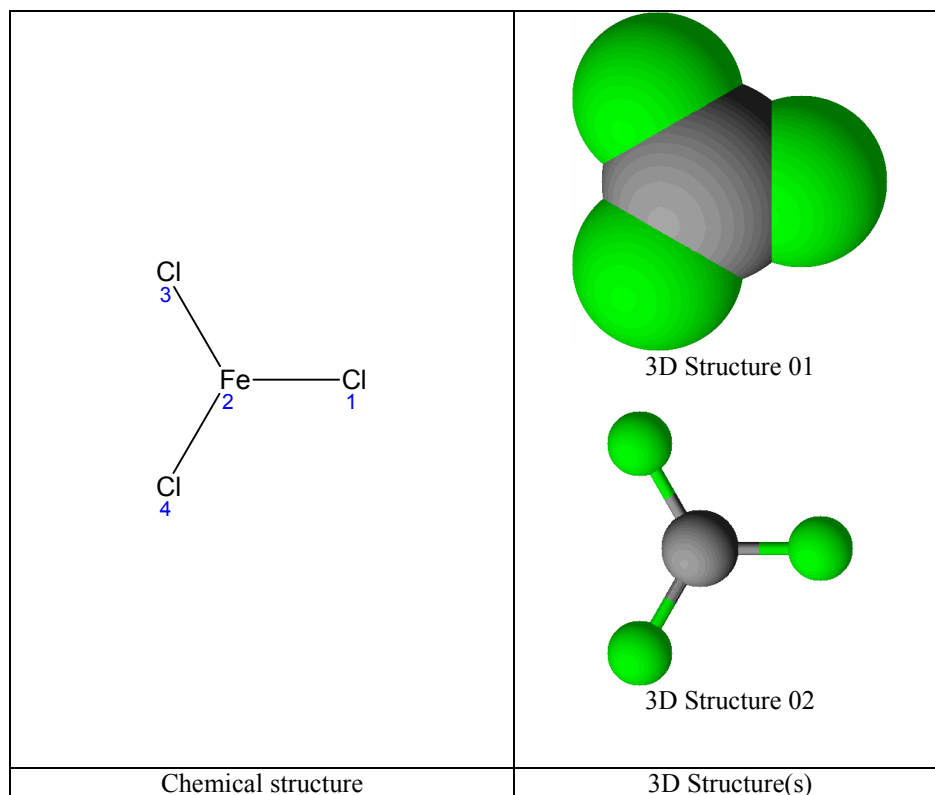
Hazards: None.

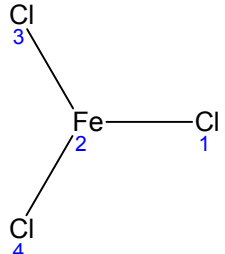
Procedure: Note: as previously mentioned, sassafras oil can be obtained from the root bark of the sassafras tree. To do this, setup a standard steam distillation apparatus, and then steam distill the root bark (grind the root bark into pieces before use). The oil and water collect in the receiver flask, where upon the oil can be seen as droplets. The oil is denser than water so it will form droplets below the water. After the steam distillation process, the oil can be collected by placing the water/oil mixture into a separatory funnel, and then recovering the lower oil layer. The collected oil layer should then be dried by mixing with it, a small amount of anhydrous calcium chloride. After filtering-off the calcium chloride, place the oil into a vacuum distillation apparatus, and vacuum distill at 100 Celsius under a vacuum of 11 millimeters of mercury (see figure below). Note: other oils containing safrole can be vacuum distilled in a similar manner. Note: because of the expense involved in purchasing vacuum distillation apparatus, try freezing the sassafras oil, or other oils that contain the safrole. Safrole has a melting point of 11 Celsius, and it may be possible to crystallize the safrole out of any oil solution by using ice baths, cold-water baths, or even a freezer. Experiment with various techniques; solvent extractions may also work.



Advanced setup, two-path vacuum distillation apparatus for collecting safrole Note: in some cases, the safrole may be the upper fraction, depending on density, and impurities.

Procedure 012: Extraction process 12: Extraction of ferric chloride from aqueous solutions (simple procedure)



 <p>1 iron atom 3 chlorine atoms</p>	FeCl_3
Structure make-up	Condensed chemical structure

Ferric chloride

Ferric chloride forms golden brownish black crystals. The crystals are readily soluble in water or alcohol, and have a melting point ranging from 37 Celsius (if hydrated), but the anhydrous form volatilizes at 300 Celsius. Aqueous solutions of ferric chloride hexahydrate are readily decomposed by boiling, forming red ferric oxide, and hydrogen chloride.

Method 1: Extraction of ferric chloride from aqueous solutions

Materials:

1. 100 grams of ferric chloride, either the anhydrous form, or the hexahydrate (150 grams if the hexahydrate is used).	3. 400 milliliters of tap water.
2. 100 grams of sucrose, table sugar	4. 1200 milliliters of isopropyl rubbing alcohol

Hazards: rubbing alcohol is flammable so use caution near open flames.

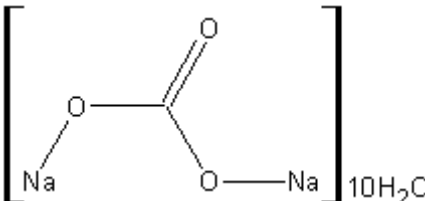
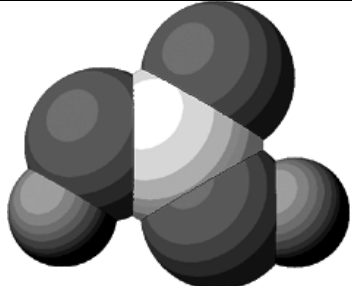
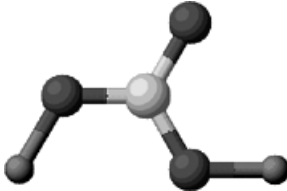
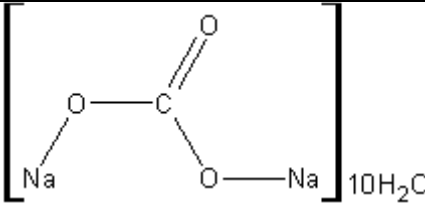
Procedure: Into a suitable beaker of capacity, pour in 400 milliliters of tap water, and then add and dissolve 100 grams of table sugar. Once the sugar has dissolved, add in 100 grams of anhydrous ferric chloride or 150 grams of ferric chloride hexahydrate, and stir the solution for about 10 minutes to assure that all solids dissolve. Now, pour this solution into a large separatory funnel, and then pour in 400 milliliters of isopropyl rubbing alcohol. Thereafter, shake the separatory funnel (make sure the stopcock is closed and a stopper is in place, and shake the separatory funnel for about 5 minutes. Then allow the funnel to sit for 10 minutes whereby a two-phase mixture will result. Now, drain-off the bottom aqueous layer containing the dissolved sugar, and then pour off the upper alcohol layer, which will be colored a yellowish brown. Now, pour the lower drained-off aqueous layer back into the same separatory funnel, and then pour in 400 milliliters of fresh rubbing alcohol, and then shake the flask for about 10 minutes. After ten minutes, allow the separatory funnel to settle for 10 minutes as before, and then drain-off the bottom aqueous layer once again. Thereafter, pour off the upper alcohol layer, and combine with the previous upper alcohol layer. Now, repeat this process one more time with 400 milliliters of additional rubbing alcohol. After this extraction process, combine all upper alcohol layers (if not already done so), and then pour the combined alcohol layers (containing the ferric chloride) into a distillation apparatus and distill-off the alcohol until no more alcohol distills over, and a golden yellowish-brown solid remains.

Chapter 7: General Lab Procedures

Now that you are familiar with mixtures and extraction techniques and procedures, it's now time to dip into the true essence of chemistry, and that involves chemical reactions. Chemical reactions are the heart behind the nature of chemistry, and include many different types of reactions and situations. Some chemical reactions can be quite time consuming and difficult, whereas some chemical reactions can be very swift and easy. Some reactions are simple, but require prolonged periods of time to complete, and on the other hand, some chemical reactions can be swift, but complicated in nature; therefore, we will primarily discuss general and basic laboratory techniques in this section. General laboratory procedures normally include basic chemicals and involving general laboratory equipment.

Procedure 01: The preparation of sodium carbonate decahydrate (classic crystallization process)

Also known as: Nevite soda, Sal soda, or Washing soda

	 <p>3D structure 1</p>  <p>3D structure 2</p>
Chemical structure	3D Structure(s)
 <p>1 carbon atom 13 oxygen atoms 2 sodium atoms 22 hydrogen atoms</p>	<p>Na₂CO₃ – 10H₂O</p>
Structure make-up	Condensed chemical structure

Sodium carbonate decahydrate

Sodium carbonate decahydrate forms transparent crystals, or a white powder. The crystals are readily soluble in water, but insoluble in alcohol and most organic solvents. Solutions of the decahydrate are very alkaline and readily decompose in the presence of acids.

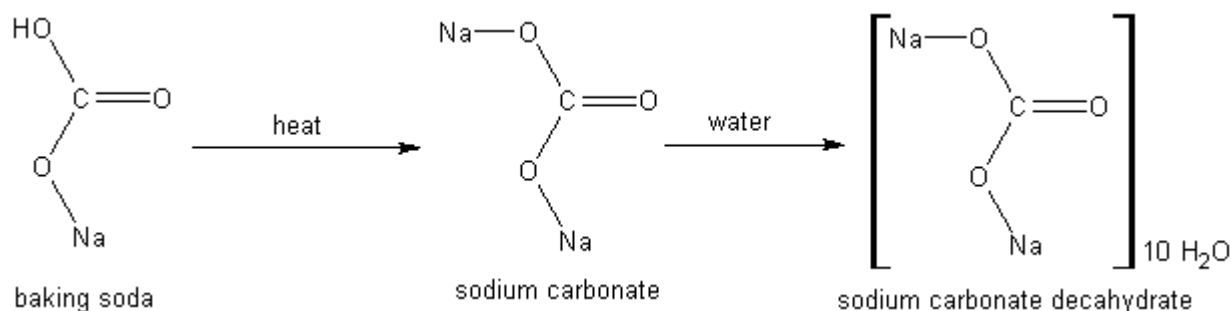
Method 1: Preparation of sodium carbonate decahydrate from baking soda

(By-products from reaction: water)

Materials:

1. 150 grams (5.2 oz.) of baking soda	2. 250 milliliters (8.5 fluid oz.) of cold tap water
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Reaction summary: Sodium carbonate decahydrate is readily prepared by roasting baking soda (sodium bicarbonate) with an open flame from a Bunsen burner or similar gas flame. During the roasting process, the baking soda will be transformed into the carbonate, and water is lost during the reaction. After the reaction, the anhydrous sodium carbonate is then dissolved in the tap water, whereupon the decahydrate crystallizes out spontaneously.



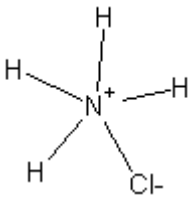

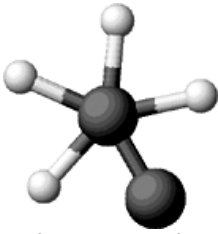
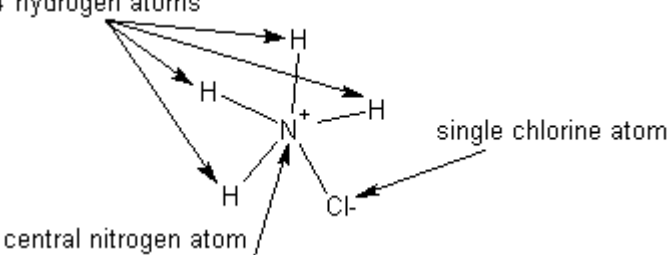
Why does this reaction happen? The reason the anhydrous sodium carbonate crystallizes out of solution is because the anhydrous form has a very high affinity for water, and hence, attracts and snatches-up the water molecules around it. When too many water molecules crowd the carbonate molecules, it causes the molecules of carbonate to crystallize out of solution along with the water of hydration.

Hazards: None.

Procedure: Into a suitable crucible or steel cup or container, place 150 grams (5.2 oz.) of baking soda. Thereafter, heat the baking soda utilizing an open flame from a Bunsen burner or gas operated stove top for about 30 to 40 minutes at high flame heat. During the heating process, the baking soda will transform into sodium carbonate, and water will be lost. After the heating process, remove the heat source, and then allow the material to cool to room temperature. Then, into a clean beaker, place 250 milliliters (8.5 fluid oz.) of cold tap water, and then add and dissolve the cooled sodium carbonate contained in the crucible or steel container. Note: rapidly stir the mixture and dissolve the sodium carbonate into the water as fast as possible. Shortly after dissolving some of the sodium carbonate, the decahydrate should spontaneously crystallize out of the water solution, forming nice transparent crystals. If for some reason, the crystals do not crystallize out of the water solution by themselves, boil the mixture until some crystals form on the surface of the water solution—where upon remove the heat source, and allow the mixture to cool to room temperature, at which time some of the decahydrate should crystallize out.

Procedure 02: The preparation of ammonium chloride

Also known as: ammonium muriate, sal ammoniac, Amchlor, or Darammon

	 <p>3D structure 1</p>  <p>3D structure 2</p>
Chemical structure	3D Structure(s)
	<p>NH₄Cl</p>
Structure make-up	Condensed chemical structure

Ammonium chloride

Ammonium chloride forms colorless crystals, or a white powder. The crystals are readily soluble in water, but have limited solubility in alcohol. The crystals are insoluble in most organic solvents. Ammonium chloride readily decomposes in the presence of alkali hydroxides and carbonates, forming ammonia.

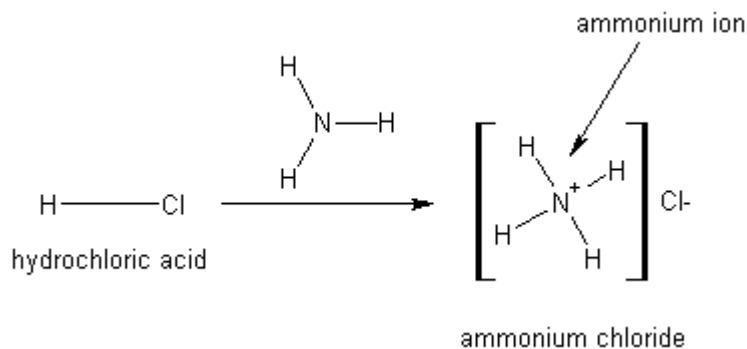
Method 1: Preparation of ammonium chloride from hydrochloric acid and bottled ammonia

(By-products from reaction: none)

Materials:

1. 1 liter (33.8 fluid oz.) of 10% store bought aqueous ammonia	2. 700 milliliters (23.6 fluid oz.) of concentrated hydrochloric acid
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Reaction summary: Ammonium chloride is readily prepared by dissolving concentrated hydrochloric acid into aqueous ammonia. The reaction is very swift, and the ammonium chloride forms spontaneously when the two ingredients are mixed. After the mixing process, the mixture is simply boiled to drive-off water and force the ammonium chloride to crystallize out. The precipitated crystals are then readily collected by filtration.



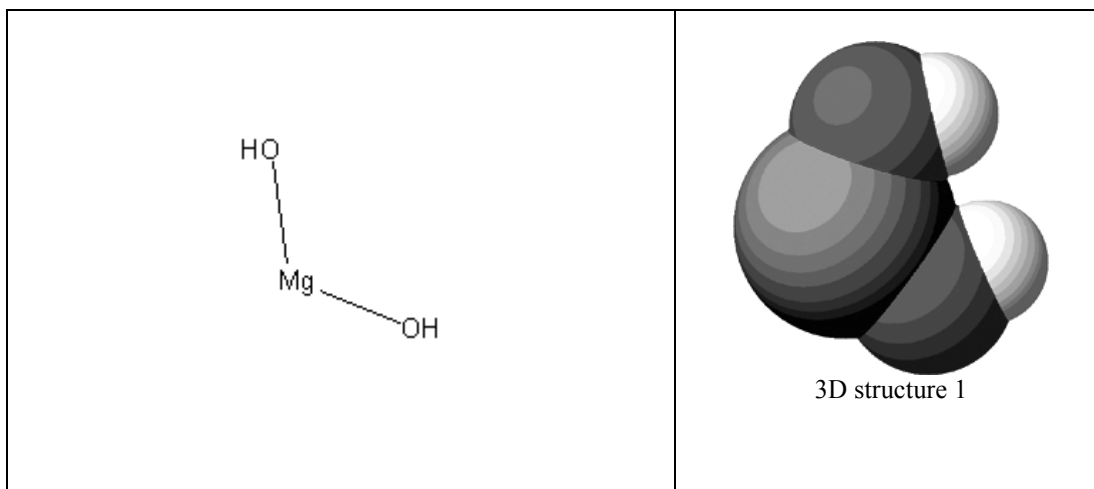
Why does this reaction happen? The reason why hydrochloric acid reacts with ammonia is because ammonia has a particular attraction for single hydrogen ions. When a single hydrogen ion (such as from a molecule of hydrochloric acid) bonds with a molecule of ammonia, an ammonium ion is formed, which is positively charged. The positively charged ammonium ions then bond with the negatively charged chlorine atoms from the hydrochloric acid.

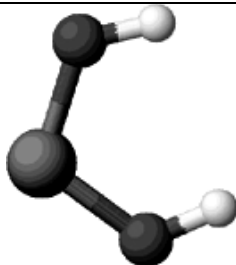
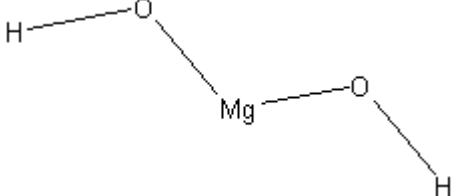
Hazards: Use caution when handling concentrated hydrochloric acid, and avoid inhalation of the fumes. Use care when handling bottled ammonia, which is highly irritating to the nose and throat.

Procedure: Into a suitable large beaker or similar container, place 1 liter (33.8 fluid oz.) of 10% store bought aqueous ammonia (crystal clear ammonia). Thereafter, rapidly add in, in one large portion, 700 milliliters (23.6 fluid oz.) of concentrated hydrochloric acid (Muriatic acid). Thereafter, boil the mixture to remove water and concentrate the mixture. Continue to boil the mixture until a few small crystals form on the surface of the mixture. When this happens, remove the heat source, and allow the mixture to cool to room temperature. As the mixture cools, ammonia chloride crystals will crystallize out of the solution. As soon as the mixture cools to room temperature, filter the mixture to recover the precipitated crystals. Thereafter, boil the filtered mixture once again to drive-off water and concentrate the solution again. Continue to boil the mixture until tiny crystals form on the surface. When this happens, remove the heat source, and allow the mixture to cool to room temperature once again. Thereafter, filter-off the precipitated crystals like before (using the same filter as previous), and then boil the filtered mixture for a third time to drive-off water, and concentrate the solution again. Continue to boil the mixture like before until tiny crystals form on the surface of the solution. When this happens for a third time, remove the heat source, and allow the solution to cool to room temperature. There again, filter the mixture using the same filter as the previous two, to remove the precipitated crystals. At this point, the filtered mixture would have been reduced quite significantly since the start, and most of the ammonium chloride would have been precipitated and collected. Finally, vacuum dry or air-dry the collected crystals of ammonium chloride, and then store the crystals in a zip lock bag or similar airtight storage container.

Procedure 03: The Preparation of Magnesium hydroxide (classic precipitation reaction)

Also known as: Magnesium hydrate



	 3D structure 2
Chemical structure	3D Structure
 1 magnesium atom 2 oxygen atoms 2 hydrogen atoms	Mg(OH) ₂
Anhydrous structure makeup	Condensed chemical structure

Magnesium hydroxide

Magnesium hydroxide forms a fine white bulky powder, which is insoluble in water and most common organic solvents. The dry solid absorbs carbon dioxide from the air, and the solid decomposes when heated forming magnesium oxide.

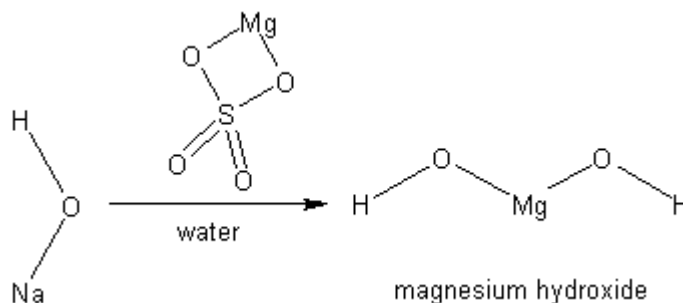
Method 1: Preparation of magnesium hydroxide from lye and Epsom salt

(By-products from reaction: sodium sulfate)

Materials:

1. 500 milliliters (16.9 fluid oz.) of warm tap water	3. 250 milliliters (8.4 fluid oz.) of cold tap water
2. 200 grams (7 oz.) of Epsom salt	4. 64 grams (2.2 oz.) of lye

Reaction summary: Magnesium hydroxide is readily prepared by mixing solutions of Epsom salt and lye. The reaction is very swift, and an insoluble precipitate of magnesium hydroxide forms immediately when the two solutions of Epsom salt and lye are mixed. The precipitated magnesium hydroxide is then readily collected by filtration.



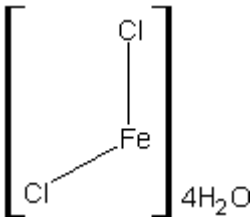
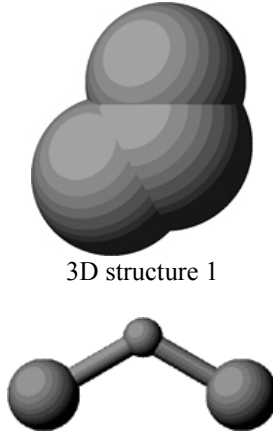
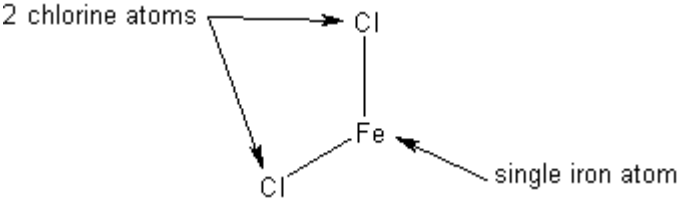
Why does this reaction happen? This reaction happens because magnesium hydroxide molecules have a very very low solubility in water. Therefore, when solutions of two water-soluble compounds such as Epsom salt and lye are mixed, the hydroxide ions bond with the magnesium ions forming magnesium hydroxide, which precipitates spontaneously. Note: if the Epsom salt and lye were mixed in a dry state, no reaction would take place.

Hazards: use caution when handling lye, which is highly corrosive and can cause severe skin irritation.

Procedure: Into a suitable beaker or similar container, place 500 milliliters (16.9 fluid oz.) of warm tap water, and then add and dissolve 200 grams (7 oz.) of Epsom salt. Thereafter, into a second beaker or similar container, place 250 milliliters (8.4 fluid oz.) of cold tap water, and then add and dissolve 64 grams (2.2 oz.) of lye. Note: Lye generates excessive heat when dissolved in water so allow the solution to cool before using. Now, pour the sodium hydroxide solution into the Epsom salt solution all at once. When both solutions are mixed, a fine milky-white precipitate of magnesium hydroxide will form instantaneously. After mixing both solutions, filter the mixture to remove the insoluble magnesium hydroxide precipitate. Vacuum filtration should be used instead of gravity filtration to speed up the process. After the filtration process, vacuum dry or air-dry the collected precipitate. Note: do not use heat to dry the solid as it will decompose.

Procedure 04: The Preparation of Ferrous chloride tetrahydrate (non-electrochemical method)

Also known as: chloride of iron

	 <p>3D structure 1</p> <p>3D structure 2</p>
Chemical structure	3D Structure
	<p>$\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$</p>
Anhydrous structure makeup	Condensed chemical structure

Ferrous chloride tetrahydrate

Ferrous chloride tetrahydrate forms greenish to pale greenish crystals, or a greenish crystalline powder. The crystals lose half their water of hydration forming the dihydrate when heated to 115 Celsius. The crystals are readily soluble in water forming green solutions. The crystals and solutions thereof are readily oxidized.

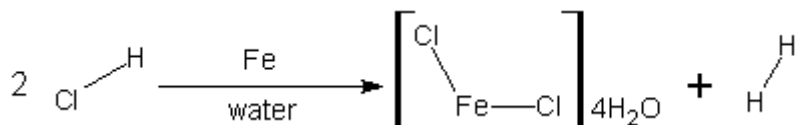
Method 1: Preparation of ferrous chloride tetrahydrate from hydrochloric acid and iron

(By-products from reaction: Hydrogen gas)

Materials:

1. 150 milliliters (5 fluid oz.) of warm tap water	4. 20 grams (0.70 oz.) of lye
2. 300 milliliters (10.1 fluid oz.) of hydrochloric acid	5. 250 milliliters (8.5 fluid oz.) of warm tap water
3. 255 grams (0.56 pounds) of iron nails, iron bolts, washers, or iron wire	

Reaction summary: Ferrous chloride tetrahydrate is readily prepared by dissolving iron metal into hydrochloric acid. Hydrogen gas is a by-product in the reaction.

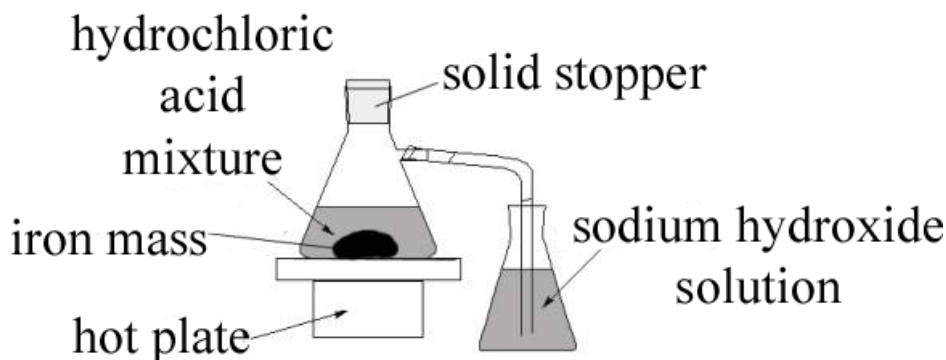


Note: On the left hand side of this reaction equation we have two molecules of hydrochloric acid, per single iron atom, represented by Fe over the arrow. On the right side of the equation we have the products of ferrous chloride tetrahydrate and hydrogen gas.

Why does this reaction happen? Hydrochloric acid is a strong acid, and has a strong affinity for metals, such as iron. Metals like iron have less electro negativities then compounds like hydrochloric acid; therefore, when hydrochloric acid reacts with iron, the chlorine of the acid bonds with the metal atoms, and since the chlorine prefers the company of the iron, the hydrogen of the acid is given off as a gas.

Hazards: carryout the reaction in a well ventilated area, as metallic like odors will be released during the reaction. Note: an apparatus should be setup utilizing lye in order to neutralize the waste gasses, which will contain non-harmful, yet noxious fumes.

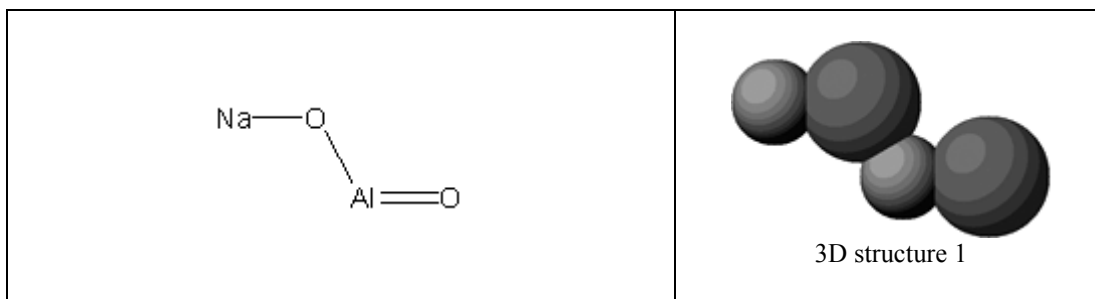
Procedure: Dissolve 20 grams (0.70 oz.) of lye into 250 milliliters of warm tap water, and then assemble the apparatus as illustrated below. Then, into a suitable flask, as illustrated in the following figure, place 150 milliliters (5 fluid oz.) of warm tap water, followed by 300 milliliters (10.1 fluid oz.) of concentrated hydrochloric acid (31 to 35% by weight). Thereafter, add in 255 grams (0.56 pounds) of iron nails, bolts, or wire. Note: try to avoid galvanized, nickel, or zinc plated iron. Then, seal the apparatus and then heat the mixture to about 50 Celsius. Thereafter, continue to heat the mixture until the evolution of hydrogen gas ceases. Actual time may vary but usually ranges from 5 to 8 hours. Once the evolution of hydrogen gas has ceased, turn off the heat and allow the mixture to cool to room temperature. Once it has, filter the mixture to remove insoluble impurities, and then recrystallize the ferrous chloride from the filtered reaction mixture. Finally, vacuum dry or air-dry the greenish crystals. Note: The crystals will turn a brownish color on standing due to oxidation, so keep them in tightly sealed containers away from moisture and air.

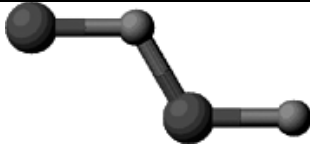
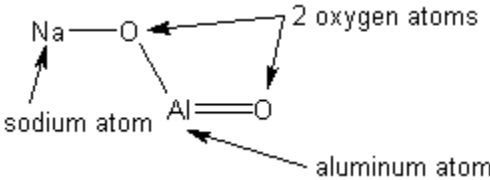


Apparatus for the preparation of ferrous chloride. The lye solution is designed to absorb noxious fumes that are produced during the reaction.

Procedure 05: The Preparation of Sodium Aluminate

Also known as: Aluminum sodium oxide



	
Chemical structure	3D Structure
	NaAlO ₂
Structure makeup	Condensed chemical structure

Sodium Aluminate

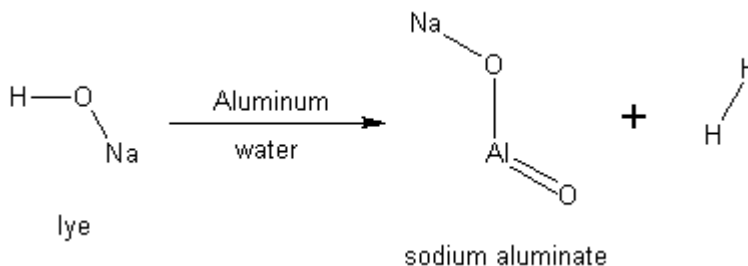
Sodium aluminate forms a white granular mass. The crystals are very soluble in water, forming a strongly alkaline solution. The crystals have a high melting of 1650 Celsius.

Method 1: Preparation of Sodium Aluminate from lye and aluminum foil

Materials:

1. 400 milliliters (13.5 fluid oz.) of water	2. 50 grams (1.7 oz.) of sodium hydroxide (lye)
3. 25 grams (0.88 oz.) of aluminum foil	

Reaction summary: Sodium aluminate is readily prepared by dissolving aluminum foil into sodium hydroxide solution. The reaction is vigorous, and generates allot of hydrogen gas. Heat is also generated during the reaction.



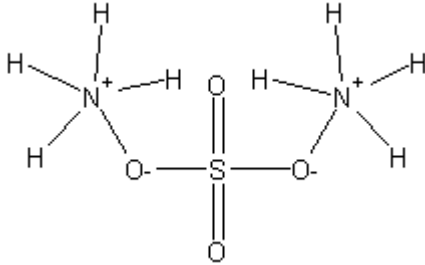
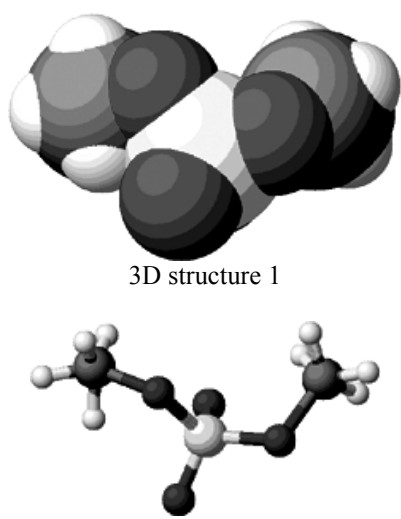
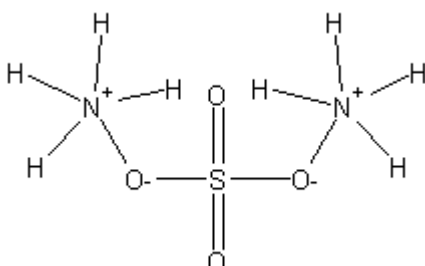
Why does this reaction happen? In the presence of water or moisture, sodium hydroxide has a strong affinity for aluminum. The primary reason is because the oxygen within the sodium hydroxide has a greater electro negativity then the aluminum, so it's very attracted to the aluminum. Because the sodium within the hydroxide has a very high affinity for oxygen as well, the aluminum and sodium both are attracted to the oxygen forming a double compound known as sodium aluminate.

Hazards: Extinguish all flames before using the reaction, as a large amount of hydrogen gas is obtained.

Procedure: Into a suitable beaker or flask, add 400 milliliters (13.5 fluid oz.) of water, and then add in 50 grams (1.7 oz.) of sodium hydroxide (lye). Note: sodium hydroxide generates excessive heat when dissolved in water; so allow the mixture to cool before using. Thereafter, add in, in small pieces, about 25 grams (0.88 oz.) of aluminum foil. Note: Do not add to much aluminum foil to the sodium hydroxide solution, as a violent evolution of gas will form. Once the aluminum has been completely dissolved, filter the reaction mixture to remove any un-reacted aluminum pieces, and to remove insoluble carbon. Thereafter, recrystallize the sodium aluminate from the mixture (re-read the recrystallization process in the lab tutorial if you need a refresher on how to recrystallize dissolved compounds. Once the sodium aluminate has been obtained, simply vacuum dry or air-dry it in the usual manner. The dry solid should be stored in airtight bottles.

Procedure 06: The Preparation of Ammonium sulfate

Also known as: Mascagnite, sulfuric acid diammonium salt

	 <p>3D structure 1</p> <p>3D structure 2</p>
Chemical structure	3D Structure
 <p>1 sulfur atom 4 oxygen atoms 2 nitrogen atoms 8 hydrogen atoms</p>	<p>(NH₄)₂SO₄</p>
Structure makeup	Condensed chemical structure

Ammonium sulfate

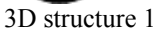
Ammonium sulfate forms colorless odorless crystals, or a white granulated powder or product. The crystals are readily soluble in water, but relatively insoluble in alcohol, acetone, and most common solvents. The crystals decompose into ammonia, sulfuric acid, and other products when heated above 300 Celsius. The crystals are stable at room temperature.

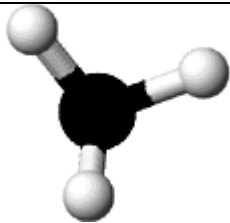
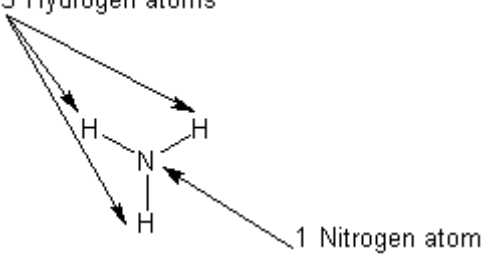
Method 1: Preparation of ammonium sulfate from bottled ammonia and concentrated sulfuric acid

Materials:

1. 1 liter (33.8 fluid oz.) of store bought crystal clear ammonia	2. 288 grams (10.1 oz; about 156 milliliters) of concentrated sulfuric acid
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Reaction summary: ammonium sulfate is readily prepared by dissolving concentrated sulfuric acid into dilute aqueous ammonia. The reaction is very swift, and afterwards, the ammonium sulfate is recrystallized from the mixture.



	 <p>3D structure 2</p>
Chemical structure	3D Structure
<p>3 Hydrogen atoms</p>  <p>1 Nitrogen atom</p>	NH ₃
Structure make-up	Condensed chemical structure

Ammonia, anhydrous

Anhydrous ammonia is a colorless gas with a very pungent odor. It has a melting point of -77 Celsius, and a boiling point of -33 Celsius. Ammonia is regarded as nonflammable, but mixtures with oxygen can ignite (when passed over red-hot platinum resulting in nitrogen dioxide—see nitric acid *vide supra*). Anhydrous ammonia is a corrosive alkaline gas that is very soluble in water. It is also soluble in alcohol, chloroform, and ether. Liquid ammonia is a good solvent for many elements and compounds. Commercial anhydrous ammonia is supplied in the form of a compressed gas in steel tanks, or in the liquid form supplied in steel tanks. Ammonia is also widely sold in water solutions. Anhydrous ammonia is the 4th largest chemical produced in the US. Anhydrous ammonia is a widely available commercial chemical. Anhydrous ammonia is prepared on an industrial scale from hydrogen and nitrogen at high pressure and temperature in the presence of platinum. The average ammonia plant produces 1000 tons of liquid ammonia per day.

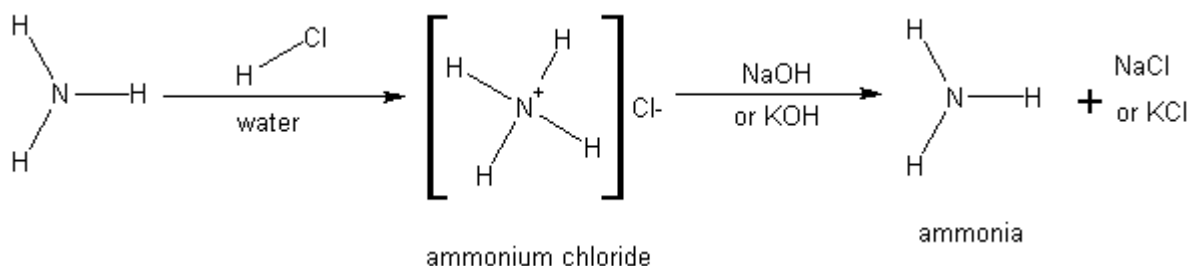
Method 1: Preparation of anhydrous ammonia from store bought commercial ammonia solution (for cleaning) with lye and acid

(By-products from reaction: sodium chloride or sodium or potassium sulfate)

Materials:

1. 156 grams (5.5 oz.) of 10% ammonia (household ammonia)	3. 38 grams (1.3 oz.) of sodium hydroxide or 53 grams (1.9 oz.) of potassium hydroxide
2. 100 grams (3.5 oz.) of 35% hydrochloric acid, or 47 grams (1.7 oz.) of 98% sulfuric acid	4. 38 milliliters (1.3 fluid oz.) of tap water or 53 milliliters (1.8 fluid oz.) of tap water

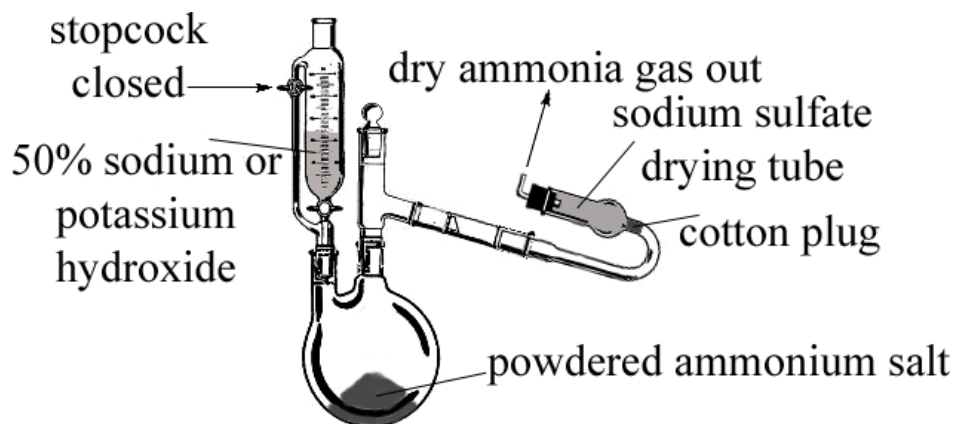
Reaction summary: Anhydrous ammonia can be prepared by treating an ammonium salt with a warm solution of sodium hydroxide, or potassium hydroxide. In the following procedure, the ammonium salt is either ammonium chloride or ammonium sulfate, which are first prepared by reaction of household ammonia with the corresponding acid. Thereafter, the resulting ammonium salt is then treated with sodium hydroxide, or potassium hydroxide to yield ammonia gas, which is then dried to yield the anhydrous gas.



Why does this reaction happen? In this situation we start with forming an ammonium salt because we are using aqueous store bought ammonia. As in the previous reaction, we simply mix the aqueous ammonia with the acid, forming the ammonium salt. Thereafter, we recover the ammonium salt by the use of recrystallization, and then we treat the ammonium salt with a strong base, such as sodium or potassium hydroxide. When an ammonium salt is treated with a strong base, free gaseous ammonia is produced because the base has a strong affinity for the chloride ion if using ammonium chloride, or the sulfate ion if using ammonium sulfate.

Hazards: Wear gloves when handling 50% sodium hydroxide, or 50% potassium hydroxide. The alkaline solutions may cause painful skin burns on prolonged exposure, and itching sensation on short exposure. Use proper ventilation when making ammonia gas, and avoid inhalation of the vapors.

Procedure: Place 156 grams (5.5 oz.) of 10% ammonia into a flask and then rapidly add 100 grams (3.5 oz.) of 35% hydrochloric acid, or 47 grams (1.7 oz.) of 98% sulfuric acid while stirring the 10% ammonia solution. After the addition of the acid, continue stirring for ten minutes, and then recrystallize the ammonium salt from solution (simply boil-off the water until crystallization begins). After all the desired ammonium salt has been collected, dry it using vacuum drying, or let air-dry. Afterwards, place the dry ammonium salt into an apparatus as illustrated below, and then prepare a sodium hydroxide solution by dissolving 38 grams (1.3 oz.) of sodium hydroxide into 38 milliliters (1.3 fluid oz.) of water, or 53 grams (1.9 oz.) of potassium hydroxide into 53 milliliters (1.8 fluid oz.) of water (much heat is produced when dissolving sodium or potassium hydroxide into water). After the sodium or potassium hydroxide solution has cooled some, add it to the dry ammonium salt, drop-wise, over a period of about thirty minutes. During the addition of the sodium hydroxide or potassium hydroxide, ammonia gas will be steadily evolved.

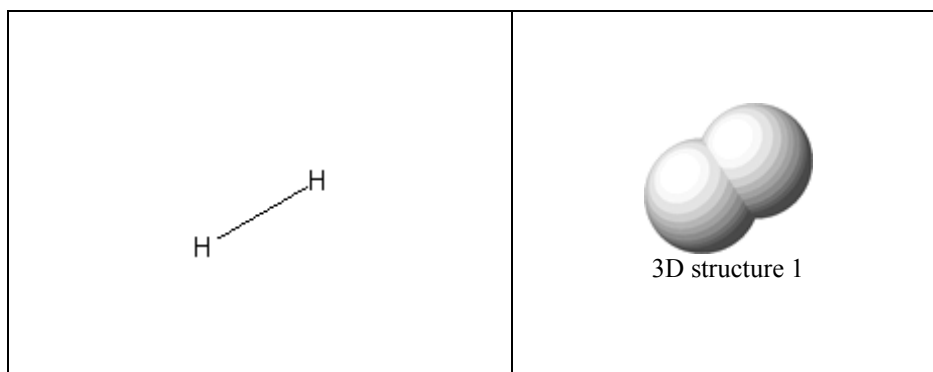


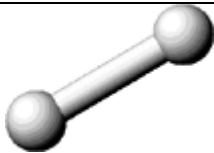
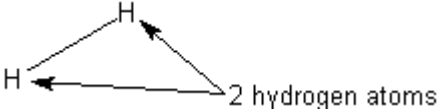
Apparatus for the preparation of anhydrous ammonia.

Final note for method 1: Like most of the apparatus illustrated in this book, the apparatus illustrated above can be replaced by any similar set-up. It should also be noted that the illustrations in this book are merely suggestive drawings, and do not represent the actual design. Design and function of apparatus can vary, and the illustrations given in this book should be used to define or describe the necessary set-up only to the point of design.

Procedure 08: The Preparation of Hydrogen gas

Also known as: Protium



	 3D structure 2
Chemical structure	3D Structure
 2 hydrogen atoms	H ₂
Structure make-up	Condensed chemical structure

Hydrogen

Hydrogen is a colorless, tasteless, highly flammable and explosive gas. Hydrogen is a strong reducing agent when in the presence of a suitable catalyst such as platinum, palladium, nickel, and the like. Hydrogen gas is very difficult to handle and store, but can be stored in compressed cylinders; these cylinders are often hard to purchase and ship, but hydrogen can easily be prepared by reacting dilute hydrochloric acid with zinc, or aluminum, and collecting the resulting hydrogen gas. Note: iron should be avoided as it contains impurities leading to the formation of metal hydrides such as arsine and stibine, which contaminate the hydrogen (gives the hydrogen a strange metallic like garlic odor). Hydrogen can also be obtained by the electrolysis of acidic or basic water solutions, or by treating a 25% sodium hydroxide (lye) solution with aluminum foil—this latter reaction is utilized by several dry drain opener products to open and clear clogged drains.

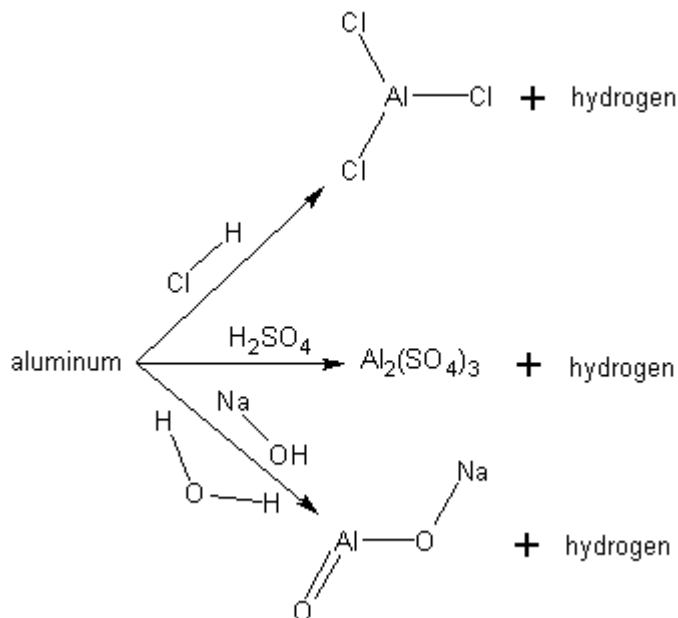
Method 1: Preparation of Hydrogen gas from hydrochloric acid or sulfuric acid and a metal, or by reaction of lye with aluminum foil

(By-products from reaction: metal chlorides, or sodium aluminate)

Materials:

1. 150 grams (5.3 oz.) of concentrated hydrochloric acid	6. 10 grams (0.35 oz.) of aluminum
2. 13 grams (1/2 oz.) of aluminum	7. 17 grams (1/2 oz.) of zinc
3. 45 grams (1.6 oz.) of zinc	8. 50 grams (1.8 oz.) of lye (sodium hydroxide),
4. 75 milliliters (2.5 fluid oz.) of ice cold tap water	9. 150 milliliters of ice-cold tap water
5. 50 grams (1.8 oz.) of concentrated sulfuric acid	10. 11 grams (0.4 oz.) of aluminum (soda pop cans or aluminum foil)

Reaction summary: Hydrogen gas is easily prepared by reacting hydrochloric acid or sulfuric acid with zinc or aluminum, or by reacting lye with aluminum foil. If using hydrochloric acid or sulfuric acid, metal foils or finely divided metals such as aluminum foil, or zinc dust, can give rise to quite violent reactions. For aluminum, aluminum pop cans house the best source, as they are cheap and readily available.



Why does this reaction happen? Hydrogen gas is readily prepared by reacting an acid, or a strong alkaline base with aluminum or sometimes zinc. The reason hydrogen is liberated is because the acids and bases have a high affinity for metals such as aluminum or zinc. Because the acid or base chemically reacts with the metal, the hydrogen of the acid or base has no place to go so its liberated as a gas.

Hazards: Hydrogen is highly flammable and explosive. Mixtures of hydrogen with air when confined will explode when ignited.

Procedure:

Using Hydrochloric acid:

Into the reaction flask of the below illustration, place 150 grams (5.3 oz.) of concentrated hydrochloric acid. Thereafter, slowly add, in small portions at a time, 13 grams (1/2 oz.) of aluminum, preferably aluminum pieces made by carefully cutting-up a clean aluminum can, or 45 grams (1.6 oz.) of zinc. After adding each piece of aluminum or zinc, a steady stream of hydrogen gas will develop. Use a cold-water bath to maintain the temperature of the reaction mixture at a safe level. The hydrogen gas that is evolved can be used for many applications. Have some fun with the hydrogen by filling up a party balloon with the gas. Then carefully make contact with the balloon with the flame of a candle!

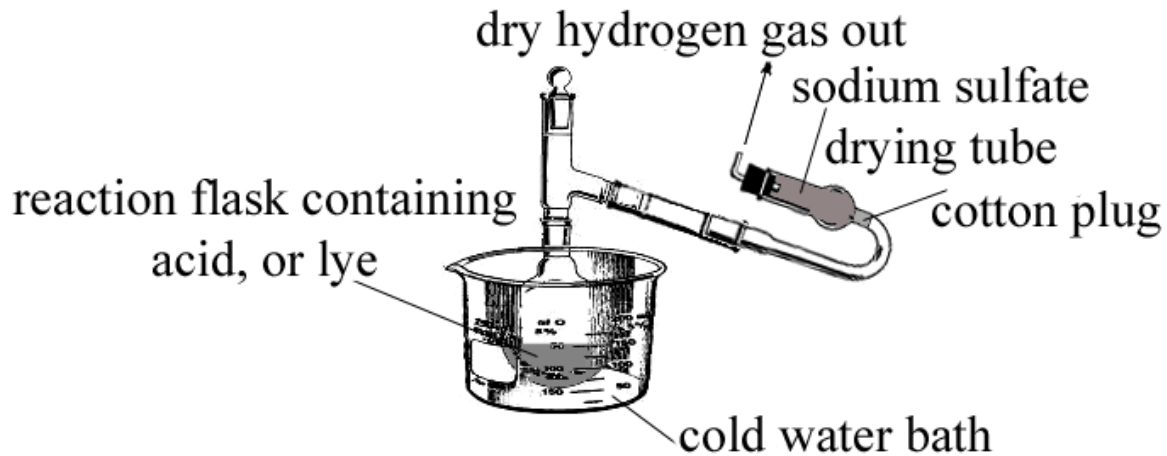
Using Sulfuric acid

Into the reaction flask of the below illustration, place 75 milliliters (2.5 fluid oz.) of ice cold tap water, and then carefully and slowly add in 50 grams (1.8 oz.) of concentrated sulfuric acid. Thereafter, allow the acid mixture to cool (as adding sulfuric acid to water generates excessive heat), and then slowly add, in small portions at a time, 10 grams (0.35 oz.) of aluminum, preferably aluminum pieces made by carefully cutting-up a clean aluminum can, or 17 grams (1/2 oz.) of zinc. After adding each piece of aluminum or zinc, a steady stream of hydrogen gas will develop. Use a cold-water bath to maintain the temperature of the reaction mixture at a safe level. The hydrogen gas that is evolved can be used for many applications. Have some fun with the hydrogen by filling up a party balloon with the gas. Then carefully make contact with the balloon with the flame of a candle!

Using Lye (sodium hydroxide)

Into a clean beaker, or other suitable glass container, place 50 grams (1.8 oz.) of lye (sodium hydroxide), followed by 150 milliliters of ice-cold tap water. Note: the addition of sodium hydroxide to water generates excessive heat, so allow the solution to cool before using. Thereafter, place the cooled alkaline mixture into the reaction flask of the below illustration. Then slowly add, in small portions at a time, 11 grams (0.4 oz.) of aluminum, preferably aluminum foil pieces. After adding each piece of aluminum, a steady stream of hydrogen gas will develop. Use a cold-water bath to maintain the temperature of the reaction

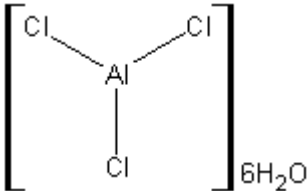
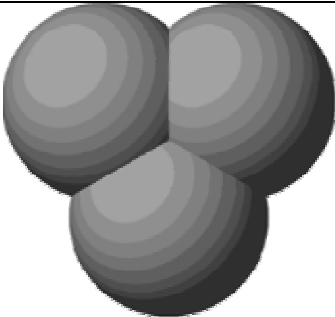
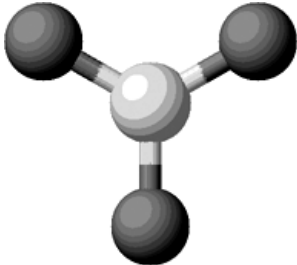
mixture at a safe level. The hydrogen gas that is evolved can be used for many applications. Have some fun with the hydrogen by filling up a party balloon with the gas. Then carefully make contact with the balloon with the flame of a candle!

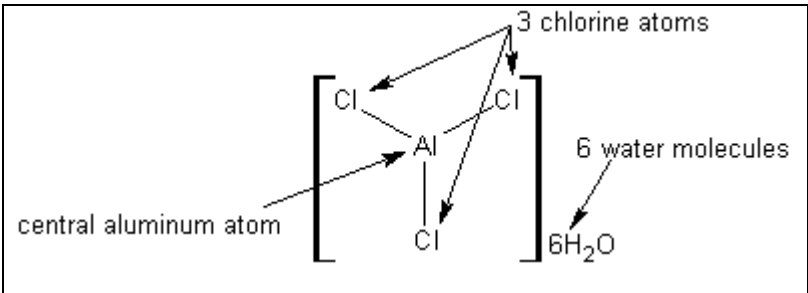


Apparatus for the generation of hydrogen gas.

Procedure 09: The Preparation of Aluminum chloride hexahydrate

Also known as: trichloro aluminum; aluminum trichloride

	 <p>3D structure 1</p>  <p>3D structure 2</p>
Chemical structure	3D Structure

	$\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$
Structure makeup	Condensed chemical structure

Aluminum chloride hexahydrate

Aluminum chloride hexahydrate forms fine colorless crystals, or a white powder. The crystals may be colored yellowish with a slight HCl odor. The crystals are readily soluble in water and soluble in alcohol.

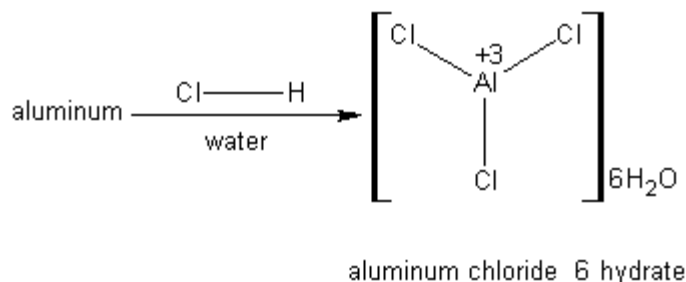
Method 1: Preparation of aluminum chloride hexahydrate from hydrochloric acid and aluminum foil

(By-products from reaction: hydrogen gas)

Materials:

1. 320 milliliters (11.8 fluid oz.) of concentrated hydrochloric acid	3. 25 grams (0.88 oz.) of aluminum foil pieces
2. 150 milliliters (5 fluid oz.) of cold tap water	

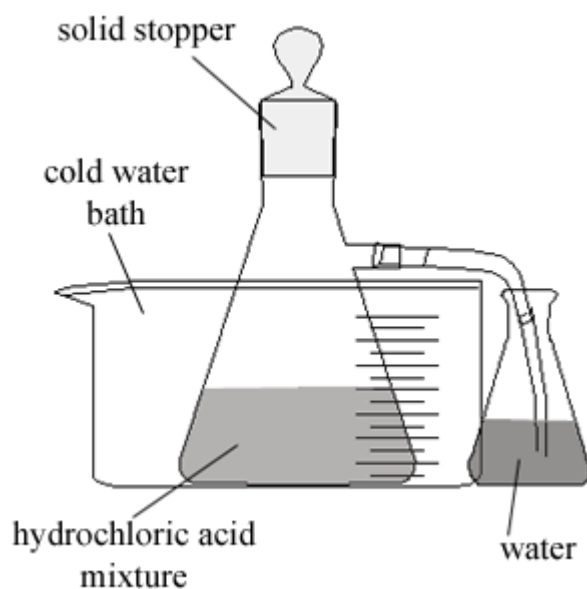
Reaction summary: Aluminum chloride hexahydrate is readily prepared by dissolving aluminum foil pieces into hydrochloric acid. After the reaction, the water is removed by evaporation, to recover the dissolved crystals of aluminum chloride.



Why does this reaction happen? As in previous reactions, aluminum has a high affinity for acids or strong bases. In this case, we dissolve aluminum foil into dilute hydrochloric acid. The aluminum bonds with the chloride of the hydrochloric acid because the electro negativity of the aluminum is much less than that of the chloride. The reason the aluminum chloride forms a hexahydrate is because the chloride has a strong affinity for water so it takes up six water molecules per molecule of aluminum chloride.

Hazards: Extinguish all flames before performing this reaction as large volumes of hydrogen gas are evolved; use proper ventilation. Do not add more than 1 gram of aluminum foil pieces to the acid at one time.

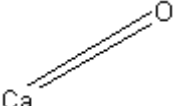
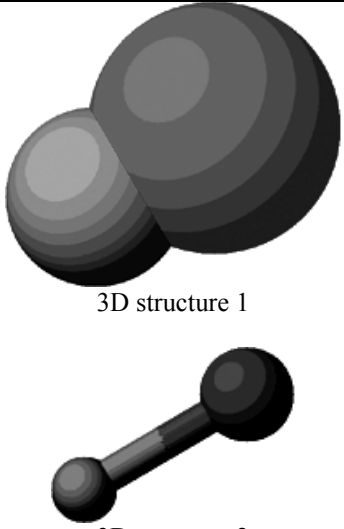
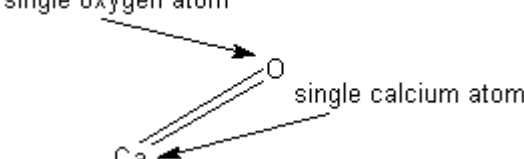
Procedure: Into a 500-milliliter flask, place 320 milliliters (11.8 fluid oz.) of concentrated hydrochloric acid, followed by 150 milliliters (5 fluid oz.) of cold tap water. Thereafter, assemble an apparatus as illustrated below, and then slowly add in small portions at a time (about 1 gram at a time) 25 grams (0.88 oz.) of aluminum foil pieces. Note: after each addition, the aluminum foil will react very vigorously with the hydrochloric acid. Use proper ventilation and extinguish all flames, as a large volume of hydrogen gas will be evolved. Note: do not add too much aluminum foil to the acid at one time, as a violent reaction can result. After the addition of all the aluminum foil has been completed, allow the mixture to stand for about 30 minutes, and then filter the mixture to remove insoluble impurities. After the filtration, the filtered mixture should be poured onto a shallow pan or tray, and allowed to evaporate to recover the dissolved crystals of aluminum chloride. Note: Do not boil the mixture to remove the water as the dissolved aluminum chloride will decompose into hydrogen chloride and aluminum oxide.



Setup for the dissolving of aluminum foil into hydrochloric acid. For each run, the solid stopper should be quickly removed, and then 1-gram of aluminum foil added, and then the stopper should be placed back airtight. Once the aluminum foil has dissolved in the hydrochloric acid, briefly remove the stopper again, and add in more aluminum foil. Repeat this process until all the aluminum foil has been added.

Procedure 10: The preparation of Calcium oxide (Lime)

Also known as: Lime, Calx, quick lime, burnt lime

	 <p>3D structure 1</p> <p>3D structure 2</p>
Chemical structure	3D Structure(s)
 <p>single oxygen atom</p> <p>single calcium atom</p>	<p>CaO</p>
Structure make-up	Condensed chemical structure

Calcium oxide (lime)

Calcium oxide forms white to grayish white granules, lumps, or powder. The material reacts readily with water forming calcium hydroxide, and generating excessive amounts of heat. The material is insoluble in most organic solvents, but has limited solubility in sugar solutions and glycerol.

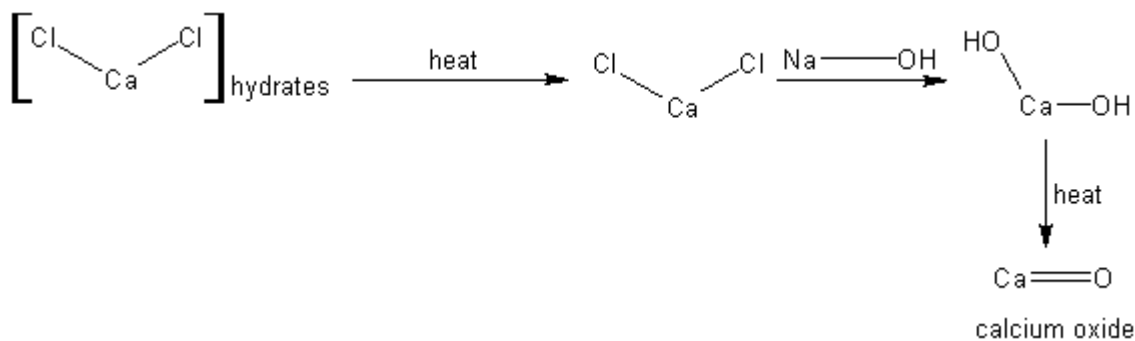
Method 1: Preparation of calcium oxide from calcium chloride and lye

(By-products from reaction: sodium chloride and water)

Materials:

1. 150 grams (5.2 oz.) of store bought calcium chloride de-icing compound	3. 300 milliliters (10.1 fluid oz.) of cold tap water
2. 350 milliliters (11.8 fluid oz.) of warm tap water	4. 71 grams (2.5 oz.) of sodium hydroxide (lye)

Reaction summary: Calcium oxide is readily prepared by heating calcium hydroxide, which is formed by reacting Lye with calcium chloride. The reaction is swift, and generates sodium chloride and water as by-products.



Why does this reaction happen? In this reaction, we start with hydrates of calcium chloride, which are available in stores sold as an ice melter. The hydrates of calcium chloride are removed by roasting at high temperatures using a Bunsen burner. The heat drives-off the water of hydration forming the anhydrous salt. Thereafter, the anhydrous calcium chloride is dissolved in water, and then treated with a solution of lye (sodium hydroxide) forming the hydroxide. Once again here we have a precipitation reaction, so when the two solutions of calcium chloride and lye are mixed, the water insoluble calcium hydroxide precipitates spontaneously. Finally, when the calcium hydroxide is roasted, it loses its hydroxide ions in the form of water, and becomes dried, forming the calcium oxide.

Hazards: Use care when handling sodium hydroxide (lye), which is highly corrosive and can cause severe burns.

Procedure:

Step 1: Roasting calcium chloride

Into a suitable crucible or similar heat resistant container, place 150 grams (5.2 oz.) of store bought calcium chloride de-icing compound (mainly hydrates of calcium chloride used to melt ice). Thereafter heat the mixture to moderate heat using an open flame from a Bunsen burner or gas burning stove top, using a medium flame. Continue to heat at this temperature for about 1 hour to ensure complete removal of water of hydration, and to form an anhydrous salt. Thereafter, remove the heat source and allow the material to cool to room temperature before removing it. Thereafter, save the cooled material of anhydrous calcium chloride for step 2.

Step 2: formation of calcium hydroxide

Into a suitable beaker or similar container, place 350 milliliters (11.8 fluid oz.) of warm tap water, and then add and dissolve 100 grams (3.5 oz.) of calcium chloride obtained in step 1. Thereafter, into a separate clean beaker or similar container, place 300 milliliters (10.1 fluid oz.) of cold tap water, and then add and dissolve 71 grams (2.5 oz.) of sodium hydroxide (lye). Note: dissolving sodium hydroxide in water generates excessive heat, so use caution, and allow the resulting solution to cool before using. Now, into the container containing the dissolved calcium chloride, pour the entire sodium hydroxide solution there into. When both solutions are mixed, a milky-white precipitate of calcium hydroxide will form instantaneously. Once both solutions

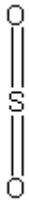
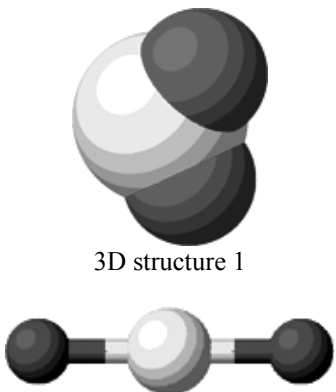
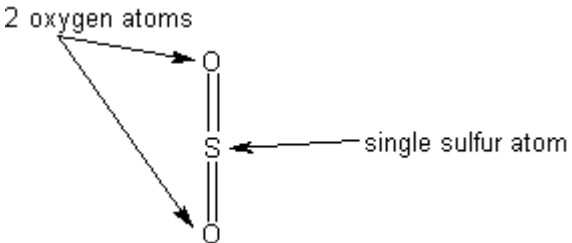
have been added to each other, filter the mixture to remove the insoluble calcium hydroxide precipitate. Note: using gravity filtration will take hours, so use vacuum filtration to speed up the process. Once the calcium hydroxide has been collected, vacuum dry or air dry the calcium hydroxide, and then save the calcium hydroxide for step 3.

Step 3: formation of calcium oxide (lime)

Into a suitable crucible or similar heat resistant container, place the calcium hydroxide obtained from step 2, and then heat the calcium hydroxide using an open flame from a Bunsen burner using a medium flame. Continue to heat the calcium hydroxide for about 1 hour to ensure proper disassociation of the hydroxide into its oxide. During the reaction, the calcium hydroxide will dehydrate losing water and forming the oxide. After heating for about one hour, remove the heat source, and allow the material to cool to room temperature. Once it has, remove it from the crucible or heat resistant container, and store it in airtight containers or bags to exclude moisture. Note: calcium oxide reacts with water with the generation of heat, i.e., see the reaction of cement.

Procedure 11: The Preparation of Sulfur dioxide gas

Also known as: dioxide of sulfur; sulfurous anhydride; sulfurous oxide

	 <p>3D structure 1</p> <p>3D structure 2</p>
Chemical structure	3D Structure 2
	<p>SO₂</p>
Structure make-up	Condensed chemical structure

Sulfur dioxide

Sulfur dioxide forms a colorless gas with a strong suffocating and irritating odor. The gas has a melting point of -72 Celsius, and a boiling point of -10 Celsius. The gas is easily condensed into a colorless liquid. The gas is soluble in water, alcohol, chloroform, and ether. Sulfur dioxide is available in gas cylinders, but it can be prepared in the lab by dripping hydrochloric acid onto excess sodium bisulfite.

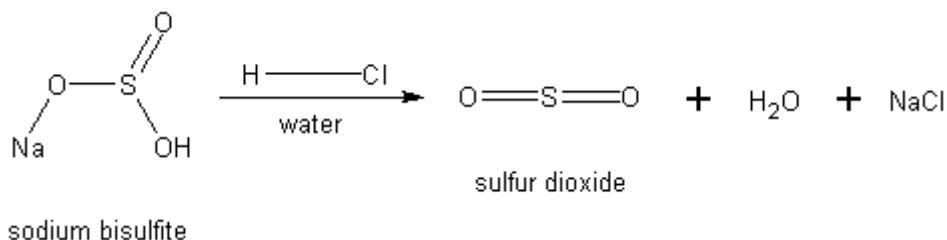
Method 1: Preparation of sulfur dioxide gas from sodium bisulfite and hydrochloric acid

(By-products from reaction: water and sodium chloride)

Materials:

1. 100 grams (3.5 oz.) of powdered sodium bisulfite	2. 100 grams (3.5 oz.) of concentrated hydrochloric acid
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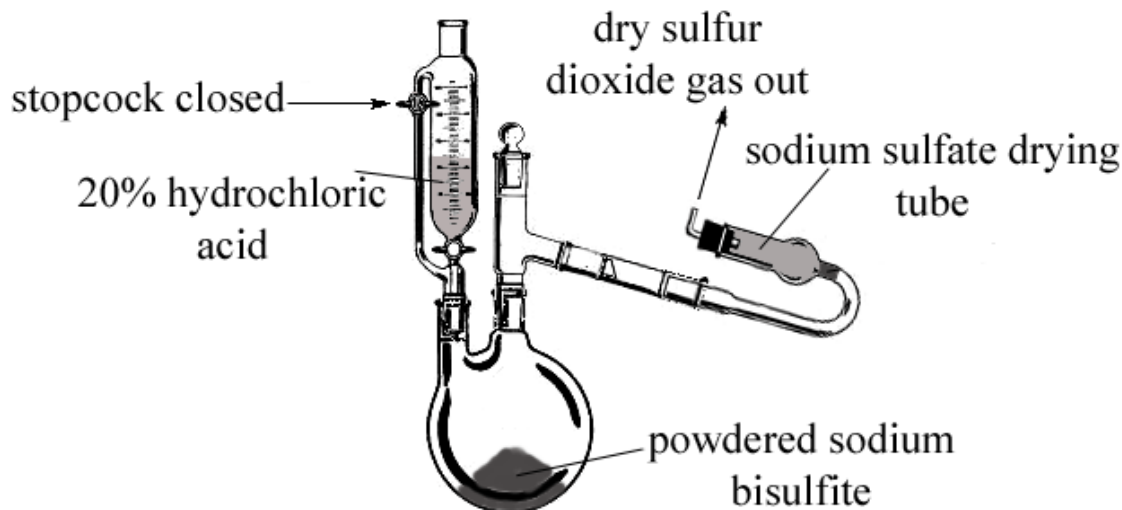
Reaction summary: Sulfur dioxide gas is easily prepared by mixing hydrochloric acid with sodium bisulfite. The reaction is very smooth and generates a steady stream of gas.



Why does this reaction happen? Sodium bisulfite has a tendency to disassociate when treated with hydrochloric acid. This disassociation is caused by the fact that the oxygen within the bisulfite molecule wants to free itself forming the more stable sulfur dioxide. The reaction is stirred on by the fact that the sodium ion of the bisulfite reacts with the chloride of the hydrochloric acid forming common table salt, i.e., sodium chloride.

Hazards: Use proper ventilation when using sulfur dioxide gas, which is very irritating to the nose and throat. Avoid inhalation of the fumes.

Procedure: Set-up the apparatus as illustrated below, and then place 100 grams (3.5 oz.) of powdered sodium bisulfite into the flask as illustrated, and then place 100 grams (3.5 oz.) of concentrated hydrochloric acid or concentrated Muriatic acid into the addition funnel as illustrated. Thereafter, gradually drip the hydrochloric acid onto the sodium bisulfite over a period of about 30 to 90 minutes. During the acid addition, the sulfur dioxide gas will be steadily evolved and will carry over.



Set-up for the generation of sulfur dioxide gas.

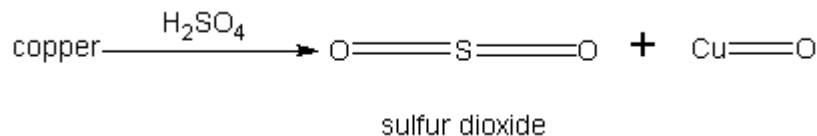
Method 2A: Preparation of sulfur dioxide gas from sulfuric acid and copper

(By-products from reaction: water and copper-II-sulfate)

Materials:

1. 150 grams (5.2 oz.) of concentrated sulfuric acid	2. 97 grams (3.4 oz.) of copper wire, pipes, pieces, or any other pure copper material
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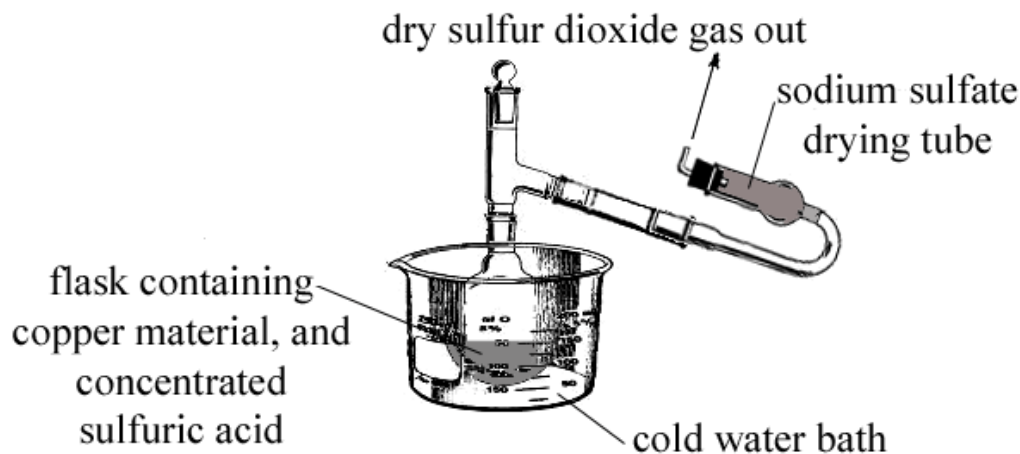
Reaction summary: Sulfur dioxide gas is readily prepared by reacting concentrated sulfuric acid with copper. The reaction is smooth and produces a steady stream of gas.



Why does this reaction happen? Sulfur dioxide can be prepared directly by reacting copper metal with concentrated sulfuric acid. The reaction occurs, because the copper acts as a reducing agent towards the sulfuric acid. First, the sulfuric acid gets reduced to sulfur dioxide, and forming copper oxide as the by-product, and second the copper oxide then reacts with another molecule of sulfuric acid forming copper sulfate.

Hazards: Use proper ventilation when using sulfur dioxide gas, and avoid inhalation of the fumes. Wear gloves when handling concentrated sulfuric acid, and use caution.

Procedure: Into the apparatus illustrated below, place 150 grams (5.2 oz.) of concentrated sulfuric acid, followed by 97 grams (3.4 grams) of copper wire, pipes, pieces, or any other pure copper material, and then seal the apparatus using a glass stopper as illustrated below. Thereafter, monitor the reaction and do not let the acid mixture get too hot. During the reaction, a steady stream of sulfur dioxide gas will be steadily evolved. The total reaction time can be anywhere from 45 minutes to 2 hours. Heat may or may not be applied to speed up the reaction.



Set-up for the preparation of sulfur dioxide gas from sulfuric acid and copper.

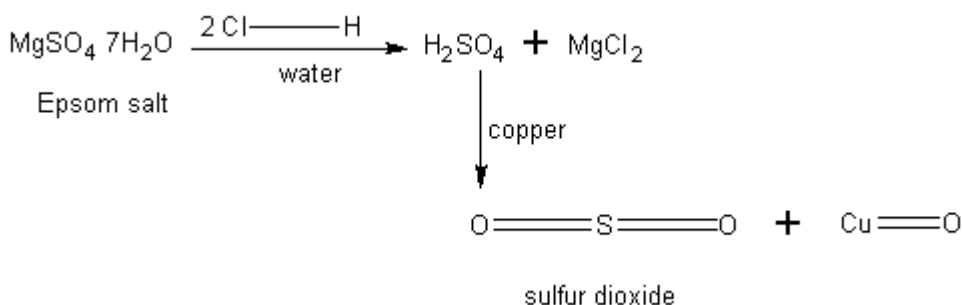
Method 2B: Preparation of sulfur dioxide gas from Epsom salt, hydrochloric acid, and copper

(By-products from reaction: water, magnesium chloride, and copper-II-sulfate)

Materials:

1. 400 grams (14.1 oz.) of Epsom salt	3. 330 grams (11.6 oz.) of concentrated hydrochloric acid or concentrated Muriatic acid
2. 1300 milliliters (1.3 quarts) of warm water	4. 103 grams (3.6 oz.) of copper wire, pipes, pieces, or any other pure copper material

Reaction summary: Sulfur dioxide gas can be readily prepared in a modified process using Epsom salt rather than sulfuric acid. In this regards, the concentrated hydrochloric acid partially reacts with the Epsom salt forming the acidic bisulfate ion, and sulfuric acid. These substances then in turn, react with copper to produce sulfur dioxide and the usual by-products.



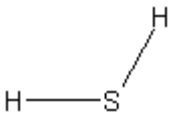


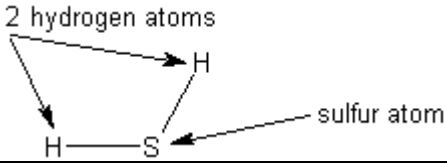
Why does this reaction happen? Epsom salt can be used as a substitute for sulfuric acid as it forms sulfuric acid when treated with hydrochloric acid. Once the sulfuric acid is formed, copper is added to the reaction mixture, and the copper reduces the sulfuric acid forming sulfur dioxide and with copper oxide as the by-product.

Hazards: Use proper ventilation when using sulfur dioxide gas, and avoid inhalation of the fumes. Wear gloves when handling concentrated hydrochloric acid, and avoid inhalation of the fumes.

Procedure: Into a large beaker or similar container, place 400 grams (14.1 oz.) of Epsom salt, followed by 1300 milliliters (1.3 quarts) of warm water, and then stir the mixture briefly to dissolve all of the Epsom salt. Thereafter, pour this Epsom salt solution into a similar apparatus as illustrated in method 2A, and then add in 330 grams (11.6 oz.) of concentrated hydrochloric acid or concentrated Muriatic acid, and then blend the mixture for about 10 minutes to form a uniform acidic mixture. Note: an exothermic reaction will proceed, lowering the temperature of the acidic solution. Thereafter add in 103 grams (3.6 oz.) of copper wire, pipes, pieces, or any other pure copper material, and then seal the apparatus using a glass stopper. Thereafter, monitor the reaction and do not let the acid mixture get to hot. During the reaction, a steady stream of sulfur dioxide gas will be steadily, yet slowly evolved. The total reaction time can be anywhere from 2 hours to 4 hours. Heat may or may not be applied to speed up the reaction.

Procedure 12: The Preparation of Anhydrous Hydrogen Sulfide gas

Also known as: sulfurated hydrogen; hydrosulfuric acid

	 <p>3D structure 1</p>  <p>3D structure 2</p>
Chemical structure	3D Structure
	<p>H_2S</p>
Structure make-up	Condensed chemical structure

Hydrogen sulfide gas

Hydrogen sulfide is a flammable, poisonous gas with disagreeable odor of rotten eggs. It can be detected by the human nose in extremely small quantities. Hydrogen sulfide has a sweetish taste. Hydrogen sulfide burns in air with a pale blue flame. It has a melting point of -85 Celsius, and a boiling point of -60 Celsius. Hydrogen sulfide is insoluble in water, and not very soluble in alcohol. It is soluble in glycerol, gasoline, kerosene, carbon disulfide, and crude oil. Hydrogen sulfide is a highly toxic gas, and inhalation can be fatal. Use maximum ventilation when handling. It is evolved from many different natural environmental sources including bacterial decomposition of vegetable and animal proteins, natural springs, natural gas deposits, and volcanoes. Hydrogen sulfide can be obtained from the distillation of petroleum. Hydrogen sulfide is prepared in the laboratory by dropping an acid (usually sulfuric or hydrochloric) onto a metal sulfide such as sodium sulfide, or calcium sulfide (calcium sulfide is prepared by roasting calcium sulfate with charcoal at 1000 Celsius). Hydrogen sulfide is commercially available, but shipping regulations may decrease sale.

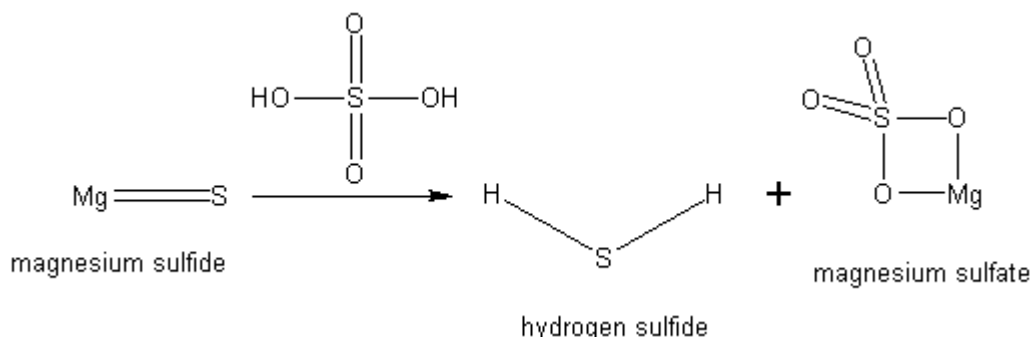
Method 1: Preparation of Anhydrous Hydrogen Sulfide gas from sulfides and sulfuric acid

(By-products from reaction: metal sulfates)

Materials:

1. 50 grams (1.8 oz.) of concentrated sulfuric acid	2. 30 to 40 grams (1.05 to 1.4 oz.) of powdered sodium sulfide, calcium sulfide, magnesium sulfide, or pulverized pyrites, or pulverized sulfide ores
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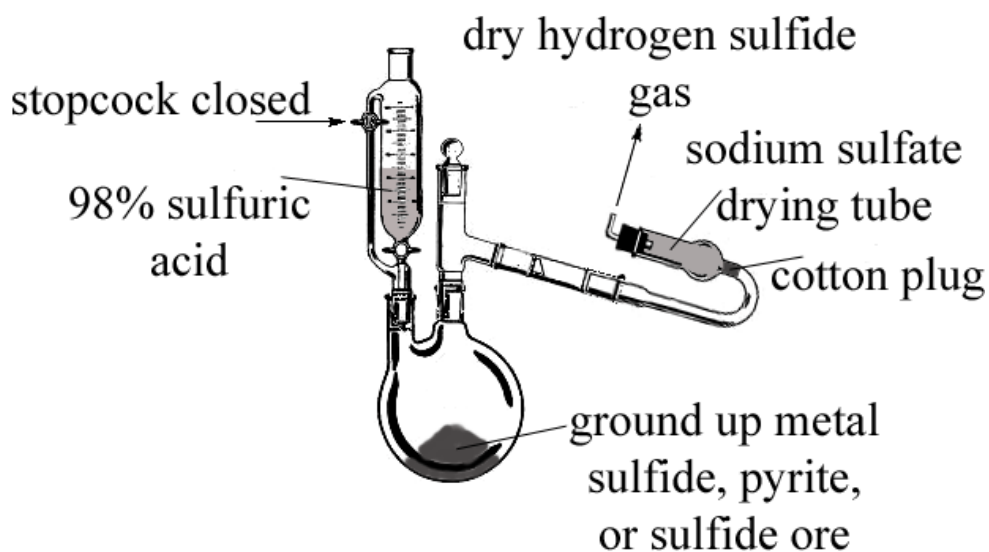
Reaction summary: Hydrogen Sulfide gas is readily prepared by reacting concentrated sulfuric acid with a sulfide material. Sulfides such as sodium or magnesium sulfide are the most commonly used sulfide materials, but pyrites, and sulfide ores can be used. **Note: Sodium, Calcium, or Magnesium sulfide can be prepared by roasting sodium sulfate, calcium sulfate (gypsum), or magnesium sulfate (Epsom salt) with powdered charcoal (natural charcoal; no added chemicals or quick light stuff) at 1000 Celsius (1800 Fahrenheit) for several hours. The resulting blackish-gray mass can then be cooled and then placed into the reaction flask for reaction with the sulfuric acid.**



Why does this reaction happen? Hydrogen sulfide is readily prepared by reacting a metal sulfide with concentrated sulfuric acid. The reaction takes place because the sulfuric acid prefers the metal ion of the sulfide compound rather than sulfur; therefore, hydrogen sulfide is produced as the by-product.

Hazards: Use proper ventilation when using hydrogen sulfide, and avoid chronic exposure, as hydrogen sulfide is toxic.

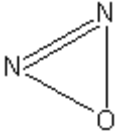


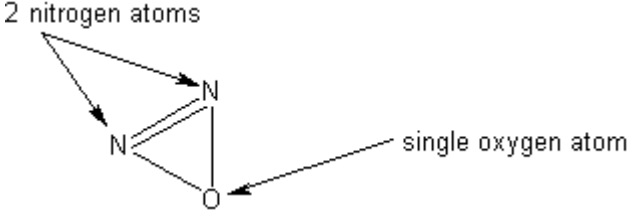
Procedure: Simply place 50 grams (1.8 oz.) of concentrated sulfuric acid into the addition funnel, and then place 30 to 40 grams (1.05 to 1.4 oz.) of powdered sodium sulfide, calcium sulfide, magnesium sulfide, or pulverized pyrites, or pulverized sulfide ores into the reaction flask. Thereafter, slowly drip the concentrated sulfuric acid onto the sulfides. During the sulfuric acid addition, hydrogen sulfide gas will be steadily evolved.



Set-up for the preparation of anhydrous hydrogen sulfide gas. Note: gently heating the reaction flask can help speed up the reaction.

Procedure 13: The Preparation of Nitrous Oxide (Laughing Gas)

Also known as: dinitrogen oxide; hyponitrous acid anhydride

	 <p>3D structure 1</p>  <p>3D structure 2</p>
Chemical structure	3D Structure
 <p>2 nitrogen atoms</p> <p>single oxygen atom</p>	<p>N_2O</p>
Structure makeup	Condensed chemical structure

Nitrous oxide

Nitrous oxide is a colorless gas that has a peculiar odor and taste. The gas supports combustion, but is relatively inert at room temperature. Nitrous oxide has a melting point of -90 Celsius, and a boiling point of -88 Celsius. The gas is readily obtained

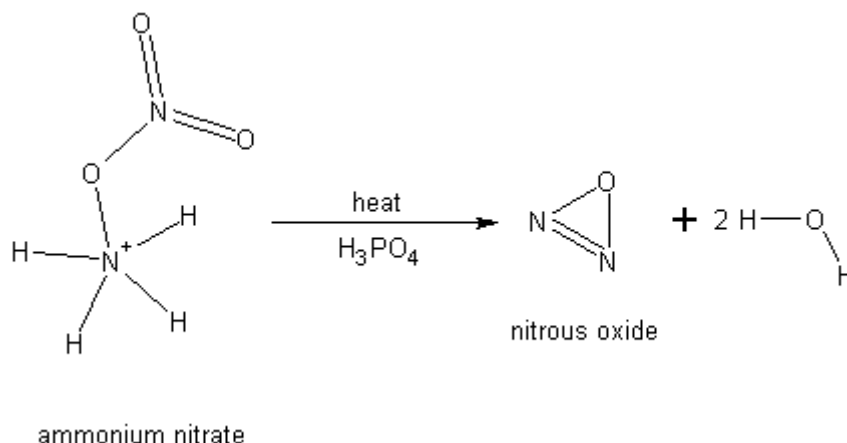
by the careful thermal decomposition of ammonium nitrate. The gas is an anesthetic when inhaled, and is capable of producing a narcotic-like high when large quantities are inhaled.

Method 1: Preparation of Nitrous Oxide from ammonium nitrate and phosphoric acid with heat

Materials:

1. 500 grams of pure ammonium nitrate	2. 5 grams of pure phosphoric acid.
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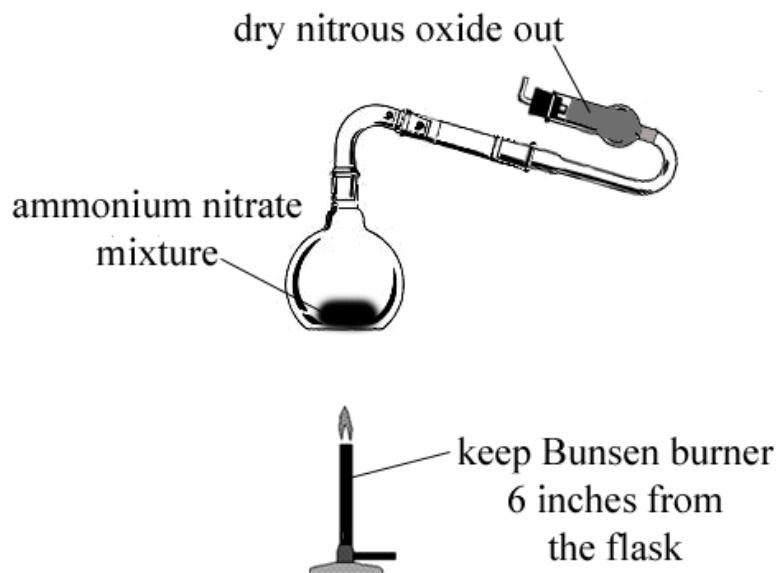
Reaction summary: Nitrous oxide is readily prepared by the thermal decomposition of ammonium nitrate. A catalyst is usually employed such as phosphorus compounds and the like. Metal salts such as iron oxide may be suitable as well. The best way to store the nitrous oxide, is to tie a balloon around the exit tube, and let the pressure inflate the balloon.



Why does this reaction happen? Nitrous oxide is formed by the thermal decomposition of ammonium nitrate because the two nitrogens of the ammonium nitrate prefer to bond to each other forming diatomic nitrogen; however, instead of forming diatomic nitrogen, the two nitrogens bond together and then bond with a single oxygen atom forming nitrous oxide. Water is the by-product and is formed because the remaining hydrogens prefer to bond with the other oxygen atoms forming the very stable and preferred water molecules.

Hazards: Use caution when using an open flame.

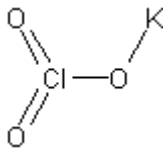
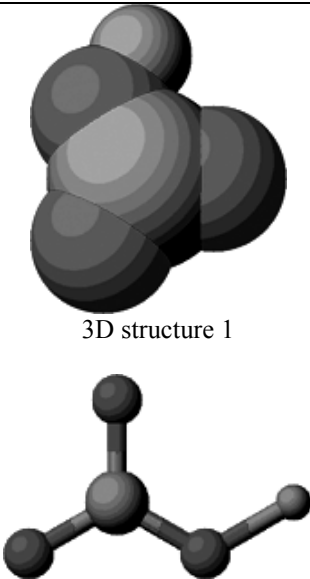
Procedure: Into a plastic zip lock bag, or any other container, place 500 grams of pure ammonium nitrate, and then add in 5 grams of pure phosphoric acid. Thereafter, shake the ingredients vigorously for about 5 to 10 minutes to form a uniform mixture. Thereafter, place the mixed ingredients into an apparatus as illustrated below, and then gently and carefully heat the mixture. During the heating process, do not keep the flame on the flask for more than 5 to 10 seconds at a time. Too much heat, and over heating can cause a pressure gap. During the on-and-off heating process, a steady stream of nitrous oxide and steam will be evolved.

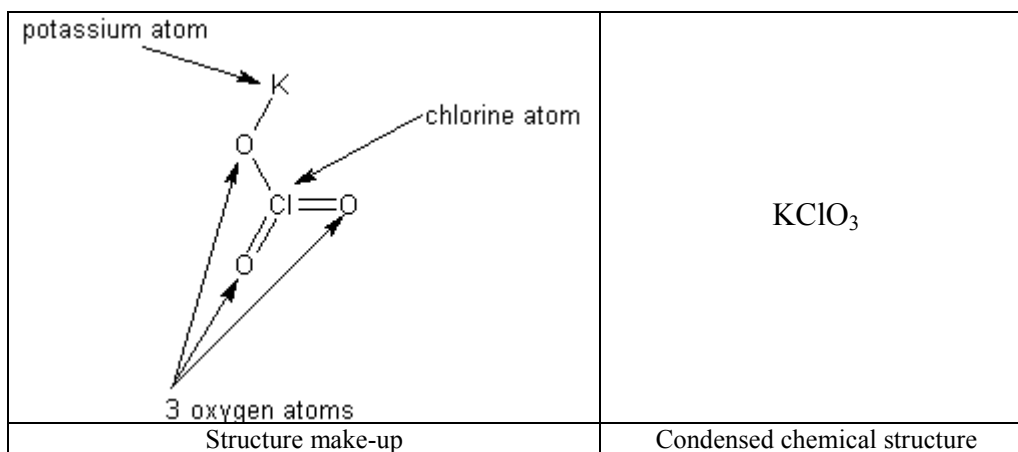


Simple setup for generating nitrous oxide.

Procedure 14: The Preparation of Potassium chlorate (classic precipitation “crystallization” reaction)

Also known as: potcrate

	 <p>3D structure 1</p> <p>3D structure 2</p>
Chemical structure	3D Structure



Potassium chlorate

Potassium chlorate forms colorless to white crystals, white granules or powder. The crystals have a melting point of 368 Celsius, and are relatively insoluble in water and most organic solvents. However, the crystals are soluble in boiling water. Potassium chlorate forms explosives mixtures with combustible materials. These mixtures readily ignite from friction, heat, flames, or sparks. Potassium chlorate should be kept away from iodides and tartaric acid.

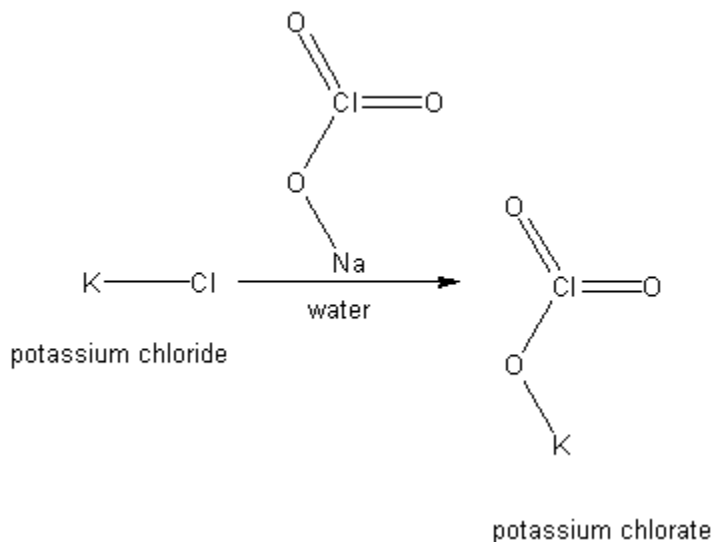
Method 1: Preparation of Potassium chlorate from sodium chlorate

(By-products from reaction: sodium chloride)

Materials:

1. 220 milliliters (7.4 fluid oz.) of warm water	3. 105 grams (3.7 oz.) of potassium chloride
2. 150 grams (5.2 oz.) of sodium chlorate	4. 250 milliliters (8.4 fluid oz.) of warm water

Reaction summary: Potassium chlorate is readily prepared by condensing potassium chloride with sodium chlorate in solution. After the initial mixing, the potassium chlorate is readily collected by filtration.



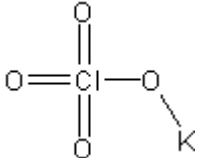

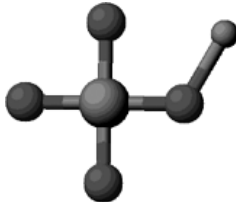
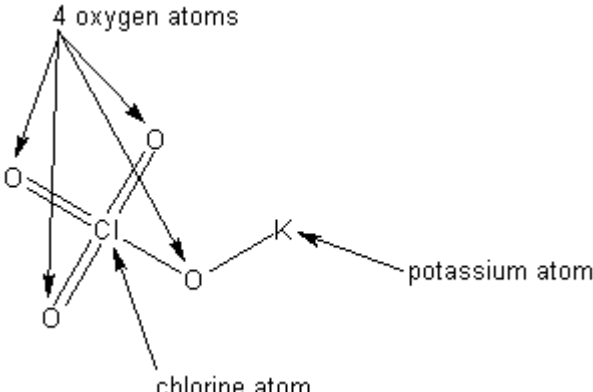
Why does this reaction happen? As with other precipitation reactions, potassium chlorate forms when two solutions of potassium chloride and sodium chlorate are mixed because the potassium chlorate is less soluble in water than the potassium chloride or sodium chlorate. Sodium chlorate is very soluble in water, yet potassium chlorate is not; therefore, when the two solutions of potassium chloride and sodium chlorate are mixed, the potassium chlorate precipitates due to its lack of solubility.

Hazards: None.

Procedure: Into a suitable beaker, flask, or any other suitable container, place 220 milliliters (7.4 fluid oz.) of warm water, and then add in 150 grams (5.2 oz.) of sodium chlorate. Thereafter, stir the mixture until all the chlorate dissolves. Now, prepare a second solution by adding and dissolving 105 grams (3.7 oz.) of potassium chloride into 250 milliliters (8.4 fluid oz.) of warm water in a suitable beaker, flask, or container. Then, quickly mix both solutions, and then stir the combined mixture for about 30 minutes. Thereafter, cool the mixture to 0 Celsius by using an ice bath, or by placing the beaker, flask, or container containing the combined mixture into a refrigerator and allow it to stand for 1 hour or so. Afterwards, filter-off the precipitated potassium chlorate, and then vacuum dry or air-dry the crystals. Afterwards, the potassium chlorate can be recrystallized from boiling water if desired, but this is not necessary in most cases as the chlorate will have a good purity as is.

Procedure 15: The Preparation of Potassium perchlorate

Also known as: Peroidin; perchloracap

	 <p>3D structure 1</p>  <p>3D structure 2</p>
Chemical structure	3D Structure
	<p>KClO₄</p>
Structure make-up	Condensed chemical structure

Potassium perchlorate

Potassium perchlorate forms colorless to white crystals, or white crystalline powder, or granules. The crystals begin to decompose when heated to 400 Celsius into oxygen and potassium chloride. Potassium perchlorate is sensitive to concussion, and forms explosive mixtures with combustible materials such as sulfur and charcoal. The crystals have a low solubility in water (1 gram per 65 milliliters of water) at room temperature, but the crystals are somewhat soluble in boiling water (1 gram in 15 milliliters). Potassium perchlorate is insoluble in the usual solvents.

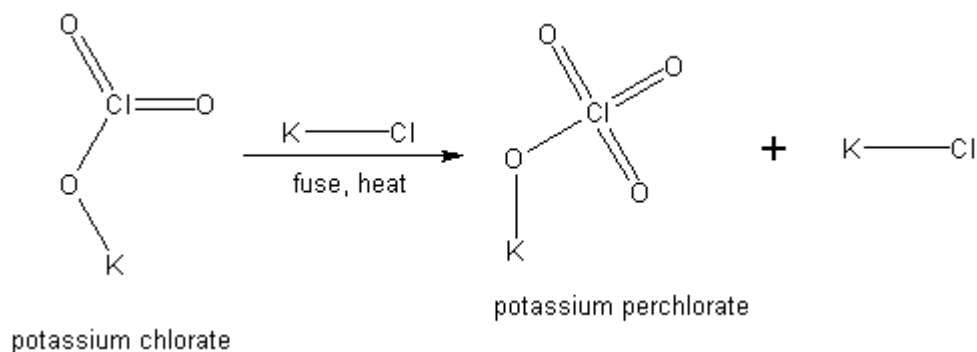
Method 1: Preparation of Potassium perchlorate from potassium chlorate

(By-products from reaction: potassium chloride)

Materials:

1. 100 grams (3.5 oz.) of potassium chlorate	3. 200 milliliters (6.7 fluid oz.) of water
2. 50 grams (1.7 oz.) of potassium chloride	

Reaction summary: Potassium perchlorate is readily prepared by fusing potassium chlorate in the presence of potassium chloride. The potassium chloride is used to decrease decomposition of the perchlorate and provide a stabilized uniform mixture. After the initial reaction, the potassium perchlorate is easily collected by thoroughly mixing the cooled mixture in water to dissolve the potassium chloride and any un-reacted chlorate, followed by filtration to recover the insoluble perchlorate.



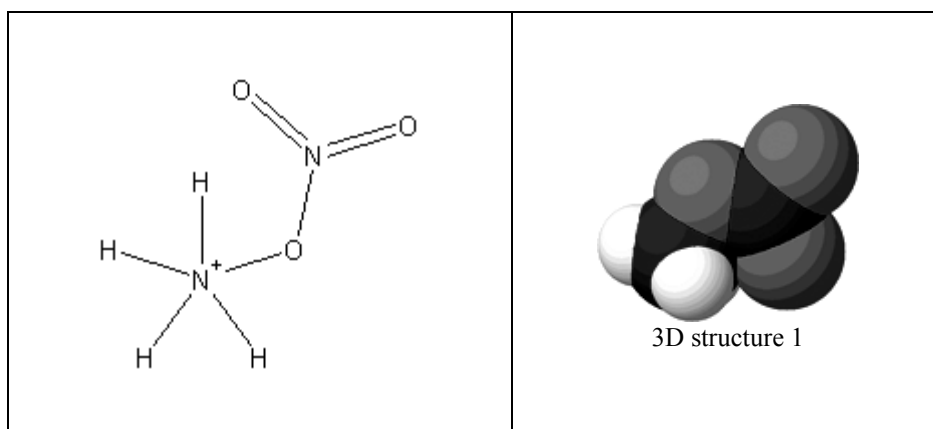
Why does this reaction happen? Potassium perchlorate is formed when a molten mixture of potassium chloride and potassium chlorate are fused to about 400 Celsius. The reason why the perchlorate is formed is because the molten potassium chlorate self oxidizes itself forming the perchlorate, and a second molecule of potassium chloride.

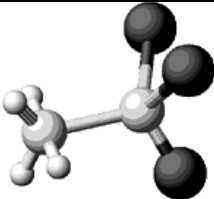
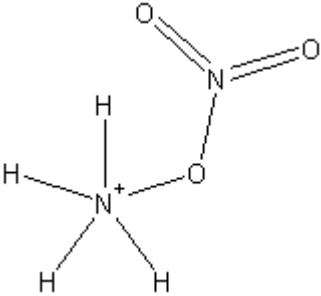
Hazards: None.

Procedure: Into a suitable crucible, place 100 grams (3.5 oz.) of potassium chlorate, followed by 50 grams (1.7 oz.) of potassium chloride. Thereafter, briefly shake the crucible to mix the two salts. Then gently and slowly heat the mixture using a Bunsen burner to about 370 Celsius. Be sure to carefully monitor the temperature using a temperature probe or equivalent. Shortly thereafter, a gentle gas evolution of oxygen will take place. Keep heating the mixture at about 380 Celsius until the evolution of gas ceases. After the evolution of gas has ceased, immediately remove the heat source, and allow the mixture to cool to room temperature. Then, using a knife or other sharp and hard instrument, fracture and pulverize the solid mass in the crucible, and then place this pulverized mass into a suitable beaker, or container. Thereafter, add in 200 milliliters (6.7 fluid oz.) of water (at room temperature), and then rapidly stir the entire mixture for about 1 hour to dissolve the potassium chloride. After stirring for 1 hour, filter-off the insoluble potassium perchlorate, and then vacuum dry or air-dry the crystals.

Procedure 16: The Preparation of Ammonium nitrate

Also known as: nitrate of ammonia



	 3D structure 2
Chemical structure	3D Structure
 4 hydrogen atoms 2 nitrogen atoms 3 oxygen atoms	 NH_4NO_3
Structure make-up	Condensed chemical structure

Ammonium nitrate

Ammonium nitrate forms odorless, transparent, hygroscopic, and deliquescent crystals or white granules. It decomposes at 210 Celsius into water and nitrous oxide (laughing gas). Ammonium nitrate is very soluble in water, and alcohol. It is widely available commercially, and is available in a number of products including fertilizers and garden products. For use in explosives, AN is commonly used admixed with fuel oil, diesel fuel, TNT, oils, and aluminum for explosives compositions. Ammonium nitrate mixed with oils and fuels are commonly called prills, and are highly stable mixtures widely used in mining and engineering operations. Because ammonium nitrate prills are quite stable for long-term storage and they yield no noxious fumes upon detonation, they are called “safety explosives”. Ammonium nitrate and TNT are commonly used as military dynamites, and blasting charges for military engineering purposes. Ammonium nitrate should be stored in tightly sealed, amber glass bottles, or non-transparent plastic containers in a well-cooled area protected from sunlight.

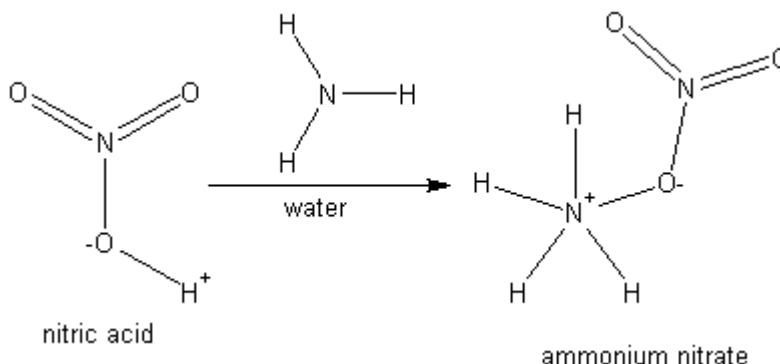
Method 1: Preparation of ammonium nitrate from ammonia and nitric acid (as seen in The Preparatory Manual of Explosives by Jared Ledgard)

(By-products from reaction: none)

Materials:

1. 206 grams (7.3 oz.) of 70% nitric acid	3. Next, bubble 40 grams (1.4 oz.) of ammonia gas or 136 grams (4.8 oz.) of 28 – 30% ammonia solution or 400 grams (14.1 oz.) of a 10% ammonia solution (household ammonia)
2. 600 milliliters (20.2 fluid oz.) of water	

Reaction summary: Ammonium nitrate is easily prepared by dissolving ammonia gas, or water solutions of ammonia into dilute nitric acid at low temperature. The ammonium nitrate is recovered by recrystallization from the water.



Why does this reaction happen? As seen in previous reactions, acids are highly reactive towards bases such as ammonia. When nitric acid reacts with ammonia, the nitrogen of the ammonia grabs the hydrogen ion from the nitric acid, forming an ammonium ion. The positively charged ammonium ion then bonds with the negatively charged nitrate ion of the nitric acid forming ammonium nitrate.

Hazards: Wear gloves when handling 70% nitric acid. 70% Nitric is highly toxic and corrosive. Use proper ventilation when handling ammonia gas, or its solutions.

Procedure: Dissolve 206 grams (7.3 oz.) of 70% nitric acid into 600 milliliters (20.2 fluid oz.) of water. Then cool the diluted nitric acid solution to 0 Celsius by means of an ice bath while stirring continuously. Next, bubble 40 grams (1.4 oz.) of ammonia gas into the nitric acid mixture over a period of one hour while stirring and maintaining the reaction mixtures temperature at 0 Celsius. Or slowly add 136 grams (4.8 oz.) of 28 – 30% ammonia solution, or 400 grams (14.1 oz.) of a 10% ammonia solution into the diluted nitric acid solution while stirring and maintaining the diluted nitric acids temperature at 0 Celsius. Upon completion of the ammonia addition, recrystallize the ammonium nitrate from the reaction mixture, and then vacuum dry or air dry the product. If a rotary evaporator is available, place the filtered reaction mixture there into, and remove the water under high vacuum. Then, recrystallize the ammonium nitrate from methanol (2 grams (0.07 oz.) ammonium nitrate dissolves in 16 milliliters (0.54 fluid oz.) methanol, and then vacuum dry or air-dry the product. The product will weigh about 180 to 188 grams and will be of 99% purity.

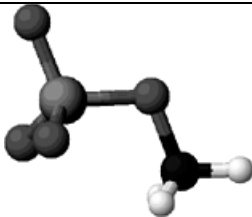
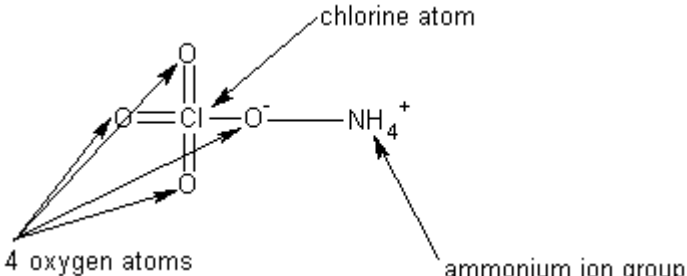
Method 2: Preparation of ammonium nitrate from sodium nitrate, ammonia, and carbon dioxide (as seen in The Preparatory Manual of Explosives by Jared Ledgard)

(By-products from reaction: sodium bicarbonate)

Materials:

1. 1000 grams (2.2 pounds) of sodium nitrate	3. 740 grams (26.1 oz.) of 28 – 30% ammonia solution or 2000 grams (4.4 pounds) of 10% ammonia solution or 200 grams (7 oz.) of anhydrous ammonia
2. 2300 milliliters (2.4 quarts) of water	4. 3530 grams (7.8 pounds) of dry carbon dioxide or 3000 grams (6.6 pounds) of dry ice

Reaction summary: Ammonium nitrate is easily prepared by dissolving sodium nitrate into water, adding ammonia, and then passing in an excess of carbon dioxide gas (dry ice may be used if desired). The resulting insoluble sodium bicarbonate is then filtered-off, and the ammonium nitrate is recovered by recrystallization from the water.

	 <p>3D structure 2</p>
Chemical structure	3D Structure
 <p>chlorine atom</p> <p>4 oxygen atoms</p> <p>ammonium ion group</p>	NH_4ClO_4
Structure make-up	Condensed chemical structure

Ammonium perchlorate

Ammonium perchlorate forms colorless orthorhombic crystals or a white powder or granules. The crystals decompose when heated into oxygen and ammonium chloride. The salt is readily soluble in water, but relatively insoluble in most organic solvents. Ammonium perchlorate is widely used in solid rocket propellants. The salt is a strong oxidizer, and should be kept away from combustible materials and sources of possible ignition. Store ammonium perchlorate in tightly sealed bottles in a cool place, and away from sunlight.

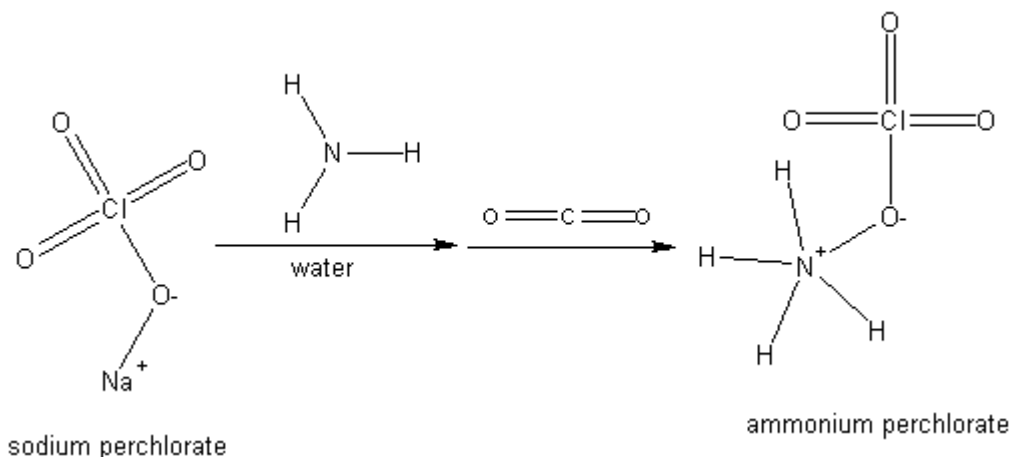
Method 1: Preparation of ammonium perchlorate from household ammonia and sodium perchlorate utilizing carbon dioxide (as seen in *The Preparatory Manual of Explosives* by Jared Ledgard)

(By-products from reaction: sodium bicarbonate)

Materials:

1. 470 grams (1 pound) of sodium perchlorate monohydrate	4. 1300 grams (2.8 pounds) of dry carbon dioxide gas
2. 1000 milliliters (1 quart) of tap water	5. or 1000 grams (2.2 pounds) of dry ice
3. 900 grams (1.9 pounds) of a household ammonia solution	

Reaction summary: Ammonium perchlorate is prepared by adding an ammonia solution to a concentrated solution of sodium perchlorate, and then passing carbon dioxide into the solution over several hours to precipitate the sodium as the water insoluble sodium bicarbonate. The ammonium perchlorate remains in solution, and the solution is then filtered to remove the water insoluble sodium bicarbonate. Thereafter, the ammonium perchlorate is then collected by recrystallization in the usual means.



Why does this reaction happen? As in the previous example, we use carbon dioxide to initiate a precipitation reaction. Usually, when sodium perchlorate and ammonia are mixed, no reaction takes place; however, if we add in carbon dioxide, a reaction takes place whereby ammonium perchlorate forms in solution, and the sodium ion of the sodium perchlorate reacts with the carbon dioxide forming the relatively water insoluble sodium bicarbonate, which precipitates leaving the ammonium perchlorate in solution.

Hazards: Wear gloves and use proper ventilation when handling ammonia.

Procedure: Dissolve 470 grams (1 pound) of sodium perchlorate monohydrate into 1000 milliliters (1 quart) of tap water. Then quickly add in 900 grams (1.9 pounds) of a household ammonia solution (obtained from grocery stores, sold as “crystal clear” ammonia), while stirring the sodium perchlorate solution. Afterwards, Cool the mixture to 0 Celsius by means of an ice bath. Then bubble 1300 grams (2.8 pounds) of dry carbon dioxide gas into the mixture over a several hour period while keeping the reaction temperature at 0 Celsius and stirring, or add in pieces, 1000 grams (2.2 pounds) of dry ice. During the carbon dioxide gas addition, sodium bicarbonate will slowly precipitate (if dry ice is used, precipitation of the sodium bicarbonate will be more rapid). After all the carbon dioxide has been added, continue to stir the reaction mixture for 90 minutes at 0 Celsius, and then filter the reaction mixture to remove the insoluble sodium bicarbonate. After filtration, recrystallize the ammonium perchlorate from the filtered mixture at 100 Celsius. Note: during the recrystallization process, do not over recrystallize. This means when most of the ammonium perchlorate has been collected by filtration, do not boil-off all the remaining water to the point where dry solid remains. Dry ammonium perchlorate may decompose if heated above 70 Celsius. If boiling the mixture at 100 Celsius during the recrystallization process causes some decomposition of the ammonium perchlorate, place the filtered reaction mixture into a shallow pan, and heat the pan at 50 Celsius while blowing air over the surface of the liquid. Do this until dry solid remains (a small portable cooling fan can be used).

Method 2: Preparation of ammonium perchlorate from dry ammonia gas and sodium perchlorate utilizing carbon dioxide (as seen in The Preparatory Manual of Explosives by Jared Ledgard)

(By-products from reaction: sodium bicarbonate)

Materials:

1. 470 grams (1 pound) of sodium perchlorate monohydrate	4. 1300 grams (2.8 pounds) of dry carbon dioxide gas
2. 1000 milliliters (1 quart) tap water	5. or 1000 grams (2.2 pounds) of dry ice
3. 96 grams (3.4 ounces) of anhydrous ammonia gas	

Reaction Summary: This procedure is identical to method 1, except the house hold ammonia is replaced with dry anhydrous ammonia gas. Ammonium perchlorate is prepared by bubbling dry ammonia gas into a concentrated solution of sodium perchlorate, and then passing carbon dioxide into the solution over several hours to precipitate the sodium as the water insoluble sodium bicarbonate. The ammonium perchlorate remains in solution, and the solution is then filtered. Thereafter, the ammonium perchlorate is then collected by recrystallization.

Why does this reaction happen? Same as in method 1.

Hazards: Wear gloves and use proper ventilation when handling dry ammonia gas, which is highly irritating to the nose and throat.

Procedure: Dissolve 470 grams (1 pound) of sodium perchlorate monohydrate into 1000 milliliters (1 quart) water. Then bubble 96 grams (3.4 ounces) of anhydrous ammonia gas into the sodium perchlorate solution while stirring the sodium perchlorate solution. Afterwards, Cool the mixture to 0 Celsius by means of an ice bath. Then bubble 1300 grams (2.8 pounds) of dry carbon dioxide gas into the mixture over a several hour period while keeping the reaction temperature at 0 Celsius and stirring, or add in pieces, 1000 grams (2.2 pounds) of dry ice. During the carbon dioxide gas addition, sodium bicarbonate will slowly precipitate (if dry ice is used, precipitation of the sodium bicarbonate will be more rapid). After all the carbon dioxide has been added, continue to stir the reaction mixture for 90 minutes at 0 Celsius, and then filter the reaction mixture to remove the insoluble sodium bicarbonate. After filtration, recrystallize the ammonium perchlorate from the filtered mixture at 100 Celsius. Note: during the recrystallization process, do not over recrystallize. This means when most of the ammonium perchlorate has been collected by filtration, do not boil-off all the remaining water to the point where dry solid remains. Dry ammonium perchlorate may decompose if heated above 70 Celsius. If boiling the mixture at 100 Celsius during the recrystallization process causes some decomposition of the ammonium perchlorate, place the filtered reaction mixture into a shallow pan, and heat the pan at 50 Celsius while blowing air over the surface of the liquid. Do this until dry solid remains (a small portable cooling fan can be used).

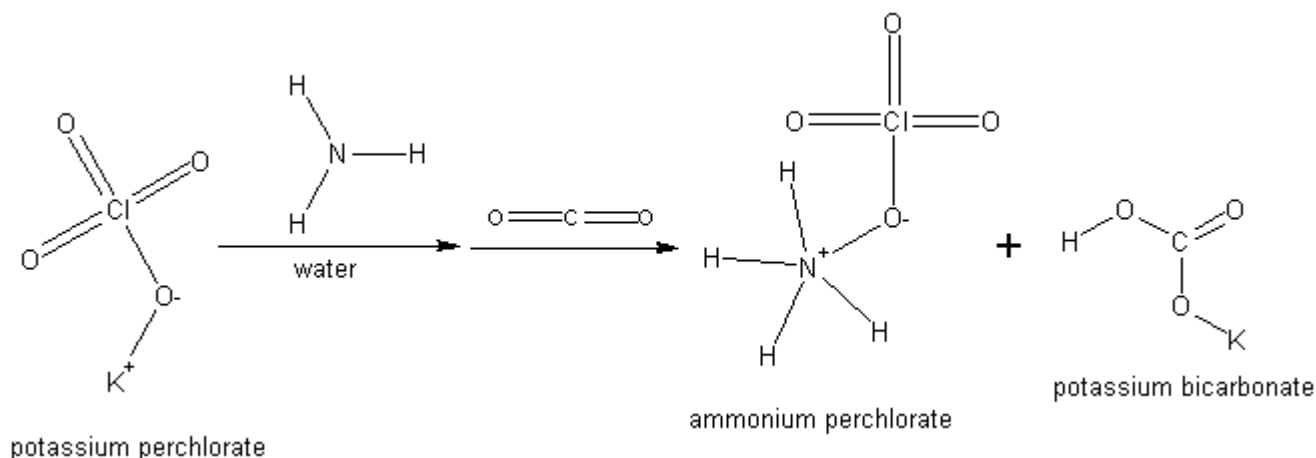
Method 3: Preparation of ammonium perchlorate from house-hold ammonia and potassium perchlorate utilizing carbon dioxide

(By-products from reaction: potassium bicarbonate)

Materials:

1. 57.6 grams (2 oz.) of potassium perchlorate	4. 200+ grams (7 oz.) of dry carbon dioxide gas
2. 1000 milliliters (1 quart) tap water	5. or 200+ grams (7 oz.) of dry ice
3. 90 grams (3.1 oz.) of a household ammonia solution	

Reaction Summary: This procedure is identical to method 1 and 2; except the sodium perchlorate monohydrate is simply replaced with potassium perchlorate. The ammonium perchlorate is prepared by adding an ammonia solution to a hot dilute solution of potassium perchlorate, and then passing carbon dioxide into the solution over several hours to form potassium bicarbonate and ammonium perchlorate. After the reaction, the mixture is chilled, and then filtered to remove the potassium bicarbonate precipitate. The ammonium perchlorate remains in solution, and is then collected by recrystallization in the usual means.



Why does this reaction happen? Same as in methods 1 and 2, except that the sodium perchlorate is replaced with potassium perchlorate.

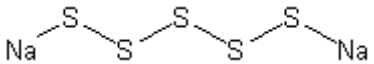

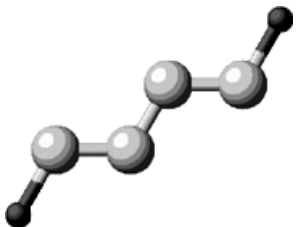
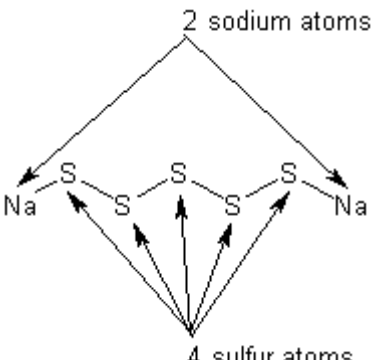
Hazards: Wear gloves and use proper ventilation when handling ammonia.

Procedure: Dissolve 57.6 grams (2 oz.) of potassium perchlorate into 1000 milliliters (1 quart) of boiling tap water. Then quickly add in 90 grams (3.1 oz.) of a household ammonia solution (obtained from grocery stores, sold as “crystal clear” ammonia), while stirring the potassium perchlorate solution. Afterwards, while boiling the solution, bubble 200+ grams (7 oz.) of dry carbon dioxide gas into the mixture over a several hour period while boiling and stirring the mixture, or add in pieces, 200+ grams (7 oz.) of dry ice. Use caution when adding dry ice to boiling water. During the carbon dioxide gas addition,

potassium bicarbonate will slowly form, and some of it will dissolve in the boiling water, and some will precipitate (if dry ice is used, the formation of potassium bicarbonate will be much faster). Note: during the boiling process, water will evaporate. As the water evaporates, more needs to be added, so maintain the volume of the water at about 1000 milliliters during the whole boiling process. After all the carbon dioxide has been added, continue to stir the reaction mixture at 100 Celsius for about 10 to 15 minutes, and then remove the heat source and allow the mixture to cool to room temperature. Thereafter, place the cooled reaction mixture into an ice bath, and chill to 0 Celsius and allow this mixture to stand at this temperature for about 90 minutes. Thereafter, filter the reaction mixture to remove the precipitated potassium bicarbonate. After filtration, recrystallize the ammonium perchlorate from the filtered mixture at 100 Celsius. Note: during the recrystallization process, do not over recrystallize. This means when most of the ammonium perchlorate has been collected by filtration, do not boil-off all the remaining water to the point where dry solid remains. Dry ammonium perchlorate may decompose if heated above 70 Celsius. If boiling the mixture at 100 Celsius during the recrystallization process causes some decomposition of the ammonium perchlorate, place the filtered reaction mixture into a shallow pan, and heat the pan at 50 Celsius while blowing air over the surface of the liquid. Do this until dry solid remains (a small portable cooling fan can be used).

Procedure 18: The Preparation of Sodium polysulfides

Also known as: disodium pentasulfide

	 3D structure 1  3D structure 2
Chemical structure	3D Structure
	N_2S_4
Structure make-up	Condensed chemical structure

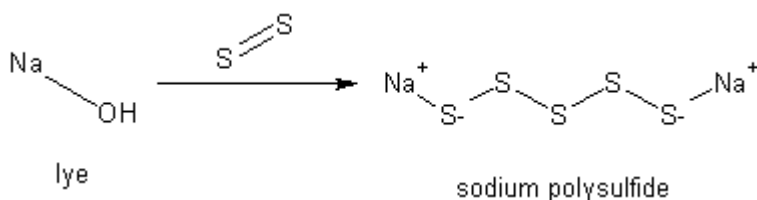
Method 1: Preparation of sodium polysulfides from sulfur and lye

(By-products from reaction: sodium thiosulfate)

Materials:

1. 250 milliliters (8.5 fluid oz.) of cold water	3. 50 grams (1.7 oz.) of powdered sulfur
2. 51 grams (1.8 oz.) of sodium hydroxide	

Reaction summary: Sodium polysulfide is readily prepared by treating a sodium hydroxide solution with excess sulfur. The reaction produces a dark brownish red liquid.



Why does this reaction happen? Sodium hydroxide is a very reactive compound and is capable of reacting with many different compounds. Sulfur, being one of them, readily dissolves in sodium hydroxide solutions forming sodium sulfide and sodium thiosulfate. When more sulfur is added to the mixture, sodium polysulfides form, turning the reaction mixture a dark reddish color.

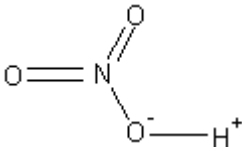

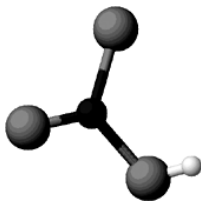
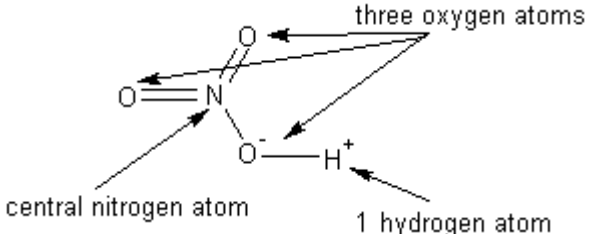
Hazards: Use caution when handling sodium hydroxide, which is capable of producing skin burns.

Procedure: Into a suitable beaker, flask, ect., place 250 milliliters (8.5 fluid oz.) of cold water, and then add and dissolve 51 grams (1.8 oz.) of sodium hydroxide. Note: sodium hydroxide generates excessive heat when dissolved in water so use caution. Thereafter, add in, in small portions at a time, 50 grams (1.7 oz.) of powdered sulfur, and stir the mixture vigorously during the addition. Note: more sulfur may be added if desired. Thereafter, continue to stir the mixture for about 30 minutes. After 30 minutes the mixture is ready for use. Even though the mixture will be contaminated with sodium thiosulfate, it can be used directly in the reduction of nitro compounds.

This method of preparing sodium polysulfides can be used to purify sulfur to high degree. When hydrochloric acid is added to the mixture, pure sulfur precipitates.

Procedure 19: The Preparation of Nitric Acid

Also known as: Aquafortis

	 <p>3D structure 1</p>  <p>3D structure 2</p>
Chemical structure	3D Structure
	<p>HNO_3</p>
Structure make-up	Condensed chemical structure

70% Nitric acid (65 to 68% acid) *Aqua fortis*; *Salpetersaure*

70% Nitric acid is referred to as concentrated nitric acid, and has a concentration of 65 to 71% nitric acid by weight (simply called 70% nitric acid). 70% nitric acid is a colorless liquid, which fumes in moist air yielding a choking vapor. Its melting point is -41 Celsius with a boiling point of 85 Celsius. When boiling 70% nitric acid, it forms a constant boiling mixture with water forming 68% nitric acid with a boiling point of 120 Celsius. 70% nitric acid should be stored in airtight glass bottles away from sunlight. It stains fabrics, and animal tissue a bright yellow. In contact with the skin, it produces a yellow stain. 70% Nitric acid is a powerful oxidizing acid. It reacts violently with many different substances including alcohols, turpentine, charcoal, and organic refuse. 70% Nitric acid begins to turn yellow in contact with strong light sources such as sunlight or magnesium light. 70% Nitric acid is a widely available commercial acid. It is the 7th largest manufactured chemical in the US. Many chemical dealers will not sell 70% nitric acid to unlicensed persons because of its use in manufacturing high explosives. Concentrated nitric acid is prepared on an industrial scale by the oxidation of ammonia with air in a special ammonia burner, utilizing platinum as catalyst. ***Wear gloves and use proper ventilation when handling 70% nitric acid. 70% Nitric acid can cause skin burns and can chemically react with clothing. Use caution when handling.***

90% fuming nitric acid

90% Nitric acid is a yellow to brownish-red, strongly fuming liquid. It is a very poisonous and corrosive liquid, which evolves poisonous, choking fumes of nitrogen oxides. It is a powerful oxidizer, and should be kept stored in glass, or Teflon containers, in a cool dry place and away from light (preferably in a refrigerator). 90% Nitric acid reacts violently with many substances. The acid is commercially available, but shipping regulations and dealer restrictions may limit its sale. In most cases, 90% nitric acid is not available to the general public. ***Wear gloves and use maximum ventilation when handling this substance.***

99% Nitric Acid

99% nitric acid is a colorless (when freshly prepared, i.e., white fuming acid), highly fuming, and poisonous liquid. It turns yellow to dark-red on standing, and has a melting point of -41.59 Celsius. 99% nitric acid is a powerful oxidizer, and it reacts violently with many substances. For the most part, the acid should be used right after it's preparation, but the fuming acid can be stored in tightly sealed amber glass bottles in a cool place for a week or more. 99% Nitric acid is commercially available, but shipping regulations and dealer restrictions will definitely restrict its sale and shipment to most people. ***Warning! 99% Nitric acid is a very poisonous and corrosive liquid, which evolves large amounts of poisonous fumes. Wear gloves and proper laboratory clothing (lab coat; boots, face shield) when handling this substance and use maximum ventilation.***

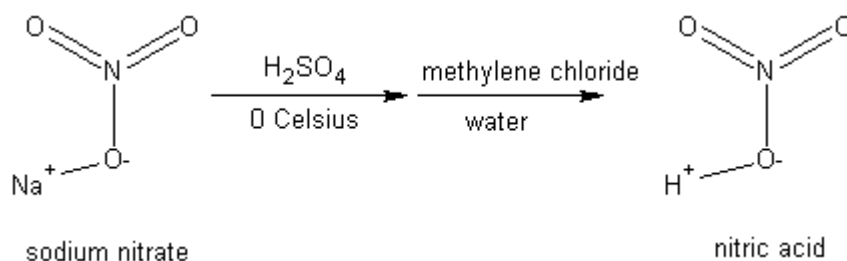
Method 1: Preparation of 70% nitric acid from potassium or sodium nitrate utilizing sulfuric acid and extraction with methylene chloride

(By-products from reaction: sodium or potassium sulfate)

Materials:

1. 194 grams (6.8 oz.) of 98% sulfuric acid	4. 1050 milliliters of methylene chloride (2.2 pints)
2. 100 grams (3.5 oz.) of potassium nitrate or 84 grams (3 oz.) of sodium nitrate	5. 27 milliliters (1 fluid oz.) of tap water
3. 260 milliliters (8.8 fluid oz.) of cold water	

Reaction summary: 70% Nitric acid can be obtained by treating potassium or sodium nitrate with sulfuric acid, mixing with water, and then extracting the acid mixture with methylene chloride. The result is a methylene chloride solution containing 99% nitric acid. This methylene chloride/99% nitric acid solution is then mixed with a calculated amount of water, and then carefully distilled to remove the methylene chloride and leave behind 70% nitric acid.



Why does this reaction happen? Sulfuric acid reacts with sodium nitrate forming nitric acid and sodium sulfate. The reaction mixture is then extracted with methylene chloride, whereby the nitric acid is taken up into the methylene chloride forming a nitric acid solution in methylene chloride. Water is then added, and the mixture is then distilled to remove the methylene chloride. The reason why sulfuric acid reacts with sodium nitrate is because the sulfuric acid has a higher affinity for the sodium of the nitrate.

Hazards: Methylene chloride/99% nitric acid mixture is very volatile and dangerous—wear gloves, and use proper ventilation. 99% nitric acid is highly toxic, and corrosive, which evolves poisonous fumes—use maximum ventilation. Perform the distillation using proper ventilation, and protect from direct sun light.

Procedure: Place 194 grams (6.8 oz.) of 98% sulfuric acid into a beaker, and then place the beaker in a ice bath and cool to 0 Celsius. When the sulfuric acid reaches a temperature of 0 Celsius, slowly add in portions, 100 grams (3.5 oz.) of potassium nitrate or 84 grams (3 oz.) of sodium nitrate over a period of 1 hour while stirring the sulfuric acid and maintaining its temperature at 0 Celsius. After the addition of the potassium or sodium nitrate, slowly add over a period of one hour, 260 milliliters (8.8 fluid oz.) of cold tap water while continuously stirring the sulfuric acid mixture and maintaining its temperature at 0 Celsius (note: a precipitate may form before or after the addition of the water—if a precipitate forms, never mind it). Afterwards, remove the ice bath and then extract the acid mixture with seven 150-milliliter portions (seven 5.0 fluid oz. portions) of methylene chloride. Then combine all seven portions of methylene chloride (if not already done so), and then add 27 milliliters (1 fluid oz.) of tap water to the methylene chloride. Next, place the entire methylene chloride (two phase) mixture into a distillation apparatus and carefully distill at 40 Celsius until no more methylene chloride is collected in the receiver flask. *Note: After all the methylene chloride has been removed, the 70% nitric acid may have a slight yellowish to red tint. If this is the case, don't worry—go ahead and use this nitric acid as is.*

Method 2: Preparation of 90% nitric acid from potassium or sodium nitrate utilizing sulfuric acid and extraction with methylene chloride

(By-products from reaction: sodium or potassium sulfate)

Materials:

1. 194 grams (6.8 oz.) of 98% sulfuric acid	3. 260 milliliters (8.8 fluid oz.) of cold tap water
2. 100 grams (3.5 oz.) of potassium nitrate or 84 grams (3 oz.) of sodium nitrate	4. 1050 milliliters of methylene chloride (2.2 pints)

Reaction summary: 90% Nitric acid can be obtained by treating potassium or sodium nitrate with sulfuric acid, mixing with water and then extracting the acid mixture with methylene chloride. The result is a methylene chloride solution containing 99% nitric acid.

This methylene chloride/99% nitric acid solution is then mixed with a calculated amount of water, and then distilled to remove the methylene chloride and produce 90% nitric acid.

Why does this reaction happen? Same as in method 1.

Hazards: Take caution when distilling and use proper ventilation because decomposition of the nitric acid might take place forming reddish-brown fumes of nitrogen oxides. If the nitric acid begins to decompose during the distillation, don't worry and continue the distillation. After all the methylene chloride has been removed, the result might be a reddish-brown highly fuming liquid. This reddish-brown fuming liquid is suitable for use as 90% nitric acid. Carryout the distillation with extreme caution. Do not heat the methylene chloride/nitric acid mixture above 40 Celsius, and use proper ventilation. ***Nitrogen oxide gases will develop so be prepared. Carryout the distillation away from direct sun light.***

Procedure: Place 194 grams (6.8 oz.) of 98% sulfuric acid into a beaker, and then place the beaker in an ice bath and cool to 0 Celsius. When the sulfuric acid reaches a temperature of 0 Celsius, slowly add in portions, 100 grams (3.5 oz.) of potassium nitrate or 84 grams (3 oz.) of sodium nitrate over a period of 1 hour while stirring the sulfuric acid and maintaining its temperature at 0 Celsius. After the addition of the potassium or sodium nitrate, slowly add over a period of one hour, 260 milliliters (8.8 fluid oz.) of cold tap water while continuously stirring the sulfuric acid mixture and maintaining its temperature at 0 Celsius (note: a precipitate may form before or after the addition of the water. If this happens, never mind it). Then remove the ice bath and then extract the acid mixture with seven 150-milliliter portions (seven 5.07 fluid oz. portions) of methylene chloride. Afterwards, combine all seven portions of methylene chloride (if not already done so), and then add 7 milliliters (0.23 fluid oz. or 1.4 tsp) of water to the methylene chloride. Then place the entire methylene chloride (two phase) mixture into a distillation apparatus (see the illustration in method 3 below), and carefully distill at 40 Celsius until no more methylene chloride is collected in the receiver flask.

Method 3: Preparation of 99% nitric acid from 70% Nitric acid or lower concentrations of nitric acid utilizing sulfuric acid and extraction with methylene chloride

(By-products from reaction: none)

Materials:

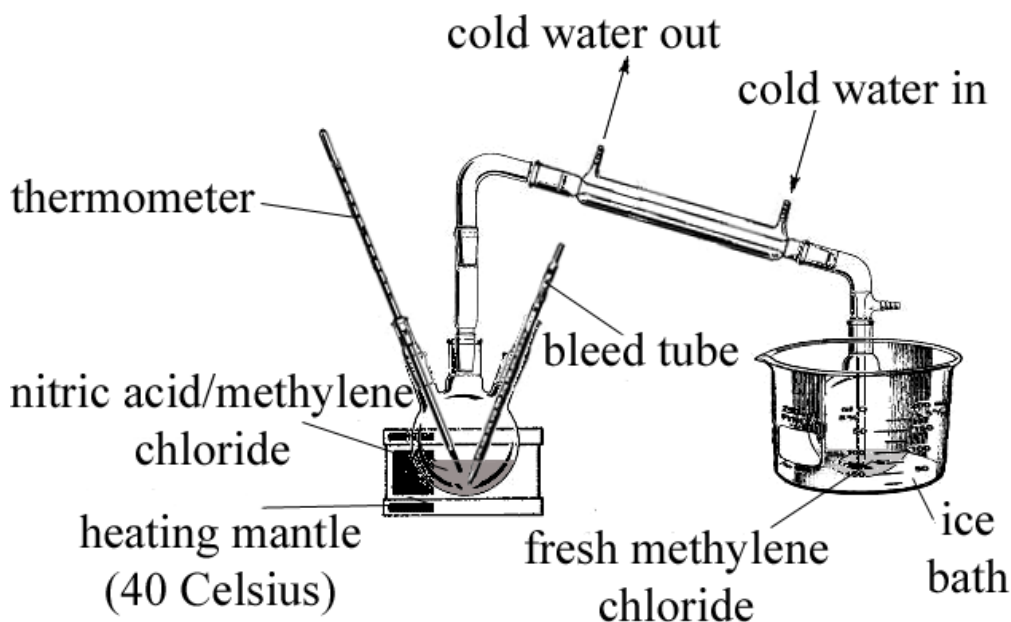
1. 94 milliliters (3.1 fluid oz.) of 98% sulfuric acid	3. 1400 milliliters of methylene chloride (3 pints)
2. 106 milliliters (3.6 fluid oz.) of 70% nitric acid	

Process summary: 99% Nitric acid can be prepared from 70% nitric acid or any other lower concentration of nitric acid by adding the nitric acid to excess sulfuric acid, and then extracting with methylene chloride. The upper methylene chloride layer is recovered, and then carefully distilled at 40 Celsius to remove the methylene chloride and recover the 99% nitric acid.

Why does this reaction happen? N/A

Hazards: Carryout the distillation with extreme caution. Do not heat the methylene chloride/nitric acid mixture above 40 Celsius, and use proper ventilation. *Nitrogen oxide gases will develop so be prepared. Carryout the distillation away from direct sun light.*

Procedure: Into an appropriate sized beaker place 94 milliliters (3.1 fluid oz.) of 98% sulfuric acid, and then add in 106 milliliters (3.6 fluid oz.) of 70% nitric acid. Then extract this acid mixture with seven 200-milliliter portions (seven 6.7 fluid oz. portions) of methylene chloride. Afterwards, combine all seven portions of methylene chloride (if not already done so). The result will be a 99% nitric acid solution in methylene chloride. This methylene chloride/nitric acid mixture can be used directly in general chemistry applications (if desired), or separated to recover pure 99% nitric acid. To separate the mixture, place the mixture into a distillation apparatus (as illustrated below), and then carefully distill at 40 Celsius until no more methylene chloride is collected in the receiver flask. *Note: Take caution when distilling and use proper ventilation because decomposition of the nitric acid might result forming reddish-brown fumes of nitrogen oxides. If the nitric acid begins to decompose during the distillation, don't worry and continue the distillation. After all the methylene chloride has been removed, the result might be a reddish-brown highly fuming liquid. This reddish-brown fuming liquid is suitable for use as 99% nitric acid.*



Apparatus for the distillation of methylene chloride to collect pure nitric acid. The bleed tube is inserted in order to keep pressure from reducing inside the reaction flask. The receiver flask should contain some fresh methylene chloride so as to form an air-tight seal by submerging the exit tube below the liquid surface. When the methylene chloride has been removed, the pure nitric acid will remain in the distillation flask.

Note: Any concentration of nitric acid can be used instead of just 70% nitric acid. When using nitric acid concentrations below 70% by weight, simply mix the dilute nitric acid with excess sulfuric acid. For example, mix 57 grams (2.01 oz.) of 98% sulfuric acid with 53 milliliters (1.8 fluid oz.) of 60% nitric acid, mix 67 grams (2.4 oz.) of 98% sulfuric acid with 53 milliliters (1.8 fluid oz.) of 50% nitric acid, mix 77 grams (2.7 oz.) of 98% sulfuric acid with 53 milliliters (1.8 fluid oz.) of 40% nitric acid, mix 87 grams (3.06 oz.) of sulfuric acid with 53 milliliters (1.8 fluid oz.) of 30% nitric acid, mix 97 grams (3.4 oz.) of 98% sulfuric acid with 53 milliliters (1.8 fluid oz.) of 20% nitric acid, or mix 107 grams (3.8 oz.) of 98% sulfuric acid with 53 milliliters (1.8 fluid oz.) of 10% nitric acid. After anyone of these mixings, extract the acid mixture with seven 100-milliliter portions (seven 3.4 fluid oz. portions) of methylene chloride. Thereafter, the result will be the same as in the above procedure. To isolate the 99% nitric acid, follow the directions in the above procedure.

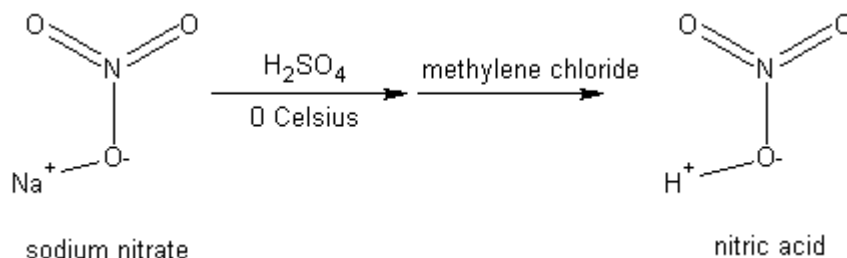
Method 4: Preparation of 99% nitric acid from potassium or sodium nitrate utilizing sulfuric acid and extraction with methylene chloride

(By-products from reaction: sodium or potassium sulfate)

Materials:

1. 194 grams (6.8 oz.) of 98% sulfuric acid	3. 260 milliliters (8.8 fluid oz.) of cold tap water
2. 100 grams (3.5 oz.) of potassium nitrate or 84 grams (3 oz.) of sodium nitrate	4. 1050 milliliters of methylene chloride (2.2 pints)

Reaction summary: 99% Nitric acid can also be obtained by treating potassium or sodium nitrate with sulfuric acid, adding water, and then extracting the acid mixture with methylene chloride to form a solution of 99% nitric acid in methylene chloride. The methylene chloride is then carefully distilled off to recover the pure 99% nitric acid.



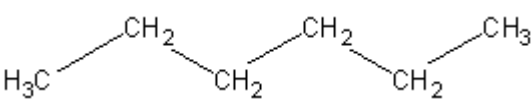
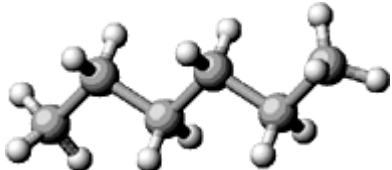
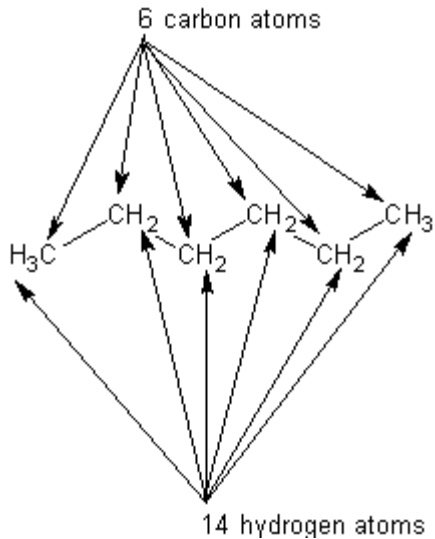
Why does this reaction happen? As in method 1, Sulfuric acid reacts with sodium nitrate forming nitric acid and sodium sulfate. The reaction mixture is then extracted with methylene chloride, whereby the nitric acid is taken up into the methylene chloride forming a nitric acid solution in methylene chloride. The mixture is then directly distilled to remove the methylene chloride and leave behind pure nitric acid.

Hazards: Take caution when distilling and use proper ventilation because decomposition of the nitric acid might take place forming reddish-brown fumes of nitrogen oxides. If the nitric acid begins to decompose during the distillation, don't worry and continue the distillation. After all the methylene chloride has been removed, the result might be a reddish-brown highly fuming liquid. This reddish-brown fuming liquid is suitable for use as 99% nitric acid. Carryout the distillation with extreme caution. Do not heat the methylene chloride/nitric acid mixture above 40 Celsius, and use proper ventilation. ***Nitrogen oxide gases will develop so be prepared. Carryout the distillation away from direct sun light.***

Procedure: Place 194 grams (6.8 oz.) of 98% sulfuric acid into a beaker, and then place the beaker in a ice bath and cool to 0 Celsius by means of an ice bath. When the sulfuric acid reaches a temperature of 0 Celsius, slowly add in portions, 100 grams (3.5 oz.) of potassium nitrate or 84 grams (3 oz.) of sodium nitrate over a period of 1 hour while stirring the 98% sulfuric acid and maintaining its temperature at 0 Celsius. After the addition of the potassium or sodium nitrate, slowly add over a period of one hour, 260 milliliters (8.8 fluid oz.) of cold tap water while continuously stirring the 98% sulfuric acid mixture and maintaining its temperature at 0 Celsius. Afterwards, remove the ice bath and then extract the acid mixture with seven 150-milliliter portions (seven 5.07 fluid oz. portions) of methylene chloride. Then combine all seven portions of methylene chloride (if not already done so), and then place the methylene chloride into the distillation apparatus as illustrated in method 3, and carefully distill at 40 Celsius until no more methylene chloride is collected in the receiver flask.

Procedure 20: The Preparation of Hexanes; the distillation of gasoline

Also known as: N/A

	 Mixed isomers
Chemical structure	3D Structure
	C_6H_{14}
Structure make-up	Condensed chemical structure

Hexanes

Hexanes are a colorless, very volatile liquids with a faint, peculiar odor. It is rarely sold as n-hexane but usually admixed with hexane isomers simply called “hexanes”, but marketed as “hexane”. Hexane has a boiling point of 69 Celsius, and a melting point of -100 Celsius. It is insoluble in water, but miscible with alcohol, chloroform, and ether. Hexane is a major component of gasoline, and can be distilled from the gasoline using a multiple-path distillation apparatus. Hexane is obtained commercially from petroleum, and is a widely available commercial chemical.

Method 1: Preparation of mixed hexanes by the distillation of gasoline

(By-products from reaction: complex mixture of hydrocarbons and gasoline additives)

Materials:

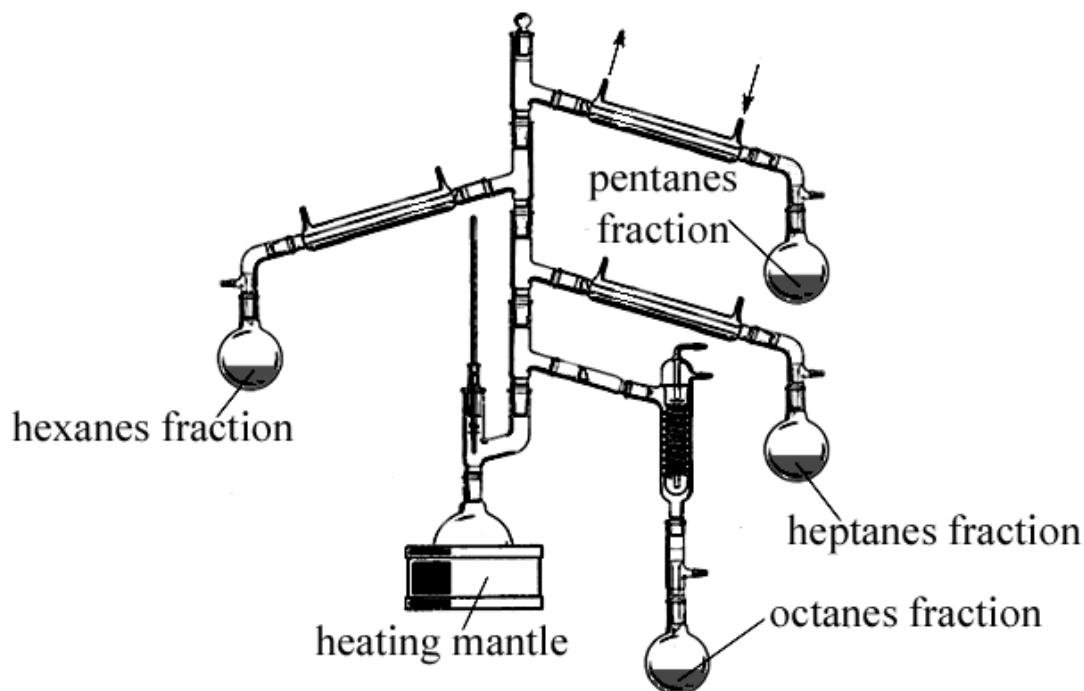
1. 1 to 4 liters (1 to 4.2 quarts) of unleaded premium gasoline	
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Process summary: Gasoline can be distilled to yield a variety of products, one of which is the desired hexanes. Hexanes are widely used as a solvent, and are a major component of gasoline.

Hazards: Gasoline is extremely flammable, so use caution.

Procedure: Mixed hexanes are prepared by distilling unleaded premium gasoline using a special multi-path distillation apparatus. To proceed, setup an apparatus as illustrated below. Then fill the distillation flask with 4 liters or less of unleaded premium gasoline, and then distill the mixture at 70 Celsius. This method of distillation takes advantage of the vapor densities of the various components of the gasoline. The vapors of the liquids with the lower densities will condense higher up in the

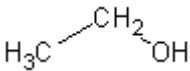

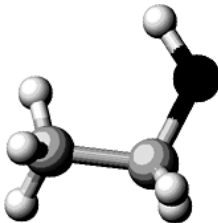
apparatus, and the vapors with the higher densities, such as octane condense lower down in the apparatus. The octane, and heptane fractions will distill over first, followed by the hexane fraction at the second highest condenser mark. The pentane fraction will condense at the highest mark, due to its density being less than the hexane, heptane, and octane fractions. Dissolved gases within the gasoline will carry over and will be vented through the top of the system, so the distillation should be carried to in a well ventilated area away from any source of ignition.



Distillation apparatus for the distillation of gasoline.

Procedure 21: The Preparation of Ethyl Alcohol; Ethanol

Also known as: Absolute alcohol; Grain alcohol; Ethyl hydrate

	 <p>3D structure 1</p>  <p>3D structure 2</p>
Chemical structure	3D Structure

	C_2H_6O
Structure make-up	Condensed chemical structure

95% Ethyl alcohol

95% Ethanol is a clear, colorless, very mobile, flammable liquid with a pleasant odor, and a pungent, burning taste. It has a boiling point of 78 Celsius and a melting point of -114 Celsius. 95% Ethanol slowly absorbs water from the air, and dilute ethanol solutions are slowly oxidized by air forming brown colored solutions containing mixtures of aldehydes, and carboxylic acids; mainly acetic acid. 95% Ethanol is miscible with water, and many organic solvents. 95% ethyl alcohol is called absolute ethanol because ethyl alcohol forms a binary azeotrope containing 95.57% ethyl alcohol by weight with a boiling point of 78 Celsius. Distillations cannot produce 99% ethanol because of this azeotrope. Ethyl alcohol is usually sold as denatured ethyl alcohol (mixed with small amounts of toxic chemicals to make non-drinkable) due to US government tax regulations. 95% Ethyl alcohol is toxic, and ingestion can cause alcohol poisoning. Dilute mixtures of ethyl alcohol (Vodka, Gin, Rum, Jack Daniels, beer, wine) produce intoxicating effects when ingested (these intoxicating effects can be increased if the dilute ethanol mixture is injected). 95% ethanol can be made by fermenting starch or sugars with yeast, followed by double distillation. 95% Ethanol is manufactured on an industrial scale by the petroleum industry from ethylene gas, sulfuric acid, and water. 95% Ethanol is a widely available commercial chemical sold under a variety of names. 95% ethanol can be obtained from double distillation of alcoholic beverages such as vodka, gin, or rum.

99% Ethyl alcohol

99% Ethyl alcohol is a colorless, very mobile and flammable liquid with a pleasant odor. Pure ethanol is tasteless. It rapidly absorbs water from the air, from which it forms an azeotrope of 95% ethyl alcohol. It is miscible with water, alcohol, ether, and many common organic solvents. 99% ethanol is toxic, and ingestion can cause poisoning. It is prepared by reacting ethylene gas with sulfuric acid, followed by distillation in the presence of minute amounts of water. It can also be made by double distillation of fermented cocktails, followed by treatment with metallic sodium to remove the water of azeotrope. 99% Ethyl alcohol can be obtained by salting out vodka (see 91% isopropyl alcohol), treating the recovered upper ethyl alcohol layer with large amounts of anhydrous magnesium sulfate, filtering, and then distilling the mixture to recover 99% ethyl alcohol.

Method 1: Preparation of 95% ethyl alcohol from liquor, wine, or beer

(By-products from reaction: none)

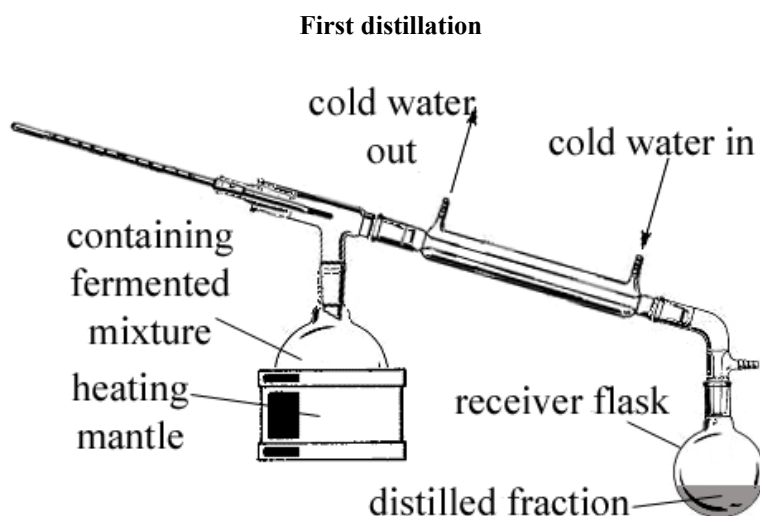
Materials:

1. 1 liter or more (1.86 quarts or more) of 60 to 180 proof liquors (any kind will work, but you should use the cheap stuff to save money)	3. or 5 liters or more (1.3 gallons or more) of any beer, ail, brew, or malt liquor
2. or 2.5 liters or more (2.64 quarts or more) of wine (any will work) with an alcohol concentration of at least 7%	4. 50 grams (1.7 oz.) of baking soda

Process summary: 95% ethanol can be prepared by double distilling liquors, wines, or beers. After the first distillation, the crude ethyl alcohol (which will contain up to 90%+ ethyl alcohol), is treated with baking soda to remove odors, filtered, and then fractionally re-distilled producing 95% ethanol.

Procedure: If using liquors, place 1 liter or more (1.86 quarts or more) of 60 to 180 proof liquors (any kind will work, but you should use the cheap stuff to save money), into a clean distillation apparatus or similar as illustrated below. If using wines, place 2.5 liters or more (2.64 quarts or more) of wine (any will work) with an alcohol concentration of at least 7%, into a distillation apparatus (similar to the one illustrated below). If using beer, place, 5 liters or more (1.3 gallons or more) of any beer, ail, brew, or malt liquor into a distillation apparatus or similar device as illustrated below. Then, despite which alcoholic

beverage you use, distill your alcoholic beverage or concoction at 90 Celsius until no more liquid passes into the receiver flask (will take 4 to 5 hours for the liquors, 4 to 8 hours for the wines, and 6 to 10 hours for the beers). When no more liquid passes into the receiver flask, remove the heat source, and then remove the receiver flask from the distillation apparatus. Then place 50 grams (1.7 oz.) of baking soda into the receiver flask, and then boil the contents to 100 Celsius for about 5 minutes. Thereafter, remove the heat source, and allow the contents to cool to room temperature. Afterwards, filter the mixture to remove the insoluble baking soda, and then place this filtered mixture into a clean fractional distillation apparatus. Then distill at 80 Celsius until no more liquid passes into the receiver flask.



Simple distillation apparatus for the distillation of liquors, wines, or beers to yield 90%+ ethyl alcohol.

Final note for method 1: None

Method 2A: Preparation of 95% ethyl alcohol from table sugar or common food grade starch (corn starch)

(By-products from reaction: too complex for discussion here)

Materials:

1. 1 kilogram (2.2 pounds) of table sugar	7. or 2 liters (2.1 quarts) of hot tap water
2. 3 liters (3.2 quarts) of tap water	8. or 10 drops of concentrated hydrochloric acid (muriatic acid will work).
3. 5 drops of concentrated hydrochloric acid (muriatic acid will work) or 5 drops of concentrated sulfuric acid (plumbers grade will work).	9. or 1 small spoon full of baking soda
4. 115 grams (4 oz.) of baking soda	10. or 5 to 10 grams (just under ½ oz.) of regular yeast (brewers yeast—not the supplement, but the pure enzyme, which can be purchased from wine making supply companies)
5. 5 to 10 grams (just under ½ oz.) of regular yeast (brewers yeast—not the supplement, but the pure enzyme, which can be purchased from wine making supply companies)	11. 50 grams (1.8 oz.) of baking soda
6. or 500 grams (17.6 oz.) of regular store bought food starch not the "starch" used for clothing	12. Charcoal (store bought charcoal will work, but only ordinary charcoal—nothing added, i.e., no quick light stuff, ect., ect.,

Reaction summary: 95% Ethyl alcohol can be obtained on a lower yield by hydrolyzing table sugar with dilute acid, then fermenting the resulting mixture with yeast to form an ethyl alcohol solution. This ethyl alcohol is then distilled to obtain a crude ethyl alcohol product. This crude product will be contaminated heavily with by-products so multiple distillations and

treatments with baking soda will be needed in order to fulfill proper purification. Baking soda is mixed with the distilled liquid to absorb odors and the like. 95% Ethyl alcohol can also be obtained by the fermentation of glucose obtained from starch. In this case regular food starch is hydrolyzed with acid to form a glucose solution, which is then fermented in the usual manner. The fermented mixture is then double distilled under the usual techniques.

Why does this reaction happen? Enzymes have a particular attraction to substances like carbohydrates. When carbohydrates such as glucose are subjected to enzymes in the presence of water, the enzymes catalytically break down the glucose molecules forming ethyl alcohol and carbon dioxide.

Procedure:

If using table sugar:

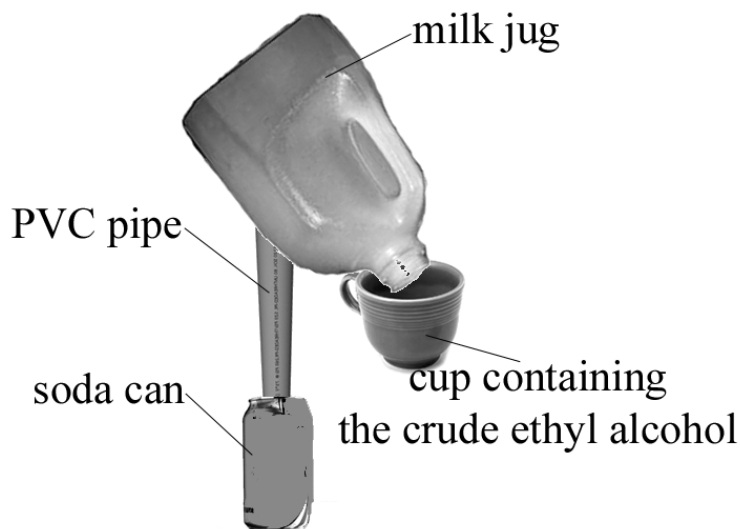
Dissolve 1 kilogram (2.2 pounds) of table sugar into 3 liters (3.2 quarts) of tap water. Then rapidly stir this sugar mixture to dissolve all solids, and thereafter, heat this sugar solution (using a hot plate, stovetop, Bunsen burner, ect.,) to 80 Celsius. When the sugar solution reaches about 80 Celsius, stir the sugar solution and add in 5 drops of concentrated hydrochloric acid or 5 drops of concentrated sulfuric acid (hydrochloric acid works best), and then continue heating and stirring the sugar solution for thirty minutes (at the end of 15 to 30 minutes, the sugar solution should have a slight grape sugar like odor). After thirty minutes, remove the heat source, and allow the sugar mixture to cool to room temperature. Then add in 5 grams (2/10th of an oz.) of baking soda to neutralize the acid. Afterwards, pour the sugar solution into an empty bottle (such as a clean empty plastic milk jug), and then add in 5 to 10 grams (just under ½ oz.) of regular yeast (brewers yeast—not the supplement, but the pure enzyme, which can be purchased from wine making supply companies). Note: the yeast must be finely ground into a small amount of warm water before adding to the sugar solution. After adding the yeast slurry, stir the mixture for several minutes to insure good dispersion of the yeast. Then plug the bottles opening with cotton or similar materials (but not air-tight), and then place the bottle into a cool place away from light. Then allow the sugar mixture to ferment for about 2 to 3 weeks. After about 2 to 3 weeks, remove the cotton or similar material from the bottles opening, and then pour the contents of the bottle into a distillation apparatus (similar to the one illustrated in method 1). Then distill at 100 Celsius for 3 to 4 ½ hours. After which, remove the heat source, and then recover the crude ethyl alcohol mixture from the receiver flask. Then pour this crude ethyl alcohol mixture into a clean beaker or other container, and then add in 50 grams (1.8 oz.) of baking soda. Thereafter, boil this mixture for about 5 minutes, and thereafter, remove the heat source, and allow the mixture to cool to room temperature. Afterwards, filter the mixture to remove any insoluble baking soda, and then place the filtered mixture into a regular clean distillation apparatus, and distill at 80 Celsius until no more liquid passes into the receiver flask (takes about 3 to 4 hours). When no more liquid passes into the receiver flask, remove the heat source, and then recover the crude ethyl alcohol from the receiver flask just like before. Finally, place this crude ethyl alcohol mixture into a clean fractional distillation apparatus followed by 50 grams (1.8 oz.) of baking soda, and then distill the mixture at 80 Celsius until no more ethyl alcohol passes into the receiver flask (takes about 3 to 4 hours). Once the ethyl alcohol has distilled over for this final time, recover the ethyl alcohol from the receiver flask, and then filter it through a filter (gravity filtration) containing a layer of charcoal. Note: simply place ordinary charcoal briquettes (crush them up into pieces if necessary) into the bottom of the filter paper (store bought charcoal will work, but only ordinary charcoal—nothing added, i.e., no quick light stuff, ect., ect.). Pass the ethyl alcohol through this filter several times to purify it—the charcoal will help remove impurities, odors, and the like. After the charcoal filtration process, your 95% ethyl alcohol is ready for use. Note: if desired, this alcohol can be used to prepare ether, or diluted to 100 to 80 proof for consumption!

If using starch

Into a large beaker or other container, place 500 grams (17.6 oz.) of regular store bought food starch (not the "starch" used for clothing). Thereafter, add in 2 liters (2.1 quarts) of hot tap water, and then add in 10 drops of concentrated hydrochloric acid. Thereafter, heat this starch suspension for about 2 hours at the boiling temperature of water. During the heating process, stir the mixture as much as possible. Keep heating the mixture until the bulk of the starch dissolves. Note: The purpose for the acid is to hydrolyze the starch into glucose. During the heating process, the insoluble starch suspension will slowly disappear to give a clear solution; this is because of the starch being broken down into glucose, which is very soluble in water. When most of the insoluble starch has been dissolved, remove the heat source, and allow the resulting solution to cool to room temperature. When the solution cools, add in 1 small spoon full of baking soda (to neutralize the acid), and then pour the entire mixture into a clean empty plastic milk jug. At this point, you should now have a plastic milk jug filled with just over 2 liters of glucose solution, containing a small amount of baking soda. Now, pour in 5 to 10 grams (just under ½ oz.) of regular yeast (brewers yeast—not the supplement, but the pure enzyme, which can be purchased from wine making supply companies). Note: the yeast needs to be finely ground in a small amount of warm water to form a slurry. Thereafter, when the yeast has been added, don't shake the container; just allow the yeast to settle on its own. After adding in the yeast, plug the top of the plastic milk container with a cloth, cotton, or any thing that is not airtight, but will keep dust out. Thereafter, place your milk jug into a cool dark place, and let it ferment for about 2 to 3 weeks. After 2 to 3 weeks, your home brew is finished. In some cases, it may take

as long as 4 weeks to complete the fermentation. Note: During the fermentation process, the yeast catalytically reacts with the glucose forming ethyl alcohol, and carbon dioxide. The carbon dioxide bubbles to the surface, and the alcohol remains in solution. Once the fermentation process is complete, it's now time for the hard part—the mixture has to be distilled. To do this, assemble an apparatus similar to the one in method 1, and then distill at 100 Celsius for 4 ½ hours. After which, remove the heat source, and then recover the crude ethyl alcohol mixture from the receiver flask. Then pour this crude ethyl alcohol mixture into a clean beaker or other container, and then add in 50 grams (1.8 oz.) of baking soda. Thereafter, boil this mixture for about 5 minutes, and thereafter, remove the heat source, and allow the mixture to cool to room temperature. Afterwards, filter the mixture to remove any insoluble baking soda, and then place the filtered mixture into a clean fractional distillation apparatus, and distill at 80 Celsius until no more liquid passes into the receiver flask (takes about 3 to 4 hours). When no more liquid passes into the receiver flask, remove the heat source, and then recover the ethyl alcohol from the receiver flask. Then, filter it through a filter (gravity filtration) containing a layer of charcoal. Note: simply place ordinary charcoal briquettes (crush them up into pieces if necessary) into the bottom of the filter paper (store bought charcoal will work, but only ordinary charcoal—nothing added, i.e., no quick light stuff, ect., ect.). Pass the ethyl alcohol through this filter several times to purify it—the charcoal will help remove impurities, odors, and the like. After the charcoal filtration process, your 95% ethyl alcohol is ready for use. Note: if desired, this alcohol can be used to prepare ether, or diluted to 100 or 80 proof for consumption!

NOTE: If you don't have access to laboratory glassware (which most people don't), you will need to use your imagination and come up with a homemade "still" of your own. Use your creativity to design a cheap home made "still". One method is screwing a PVC pipe of appropriate size into the opening of a clean soda can, and then inserting the other end of the PVC pipe into an empty plastic container (such as an empty milk jug). Note: Do not drink the distilled alcohol fraction as is; its 90% alcohol and ingestion can cause poisoning, and it should be diluted before consumption.



Crude setup for the distillation of ethyl alcohol. In the above illustration, the fermented mixture is placed into the soda can and a PVC pipe is then inserted into the mouth opening. Plumbers putty or hot glue can be used to seal the PVC pipe to the soda can. A milk jug is then attached to the top of the PVC pipe, Plumbers putty or hot glue can be used to secure the PVC pipe to the milk jug. As heat is applied to the soda can, the ethyl alcohol will boil-off, and its vapors will condense on the roof of the milk jug. The condensate will then drip down into the cup, where the alcohol fraction can be obtained. Instead of using a fermented mixture, cooking wine can be used as well, but should be diluted before consumption.

Final note for method 2: None

Method 2B: Preparation of 95% ethyl alcohol from table sugar, brown sugar, molasses, or juice (improved process)

(By-products from reaction: to complex for discussion here)

Materials:

1. 1 kilogram of sugar (2.2 pounds) or 1 kilogram (2.2 pounds) of brown sugar	5. 2 to 4 liters (2.1 to 4.2 quart) container of grape juice, apple juice, or apricot juice
2. 3 liters (3.1 quarts) of warm tap water	6. 1 kilogram (2.2 pounds) of molasses
3. 5 to 10 grams (just under ½ oz.) of regular yeast (brewers	7. 3 liters (3.1 quarts) of warm water

yeast—not the supplement, but the pure enzyme, which can be purchased from wine making supply companies)	
4. 200 grams (7 oz.) of pickling salt	

Reaction summary: Ethyl alcohol can be prepared by fermenting sugar, brown sugar, juice, or molasses using yeast in a similar manner as in method 2A. In this improved process, the yeast is thoroughly ground into a small amount of warm water prior to mixing with the sugar, brown sugar, juice, or molasses mixture. The fermentation is rapid and only takes about 2 weeks under the normal conditions. After the fermentation process, in either case, the fermented mixture is distilled to recover a crude alcohol product. This crude alcohol product is then salted out by mixing it thoroughly with pickling salt to form a two-phase mixture. This two-phase mixture is then separated, and the alcohol fraction is then re-distilled to recover a refined alcohol fraction.

Why does this reaction happen? Enzymes have a particular attraction to substances like carbohydrates. When carbohydrates such as glucose are subjected to enzymes in the presence of water, the enzymes catalytically break down the glucose molecules forming ethyl alcohol and carbon dioxide.

Hazards: None.

Procedure: 95% Ethyl alcohol can be prepared by fermenting sugar, brown sugar, molasses, or juice using yeast in a similar manner to method 2A. However, in the case of sugar, instead of hydrolyzing the sugar, it is fermented directly using brewers yeast used for wine making. Now, if using sugar or brown sugar, dissolve 1 kilogram of sugar (2.2 pounds) or 1 kilogram (2.2 pounds) of brown sugar into 3 liters (3.1 quarts) of warm tap water contained in a washed out empty milk gallon jug, and then prepare a yeast base mixture by doing the following: 1. take 1 packet brewers yeast—7 grams or 1/4th of an ounce, and place it into 1/4th of a cup of warm water, and then using a spoon or similar utensil, grind and thoroughly mix the yeast into the water until a uniform mixture results, which is free from clumps, chunks, or the like. Thereafter, pour the yeast mixture into the sugar solution, and then place a paper towel or cloth over the mouth of the gallon jug, and then allow the mixture to ferment for about 2 weeks at room temperature. Note: once the yeast gets going, in about an hour or so the evolution of carbon dioxide will become vigorous. The total amount of time for the fermentation process ranges from about 14 to 16 days. When the evolution of carbon dioxide gas bubbles slows down and drops-off to a trickle, the fermentation process is relatively complete. After the fermentation process, the mixture needs to be distilled using a similar apparatus as in method 1. The distillation process will take about 5 to 6 hours for completion. After the distillation process, the crude alcohol mixture should then be “Salted out” using the following process: 1. Place the crude alcohol mixture into a separatory funnel (see extraction), and then add in 200 grams (7 oz.) of pickling salt, and then shake the mixture vigorously for about 5 to 10 minutes. Thereafter, allow the mixture to settle and stand for about 15 minutes, whereupon a two-phase mixture will develop. The upper layer will be the alcohol layer, and the water layer containing dissolved pickling salt and other impurities, will be the lower layer. Now, simply drain-off the lower water layer, and discard, or you can keep it to recover the dissolved salt, and then drain-off or pour out the upper alcohol layer. Then place this alcohol layer into a clean distillation apparatus of the usual design, as illustrated in method 1, and then distill the alcohol at 78 Celsius. The resulting alcohol may not be exactly 95%, but is well suitable for use in applications as a solvent.

If using molasses, or juice, simply prepare a yeast mixture by taking 1 packet of brewers yeast—7 grams or 1/4th of an ounce, and place it into 1/4th of a cup of warm water, and then using a spoon or similar utensil, grind and thoroughly mix the yeast into the water until a uniform mixture results, which is free from clumps, chunks, or the like, and then pour this yeast mixture directly into a large (2 to 4 liter, 2.1 to 4.2 quart) container of grape juice, apple juice, apricot juice, or a molasses solution, and then allow the mixture to ferment for about 2 weeks. As in the above process, once the yeast gets going, in about an hour or so the evolution of carbon dioxide will become vigorous. The total amount of time for the fermentation process ranges from about 14 to 17 days. When the evolution of carbon dioxide gas bubbles slows down and drops-off to a trickle, the fermentation process is relatively complete. Note: if using juice, pour out some of juice before adding the yeast to create a gap or space between the juice and the mouth of the container to prevent foaming and frothing from escaping the bottle once the yeast kicks in. Second not: If using molasses, dissolve 1 large container of molasses (about 1 kilogram, 2.2 pounds), into 3 liters (3.1 quarts) of warm water contained in a clean empty milk jug or equivalent. If using molasses, you will want to carryout the procedure in a well-ventilated area as the odor of molasses will be heavy. After the fermentation process, the fermented juice or molasses mixture needs to be distilled using a similar apparatus as in method 1. Then, place the crude ethyl alcohol mixture into a clean fractional distillation apparatus and then distill at 80 Celsius. The distillation process will take about 5 to 6 hours for completion. After the distillation process, the purified alcohol will be exactly 95%, and is well suitable for use in applications as a solvent, making ether, or for consumption (dilute to 100 or 80 proof for consumption!).

Method 3: Preparation of 99% ethyl alcohol from 95% ethyl alcohol and metallic sodium

(By-products from reaction: sodium hydroxide and a small amount of sodium ethoxide)

Materials:

1. 500 milliliters (17 fluid oz.) of 95% ethyl alcohol	2. 40 grams (1.4 oz.) of metallic sodium
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Reaction summary: 99% ethyl alcohol is obtained by treating 95% ethyl alcohol with metallic sodium. The metallic sodium reacts with the water in the binary azeotrope, freeing the alcohol. Once the water has been neutralized (forming sodium hydroxide and sodium ethoxide), the mixture is distilled to recover the anhydrous ethyl alcohol.

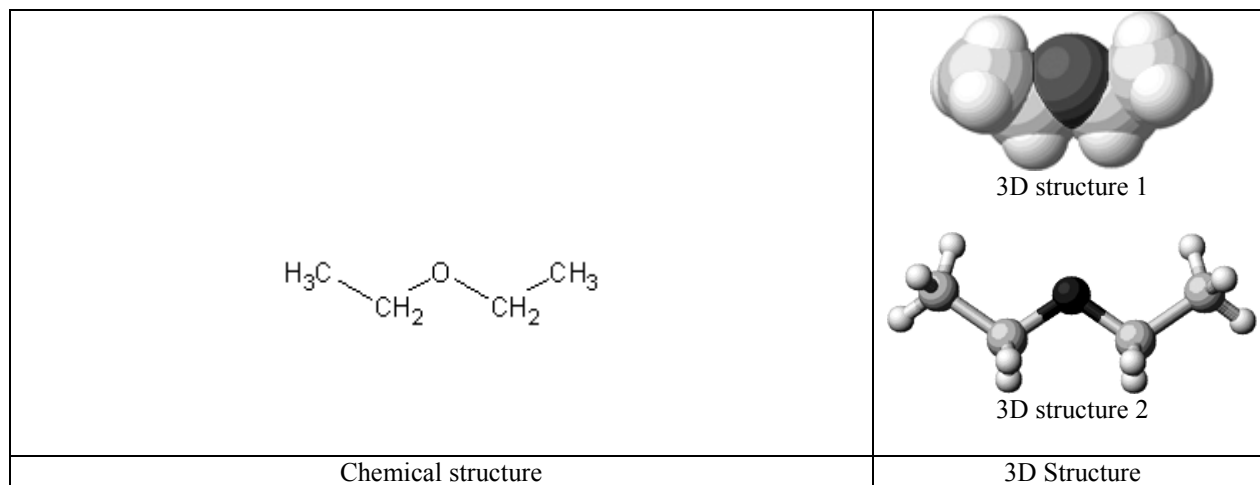
Why does this reaction happen? In order to obtain pure ethyl alcohol, concentrated ethyl alcohol, known as absolute alcohol, or 95% ethyl alcohol, must be reacted with a compound that removes water completely. In order to do this, we mix metallic sodium with the 95% ethyl alcohol. The metallic sodium reacts with the water present, thereby removing it from the system. Metallic sodium has a great affinity for water, reacting with it to produce sodium hydroxide and hydrogen gas.

Hazards: Wear gloves when handling metallic sodium, and avoid skin contact and ingestion. Metallic sodium reacts violently with water and other chemicals, so use caution.

Procedure: Into a suitable beaker or container, place 500 milliliters (17 fluid oz.) of 95% ethyl alcohol, and then place the beaker or container into an ice bath, and chill to about 5 to 10 Celsius. Thereafter, slowly add in, in very small portions, 40 grams (1.4 oz.) of metallic sodium over a period of about 1 hour. After the addition of the metallic sodium, stir the entire alcohol mixture for about 30 minutes, and then place it into a standard distillation apparatus, and distill the ethyl alcohol at 80 Celsius. Note: your distillation apparatus should be completely dry before proceeding. When no more ethyl alcohol passes into the receiver flask, remove the heat source, and then recover the ethyl alcohol from the receiver flask. The resulting alcohol will be about 99% pure, and should be stored in a suitable container protected from moisture. Note: never consume 99% ethyl alcohol as death may occur. 99% ethyl alcohol is never used to make alcoholic beverages and is primarily used as a solvent in organic reactions. Note: 99% ethyl alcohol is a controlled substance according to the US government.

Procedure 22: The Preparation of Diethyl Ether

Also known as: Ether, Ethyl oxide, Anesthetic, Ethoxyethane



	$C_4H_{10}O$
Structure make-up	Condensed chemical structure

Diethyl Ether, other wise known as just ether, is a mobile, very volatile, highly flammable liquid, which produces explosive vapors. It has a sweetish, pungent odor, and a burning taste. Ether forms explosive peroxides when exposed to air—ether containing peroxides will detonate if heated, shattering the glass vessel. Before heating mixtures containing ether, the peroxide test should be conducted. To test for peroxides, add five drops of ferrous chloride solution to the ether mixture. If a red or black color appears, peroxides are present. *Note: This test will not work properly if there are oxidizing agents in the ether mixture.* Ether can be stabilized by the addition of small amounts of naphthols, but this does not protect ether 100% from peroxide formation. Ether has a melting point of -116 Celsius, and a boiling point of 35 Celsius. Ether and air mixtures are explosive, so extinguish all flames and do not smoke when handling it. Protect ether from static electricity, which can also cause fire. Ether is insoluble in water, but miscible with alcohol, benzene, chloroform, and many oils. Do not mix 99% nitric acid with ether, as detonation will take place. Inhalation of ether vapors can produce intoxicating effects. These effects include feelings of euphoria, well-being, relaxation, and a general state of high. These effects can also lead to feelings of drunkenness. Ether is a narcotic in high concentrations, but is not habit forming. Store ether in tightly sealed bottles in a cool place (preferably in a refrigerator). For prolonged storage, store ether over sodium sulfite and keep in a bottle filled to the top (to minimize the air space). Ether can be prepared by heating 95% ethanol and 98% sulfuric acid (1 to 1 ratio) to 100 Celsius, and simultaneously condensing the distilled-off vapors of ether. The ether is then purified by re-distillation. Ether is a widely available commercial chemical, but its status as an anesthetic may limit its sale to the general public.

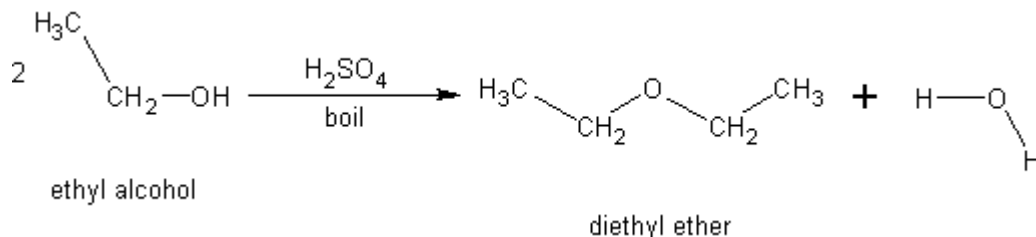
Method 1: Preparation of diethyl ether from vodka and concentrated sulfuric acid

(By-products from reaction: none)

Materials:

1. 1250 milliliters (42.2 fluid oz.) of store bought vodka, usually 80 proof (use the cheap stuff, i.e., Popov, Kirov, Skoll, ect.,)	2. 900 milliliters (30.4 fluid oz.) of concentrated sulfuric acid (available at several places, but is readily available at janitorial and plumbing supply stores, where it sold as 93% to 98% purity. The 93% acid is 98% concentrated acid, but with iron, and can be used with satisfaction).
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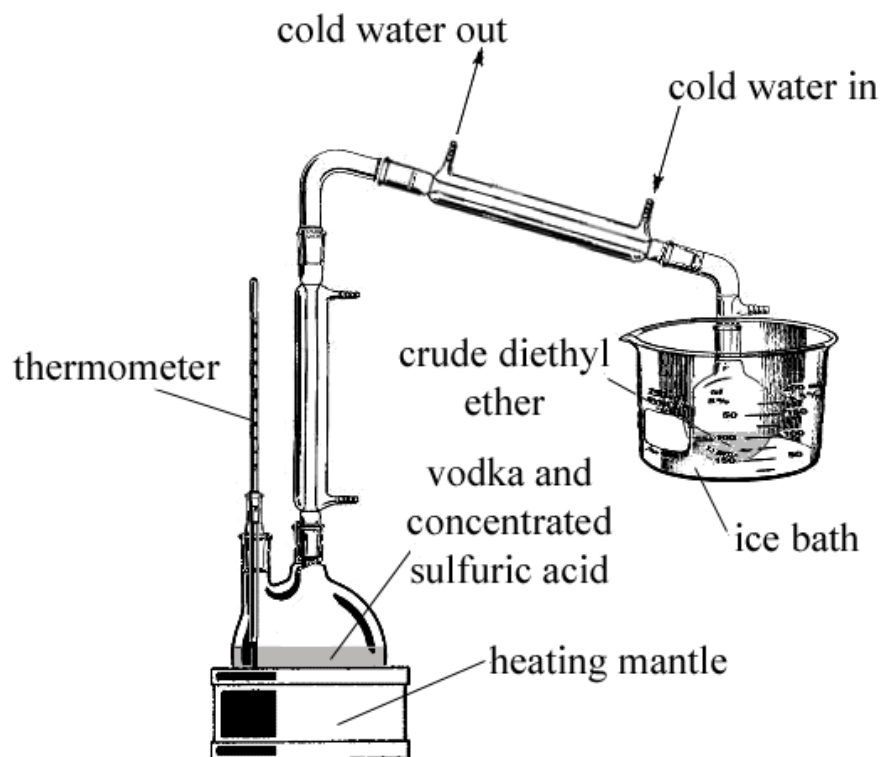
Reaction summary: Diethyl ether can be conveniently prepared by distilling a mixture of vodka with sulfuric acid. The reaction takes appreciable amounts of time, ranging from how much vodka you use to begin with, but produces a steady stream of diethyl ether, which distills over during the reaction. After the initial reaction, the recovered diethyl ether is re-distilled for quality and purity.



Why does this reaction happen? Sulfuric acid has a very strong affinity for water, and therefore it can extract it from many substances. Sulfuric acid can char wood and many organic substances. In this procedure, we use sulfuric acid as the catalyst for extracting a molecule of water from two molecules of ethyl alcohol. Because two molecules of ethyl alcohol give rise to one free molecule of water, both the ethyl alcohol molecules bond together forming the ether.

Hazards: Wear gloves when handling concentrated sulfuric acid, and avoid skin contact, contact with clothing, and ingestion at all cost. Concentrated sulfuric acid will corrode, char, and dissolve many substances including some plastics.

Procedure: Into a suitable distillation apparatus, place 1250 milliliters (42.2 fluid oz.) of store bought vodka (use the cheap stuff, i.e., Popov, Kirov, Skoll, ect.), followed by slowly and carefully adding in 900 milliliters (30.4 fluid oz.) of concentrated sulfuric acid. The concentrated sulfuric acid can be of any brand, and you may use plumbers brand concentrated sulfuric acid with satisfactory results. Note: The addition of the concentrated sulfuric acid to the vodka generates excessive heat, so use caution. After adding in all the concentrated sulfuric acid, boil the mixture at 100 Celsius for about 6 to 8 hours, and allow the mixture to distill over during this time. After 6 to 8 hours, remove the heat source, and allow the sulfuric acid mixture in the distillation flask or pot to cool to room temperature. The diethyl ether can then be recovered from the receiver flask, and then re-distilled using a fractional distillation apparatus, and distilled at 40 Celsius. The result after re-distillation will be around 500 milliliters of pure ether. Note: the sulfuric acid mixture, which may be colored blood red if iron was present in the acid (plumbers grade, 93% sulfuric acid), can be boiled at 100 Celsius in a suitable glass container to boil-off some of the water to concentrate the sulfuric acid. The point is, the sulfuric acid can be recycled over and over again (even if it is colored blood red or contaminated with other impurities), as it is only a catalyst in the reaction, and is not consumed or chemically changed in any way. Note: when boiling the sulfuric acid mixture, white fumes begin to form, remove the heat source, and allow the now concentrated sulfuric acid to cool to room temperature—at this point the sulfuric is recycled and can be used for another run of ether formation.



This apparatus can be replaced with any standard distillation apparatus, or standard fractional distillation apparatus; Alcohol stills can also be used, but may undergo corrosion in the distillation pot due to the presence of sulfuric acid. The diethyl ether should be re-distilled using a standard fractional distillation apparatus, or equivalent. After re-distillation, the ether should be stored in a proper container such as a metal can, or glass container, sealed air-tight, and filled to the top (to minimize air gaps), and these containers should be stored in a refrigerator until use. Note: the heating mantle portrayed in the illustration can be replaced by a Bunsen burner, hot plate, stovetop, steam bath, or oil bath if desired.

Final note for method 1: None

Method 2: Preparation of diethyl ether from vodka and battery acid

(By-products from reaction: none)

Materials:

1. 1250 milliliters (42.2 fluid oz.) of store bought vodka, usually 80 proof (use the cheap stuff, i.e., Popov, Kirov, Skoll, ect.,)	2. 1500 milliliters (50.7 fluid oz.) of fresh battery acid
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Reaction summary: Diethyl ether can be conveniently prepared by distilling a mixture of vodka with sulfuric acid. The reaction takes appreciable amounts of time, ranging from how much vodka you use to begin with, but produces a steady stream of diethyl ether, which distills over during the reaction. After the initial reaction, the recovered diethyl ether is re-distilled for quality and purity.

Why does this reaction happen? Same as in method 1.

Hazards: Wear gloves when handling battery acid, and avoid skin contact, contact with clothing, and ingestion. Battery acid contains about 25% sulfuric acid by weight, and is very corrosive.

Procedure: First, into a suitable sized thick walled Pyrex low form beaker, place 1500 milliliters (50.7 fluid oz.) of battery acid, and then boil the mixture by heating it to 150 Celsius until white fumes start to evolve. When white fumes start to evolve, turn off the heat source and allow the now concentrated sulfuric acid to cool to room temperature. Once it has, place it into a distillation apparatus as in method 1, and then add in all at once 1250 milliliters (42.2 fluid oz.) of vodka, and then distill the mixture at 100 Celsius for about 6 to 8 hours. After 6 to 8 hours, remove the heat source, and then recover the diethyl ether from the receiver flask or receiver container. Then re-distill this diethyl ether using a standard fractional distillation apparatus or equivalent and distill at 40 Celsius. Note: the sulfuric acid mixture, which may be colored blood red if iron was present in the acid (plumbers grade, 93% sulfuric acid), can be boiled at 100 Celsius in a suitable glass container to boil-off some of the water to concentrate the sulfuric acid. The point is, the sulfuric acid can be recycled over and over again (even if it is colored blood red or contaminated with other impurities), as it is only a catalyst in the reaction, and is not consumed or chemically changed in any way. Note: when boiling the sulfuric acid mixture, white fumes begin to form, remove the heat source, and allow the now concentrated sulfuric acid to cool to room temperature—at this point the sulfuric is recycled and can be used for another run of ether formation.

Final note for method 2: None

Method 3: Preparation of diethyl ether from distilled ethyl alcohol (95% alcohol; ABS alcohol; ever clear), with concentrated sulfuric acid or battery acid

(By-products from reaction: none)

Materials:

1. 500 milliliters (17 fluid oz.) of 95% ethyl alcohol; ABS alcohol; ever clear	3. or 500 milliliters (17 fluid oz.) of distilled ethyl alcohol (95% ethyl alcohol; ABS alcohol; ever clear
2. 250 milliliters (8.4 fluid oz.) of concentrated sulfuric acid	4. 1500 milliliters (50.7 fluid oz.) of battery acid

Reaction summary: Diethyl ether can be prepared by reacting 95% ethyl alcohol with concentrated sulfuric acid, or battery acid. The 95% ethyl alcohol is commonly called ABS alcohol, or ever clear. The alcohol is simply mixed with concentrated sulfuric acid, and then distilled in the usual manner. The diethyl ether that forms, slowly distills over in the usual manner.

Why does this reaction happen? Same as in methods 1 or 2.

Hazards: Wear gloves when handling concentrated sulfuric acid, and avoid skin contact, contact with clothing, and ingestion at all cost. Concentrated sulfuric acid will corrode, char, and dissolve many substances including some plastics. Wear gloves when handling battery acid, and avoid skin contact, contact with clothing, and ingestion. Battery acid contains about 25% sulfuric acid by weight, and is very corrosive.

Procedure: If using concentrated sulfuric acid, place 500 milliliters (17 fluid oz.) of distilled ethyl alcohol (95% ethyl alcohol; ABS alcohol; ever clear), into a standard distillation apparatus, as illustrated in method 1 or similar, and then slowly and carefully add in 250 milliliters (8.4 fluid oz.) of concentrated sulfuric acid (plumbers grade works great). Thereafter, boil this

mixture at 100 Celsius for about 6 to 8 hours, and allow the mixture to distill during this time. After 6 to 8 hours, remove the heat source, and then recover the diethyl ether from the receiver flask or receiver container. Then re-distill this diethyl ether using a standard fractional distillation apparatus or equivalent in the same manner as in method 1 (distill at 40 Celsius). Note: the sulfuric acid mixture left over, can be recycled, but it should be boiled to drive-off some of the water.

If using battery acid, place into a suitable sized thick walled Pyrex low form beaker, 1500 milliliters (50.7 fluid oz.) of battery acid, and then boil the mixture by heating it to 150 Celsius until white fumes start to evolve. When white fumes start to evolve, turn off the heat source and allow the now concentrated sulfuric acid to cool to room temperature. Once it has, place it into a distillation apparatus as in method 1, and then add in slowly, 1500 milliliters (17 fluid oz.) of distilled ethyl alcohol (95% ethyl alcohol; ABS alcohol; ever clear), as illustrated in method 1 or similar. Thereafter, boil this mixture at 100 Celsius for about 6 to 8 hours. After 6 to 8 hours, remove the heat source, and then recover the diethyl ether from the receiver flask or receiver container. Then re-distill this diethyl ether using a standard fractional distillation apparatus or equivalent and distill at 40 Celsius. Note: the sulfuric acid mixture, which may be colored blood red if iron was present in the acid (plumbers grade, 93% sulfuric acid), can be boiled at 100 Celsius in a suitable glass container to boil-off some of the water to concentrate the sulfuric acid. The point is, the sulfuric acid can be recycled over and over again (even if it is colored blood red or contaminated with other impurities), as it is only a catalyst in the reaction, and is not consumed or chemically changed in any way. Note: when boiling the sulfuric acid mixture, white fumes begin to form, remove the heat source, and allow the now concentrated sulfuric acid to cool to room temperature—at this point the sulfuric is recycled and can be used for another run of ether formation.

Final note for method 3: None

Method 4: Preparation of diethyl ether from denatured alcohol with concentrated sulfuric acid or battery acid

(By-products from reaction: none)

Materials:

1. 550 milliliters (18.5 fluid oz.) of denatured alcohol—ethyl alcohol (available in most hardware stores, paint stores, and automotive stores).	3. or 550 milliliters (18.5 fluid oz.) of denatured alcohol
2. 250 milliliters (8.4 fluid oz.) of concentrated sulfuric acid	4. 1500 milliliters (50.7 fluid oz.) of battery acid

Reaction summary: Diethyl ether can be prepared by reacting denatured alcohol with concentrated sulfuric acid, or battery acid. The alcohol is simply mixed with concentrated sulfuric acid or battery acid, and then distilled in the usual manner. The diethyl ether that forms, slowly distills over. The diethyl ether should then be re-distilled using a fractional distillation apparatus for purity.

Why does this reaction happen? Same as in methods 1, 2, or 3.

Hazards: Wear gloves when handling concentrated sulfuric acid, and avoid skin contact, contact with clothing, and ingestion at all cost. Concentrated sulfuric acid will corrode, char, and dissolve many substances including some plastics. Wear gloves when handling battery acid, and avoid skin contact, contact with clothing, and ingestion. Battery acid contains about 25% sulfuric acid by weight, and is very corrosive.

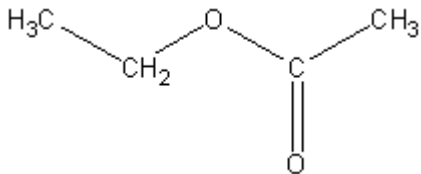
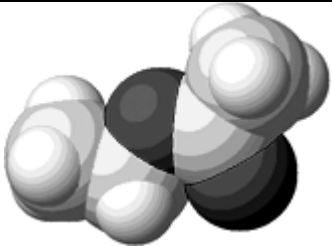
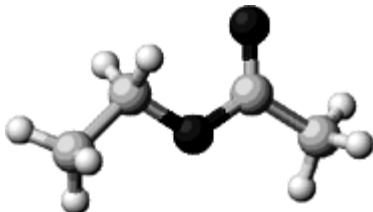
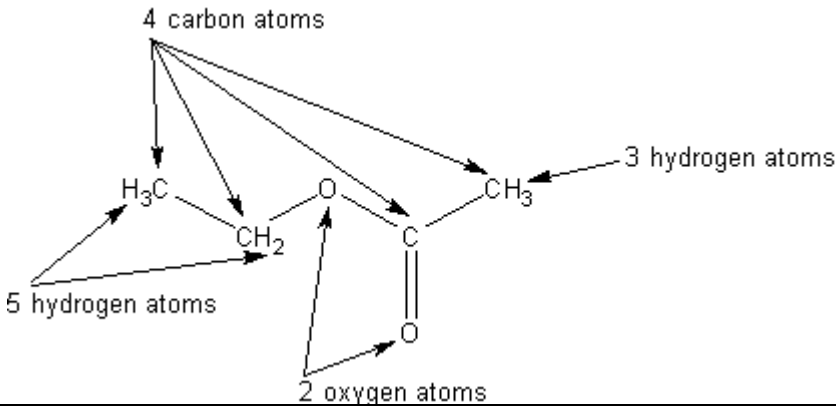
Procedure: If using concentrated sulfuric acid, place 550 milliliters (18.5 fluid oz.) of denatured alcohol into a standard distillation apparatus as illustrated in method 1 or similar, and then slowly and carefully add in 250 milliliters (8.4 fluid oz.) of concentrated sulfuric acid (plumbers grade works great). Thereafter, boil this mixture at 100 Celsius for about 6 to 8 hours, and allow the mixture to distill during this time. After 6 to 8 hours, remove the heat source, and then recover the crude diethyl ether from the receiver flask or receiver container. Finally, place this crude ether into a clean fractional distillation apparatus, and distill the ether at 40 Celsius, as in method 1. Note: the sulfuric acid mixture, which may be colored blood red if iron was present in the acid (plumbers grade, 93% sulfuric acid), can be boiled at 100 Celsius in a suitable glass container to boil-off some of the water to concentrate the sulfuric acid. The point is, the sulfuric acid can be recycled over and over again (even if it is colored blood red or contaminated with other impurities), as it is only a catalyst in the reaction, and is not consumed or chemically changed in any way. Note: when boiling the sulfuric acid mixture, white fumes begin to form, remove the heat source, and allow the now concentrated sulfuric acid to cool to room temperature—at this point the sulfuric is recycled and can be used for another run of ether formation.

If using battery acid, place into a suitable sized thick walled Pyrex low form beaker, place 1500 milliliters (50.7 fluid oz.) of battery acid, and then boil the mixture by heating it to 150 Celsius until white fumes start to evolve. When white fumes start to

evolve, turn off the heat source and allow the now concentrated sulfuric acid to cool to room temperature. Now, place 550 milliliters (18.5 fluid oz.) of denatured alcohol into a standard distillation apparatus, as illustrated in method 1 or similar, and then add in the concentrated battery acid previously prepared. Thereafter, boil this mixture at 100 Celsius for about 6 to 8 hours, and allow the mixture to distill during this time. After 6 to 8 hours, remove the heat source, and then recover the crude diethyl ether from the receiver flask or receiver container. Finally, place this collected crude ether layer into a clean fractional distillation apparatus, and distill the ether at 40 Celsius. Note: the sulfuric acid mixture, which may be colored blood red if iron was present in the acid (plumbers grade, 93% sulfuric acid), can be boiled at 100 Celsius in a suitable glass container to boil-off some of the water to concentrate the sulfuric acid. The point is, the sulfuric acid can be recycled over and over again (even if it is colored blood red or contaminated with other impurities), as it is only a catalyst in the reaction, and is not consumed or chemically changed in any way. Note: when boiling the sulfuric acid mixture, white fumes begin to form, remove the heat source, and allow the now concentrated sulfuric acid to cool to room temperature—at this point the sulfuric is recycled and can be used for another run of ether formation.

Procedure 23: The Preparation of Ethyl acetate

Also known as: acetic acid ethyl ester; acetic ether

	 <p>3D structure 1</p>  <p>3D structure 2</p>
Chemical structure	3D Structure
 <p>4 carbon atoms</p> <p>5 hydrogen atoms</p> <p>3 hydrogen atoms</p> <p>2 oxygen atoms</p> <p>Structure make-up</p>	<p>$C_4H_8O_2$</p>
Structure make-up	Condensed chemical structure

Ethyl acetate

Ethyl acetate is a clear, volatile, and flammable liquid with a pleasant, fruity odor. It has a pleasant taste when diluted. Ethyl acetate slowly decomposes by moisture, so it should be kept in air-tight bottles and in a cool dry place. It has a boiling point of 77 Celsius, and a melting point of -83 Celsius. Ethyl acetate is moderately soluble in water (1 milliliter in 10 milliliters of water), but is miscible with alcohol, acetone, chloroform, and ether. It forms an azeotropic mixture with water (6% by weight

with a boiling point of 70 Celsius). Ethyl acetate can be prepared by distilling a mixture of ethanol and acetic acid in the presence of a few drops of sulfuric acid. Ethyl acetate is a valuable solvent for many chemical reactions.

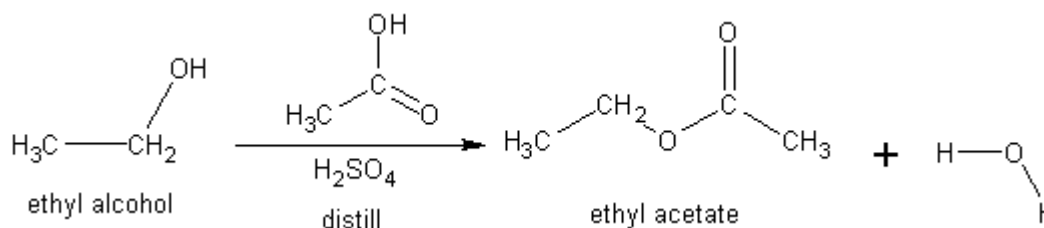
Method 1: Preparation of ethyl acetate from Vodka, battery acid, and vinegar

(By-products from reaction: none)

Materials:

1. 1000 milliliters (1 quart) of standard 5% acidity distilled crystal clear vinegar	4. 5 drops of concentrated sulfuric acid or 10 to 15 drops of battery acid
2. 180 milliliters (6 fluid oz.) of concentrated vinegar of 30% acidity	5. 15 grams (1/2 oz.) of anhydrous magnesium sulfate
3. 120 milliliters (4 fluid oz.) of cheap Vodka of 80 proof	

Reaction summary: Ethyl acetate is readily and easily prepared by distilling a mixture of vinegar and ethyl alcohol in the presence of a small amount of sulfuric acid. The sulfuric acid is the catalyst. The resulting distilled product is then treated with anhydrous magnesium sulfate and re-distilled to remove water.



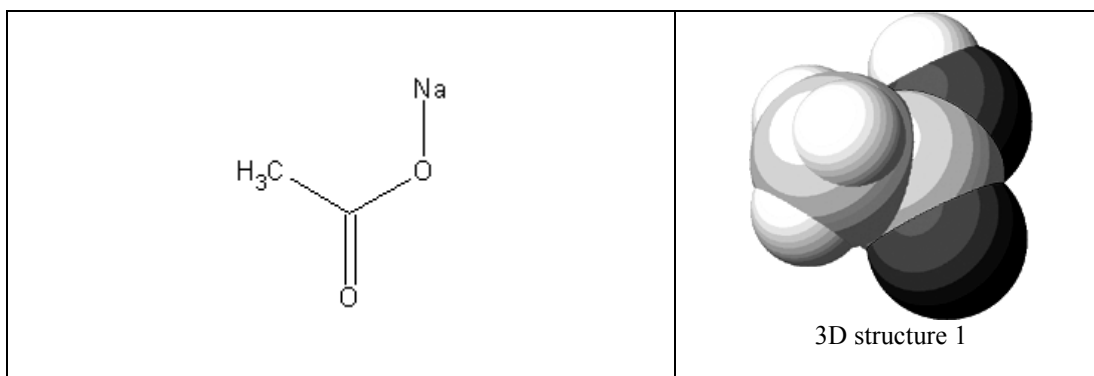
Why does this reaction happen? Alcohols and acids, such as ethyl alcohol and acetic acid, have an attraction towards each other when sulfuric acid is present. When these two compounds react, they form an ester, in this case ethyl acetate. The sulfuric acid acts as the catalyst in the reaction, and is not consumed or used up. Almost any alcohol and acid can be used in this type of reaction, and water is always the byproduct. The reason why the reaction takes place is because the sulfuric acid extracts a molecule of water from the ethyl alcohol and acetic acid.

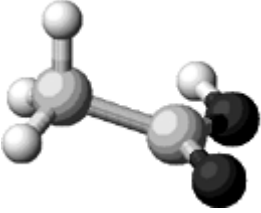
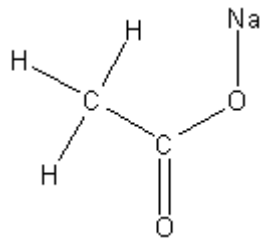
Hazards: Use caution when handling sulfuric acid.

Procedure: Into a large distillation apparatus, or similar distillation apparatus of any desired type or design, place 1000 milliliters (1 quart) of standard 5% acidity distilled crystal clear vinegar or 180 milliliters (6 fluid oz.) of concentrated vinegar of 30% acidity, followed by 120 milliliters (4 fluid oz.) of cheap Vodka of 80 proof, and then followed by 5 drops of concentrated sulfuric acid, or 10 to 15 drops of battery acid. Thereafter, heat and distill the mixture at 77 Celsius until no more ethyl acetate distills over—the time may vary from 1 to 3 hours. After the distillation period, the crude ethyl acetate should be thoroughly mixed with about 15 grams (1/2 oz.) of anhydrous magnesium sulfate, and stirred for about 30 minutes at room temperature. Finally, filter-off the magnesium sulfate, and then fractionally distill the filtered liquid at 77 Celsius to obtain a refined product of at least 99% ethyl acetate.

Procedure 24: The Preparation of Sodium acetate

Also known as: N/A



	 <p>3D structure 2</p>
Chemical structure	3D Structure
 <p>2 carbon atoms 3 hydrogen atoms 2 oxygen atoms 1 sodium atom</p>	C ₂ H ₃ O ₂ Na
Structure makeup	Condensed chemical structure

Sodium acetate

Sodium acetate forms a trihydrate, which forms transparent crystals or granules, or a white powder. The crystals have a melting point of about 58 Celsius, and become anhydrous when heated to 120 Celsius. The crystals are readily soluble in water, but relatively insoluble in alcohol, and most common organic solvents. The anhydrous form is widely used in organic synthesis as an acetylation agent.

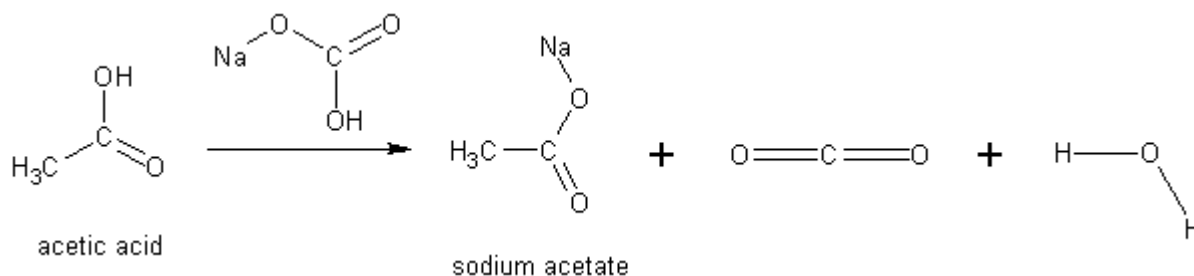
Method 1: Preparation of sodium acetate from vinegar and baking soda

(By-products from reaction: carbon dioxide gas)

Materials:

1. 1 gallon (3.73 liters) of 5% acidity regular colorless vinegar	2. 300 grams (10.5 oz.) of baking soda
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Reaction summary: Sodium acetate is readily prepared by reacting baking soda with regular old vinegar. You may have mixed these two ingredients before when you were a kid to form a volcano or for some other school project.



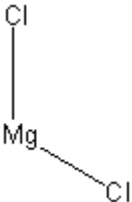
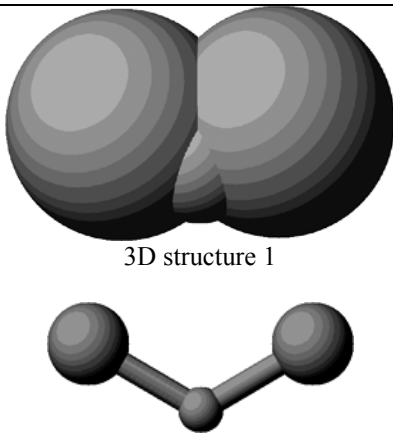
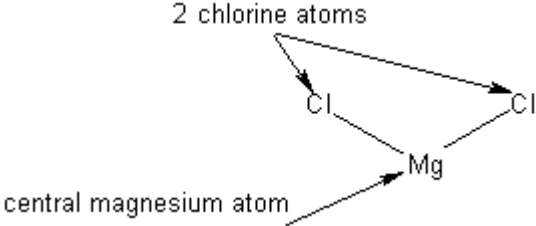
Why does this reaction happen? Baking soda, like other bases, is strongly attracted to acids such as acetic acid. When baking soda reacts with acetic acid, the acid vigorously reacts breaking the baking soda down into carbon dioxide and water. The sodium ion bonds with the acetic acid because the sodium ion is very attractive to the free oxygen of the acetic acid.

Hazards: None.

Procedure: Into a suitable large beaker or Pyrex container, place 1 gallon (3.73 liters) of 5% acidity regular colorless vinegar (nothing added). Thereafter, slowly add in, in small portions at a time, a total of about 300 grams (10.5 oz.) of baking soda over a period of about 45 minutes to one hour. During each addition of the baking soda, the mixture will foam vigorously as the acetic acid in the vinegar reacts with the base of sodium bicarbonate (baking soda). After all the baking soda has been added, the mixture should be immediately filtered to remove any insoluble materials. Thereafter, boil the filtered mixture to drive-off water and concentrate the mixture down to about 800 to 900 milliliters in total volume. When enough water has been boiled-off to reduce the mixture to about 800 to 900 milliliters in total volume, remove the heat source, and allow the mixture to cool to room temperature. Thereafter, quickly filter the mixture to remove any insoluble impurities, and then recrystallize the dissolved sodium acetate from the filtered mixture. See recrystallization for information on recrystallization processes. Note: vacuum filtration should be used to remove the precipitated crystals of sodium acetate as the recrystallization process proceeds. Second note: the final recovered crystals of the sodium acetate may have a slight brownish tint due to carbonous impurities. If desired, this product should be recrystallized from fresh water or alcohol for purity, but this is not necessary in the long run.

Procedure 25: The Preparation of anhydrous magnesium chloride

Also known as: chloride of magnesia

	 <p>3D structure 1</p> <p>3D structure 2</p>
Chemical structure	3D Structure
	<p>MgCl₂</p>
Structure makeup	Condensed chemical structure

Anhydrous magnesium chloride

Anhydrous magnesium chloride forms soft white, to colorless crystals. The crystals have a very high affinity for water, and must be kept in moisture free containers. The crystals melt when rapidly heated to 712 Celsius, but when slowly heated to 300 Celsius, chlorine gas is slowly released. The crystals are soluble in water and alcohol.

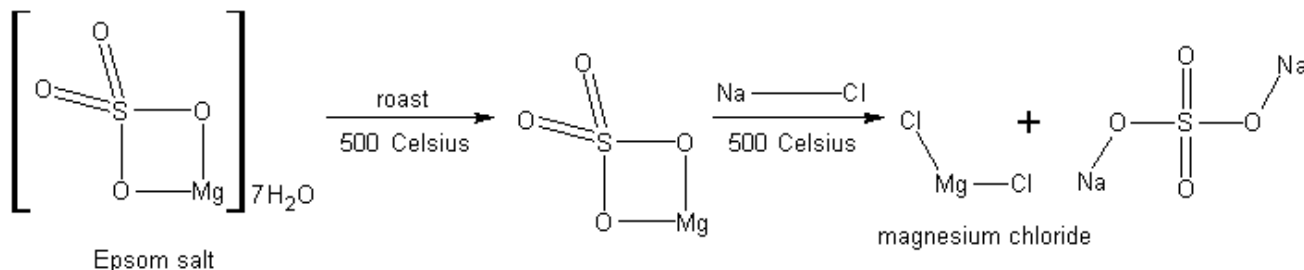
Method 1: Preparation of anhydrous magnesium chloride from Epsom salt and pickling salt

(By-products from reaction: sodium sulfate)

Materials:

1. 200 grams (7 oz.) of store bought Epsom salt	3. 250 milliliters (8.4 fluid oz.) of 99% isopropyl alcohol or 180 milliliters (6 fluid oz.) of 99% methanol
2. 100 grams (3.5 oz.) of regular store bought pickling salt	

Reaction summary: Anhydrous magnesium chloride is prepared in a four step process starting with the roasting of two salts, Epsom salt and pickling salt. During the roasting processes, water is removed. Once both salts have been roasted to drive-off any water, the two dry salts are then mixed and then heated together to form anhydrous magnesium chloride and sodium sulfate. Thereafter, the magnesium chloride is recovered by extracting it into alcohol, leaving the insoluble sodium sulfate behind. After that, the anhydrous magnesium chloride is then recovered by distilling-off the alcohol at the appropriate temperature. The anhydrous magnesium chloride is then readily recovered.



Why does this reaction happen? In order for the reaction to proceed, the magnesium sulfate and sodium chloride must be free of water; therefore, the two compounds are roasted prior to reaction to drive-off any water. Thereafter, the magnesium sulfate and sodium chloride are then mixed, and then roasted using a Bunsen burner. The reaction proceeds fast because the sodium ions of the sodium chloride prefer to bond with the oxygens of the magnesium sulfate; therefore, the magnesium ions and sodium ions swap each other.

Hazards: If using methanol, use caution and avoid skin contact and inhalation of the vapors, as methanol is poisonous.

Procedure:

Step 1: Roasting Epsom salt to form the anhydrous salt

Into a suitable crucible or steel cup or container, place 200 grams (7 oz.) of store bought Epsom salt (magnesium sulfate heptahydrate), and then roast the salt at high heat using a Bunsen burner or free flame from a gas operated stove for about 40 to 50 minutes or until no more water vapor is released. To determine when no more water is released, hold a mirror over the heated salt. If the mirror fogs up, water vapor is still being liberated from the salt. Once all the water has been driven off, the salt is now anhydrous magnesium sulfate. At this point, remove the heat source and allow the roasted salt to cool to room temperature before removing. Then save the remaining 100+ grams of the anhydrous magnesium salt for step 3. Note: the anhydrous salt will have to be broken up and pulverized once it has cooled. Note: all traces of water must be driven-off. If any moisture is present in the Epsom salt, the formation of anhydrous magnesium chloride will not take place.

Step 2: Roasting pickling salt to drive-off any water

Into the same crucible as used in step 1 (make sure you clean it with tap water first), or into a separate clean crucible or steel cup or container, place 100 grams (3.5 oz.) of regular store bought pickling salt, and then roast the salt at high heat using the free flame of a Bunsen burner or open flame from a gas operated stove or equivalent for about 20 to 30 minutes. Thereafter, remove the heat source and allow the salt to cool to room temperature. Thereafter, save this salt for step 3.

Step 3: formation of anhydrous magnesium chloride

First, into a clean blender or mincing blender (used to chop up vegetables or other solids), place 100 grams of the pulverized anhydrous Epsom salt prepared in step 1, followed by 97 grams of the roasted pickling salt as prepared in step 2. Thereafter, blend the mixture for about 90 to 120 seconds to thoroughly blend the mixture. If you don't have a mixing blender or similar device, place the ingredients into a zip lock bag, seal the bag airtight, and then shake the bag vigorously for about 10 minutes to

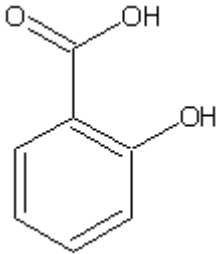

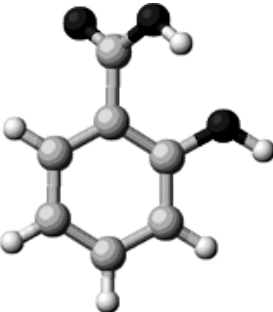
thoroughly mix the two solids. Now, into the same crucible as used in step 1 or 2, after cleaning it out with tap water and drying thoroughly before using, or into a separate clean and dry second or third crucible or steel cup or container, place the thoroughly blended mixture containing the two salts, and then roast the salts using a open flame from a Bunsen burner or gas operated stove for about 45 to 50 minutes. During the heating process, a simple chemical reaction will take place, and the anhydrous magnesium chloride will form. After the heating process, remove the heat source, and allow the mixture to cool to room temperature. Thereafter, remove the entire mass, pulverize it into a fine grain mass, and then save it for step 4.

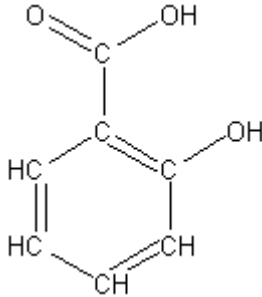
Step 4: recovering the anhydrous magnesium chloride by extraction into alcohol

Into a clean beaker, place the pulverized mass from step 3, and then add in 250 milliliters (8.4 fluid oz.) of 99% isopropyl alcohol, and then blend the mixture on high speed for about 10 minutes to thoroughly dissolve the anhydrous magnesium chloride, leaving the sodium sulfate un-dissolved. Note: anhydrous magnesium chloride can form addition compounds with alcohol, and this may interfere with the extraction process. In some cases, substitute the rubbing alcohol with 180 milliliters (6 fluid oz.) of 99% methanol. After the blending operation, filter the entire mixture to remove the insoluble sulfate, and to recover the alcohol solution. Thereafter, place the filtered alcohol mixture into a clean dry distillation apparatus, and distill-off the alcohol at 70+ Celsius, if you used methanol, or at 80+ Celsius if you used isopropyl alcohol. Continue to distill-off the alcohol until dry solid remains. When dry solid remains, remove the heat source and allow the remaining dry solid to cool to room temperature. Thereafter, recover the anhydrous magnesium chloride. Note: you may have to break-up and pulverize the anhydrous magnesium chloride using a metal stirring rod to remove it from the distillation flask. Store the anhydrous magnesium chloride in a zip lock bag sealed tight to protect it from moisture.

Procedure 26: The Preparation of Salicylic acid

Also known as: 2-Hydroxy benzoic acid; Verrugon

	 <p>3D structure 1</p>  <p>3D structure 2</p>
Chemical structure	3D Structure

 <p>7 carbon atoms 6 hydrogen atoms 3 oxygen atoms</p>	$C_6H_6O_3$
Structure makeup	Condensed chemical structure

Salicylic acid

Salicylic acid forms colorless to white crystals or crystalline powder, with a melting point of 159 Celsius. The pure crystals slowly discolor in the presence of bright light or sunlight. When the crystals are rapidly heated, they decompose into phenol and carbon dioxide. Salicylic acid is virtually insoluble in water, but soluble in alcohol, acetone, and ether.

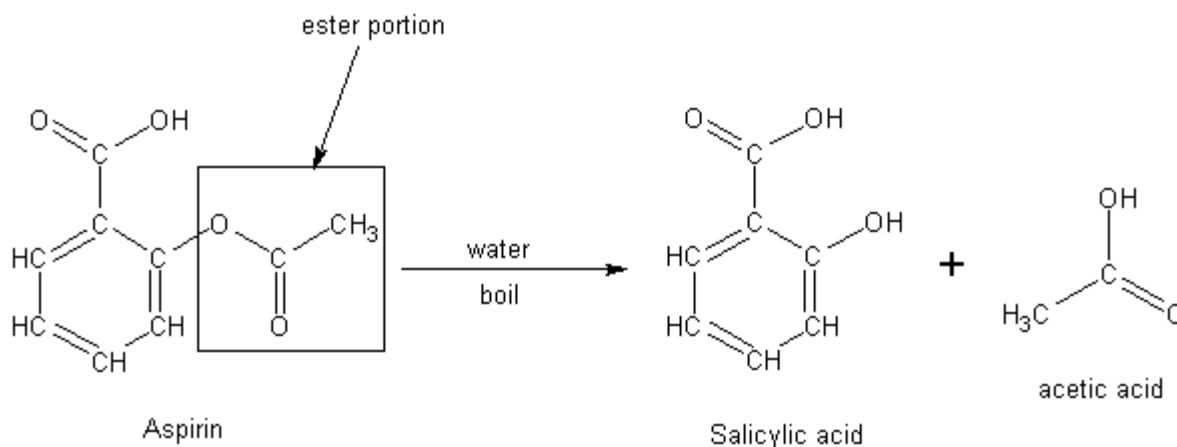
Method 1: Preparation of salicylic acid from aspirin and boiling water

(By-products from reaction: acetic acid)

Materials:

1. 300 grams (10.1 oz.) of regular store bought aspirin tablets	3. 1 liter (33.8 fluid oz.) of rubbing alcohol containing 70% isopropyl alcohol by volume
2. 500 milliliters (17 fluid oz.) of cold tap water	4. 100 to 150 grams (3.5 oz., or 5.3 oz.) of pickling salt

Reaction summary: Salicylic acid is readily formed by boiling a suspension of aspirin in water. The reaction is quit simple, and the boiling water breaks down the aspirin molecules into salicylic acid and acetic acid. The salicylic acid is then recovered by dissolving it into alcohol, and then recovering the salicylic acid through recrystallization.



Why does this reaction happen? Aspirin is unstable to heat, and breaks down into salicylic acid and acetic acid when heated and boiled in water. The reaction takes place because the ester portion of the aspirin molecule prefers to bond with hydrogen rather than with carbon; therefore, when energy is applied to the aspirin molecule in the presence of water, the ester portion splits off reacting with the boiling water and forming acetic acid and salicylic acid.

Hazards: None.

Procedure:

Step 1: Decomposing aspirin to form a salicylic acid mass

Into a coffee grinder or similar device place 300 grams (10.1 oz.) of regular store bought aspirin tablets. Thereafter, grind up the tablets until a fine powdery substance is obtained. Thereafter, suspend this powdered material into 500 milliliters (17 fluid oz.) of cold tap water, and then bring this mixture to a boil. As the water boils, it will decompose the aspirin molecules into salicylic acid and acetic acid. Note: never mind any fillers or additives that exist in the original aspirin tablets. Continue to boil the mixture for about 2 to 3 hours to fully break the aspirin molecules down. After about 2 to 3 hours, remove the heat source, and allow the mixture to cool to room temperature. Thereafter, filter the cooled mixture using either gravity filtration or preferably vacuum filtration to remove the insoluble materials. Then set these insoluble materials aside for the moment. Note: the filtered aqueous solution will contain dissolved acetic acid, which can be recovered by boiling-off the water at 100 Celsius until a concentrated acetic acid mixture is obtained.

Step 2: Salting out some rubbing alcohol

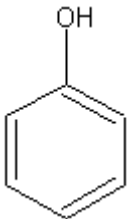

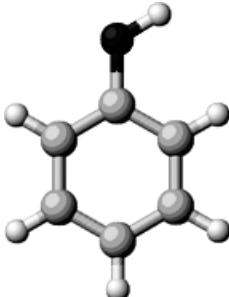
Now, its time to salt out some rubbing alcohol, so into a separatory funnel of at least 1500 milliliter capacity, place 1 liter (33.8 fluid oz.) of rubbing alcohol containing 70% isopropyl alcohol by volume. Thereafter, add in an excess of pickling salt, say about 100 to 150 grams (3.5 oz., or 5.3 oz.) of pickling salt, and then vigorously shake the separatory funnel for about 5 minutes. Then allow the separatory funnel to stand, and allow a two-phase mixture to result. Thereafter, drain-off the bottom aqueous layer, and then pour-off or drain-off the upper alcohol layer.

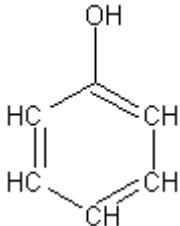
Step 3: Recrystallization of the salicylic acid

Now, into a clean beaker of at least 1000 to 1500 milliliter capacity, place at least 700 milliliters (23.6 fluid oz.) of the collected isopropyl alcohol (upper layer), and then add in the filtered-off material obtained in step 1. Thereafter, blend the mixture at room temperature for about 30 minutes to dissolve the salicylic acid. After the 30-minute period, most of the salicylic acid would have dissolved into the isopropyl alcohol. At this point, filter the alcohol mixture to remove the insoluble fillers and additives from the original aspirin tablets. Then recrystallize the salicylic acid from the filtered alcohol mixture, see recrystallization for information on how to carryout a recrystallization process.

Procedure 27: The Preparation of Phenol

Also known as: Hydroxy benzene

 <chem>Oc1ccccc1</chem>	 3D structure 1  3D structure 2
Chemical structure	3D Structure

 <p>6 carbon atoms 6 hydrogen atoms 1 oxygen atom</p>	C_6H_6O
Structure makeup	Condensed chemical structure

Phenol

Phenol forms colorless crystals or white powder, or crystalline masses. Pure phenol has a melting point of 43 Celsius. Phenol is very soluble in alcohol, chloroform, and ether. The crystals have a powerful burning sensation when exposed to the skin. Phenol is highly toxic, and skin absorption can lead to poisoning. Always use proper ventilation and wear proper gloves when handling phenol. The crystalline mass liquefies when slurried with 8% water by weight.

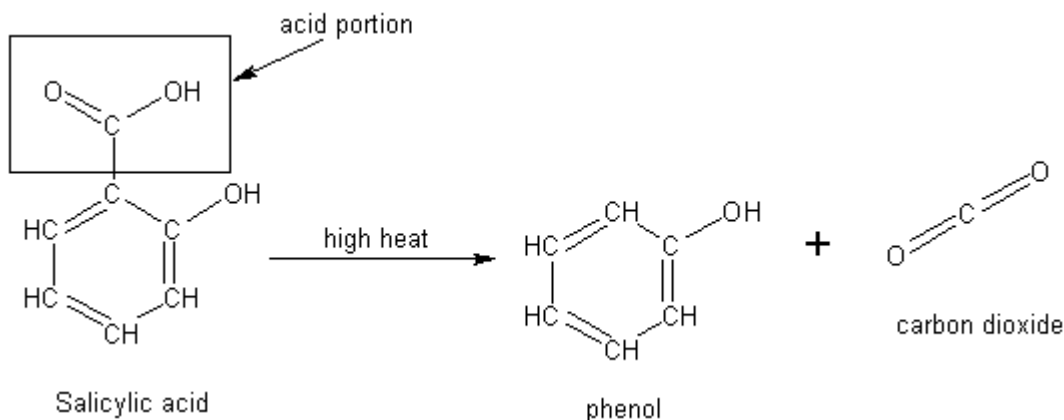
Method 1: Preparation of phenol from salicylic acid

(By-products from reaction: carbon dioxide gas)

Materials:

1. 25 grams (0.88 oz.) of salicylic acid	
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Reaction summary: Phenol is readily prepared by rapidly heating salicylic acid with a free flame. During the reaction carbon dioxide gas is evolved at a steady rate.

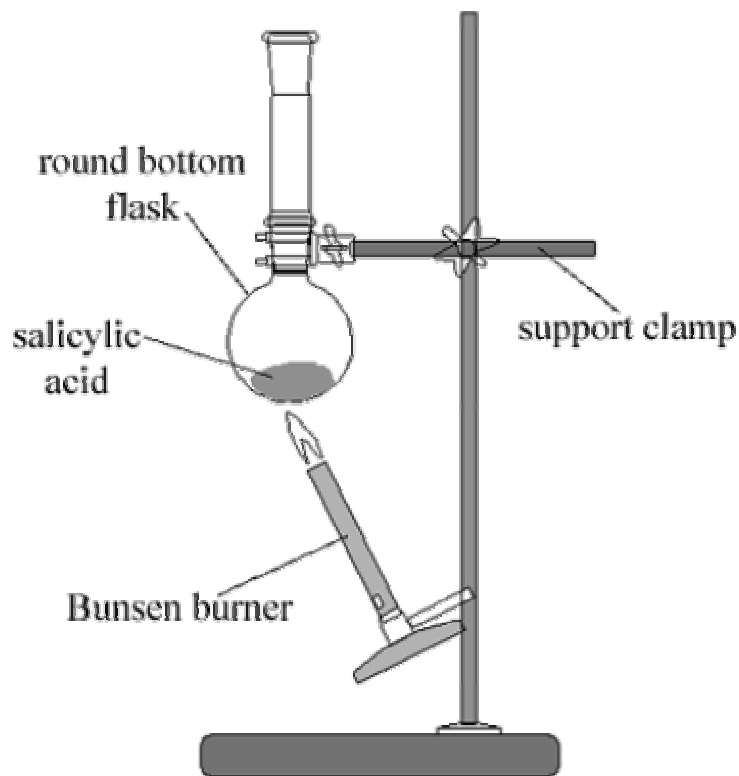


Why does this reaction happen? Similar to aspirin, salicylic acid is unstable towards heat as well, but in a different manner. When salicylic acid is rapidly heated using an open flame, it breaks down into phenol and carbon dioxide. The reaction happens because the acid portion prefers to be bonded directly to oxygen instead of both carbon and oxygen, thus forming carbon dioxide.

Hazards: Use proper ventilation when carrying out this procedure. Phenol is very toxic so use extreme caution when handling. Avoid inhalation of phenol vapors, and avoid skin contact. Phenol is rapidly absorbed by the skin, so use extreme caution.

Procedure: Into a suitable apparatus as illustrated below, place 25 grams (0.88 oz.) of salicylic acid. Thereafter, rapidly heat the mixture using a Bunsen burner, but apply the heat in small increments, meaning heat the salicylic acid for 5 to 6 seconds at a time, pause for 10 to 15 seconds, and then heat the salicylic acid for another 5 to 6 seconds. Repeat this process of heating and pausing for 10 to 15 minutes. During the rapid heating process, the salicylic acid will break down forming phenol and carbon dioxide. The carbon dioxide gas merely dissipates away. Note: use proper ventilation and wear gloves when carrying out this process. After the heating process, allow the mixture to cool to room temperature, and then add to the flask 100 milliliters of methylene chloride, and then swirl the flask for about 5 minutes to dissolve the phenol. Thereafter, place this

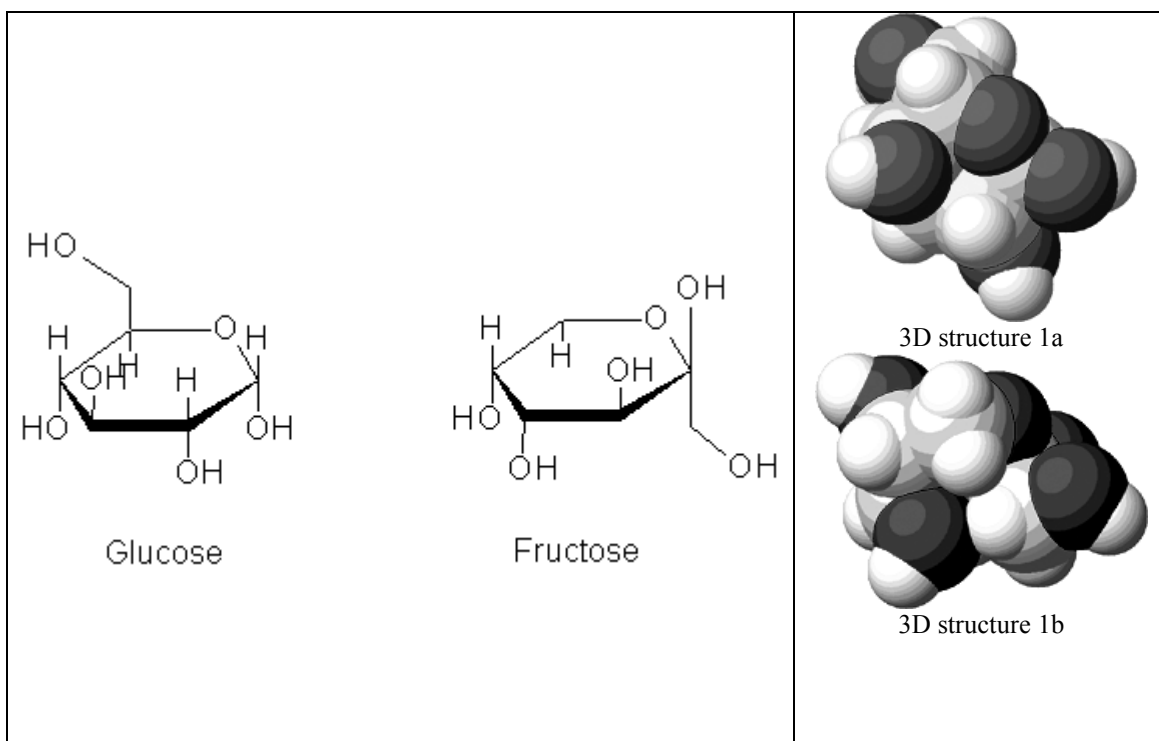
methylene chloride mixture into a suitable distillation apparatus, and remove the methylene chloride by distillation at 40 Celsius. Once all the methylene chloride has been removed, the remaining residue will be composed of phenol with a purity of 95% to 98%.

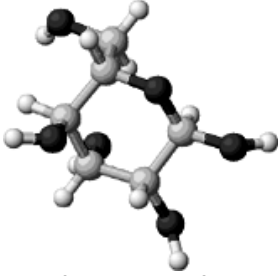
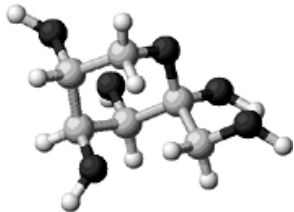
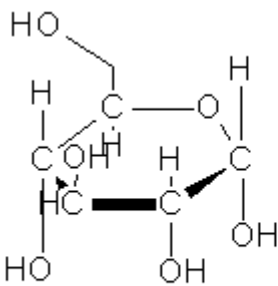
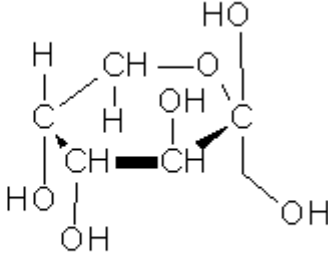


Set-up for the preparation of phenol from salicylic acid using a free flame.

Procedure 28: The Preparation of “Invert sugar”

Also known as: Fructose and glucose



	 3D structure 2a  3D structure 2b
Chemical structure	3D Structure
 Glucose  Fructose	$C_6H_{12}O_6$ $C_6H_{12}O_6$
Structure makeup	Condensed chemical structure

Invert sugar

Invert sugar is mixture of 50% glucose and 50% fructose. The product is obtained by the hydrolysis of sucrose (table sugar) using acids, bases, or enzymes, but mostly acids. Invert sugar is a syrupy colorless liquid, which is more sweeter than sugar.

Method 1: Preparation of “invert sugar” from table sugar using dilute hydrochloric acid

(By-products from reaction: None)

Materials:

1. 500 milliliters (17 fluid oz.) of warm tap water	3. 500 grams (17.6 oz.) of table sugar
2. 10 milliliters of concentrated hydrochloric acid	4. 10 grams of baking soda

Reaction summary: Invert sugar is readily prepared by hydrolysis of table sugar using hydrochloric acid. The reaction is very simple and is carried out under mild heat.

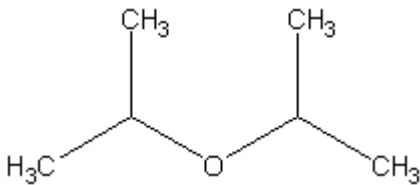
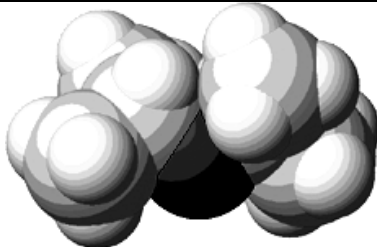
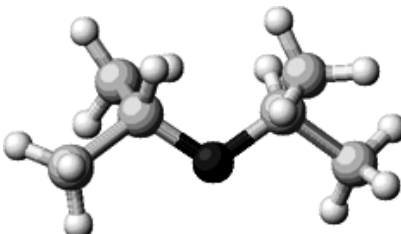
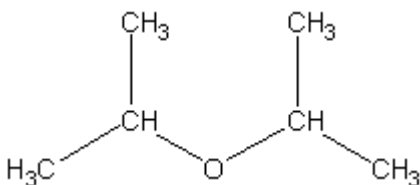
Why does this reaction happen? Sucrose is a rather large molecule, and tends to break down into smaller molecules of glucose and fructose. In some cases, boiling water alone can break sucrose down into the smaller molecules of glucose and fructose. In most cases however, a little bit of acid is used to catalyze and speed up the reaction.

Hazards: Wear gloves when handling concentrated hydrochloric acid and avoid inhalation of the fumes.

Procedure: Into a large beaker or similar glass container, place 500 milliliters (17 fluid oz.) of warm tap water or bottled water, and then add and dissolve 500 grams (17.6 oz.) of table sugar. Thereafter, while blending the mixture, heat the mixture to about 70 to 80 Celsius, and when the temperature of the sugar solution reaches the desired temperature, add in 10 milliliters of concentrated hydrochloric acid, and then continue to heat and blend the sugar mixture at 70 to 80 Celsius for about 30 to 40 minutes. Note: during the heating process, the solution may slowly turn a light yellowish color due to slight carbocation of the sugar. After heating and blending the mixture for about 30 to 40 minutes, remove the heat source, and allow the mixture to cool to room temperature. Thereafter, add in about 10 grams of baking soda, and then blend the mixture for about 15 minutes at room temperature. Thereafter, filter the mixture briefly to remove any insoluble impurities. After the filtration process, your invert sugar is ready for use.

Procedure 29: The Preparation of Isopropyl ether

Also known as: 2,2'Oxybispropane; 2-isopropoxypropane

	 <p>3D structure 1</p>  <p>3D structure 2</p>
Chemical structure	3D Structure
 <p>6 carbon atoms 14 hydrogen atoms 1 oxygen atom</p>	<p>$C_6H_{14}O$</p>
Structure makeup	Condensed chemical structure

Isopropyl ether

Isopropyl ether forms a colorless highly volatile liquid. The liquid has a boiling point of 68 Celsius. Isopropyl ether is highly flammable and readily forms peroxides when exposed to oxygen. The liquid should be stored in air-tight containers, and these containers should be filled to the top to minimize the air-gap from the top of the liquid to the bottles opening. After isopropyl ether has been stored for prolonged periods of time, it should not be used unless distilled in the presence of a small amount of concentrated sulfuric acid to destroy any potential peroxides that may have formed during the storage period.

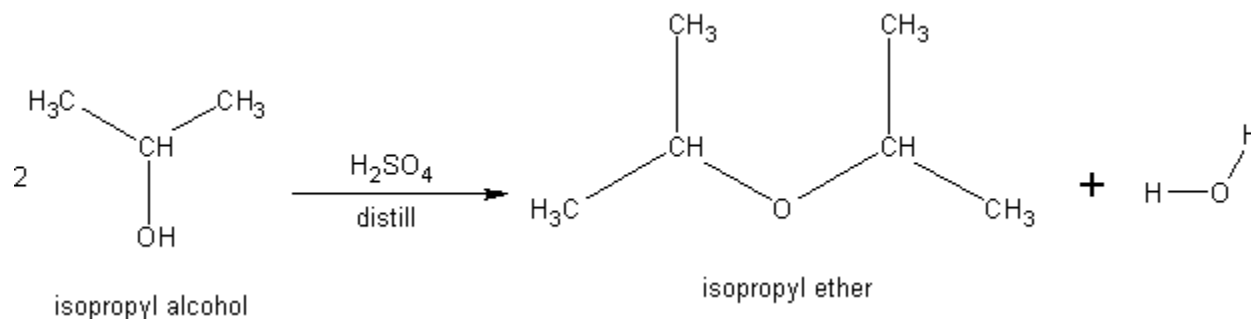
Method 1: Preparation of isopropyl ether from rubbing alcohol and concentrated sulfuric acid

(By-products from reaction: propylene gas, and water)

Materials:

1. 1 liter (33.9 fluid oz.) of regular store bought rubbing alcohol	2. 350 milliliters (11.8 fluid oz.) of concentrated sulfuric acid
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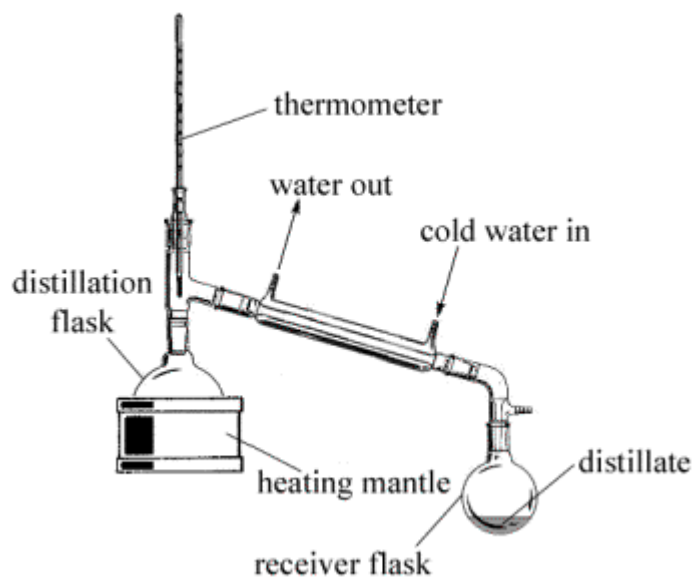
Reaction summary: Isopropyl ether is readily prepared by distilling a mixture of rubbing alcohol with concentrated sulfuric acid. As the mixture distills, the isopropyl ether will carry over and can be collected. Once the initial reaction and subsequent distillation is over, the isopropyl ether is then fractionally distilled to recover the ether in high purity.



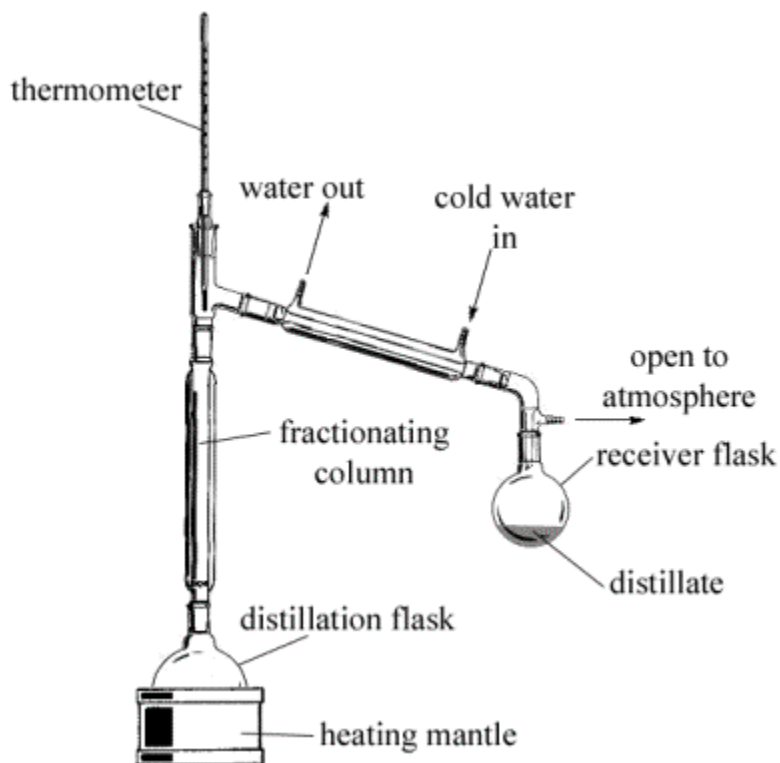
Why does this reaction happen? As seen in earlier reactions, sulfuric acid has a very strong affinity for water, and it can extract it from numerous materials. In the case of isopropyl alcohol, the sulfuric acid extracts a single water molecule from two molecules of isopropyl alcohol. When the acid extracts a water molecule from two molecules of isopropyl alcohol, the two molecules of isopropyl alcohol then bond to each other forming isopropyl ether.

Hazards: Isopropyl ether is highly flammable; so extinguish all flames before using. Wear gloves and use caution when handling concentrated sulfuric acid, which is capable of causing severe burns.

Procedure: Into a suitable apparatus, as illustrated below, place 1 liter (33.9 fluid oz.) of regular store bought rubbing alcohol containing 70% isopropyl alcohol by volume. Thereafter, slowly and carefully pour in 350 milliliters (11.8 fluid oz.) of concentrated sulfuric acid of any quality or grade, and preferably cheap store bought concentrated acid used for drain opening. Thereafter, distill the mixture at 100 Celsius for about 4 to 6 hours to recover all the isopropyl ether as possible. Thereafter, place the collected isopropyl ether into a fractional distillation apparatus, followed by a small amount of ferric chloride hexahydrate. Note: a small amount of ferric chloride hexahydrate is added to destroy any peroxides that may have formed in the ether. Peroxides are explosive compounds that ethers form when they sit around in the open air for prolonged periods of time. Thereafter, fractionally distill the mixture at 68 to 70 Celsius for about 6 hours to collect a ether product of about 96 to 97% purity. **Note:** the sulfuric acid mixture after the initial removal of the ether can be recycled over and over again for about 10 to 12 more operations for the production of isopropyl ether. However, after about the tenth to twelfth time of using, the acid concentration will have become too weak due to the formation of water as a by-product in the reaction. If the acid concentration is too weak, it will be no good for use, as the sulfuric acid acts as the catalyst in the formation of the ether to begin with, but weak acid solutions have no catalyst effect.



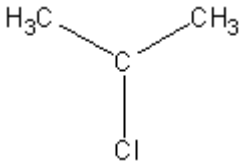
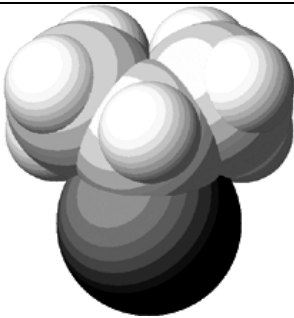
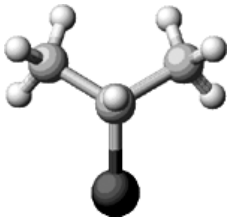
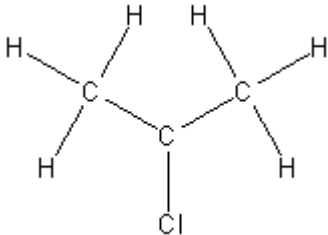
Simple distillation apparatus for the preparation of isopropyl ether. Note: the receiver flasks should be kept in a cold-water bath to prevent the vapors of the ether from evaporating away.



Classic fractional distillation apparatus for fractionally distilling the ether. Note: the receiver flask should be kept in an ice bath to prevent the ether vapors from evaporating away.

Procedure 30: The Preparation of Isopropyl chloride

Also known as: 2-chloropropane

	 <p>3D structure 1</p>  <p>3D structure 2</p>
Chemical structure	3D Structure
 <p>3 carbon atoms 6 Hydrogen atoms 1 chlorine atom</p>	<p>C_3H_7Cl</p>
Structure makeup	Condensed chemical structure

Isopropyl chloride

Isopropyl chloride forms a colorless highly volatile liquid, with a boiling point of 36 Celsius, and a melting point of -117 Celsius. The liquid is very soluble in alcohol and ether, but relatively insoluble in water.

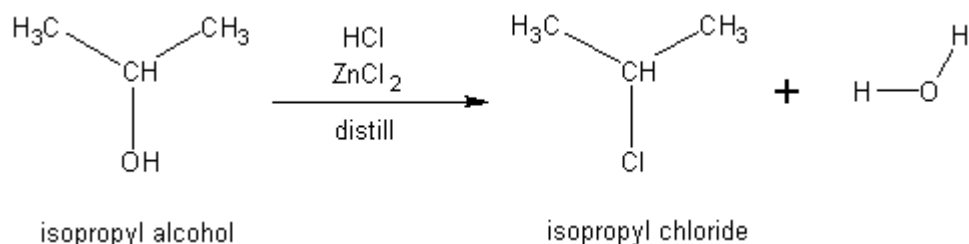
Method 1: Preparation of isopropyl chloride from rubbing alcohol and concentrated hydrochloric acid

(By-products from reaction: water)

Materials:

1. 250 milliliters (8.5 fluid oz.) of rubbing alcohol	3. 50 grams (1.8 oz.) of pickling salt
2. 200 milliliters (6.7 fluid oz.) of concentrated hydrochloric acid (Muriatic acid).	4. 100 grams (3.5 oz.) of zinc chloride

Reaction summary: Isopropyl chloride is readily prepared by reacting isopropyl alcohol with concentrated hydrochloric acid in the presence of zinc chloride as a catalyst. After the reaction, the reaction mixture is distilled to recover the desired product of isopropyl chloride.



Why does this reaction happen? In this reaction, zinc chloride acts as the catalyst in the dehydration of isopropyl alcohol. The dehydration takes place because the zinc chloride causes the extraction of a single molecule of water from the alcohol. The remaining isopropyl group then bonds with the chlorine of the hydrochloric acid forming isopropyl chloride.

Hazards: Use caution when handling isopropyl chloride, which is highly volatile. Avoid skin contact and inhalation of the vapors or liquid. Wear gloves when handling concentrated hydrochloric acid, and use proper ventilation.

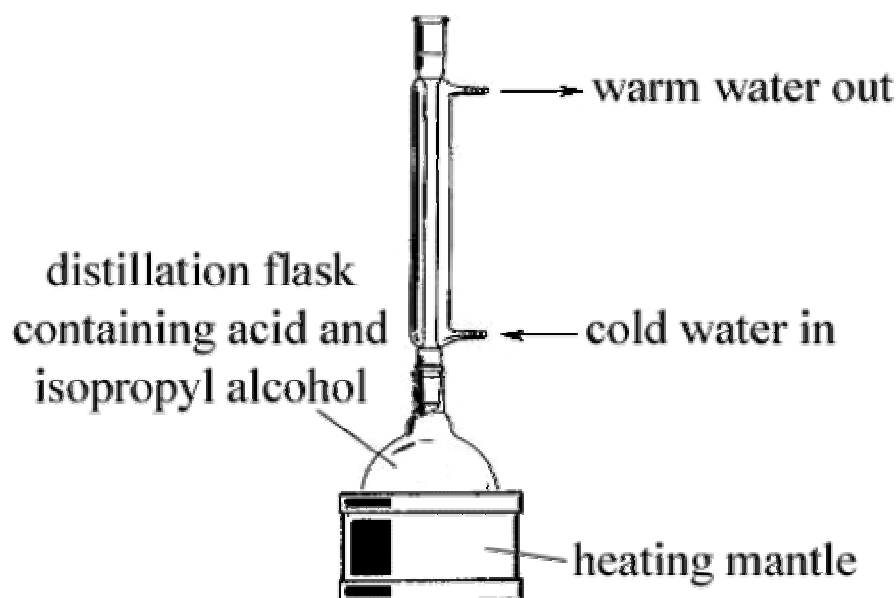
Procedure:

Step 1: Salt out rubbing alcohol

Into a separatory funnel, or suitable means, place 250 milliliters (8.5 fluid oz.) of rubbing alcohol containing 70% isopropyl alcohol by volume. Thereafter, add in about 50 grams (1.8 oz.) of pickling salt, and then shake the mixture vigorously for about 5 minutes. Thereafter, allow the mixture to settle, during which time a two-phase mixture will result. Thereafter, simply drain-off the bottom layer, and then drain-off the upper layer into a clean suitable container. This upper layer will contain the isopropyl alcohol. Save this upper layer for step two.

Step 2: Preparation of isopropyl chloride

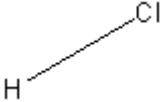
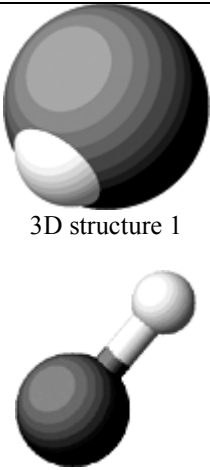
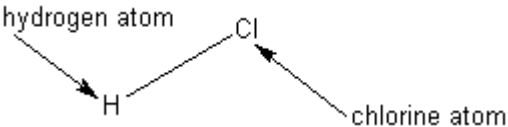
Into a suitable reflux apparatus, as illustrated below, place 140 milliliters of salted out rubbing alcohol, as prepared in step 1, followed by 200 milliliters (6.7 fluid oz.) of concentrated hydrochloric acid (Muriatic acid). Thereafter, add in 100 grams (3.5 oz.) of zinc chloride, and then reflux the mixture for about 4 hours at 100 Celsius. During the reflux process, the mixture will boil, whereby a chemical reaction will result. During this chemical reaction, hydrochloric acid will react with the isopropyl alcohol forming isopropyl chloride. The zinc chloride acts as a catalyst, and allows the reaction to take place. After the 4-hour reflux period, setup a distillation apparatus, and then distill the mixture at 80 to 90 Celsius to free the isopropyl chloride from the reaction mixture. After the distillation process, place the crude collected product into a fractional distillation apparatus, and fractionally distill the mixture at 40 to 50 Celsius, to collect a high quality isopropyl chloride product of 95 to 98% purity.



Reflux apparatus for the preparation of isopropyl chloride. After the reflux process, the reaction mixture should be first distilled using a typical distillation apparatus, which you should be familiar with at this point. Thereafter, use a typical fractional distillation apparatus to fractionally distill the crude chloride.

Procedure 31: The Preparation of Anhydrous Hydrogen Chloride gas

Also known as: Anhydrous hydrochloric acid

	
Chemical structure	3D Structure
	HCl
Structure make-up	Condensed chemical structure

Anhydrous Hydrogen chloride

Anhydrous hydrogen chloride is a very corrosive, non-flammable gas, with a highly irritating vapor. Hydrogen chloride is very soluble in water, forming a fuming liquid; vide supra, hydrochloric acid. It has a melting point of -114 Celsius, and a boiling point of -85 Celsius. It is prepared by the reaction of concentrated sulfuric acid upon table salt (preferably pickling salt), or by the action of chlorine upon organic compounds. The latter being the chief source of hydrochloric acid.

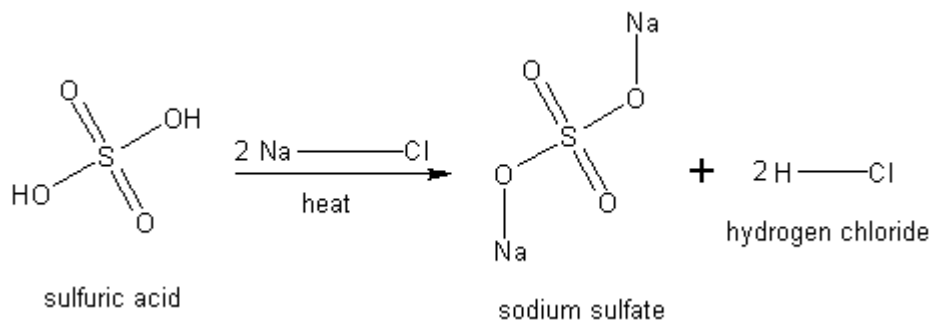
Method 1: Preparation of Anhydrous Hydrogen Chloride gas from pickling salt (sodium chloride) and sulfuric acid

(By-products from reaction: sodium sulfate, and sodium bisulfate)

Materials:

1. 50 grams (1.8 fluid oz.) of concentrated sulfuric acid	2. 60 grams (2.1 oz.) of pickling salt
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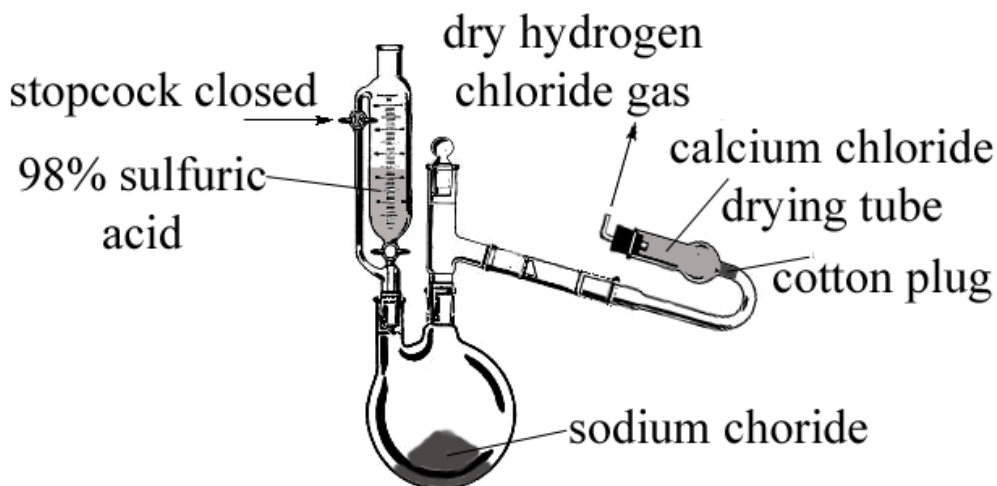
Reaction summary: Anhydrous hydrogen chloride is readily prepared by reacting concentrated sulfuric acid with pickling salt. During the reaction, hydrogen chloride gas is steadily evolved.



Why does this reaction happen? Sulfuric acid reacts with sodium chloride because the sodium ions of the sodium chloride prefer to bond with the oxygens of the sulfuric acid, thereby forming sodium sulfate and liberating hydrogen chloride gas.

Hazards: Perform the reaction in a well-ventilated area, as hydrogen chloride vapors are highly irritating to the nose and throat.

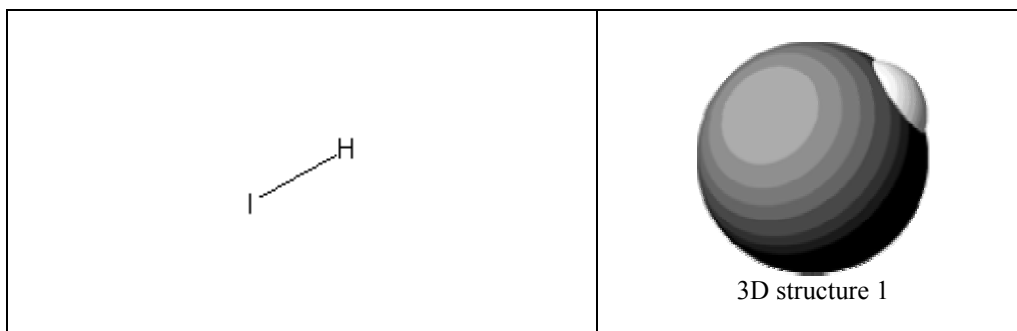
Procedure: Simply place 50 grams (1.8 fluid oz.) of concentrated sulfuric acid into the addition funnel, and then place 60 grams (2.1 oz.) of pickling salt, into the reaction flask. Thereafter, slowly drip the concentrated sulfuric acid onto the pickling salt. During the sulfuric acid addition, hydrogen chloride gas will be steadily evolved. Once the gas has been dried, it can be used in the manufacture of anhydrous aluminum chloride, in the preparation of hydrochloride salts, and/or the preparation of anhydrous metal chlorides.


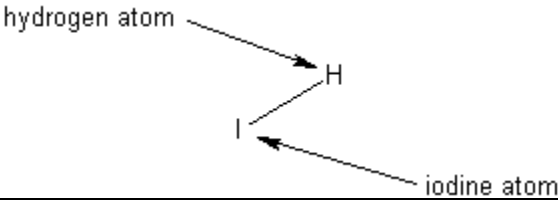


Set-up for the preparation of anhydrous hydrogen chloride gas. Note: gently heating the reaction flask can help speed up the reaction.

Procedure 32: The Preparation of 46% Hydroiodic acid

Also known as: Aqueous hydrogen iodide



	
Chemical structure	3D structure 2
	HI
Structure make-up	Condensed chemical structure

Hydroiodic acid

Hydroiodic acid is a colorless to yellowish to brownish liquid. It is colorless when freshly prepared, but rapidly turns yellow due to oxidation. Hydroiodic acid should be used within 2 weeks of its preparation, and it will turn brown on standing due to oxidation; however, these brownish solutions can be regenerated by adding in hypophosphorus acid. Hydroiodic acid forms an azeotrope with water containing 57% acid by weight with a constant boiling temperature of 127 Celsius. Hydroiodic acid is readily prepared by bubbling hydrogen sulfide gas into a suspension of iodine in water, and then filtering-off the precipitated sulfur.

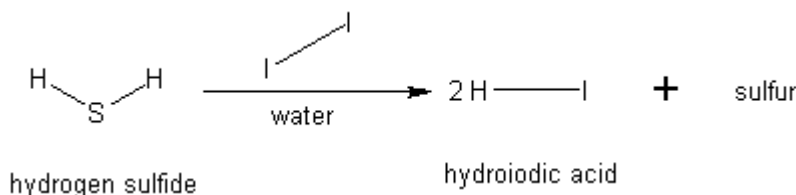
Method 1: Preparation of 46% Hydroiodic acid from Iodine and Hydrogen Sulfide gas

(By-products from reaction: sulfur)

Materials:

1. 37 grams of iodine crystals (1.3 oz.)	3. 5 grams (0.17 oz.) of hydrogen sulfide gas
2. 36 milliliters (1.2 fluid oz.) of water	

Reaction summary: Concentrated hydroiodic acid can be prepared by reacting iodine crystals with hydrogen sulfide gas in the presence of water. The acid should be used within a week of preparation.

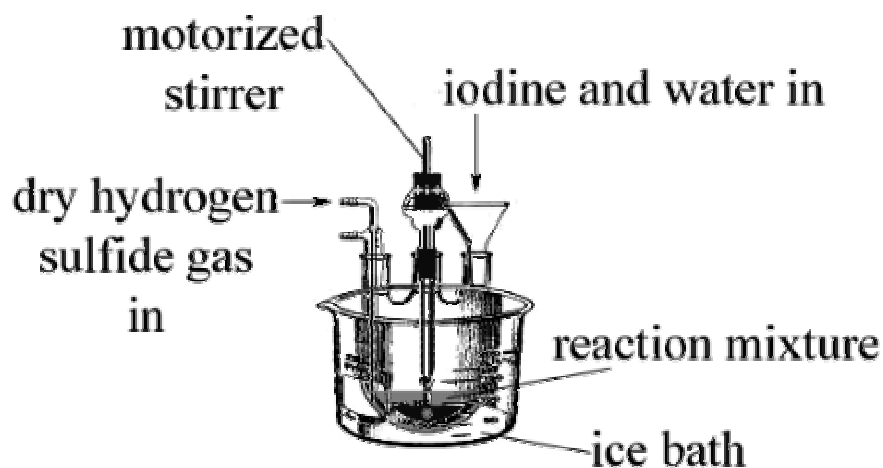


Why does this reaction happen? Hydroiodic acid is formed when hydrogen sulfide is passed into a suspension of iodine in water. The reaction takes place because hydrogen sulfide is oxidized by the iodine forming hydrogen iodide and freeing sulfur. Because water is present, the hydrogen iodide remains dissolved in the water forming the acid hydroiodic acid.

Hazards: Hydrogen sulfide is highly toxic, and inhalation should be avoided. Hydroiodic acid is corrosive and can be absorbed through the skin; wear gloves when handling.

Procedure: Into a suitable reaction flask (quipped with motorized stirrer or other stirring means—see the following illustration), and gas inlet tube, place 32 grams (1.1 oz.) of iodine crystals, followed by 36 milliliters (1.2 fluid oz.) of water. Then place this reaction flask into an ice bath, and chill to about 0 Celsius. Thereafter, bubble into this iodine mixture, 5 grams (0.17 oz.) of hydrogen sulfide gas. During the addition of the hydrogen sulfide gas, rapidly stir the iodine mixture and maintain its temperature at 0 Celsius. After the addition of the hydrogen sulfide gas, continue to rapidly stir the entire reaction mixture for about 30 minutes at 0 Celsius, and then filter-off the precipitated sulfur. Now, add to this filtered reaction mixture, 5 grams

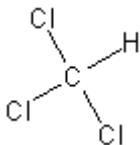

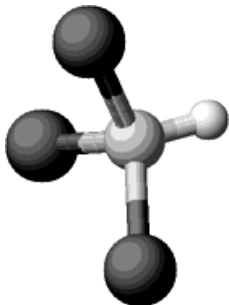
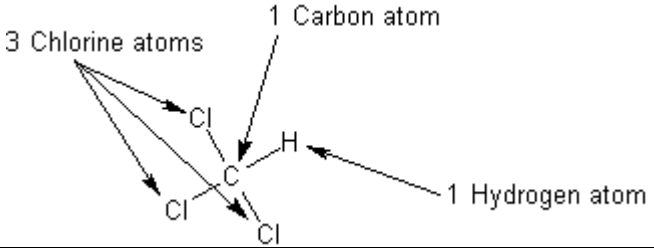
(0.17 oz.) of iodine crystals, and then store this reaction mixture (which will be the 47% hydroiodic acid) in a refrigerator at 5 Celsius until use.



Set-up for the preparation of concentrated hydroiodic acid.

Procedure 33: The preparation of Chloroform

Also known as: Trichloromethane, formyl trichloride

	 <p>3D structure 1</p>  <p>3D structure 2</p>
<p>Chemical structure</p>	<p>3D Structure(s)</p>
	<p>CHCl₃</p>
<p>Structure make-up</p>	<p>Condensed chemical structure</p>

Chloroform

Chloroform is a highly refractive, nonflammable, heavy, very volatile, and sweet-tasting liquid with a peculiar odor. It has a boiling point of 62 Celsius, and a melting point of -64 Celsius. Chloroform forms a constant boiling mixture with alcohol containing 7% alcohol, and boiling at 59 Celsius. Commercial chloroform contains a very small amount of ethanol as stabilizer. It is insoluble in water, but miscible with alcohol, benzene, ether, petroleum ether, and carbon disulfide. Pure chloroform is light sensitive, so store in amber glass bottles in a cool place. Chloroform is a suspected light carcinogen, so use proper ventilation when handling. Over exposure to chloroform vapors causes dizziness, and headache. **Note:** Distilling mixtures containing chloroform mixed with one or more strong base (lithium, sodium, or potassium hydroxide) can result in explosion or violent reaction. Always neutralize any base, or extract the chloroform before distilling.

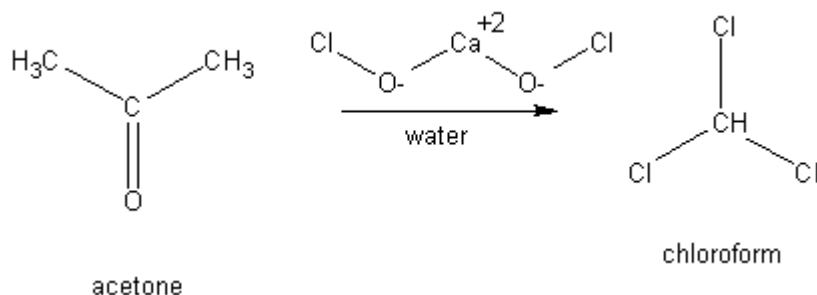
Method 1: Preparation of chloroform from acetone and bleaching powder

(By-products from reaction: Calcium acetate, calcium chloride, and calcium hydroxide)

Materials:

1. 100 milliliters of tap water (3.4 fluid oz.).	3. 1 milliliter of 95% ethyl alcohol (see entry).
2. 100 grams of acetone (3.5 oz.). Readily available in any hardware store.	4. 300 milliliters (10.1 fluid oz.) of benzene, toluene, or xylene. Toluene and xylene should be available at most hardware stores.
2. 1181 grams (2.6 pounds) of 65 to 70% calcium hypochlorite (bleaching powder) (commercially available; sold under a variety of brand names for use in swimming pools and hot tubs). Can also be found online at many pool and spa chemical suppliers and can be purchased without hassles.	5. 15 grams of anhydrous magnesium sulfate (obtained by heating Epsom salt in an oven, microwave, or Bunsen burner).

Reaction summary: Chloroform is prepared by reacting acetone with calcium hypochlorite (bleaching powder), and then extracting the mixture with benzene, toluene, or xylene. After extraction, the solvent/chloroform mixture is then distilled to collect the chloroform, which is then re-distilled. After collecting the chloroform after re-distillation, it is mixed with a small amount of 95% ethanol to act as a stabilizing agent.



Why does this reaction happen? The reaction of acetone with a strong oxidizer such as calcium hypochlorite is a well-known reaction, but the mechanism for the reaction is rather complex. In short, the reaction takes place because the oxygen of the acetone prefers to bond with the calcium of the calcium hypochlorite, and the chlorine atoms of the hypochlorite prefer to bond with a single carbon from the acetone.

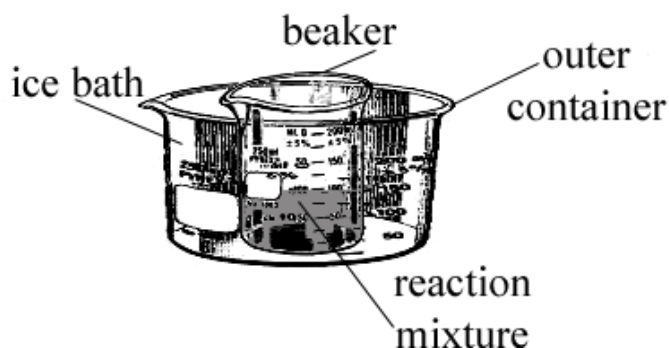
Hazards: Extinguish all flames before using acetone, which is highly volatile and flammable. *Calcium hypochlorite is a powerful oxidizer, and should never be mixed with concentrated sulfuric acid; explosions will result.* Chloroform inhalation should be avoided, but is not threatening in mild conditions. Benzene, toluene, and xylene are suspected carcinogens so avoid prolonged exposure to fumes and vapors.

Procedure: Place 100 milliliters of tap water (3.4 fluid oz.) and 100 grams of acetone (3.5 oz.) into a beaker or any suitable container, and then cool this mixture to 0 Celsius using a standard ice bath. Thereafter, slowly add in small portions, 1181 grams (2.6 pounds) of 65 to 70% calcium hypochlorite (commercially available; sold under a variety of brand names for use in swimming pools and hot tubs) over a period of 1 hour while stirring the acetone solution and maintaining its temperature at 0 Celsius. During the addition of the calcium hypochlorite, rapidly stir the acetone/water mixture, and maintain its temperature

below 20 Celsius. After the addition of the 65 to 70% calcium hypochlorite, continue to stir the reaction mixture at 0 Celsius for an additional thirty minutes. Afterwards, stop stirring and then extract the reaction mixture with four 75-milliliter portions (four 2.5 fluid oz. portions) of benzene, toluene, or xylene. After extraction, combine all four portions (if not already done so), and then dry this combined solvent portion by adding to it, 15 grams of anhydrous magnesium sulfate (to absorb water). After adding in the magnesium sulfate, stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Thereafter, place this filtered dried solvent portion into a distillation apparatus (as illustrated below) and then distill at 65 Celsius until no more chloroform passes into the receiver flask. When no more chloroform passes into the receiver flask, stop the distillation, and then remove the receiver flask from the distillation apparatus. Then place this distilled crude chloroform in a clean fractional distillation apparatus (as illustrated below) and distill at 62 Celsius until no more chloroform passes into the receiver flask. When no more chloroform passes into the receiver flask, stop the distillation, and then remove the chloroform from the receiver flask and then add to it, 1 milliliter of 95% ethyl alcohol. Then store this chloroform in an amber glass bottle in a cool dry place.

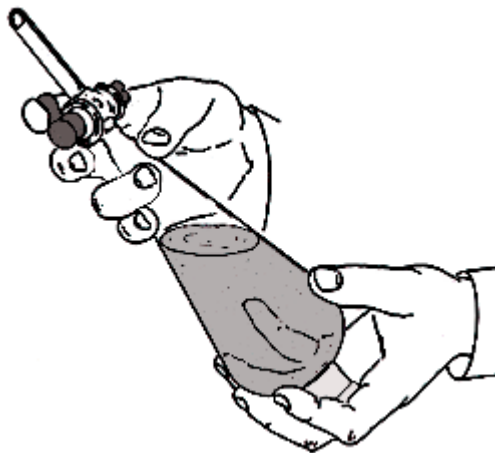
Note: the benzene, toluene, or xylene used in the extraction, can be recovered after the first distillation process.

Step 1: Initial reaction of calcium hypochlorite with acetone.



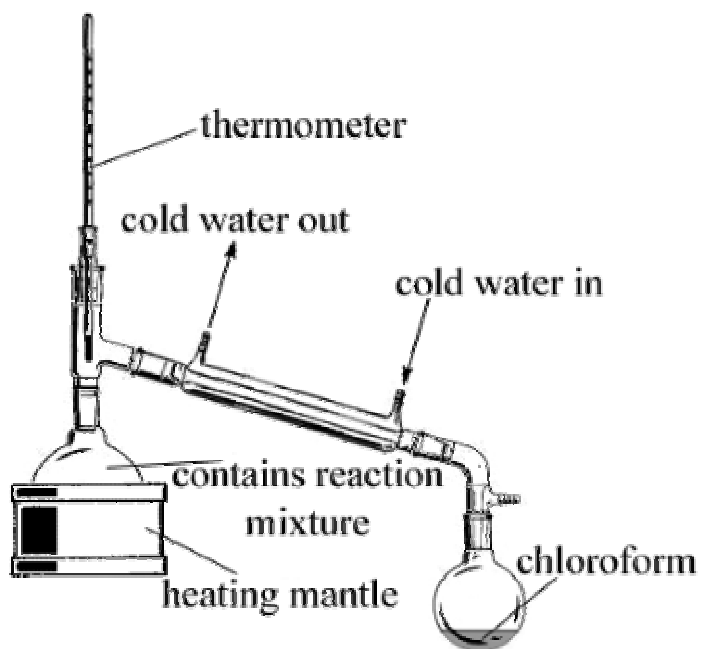
Set-up with ice bath for cooling the reaction mixture. The beaker can be re-placed with a flask or other suitable container. However, the container should not be made of plastic or other polymer that may corrode or dissolve by the acetone or chloroform. The outer container for use as the ice bath, can be glass, plastic, metal, or any other similar container.

Step 2: Extraction process



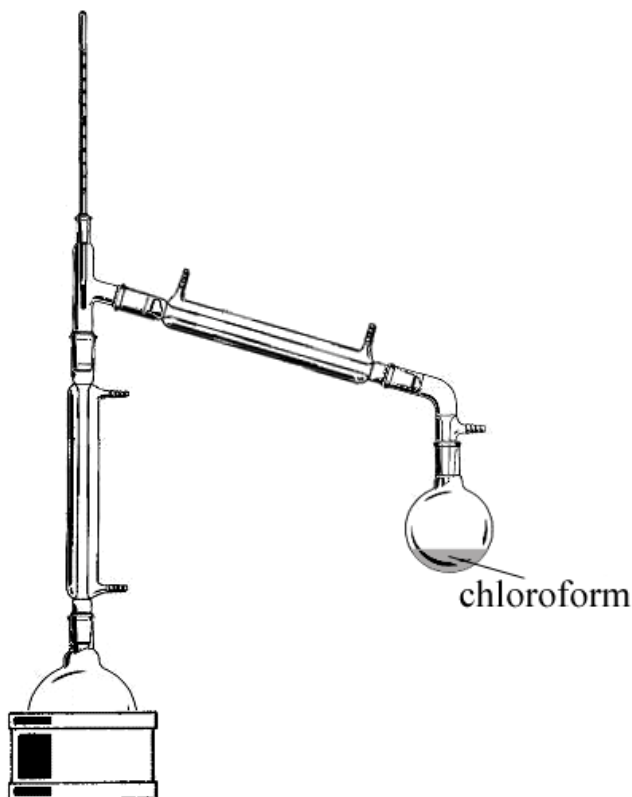
Extract the reaction mixture with benzene, toluene, or xylene.

Step 3: Distillation process to recover the chloroform



Distillation apparatus for collecting the chloroform. The heating mantle can be replaced with a Bunsen burner, but the flame should not come into direct contact with the distillation flask (as bumping and foaming may result). The chloroform should be re-distilled using a fractional distillation apparatus for quality and purity.

Step 4: Fractional distillation apparatus for purifying the chloroform



Fractional distillation apparatus for the fractional distillation of chloroform. The heating mantle can be replaced with a Bunsen burner, but the flame should not touch the glass. A hot plate or stovetop can also be used as a heat source if desired.

Final note for method 1

The by-products of calcium acetate, calcium chloride, and calcium hydroxide can be recovered as follows:

1. Filter the extracted reaction mixture (after the extraction process), to filter-off the insoluble calcium hydroxide.
2. Recover the acetate by treating the filtered reaction mixture with dilute sulfuric acid, and then filter-off the precipitated calcium sulfate. Then, distill the mixture at 110 Celsius to recover the acetic acid formed by the addition of sulfuric acid. **Note:** if using hydrochloric acid instead of sulfuric acid, evaporate the left over reaction mixture to dryness after the distillation (to remove acetic acid), so as to recover the calcium chloride. Calcium chloride can be heated using a Bunsen burner so as to form anhydrous calcium chloride, which makes a powerful drying agent. Second note: acetic acid is a useful by-product and can be used in a variety of applications.

Note: If desired, the bleaching powder can be replaced with Clorox bleach or other Clorox like bleaches (that contain sodium hypochlorite only); however, because most bleach products only contain 5% or less of sodium hypochlorite it would take astronomical amounts of bleach to carryout the reaction, but nonetheless, if you would like to try this technique—by all means; just remember to extract the entire huge reaction mixture with extra amounts of benzene, toluene, or xylene to properly recover all of the chloroform)

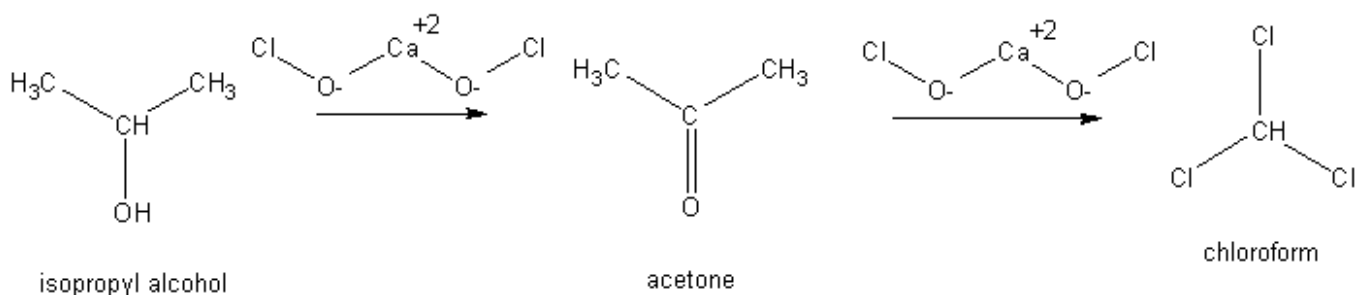
Method 2: Preparation of chloroform from Rubbing alcohol and bleaching powder

(By-products from reaction: Calcium acetate, calcium chloride, and calcium hydroxide)

Materials:

1. 100 milliliters of tap water (3.4 fluid oz.)	4. 300 milliliters (10.1 fluid oz.) of benzene, toluene, or xylene. Toluene and xylene should be available at most hardware stores.
2. 150 grams (5.3 oz.) of 70% rubbing alcohol (just regular rubbing alcohol, no fancy colored stuff or fancy additives—just plain old rubbing alcohol).	5. 40 to 50 grams (1.4 to 1.8 oz.) of pickling salt (available in any grocery store).
2. 1361 grams (3 pounds) of 65 to 70% calcium hypochlorite (commercially available; sold under a variety of brand names for use in swimming pools and hot tubs	6. 15 grams of anhydrous magnesium sulfate
3. 1 milliliter of 95% ethyl alcohol	

Reaction summary: Chloroform can be made in an identical manner as in method 1; however, we replace the acetone with rubbing alcohol. First of all, the rubbing alcohol needs to be purified, so the salting out process is used. After the salting out process, the refined alcohol, also known as isopropyl alcohol, is then reacted with bleaching powder in a familiar manner. The bleaching powder first reacts with the alcohol, oxidizing it to acetone, from where additional bleaching powder then converts it into chloroform.



Why does this reaction happen? This reaction is similar to the one in method 1, but we start with alcohol instead of acetone. First of all the alcohol gets oxidized to acetone, and then the reaction proceeds from there to form the chloroform.

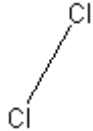

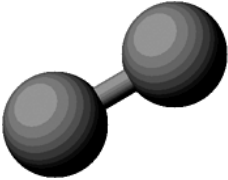
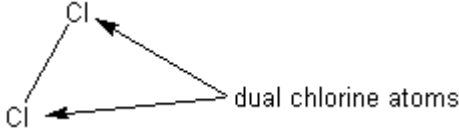
Hazards: *Calcium hypochlorite is a powerful oxidizer, and should never be mixed with concentrated sulfuric acid; explosions will result.* Chloroform inhalation should be avoided, but is not threatening in mild conditions. Benzene, toluene, and xylene are suspected carcinogens so avoid prolonged exposure to fumes and vapors.

Procedure: Into a suitable separatory funnel, place 150 grams (5.3 oz.) of 70% rubbing alcohol (just regular rubbing alcohol, no fancy colored stuff or fancy additives—just plain old rubbing alcohol), followed by 40 to 50 grams (1.4 to 1.8 oz.) of pickling salt. Note: make sure the stopcock on the separatory funnel is closed before starting. Thereafter, stopper the separatory funnel, and then shake the entire funnel vigorously for about 5 minutes. Then allow the separatory funnel to stand (upright) for about 30 minutes. Thereafter, drain-off the bottom brine layer (and any excess salt), and thereafter, recover the upper isopropyl alcohol layer. Now, place this recovered upper isopropyl alcohol layer into a suitable container, and then add in 100 milliliters (3.4 fluid oz.) of tap water, and then cool this mixture to 0 Celsius using a standard ice bath. Thereafter, slowly add in small portions, 1361 grams (3 pounds) of 65 to 70% calcium hypochlorite (commercially available; sold under a variety of brand names for use in swimming pools and hot tubs) over a period of 1 hour while stirring the alcohol solution and maintaining its temperature at 0 Celsius. During the addition of about 200 to 300 grams of calcium hypochlorite, rapidly stir the now acetone/water mixture, and maintain its temperature below 20 Celsius. After the addition of the rest of the 65 to 70% calcium hypochlorite, continue to stir the reaction mixture at 0 Celsius for an additional thirty minutes. Afterwards, stop stirring and then extract the reaction mixture with four 75-milliliter portions (four 2.5 fluid oz. portions) of benzene, toluene, or xylene. After the extraction, combine all four portions (if not already done so), and then dry this combined solvent portion by adding to it, 15 grams of anhydrous magnesium sulfate (to absorb water). After adding in the magnesium sulfate, stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Thereafter, place this filtered dried solvent portion into a distillation apparatus (similar to the one in method 1) and then distill at 65 Celsius until no more chloroform passes into the receiver flask. When no more chloroform passes into the receiver flask, stop the distillation, and then remove the receiver flask from the distillation apparatus. Then place this distilled crude chloroform into a clean fractional distillation apparatus (as similar to the one in method 1) and distill at 62 Celsius until no more chloroform passes into the receiver flask. When no more chloroform passes into the receiver flask, stop the distillation, and then remove the chloroform from the receiver flask and then add to it, 1 milliliter of 95% ethyl alcohol. Then store this chloroform in an amber glass bottle in a cool dry place. See method 1 for the illustrations on extraction, distillation, ect.,

Note: the benzene, toluene, or xylene used in the extraction, can be recovered after the first distillation process.

Procedure 34: The Preparation of Chlorine gas (non electrochemical preparation)

Also known as: Halogen chlorine

	 3D structure 1  3D structure 2
Chemical structure	3D Structure
	Cl ₂
Structure make-up	Condensed chemical structure

Chlorine

Chlorine gas is a yellow gas with a suffocating, and strongly irritating odor. It has a melting point of -101 Celsius, and a boiling point of -34 Celsius. Chlorine is sold as a compressed gas in steel cylinders. It is insoluble in water and not very soluble in alcohol, but soluble in dry benzene, and toluene. Chlorine combines readily with all elements except the noble gases, hydrogen, oxygen, and nitrogen. Chlorine does not occur naturally, but occurs in combined form as chlorides. It occurs in nature (in the form of chlorides) as sodium chloride, potassium chloride, and magnesium chloride. Many finely divided metals will burn in a chlorine atmosphere. Chlorine is a toxic gas, which can be fatal if inhaled for prolonged periods of time. Inhalation of mild quantities of chlorine causes nose and throat irritation followed by excessive mucous congestion. Chlorine is a corrosive gas, which will react with many metals on contact. It is a strong oxidizer and is capable of oxidizing a great many inorganic compounds. Chlorine will explode in contact with hydrogen if direct sunlight is present. Chlorine should be protected from sunlight. It is prepared on an industrial scale from the electrolyses of sodium chloride brine in a system called the chloro-alkali process (sodium hydroxide is a useful by-product). It can be prepared in the lab by reacting hydrochloric acid with calcium hypochlorite or any other strong oxidizer.

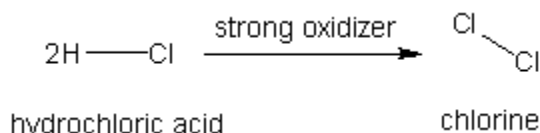
Method 1: Preparation of chlorine gas from hydrochloric acid and an oxidizing salt

(By-products from reaction: metal oxides, metal chlorides, and water)

Materials:

1. 75 grams (2.6 oz.) of manganese dioxide, or 1200 grams (2.6 pounds) of a 5% sodium hypochlorite solution (Clorox bleach), or 65 grams (2.3 oz.) of potassium permanganate, or 95 grams (3.3 oz.) of calcium hypochlorite, bleaching powder (65% available chlorine)	3. 200 milliliters (6.7 fluid oz.) of tap water
2. 150 grams (5.3 oz.) of 35 to 38% hydrochloric acid (Muriatic acid of 31% will work)	

Reaction summary: Chlorine gas is prepared by dripping hydrochloric acid onto a strong oxidizing salt. During the reaction, chlorine gas is steadily evolved.



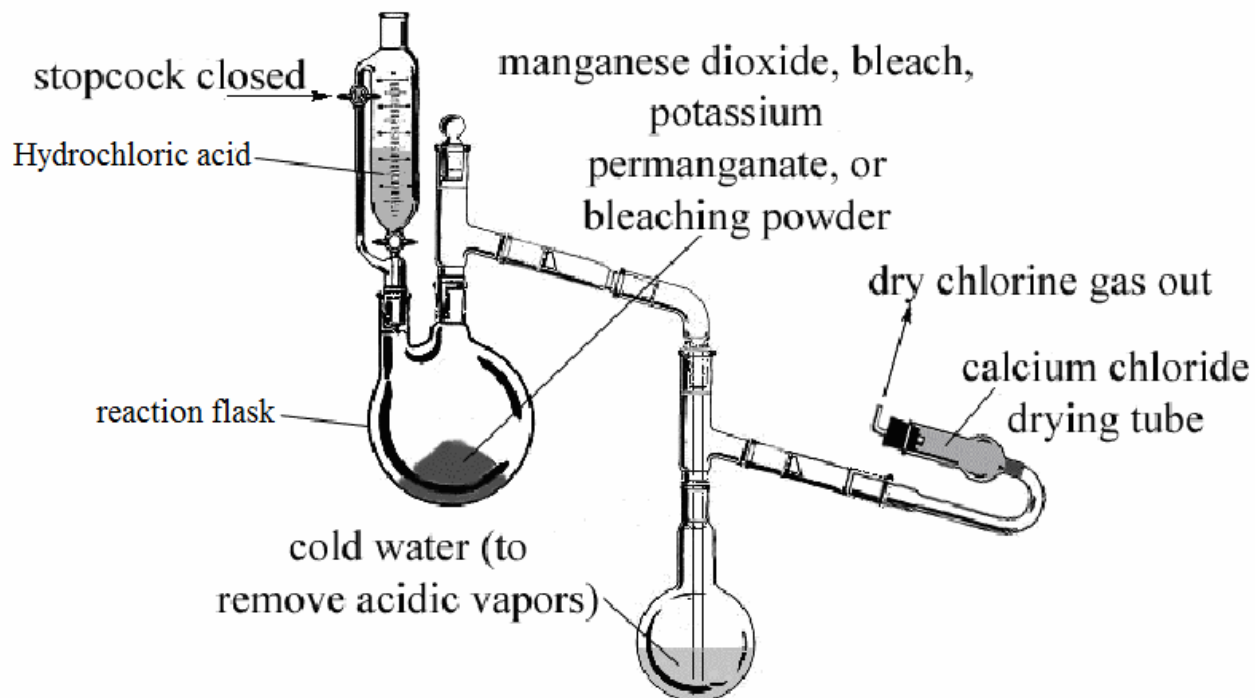
Why does this reaction happen? This reaction takes place because hydrogen chloride gets oxidized when contacted with a strong oxidizer such as manganese dioxide, a permanganate, bleach, or bleaching powder. The strong oxidizer oxidizes the hydrochloric acid forming chlorine gas. At the same time, the oxidizer gets reduced by the hydrochloric acid.

Hazards: Use care when handling strong oxidizers like manganese dioxide, potassium permanganate, sodium hypochlorite, and calcium hypochlorite, as they are all highly reactive and capable of reacting with many organic substances. Mixtures of strong oxidizers and combustible materials can ignite and burn violently. Wear gloves when handling hydrochloric acid, which can cause skin irritation. Chlorine is toxic, so handle with care and use caution.

Procedure: Setup the chlorine generating apparatus as illustrated below. Note: your chlorine generator does not have to be as fancy as the apparatus illustrated below, but your system should be similar in design. You can use latex tubing, PVC pipes and other plastic devices; as well, steel pipes can be used, but other metals should be avoided due to corrosion.

After you have the chlorine generator set-up, place into the reaction flask, 75 grams (2.6 oz.) of manganese dioxide, or 1200 grams (2.6 pounds) of a 5% sodium hypochlorite solution (Clorox bleach), or 65 grams (2.3 oz.) of potassium permanganate, or 95 grams (3.3 oz.) of calcium hypochlorite, bleaching powder (65% available chlorine). Then place into the addition funnel, a hydrochloric acid solution prepared by adding and dissolving 150 grams (5.3 oz.) of 35 to 38% hydrochloric acid (Muriatic acid of 31% will work) into 200 milliliters (6.7 fluid oz.) of tap water. Note: If using Clorox bleach, avoid brands that contain sodium hydroxide—look on the label to find out if your bleach has sodium hydroxide in it. Sodium hydroxide will screw-up the reaction, which is the main reason the companies add it in!

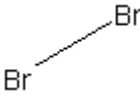
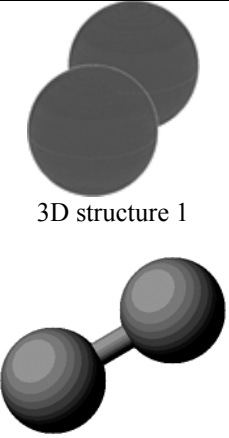
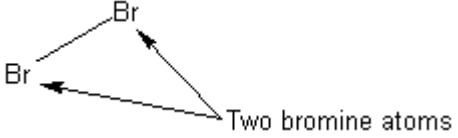
Once your chlorine generator is setup, begin slowly dripping the hydrochloric acid solution onto the oxidizer contained in the reaction flask. During the reaction, chlorine gas will be steadily evolved. Note: Obviously, you should have another apparatus set-up for reacting the chlorine with something.



Setup for the generation of chlorine gas.

Procedure 35: The Preparation of Bromine

Also known as: Halogen bromine

	 <p>3D structure 1</p> <p>3D structure 2</p>
Chemical structure	3D Structure
 <p>Two bromine atoms</p>	Br ₂
Structure make-up	Condensed chemical structure

Bromine

Bromine is a dark red, highly fuming liquid, which is very volatile. Its fumes are toxic, corrosive, and strongly irritating. Bromine has a melting point of -7 Celsius, and a boiling point of 59 Celsius. It is insoluble in water, but freely soluble in alcohol, ether, chloroform, and carbon disulfide. It is soluble in alkali bromide solutions. Bromine is less reactive than chlorine, but just as toxic. Keep bromine stored in glass stoppered bottles, and store in a cool place (refrigerator) away from sunlight. Bromine is prepared by passing chlorine gas into a solution of sodium bromide, and then simultaneously evaporating-off the bromine. The bromine vapors are then condensed. Bromine is commercially available but shipping regulations may restrict its sale.

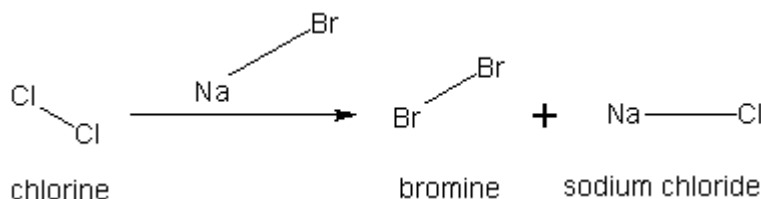
Method 1: Preparation of bromine from sodium bromide and chlorine

(By-products from reaction: sodium chloride or potassium chloride, and small amounts of monobromochloride – ClBr -)

Materials:

1. 36 grams (1.3 oz.) of potassium bromide or 31 grams of (1.09 oz.) of sodium bromide	5. 40 milliliters (1.35 fluid oz.) of tap water
2. 150 milliliters (5 oz.) of tap water	6. 40 milliliters (1.4 fluid oz.) of concentrated hydrochloric acid
3. 15 grams (1/2 oz.) of manganese dioxide, or 240 grams (8.5 oz.) of a 5% sodium hypochlorite solution (Clorox bleach), or 13 grams (1/2 oz.) of potassium permanganate, or 19 grams (0.67 oz.) of calcium hypochlorite, bleaching powder (65% available chlorine). Note: if using bleach, some recent brands of bleach now contain sodium hydroxide. Avoid these brands of bleach as they will disrupt the reaction. Generic bleach may work best.	7. 200 milliliters (7 fluid oz.) of cold tap water
4. 30 grams (1 oz.) of 35 to 38% hydrochloric acid (Muriatic acid of 31% will work)	

Reaction summary: bromine is readily prepared by bubbling chlorine gas into a solution of sodium or potassium bromide in water. The reaction is very mild, and liquid bromine is steadily evolved, forming at first, a deep red liquid that gives off red fumes. The bromine vapors are carried over and condense into liquid bromine.



Why does this reaction happen? Bromine is readily prepared by reacting chlorine gas with sodium or potassium bromide. The reason why the reaction takes place is simply because the chlorine is more reactive than bromine. Therefore, the chlorine acts as an oxidizer towards the bromine, liberating it from the sodium bromide. The chlorine has a greater attraction towards the sodium of the sodium bromide. The chlorine reacts with the sodium of the sodium bromide, liberating the bromine in free state.

Hazards: Use maximum ventilation when performing this procedure as both chlorine and bromine are toxic and very irritating to the eyes, nose, and throat—use caution.

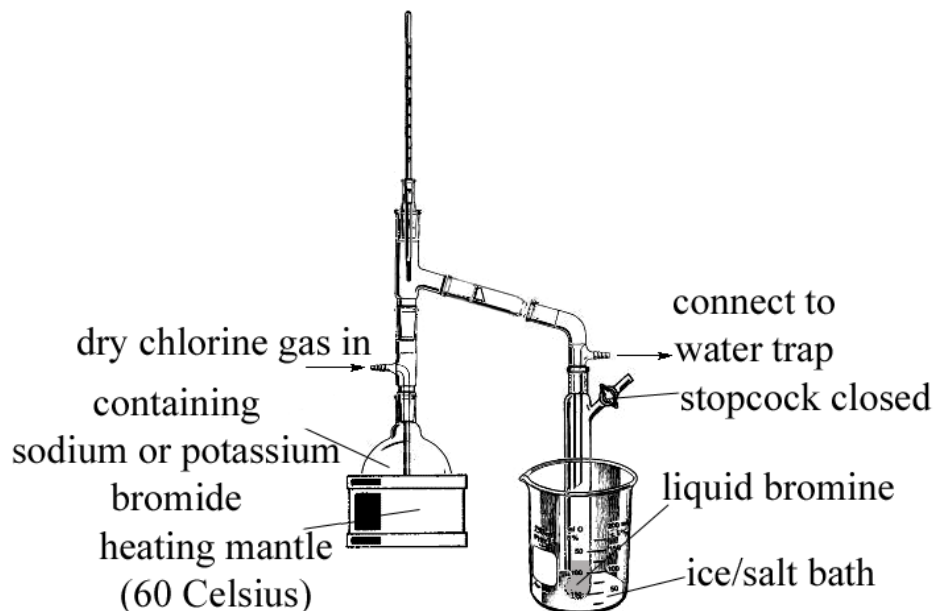
Procedure: Setup the chlorine generating apparatus as illustrated in preparation of chlorine. Note: your chlorine generator does not have to be as fancy as the apparatus illustrated, but your system should be similar in design. You can use latex tubing, PVC pipes and other plastic devices; as well, steel pipes can be used, but other metals should be avoided due to corrosion.

Now, into a suitable beaker, flask, or container, place 36 grams (1.3 oz.) of potassium bromide or 31 grams of (1.09 oz.) of sodium bromide, followed by 150 milliliters (5 oz.) of tap water. Thereafter, stir the entire mixture for about 30 minutes to dissolve all solids.

After you have the chlorine generator set-up (just like in the preparation of chlorine), place into the reaction flask, 15 grams (1/2 oz.) of manganese dioxide, or 240 grams (8.5 oz.) of a 5% sodium hypochlorite solution (Clorox bleach), or 13 grams (1/2 oz.) of potassium permanganate, or 19 grams (0.67 oz.) of calcium hypochlorite, bleaching powder (65% available chlorine). Then place into the addition funnel, a hydrochloric acid solution prepared by adding and dissolving 30 grams (1 oz.) of 35 to 38% hydrochloric acid (Muriatic acid of 31% will work) into 40 milliliters (1.35 fluid oz.) of tap water.

Once your chlorine generator is setup, begin dripping the hydrochloric acid solution, onto the oxidizer contained in the reaction flask. The chlorine gas that is evolved should then be sent to the apparatus as illustrated below for the preparation of bromine. The chlorine gas will react with the potassium or sodium bromide forming bromine. When no more chlorine gas passes over, the reaction is complete.

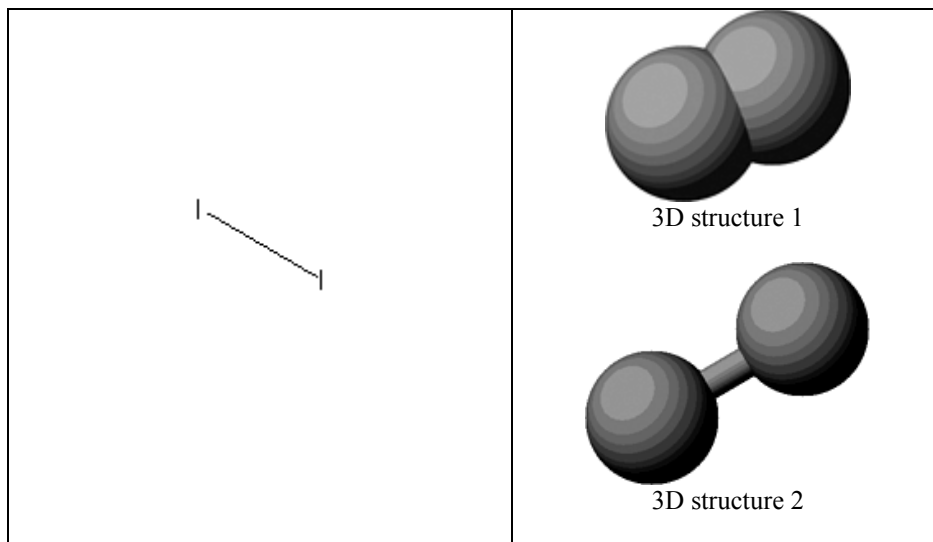
After the initial reaction, the collected liquid bromine should be fractionally distilled to free it from dissolved gases and moisture. The distilled bromine should then be stored in amber glass bottles in a cool place.

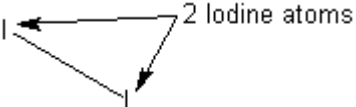


Apparatus for preparing bromine. The bromine should be fractionally distilled at 60 Celsius. Note: the apparatus should be connected to a water trap containing a sodium carbonate solution to neutralize any bromine vapors that may escape. Second note: This apparatus is merely an example, and is not intended to be the "exact" set-up.

Procedure 36: The preparation of Iodine

Also known as: Halogen iodine



Chemical structure	3D Structure
	I ₂
Structure make-up	Condensed chemical structure

Iodine

Iodine forms blackish to purplish-black plates, powder, or granules with a characteristic metallic luster and peculiar odor. It forms a purple vapor when gently heated, and readily volatilizes. It has a melting point of 114 Celsius and boiling point of 185 Celsius. Iodine is soluble in aqueous solutions of potassium or sodium iodide, and is soluble benzene, ethyl alcohol, ether, cyclohexane, and methylene chloride. Iodine is capable of producing eye, nose, and throat irritation so wear gloves when handling and use ventilation. Iodine is readily obtainable by bubbling chlorine gas into a solution of potassium or sodium iodide at room temperature, then filtering-off the precipitated iodine, followed by sublimation to purify the iodine. Iodine can also be obtained by treating solutions of potassium or sodium iodide with bleaching powder, potassium permanganate solutions, or with excess bleach (Clorox), then filtering-off the precipitated iodine crystals, followed by sublimation to purify the iodine.

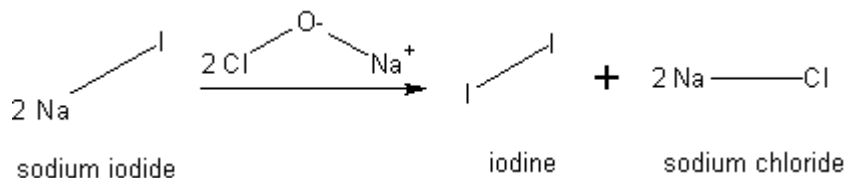
Method 1: Preparation of Iodine from potassium or sodium iodide and bleach

(By-products from reaction: sodium or potassium chloride)

Materials:

1. 50 grams (1.7 oz) of potassium iodide or 45 grams (1.6 oz) of sodium iodide (available on line).	4. 225 milliliters of methylene chloride (dichloromethane); (7.6 fluid oz.) (available on on-line)
2. 150 milliliters (5 fluid oz.) of cold tap water	5. 950 milliliters (17 fluid oz.) of warm tap water
3. 500 milliliters (17 fluid oz.) of regular household bleach (Clorox bleach).	6. 15 grams (1/2 oz.) of anhydrous magnams sulfate

Reaction summary: Iodine is readily prepared by reacting bleach with potassium or sodium iodide. The reaction is relatively swift, and produces a brownish black precipitate of crude iodine. This crude iodine can be obtained by filtration using gravity or vacuum filtration, or extracted into methylene chloride. The iodine if filtered, is washed and then dried, and then sublimed. If the reaction mixture was extracted, the methylene chloride is removed by distillation, and the resulting left over iodine is then sublimed in the same fashion. **Note: some recent brands of bleach now include sodium hydroxide. Do not buy this brand of bleach, as it will interfere with the reaction. Check the ingredients label of your bleach before purchasing. In some cases, cheap generic bleach is the better product.**



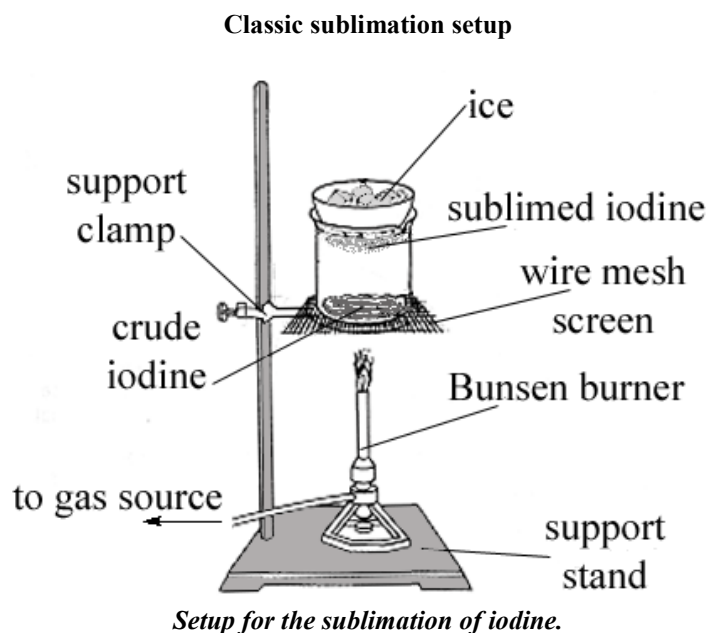
Why does this reaction happen? As with other oxidizing reactions, potassium or sodium iodide is readily oxidized with strong oxidizers such as bleach liberating free iodine. The reaction is swift and takes place primarily because the chlorine of the bleach prefers to bond with the sodium of the sodium iodide.

Hazards: Sodium hypochlorite (bleach) is a strong oxidizer and should be kept away from combustible liquids. Iodine is an irritant, so avoid skin contact, and inhalation of fumes or vapors.

Procedure: Into a beaker or other suitable container add 50 grams (1.7 oz) of potassium iodide or 45 grams (1.6 oz) of sodium iodide, followed by 150 milliliters (5 fluid oz.) of cold tap water. Thereafter, stir the entire mixture for several minutes to dissolve all solids. Thereafter, add in 500 milliliters (17 fluid oz.) of regular household bleach (Clorox bleach). Make sure the bleach is just regular household bleach and has no fancy additives, colors, or the like. The rate of addition of the household bleach to the potassium or sodium iodide solution should be slow, but should only take about 15 to 30 minutes. During the addition of the household bleach to the potassium or sodium iodide solution, casually stir the potassium or sodium iodide solution. After the addition of the household bleach, continue to stir the mixture for about 30 minutes, and then do one of two things: A. filter the mixture to recover the brownish black precipitate formed (crude iodine) using either gravity filtration or vacuum filtration, or B. extract the mixture with methylene chloride (three 75-milliliter portions, three 2.5 fluid oz. portions). If

filtering the reaction mixture, allow the brownish precipitate of iodine to collect on the filter paper, and thereafter, allow the filter paper to stand over night and partially dry, or if using vacuum filtration, vacuum dry the brownish precipitate. Thereafter, gently scrape-off the brownish precipitate from the filter paper, into a clean beaker or other suitable container, and then add in 500 milliliters (17 fluid oz.) of warm tap water. Then gently mix the iodine mixture for about 10 minutes, and then once again, filter-off the insoluble iodine using gravity filtration (this is called washing the iodine with water). Note: if using vacuum filtration, after vacuum drying the initial iodine after filtration, simply wash it several times with three 150-milliliter portions (three 5 fluid oz. portions), of warm tap water, and then vacuum dry the iodine. If using gravity filtration, after washing the iodine, and then collecting the iodine by filtration, allow the filter paper containing the iodine to stand over night to partially dry. The next day, gently scrape-off the iodine from the filter paper onto a clean piece of glass or porcelain dish (no metal), and then set this dish aside (no sunlight) for several days to allow the iodine to dry. When the iodine has been dried, either by using vacuum filtration, or by using the slow process of air-drying, place the dry iodine into a sublimation apparatus, as illustrated below, and sublime the iodine to purify it. This sublimation process should be repeated once more to produce what is called "double sublimed reagent grade iodine", which will have a purity of no less then 98% iodine

If you extracted the reaction mixture with methylene chloride, combine all methylene chloride portions once finished (if not already done so), and then dry this combined methylene chloride portion by adding to it, 15 grams (1/2 oz.) of anhydrous magnesium sulfate. Then stir the entire mixture for about 10 minutes (to absorb moisture), and then filter-off the magnesium sulfate. Once the magnesium sulfate has been filtered-off, place this methylene chloride into a standard atmospheric distillation apparatus, and gently distill-off the methylene chloride at 40 Celsius (104 Fahrenheit) until no more methylene chloride distills over. When this is the result, recycle the methylene chloride, and then recover the left over remaining brownish residue of iodine. Then place this recovered iodine into a sublimation apparatus, as illustrated below, and sublime the iodine to purify it. This sublimation process should be repeated once more to produce what is called "double sublimed reagent grade iodine", which will have a purity of no less then 98% iodine



Final note for method 1: None

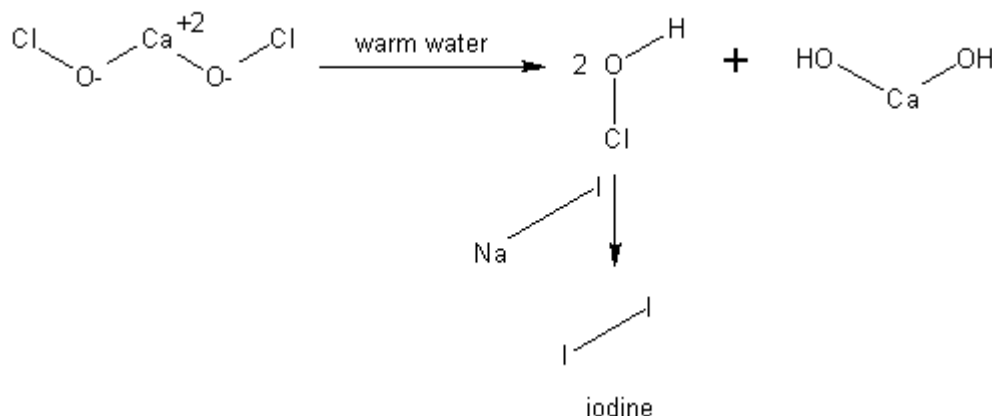
Method 2: Preparation of Iodine from potassium or sodium iodide and bleaching powder

(By-products from reaction: calcium hydroxide, and potassium or sodium chloride)

Materials:

1. 50 grams (1.7 oz) of potassium iodide or 45 grams (1.6 oz) of sodium iodide	3. 70 grams (2.5 oz.) of 65 to 70% calcium hypochlorite (bleaching powder) (commercially available; sold under a variety of brand names for use in swimming pools and hot tubs). Can also be found online at many pool and spa chemical suppliers and can be purchased without hassles.
2. 250 milliliters (15 fluid oz.) of warm tap water	4. 200 milliliters (6.8 fluid oz.) of tap water

Reaction summary: Iodine can be prepared in a modified process whereby bleaching powder is used instead of Clorox bleach. The reaction is similar, but first, a solution of hypochlorous acid is prepared by simply mixing the bleaching powder with warm water. The warm water decomposes the bleaching powder into hypochlorous acid and calcium hydroxide. This mixture is then filtered, and the resulting hypochlorous acid is then reacted with a solution of potassium or sodium iodide. After the reaction, the precipitated iodine is then recovered by filtration, washed, dried, and then sublimed to yield high purity iodine.



Why does this reaction happen? As in the previous reaction, sodium or potassium iodide is oxidized using bleach. In this reaction we use hypochlorous acid generated by mixing bleaching powder with warm water. The reaction simply takes place because the chlorine of the hypochlorous acid prefers to bond with the sodium of the sodium iodide.

Hazards: *Calcium hypochlorite is a powerful oxidizer, and should never be mixed with concentrated sulfuric acid; explosions will result. Do not mix hypochlorous acid with ammonia or ammonium salts—as the high explosive nitrogen triiodide is formed.*

Procedure: Into a suitable beaker or container, place 250 milliliters (8.4 fluid oz.) of warm tap water, followed by 70 grams (2.5 oz.) of bleaching powder. Thereafter, stir the entire mixture for about 1 hour, and then filter-off the precipitated solids (composed of calcium hydroxide). Note: vacuum filtration works best, but if all you have is gravity filtration, place a small amount of clean sand in the bottom of the filter paper before filtering. This will help aid in the filtration process, which can be a slow one. After the filtration process, you will have a yellowish relatively odorless liquid. This yellow liquid will be composed of hypochlorous acid. Now, prepare a potassium or sodium iodide solution by adding and dissolving 50 grams (1.7 oz.) of potassium iodide, or 45 grams (1.6 oz.) of sodium iodide into 200 milliliters (6.8 fluid oz.) of tap water, and then slowly add this iodide solution to your yellowish hypochlorous acid mixture over a period of about 10 to 15 minutes. After 10 to 15 minutes, stir the mixture for about 30 minutes, and then filter-off the precipitated brownish solids (composed of the crude iodine). Once the brown solids have been filtered-off, wash them with several portions of cold water (use the same technique as in method 1 for washing), and then vacuum dry or air-dry these brownish solids. Thereafter, place this dried iodine into a sublimation apparatus, as illustrated in method 1, and then sublime the iodine to purify it. This sublimation process should be repeated once more to produce what is called “double sublimed reagent grade iodine”, which will have a purity of no less than 98% iodine.

Final note for method 2: None

Method 3: Preparation of Iodine from potassium or sodium iodide and chlorine gas

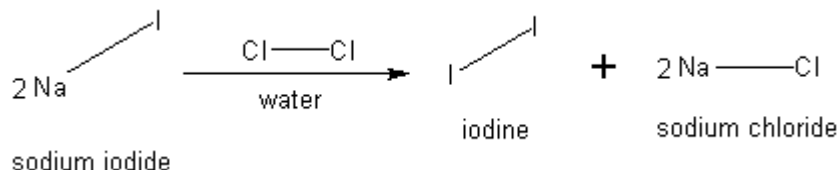
(By-products from reaction: calcium hydroxide, and potassium or sodium chloride)

Materials:

1. 50 grams (1.7 oz) of potassium iodide or 45 grams (1.6 oz) of sodium iodide.	4. 30 grams (1 oz.) of 35 to 38% hydrochloric acid (Muriatic acid of 31% will work). Sold on various online lab supply stores.
2. 240 milliliters (8 fluid oz.) of tap water	5. 40 milliliters (1.4 fluid oz.) of concentrated hydrochloric acid. (Available in hardware stores as Muriatic acid (at least 31% by weight—read label).
3. 15 grams (1/2 oz.) of manganese dioxide, or 240 grams (8.5 oz.) of a 5% sodium hypochlorite solution (Clorox	

bleach), or 13 grams (1/2 oz.) of potassium permanganate, or 19 grams (0.67 oz.) of calcium hypochlorite, bleaching powder (65% available chlorine). All are available on on-line auction sites, or other on line sites.	
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Reaction summary: Iodine can be prepared from chlorine gas instead of household bleach or bleaching powder, but it requires a more elaborate setup, and time. Nevertheless, it can be used to prepare large quantities of iodine (to produce more iodine, simply double any mass or liquid quantities involved). The chlorine gas used in the operation can be prepared by dripping hydrochloric acid onto a strong oxidizer. The liberated chlorine gas, in any case, is then bubbled into a solution of potassium or sodium iodide. After the reaction, the iodine is filtered-off, washed, dried, and then sublimed in the usual manner.



Why does this reaction happen? As in previous oxidization reactions, chlorine is a strong oxidizer and is capable of displacing other halogens such as bromine and iodine from their salts. In this reaction, chlorine oxidizes sodium or potassium iodide forming iodine and sodium chloride. As in similar reactions, the chlorine prefers to bond with the sodium or potassium of the iodide freeing iodine and forming the corresponding alkali chloride salt.

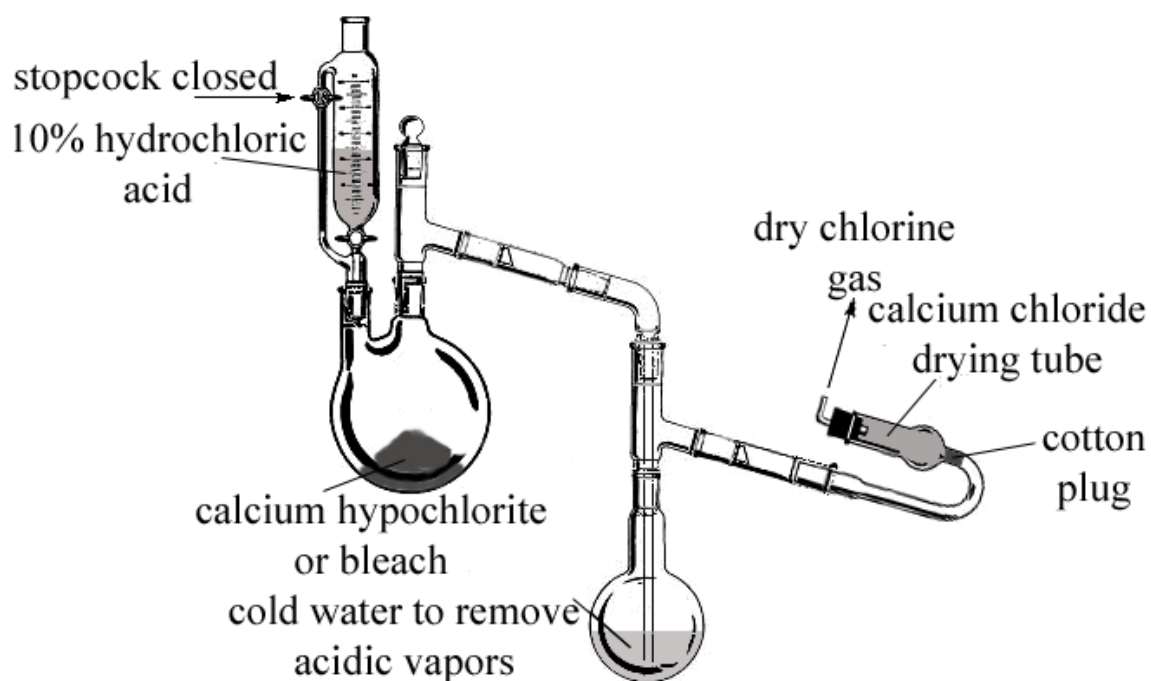
Hazards: Use care when handling strong oxidizers like manganese dioxide, potassium permanganate, sodium hypochlorite, and calcium hypochlorite, as they are all highly reactive and capable of reacting with many organic substances. Mixtures of strong oxidizers and combustible materials can ignite and burn violently. Wear gloves when handling hydrochloric acid, which can cause skin irritation.

Procedure: Setup the chlorine generating apparatus as illustrated below. Note: your chlorine generator does not have to be as fancy as the apparatus illustrated below, but your system should be similar in design. You can use latex tubing, PVC pipes and other plastic devices; as well, steel pipes can be used, but other metals should be avoided due to corrosion.

Now, into a suitable beaker, flask, or container, place 50 grams (1.7 oz.) of potassium iodide or 45 grams of (1.6 oz.) of sodium iodide, followed by 150 milliliters (5 oz.) of tap water. Thereafter, stir the entire mixture for about 30 minutes to dissolve all solids.

After you have the chlorine generator set-up, place into the reaction flask, 15 grams (1/2 oz.) of manganese dioxide, or 240 grams (8.5 oz.) of a 5% sodium hypochlorite solution (Clorox bleach), or 13 grams (1/2 oz.) of potassium permanganate, or 19 grams (0.67 oz.) of calcium hypochlorite, bleaching powder (65% available chlorine). Then place into the addition funnel, a hydrochloric acid solution prepared by adding and dissolving 30 grams (1 oz.) of 35 to 38% hydrochloric acid (Muriatic acid of 31% will work) into 40 milliliters (1.35 fluid oz.) of tap water.

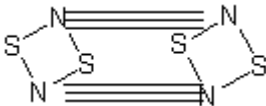

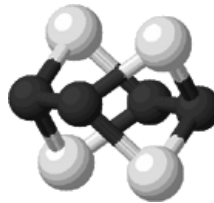
Once your chlorine generator is setup, begin dripping the hydrochloric acid solution, onto the oxidizer contained in the reaction flask. As the hydrochloric acid combines with the oxidizer a steady stream of chlorine gas will be evolved. The chlorine gas should then be passed into an apparatus as similar to the one in preparation 35 method 1, as the chlorine gas is passed into the iodide solution it will react with the potassium or sodium iodide to form iodine, which will precipitate as a brownish-black solid. When no more chlorine gas passes over, in either case, the reaction is complete. Thereafter, filter-off the precipitated brownish-black iodine solids, as usual, using either vacuum filtration or gravity filtration, and then wash them with cold water, and then vacuum dry or air-dry the solids. Once the iodine is completely dry, sublime it as illustrated in method 1. Repeat the sublimation process one more time to yield high purity reagent grade iodine.

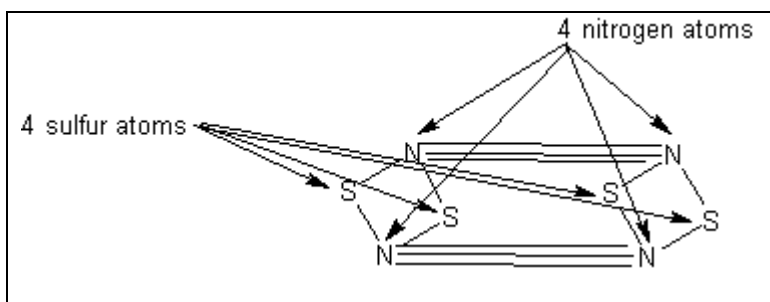


Apparatus for preparing chlorine gas—the hydrochloric acid drip method. The out let tube where the dry chlorine gas comes out, should be attached to plastic tubing, and this tubing then submerged into the potassium or sodium iodide solution.

Procedure 37: The Preparation of Sulfur Nitride

Also known as: Nitride of sulfur

	 <p>3D structure 1</p>  <p>3D structure 2</p>
Chemical structure	3D Structure

	S_4N_4
Structure make-up	Condensed chemical structure

Sulfur Nitride

Nitrogen tetrasulfide forms golden-yellow, yellowish red, or orange-red transparent prismatic crystals or orange-red needles. The crystals explode when struck; either in a mortar with a pestle, or by the blow of a hammer. The crystals burn without detonation when placed upon a hot body, and they explode when heated to 207 Celsius. The crystals may explode when heated to 160 Celsius, producing a feeble flash. Sulfur nitride may explode if rapidly heated.

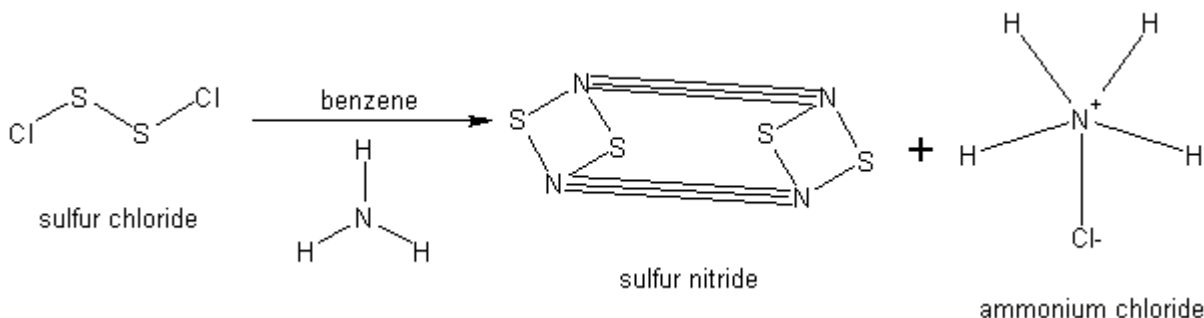
Method 1: Preparation of Sulfur Nitride from sulfur monochloride and ammonia gas (as seen in The Preparatory Manual of Explosives by Jared Ledgard)

(By-products from reaction: Ammonium chloride and sulfur)

Materials:

	1. 280 grams (9.87 oz.) of benzene or 300 grams (10.58 oz.) of toluene
	2. 50 grams (1.76 oz.) of sulfur chloride
	3. 17 grams (0.6 oz.) anhydrous ammonia gas

Reaction summary: Sulfur nitride is conveniently prepared by passing dry anhydrous ammonia gas into a benzene or toluene solution of sulfur chloride. During the reaction, ammonium chloride and sulfur precipitates, and towards the end of the reaction, red fumes will develop. When red fumes evolve, the reaction is complete. Thereafter, the reaction mixture is filtered, and then evaporated to yield dry crystals. These crystals are then recrystallized from toluene to yield high purity sulfur nitride.



Why does this reaction happen? In this particular and interesting reaction, an excess of ammonia reacts with sulfur chloride forming sulfur nitride and ammonium chloride as a by-product. The reaction takes place because excess nitrogen reacts with the sulfur chloride forming the preferred ammonium chloride and also sulfur nitride. In order for this reaction to take place, the ammonia must be in a large excess.

Hazards: Use proper ventilation when handling benzene or toluene. Benzene is a known carcinogen, and should be handled with care. Sulfur chloride is irritating to the eyes, nose, and throat. Use proper ventilation when handling. Sulfur chloride may also be corrosive to tissue, so wear gloves when handling. Use proper ventilation when handling ammonia, and avoid inhalation of the vapors.

Procedure: Into a suitable flask, place 280 grams of benzene, or 300 grams of toluene. Thereafter, add and dissolve 50 grams of sulfur chloride. Then, place the mixture into an ice/salt bath, and chill to -10 Celsius or less. Afterwards, bubble 17 grams of dry anhydrous ammonia gas into the mixture while rapidly stirring the reaction mixture. The rate of addition should be sufficient as to keep the reaction mixture below 0 Celsius at all times. Near the completion of the reaction, red fumes should

develop. If they don't, pass some more dry ammonia (1 to 2 grams) into the reaction mixture while stirring. After the addition, remove the cooling bath, and allow the reaction mixture to warm to room temperature. Thereafter, filter-off the insoluble ammonium chloride and sulfur, and then place the filtered reaction mixture into a rotary evaporator, and remove the solvent under vacuum until dry crystals remain. If a rotary evaporator is unavailable, the reaction mixture should be placed onto a shallow pan, and allowed to air evaporate; no heat. In this case, cheaper toluene should be used; for as the solvent will be evaporated into the air, and unrecoverable. The dry crystals should then be recrystallized from 300 grams of toluene. After collecting the crystals, air-dry, and then store in an amber glass bottle in a cool place away from sunlight.

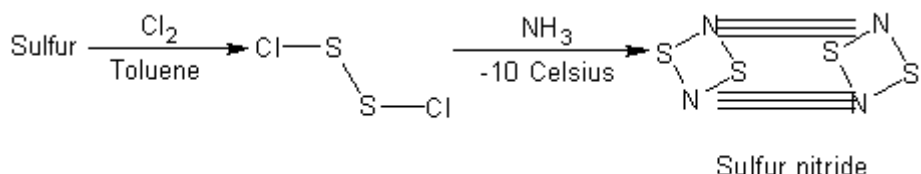
Method 2: Preparation of Sulfur Nitride from ammonia gas, sulfur, and chlorine (as seen in The Preparatory Manual of Explosives by Jared Ledgard)

(By-products from reaction: Ammonium chloride and sulfur)

Materials:

	1. 300 grams (10.58 oz.) of toluene
	2. 24 grams (0.84 oz.) powdered sulfur
	3. 22 grams (0.77 oz.) anhydrous ammonia gas
	4. 26 grams (1 oz.) dry chlorine gas

Reaction summary: Sulfur nitride is conveniently prepared by passing dry anhydrous ammonia gas into a toluene solution of sulfur chloride. In this process, the sulfur chloride is prepared directly by passing dry chlorine into a suspension of powdered sulfur in toluene. Thereafter, this sulfur chloride mixture, which also contains quantities of sulfur dichloride, and tetrachloride is treated with dry ammonia gas. During the reaction, ammonium chloride and sulfur precipitates, and towards the end of the reaction, red fumes will develop. When red fumes evolve, the reaction is complete. Thereafter, the reaction mixture is filtered, and then evaporated to yield dry crystals. These crystals are then recrystallized from toluene to yield high purity sulfur nitride.



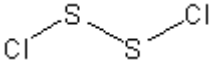


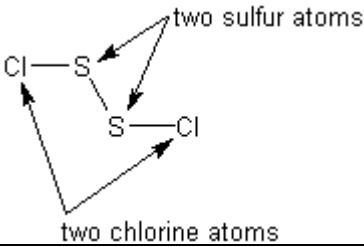
Why does this reaction happen? Same as in method 1.

Hazards: Use proper ventilation when handling chlorine gas, and avoid inhalation of vapors. Use proper ventilation when handling toluene. Sulfur chloride is irritating to the eyes, nose, and throat. Use proper ventilation when handling. Sulfur chloride may also be corrosive to tissue, so wear gloves when handling. Use proper ventilation when handling ammonia, and avoid inhalation of the vapors.

Procedure: Into an appropriate flask, place 300 grams of dry toluene, and then add in 24 grams of powdered sulfur. Thereafter, place the flask into a cold-water bath, and then pass 26 grams of dry chlorine gas into the mixture over a several hour period while rapidly stirring the reaction mixture. During the addition, keep the toluene mixture around 10 Celsius. After the addition of the chlorine gas, filter the reaction mixture to remove any insoluble materials. Note: During the filtering process, make sure no water is present. After the filtration process, place the flask into an ice/salt bath, and chill to -10 Celsius or less. Afterwards, bubble 17 grams of dry anhydrous ammonia gas into the mixture while rapidly stirring the reaction mixture. The rate of addition should be sufficient as to keep the reaction mixture below 0 Celsius at all times. Near the completion of the reaction, red fumes should develop. If they don't, pass some more dry ammonia (1 to 2 grams) into the reaction mixture while stirring. After the addition, remove the cooling bath, and allow the reaction mixture to warm to room temperature. Thereafter, filter-off the insoluble ammonium chloride and sulfur, and then place the filtered reaction mixture into a rotary evaporator, and remove the solvent under vacuum until dry crystals remain. If a rotary evaporator is unavailable, the reaction mixture should be placed onto a shallow pan, and allowed to air evaporate; no heat. The dry crystals should then be recrystallized from 300 grams of toluene. After collecting the crystals, air-dry, and then store in an amber glass bottle in a cool place away from sunlight.

Procedure 38: The Preparation of Sulfur monochloride

Also known as: Disulfur dichloride

	 <p>3D structure 1</p>  <p>3D structure 2</p>
Chemical structure	3D Structure
	<p>S_2Cl_2</p>
Structure make-up	Condensed chemical structure

Sulfur monochloride (also known as disulfur dichloride)

Sulfur monochloride forms a non-flammable, light amber to yellowish red, fuming oily liquid, which has a penetrating odor. It has a melting point of -77 Celsius, and a boiling point of 138 Celsius. It is soluble in alcohol, ether, carbon disulfide, toluene, carbon tetrachloride, and many oils. It reacts with water yielding sulfur dioxide, hydrogen chloride, and other products. Over exposure may cause skin irritation. It is conveniently prepared by passing dry chlorine into molten sulfur, and then recovering the sulfur chloride by distillation.

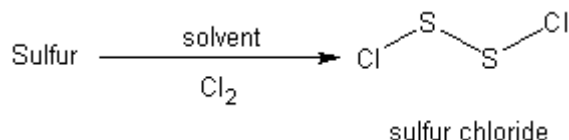
Method 1: Preparation of Sulfur monochloride from sulfur and chlorine (as seen in The Preparatory Manual of Explosives by Jared Ledgard)

(By-products from reaction: none)

Materials:

1. 300 milliliters (10 fluid oz.) of dry methylene chloride, or benzene	3. 26 grams (0.9 oz.) of dry chlorine gas
2. 24 grams (0.8 oz.) of powdered sulfur	

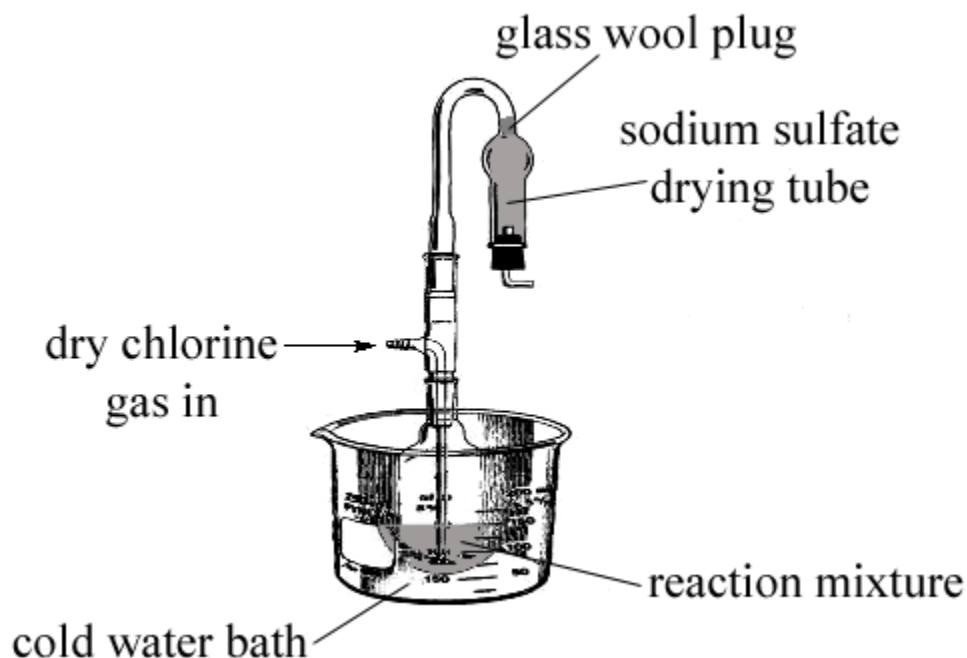
Reaction summary: Sulfur monochloride is readily prepared by reacting dry chlorine gas with powdered sulfur. The reaction is carried out in an inert solvent such as methylene chloride or benzene. After the reaction is complete, the reaction mixture is quickly filtered, and then distilled to remove the methylene chloride or benzene solvent.



Why does this reaction happen? Sulfur readily reacts with chlorine forming the addition compound sulfur monochloride. The reaction is very simple and takes place because the chlorine is a strong oxidizer and reacts readily with the sulfur forming the resulting chloride.

Hazards: Use caution when handling chlorine gas, and use proper ventilation.

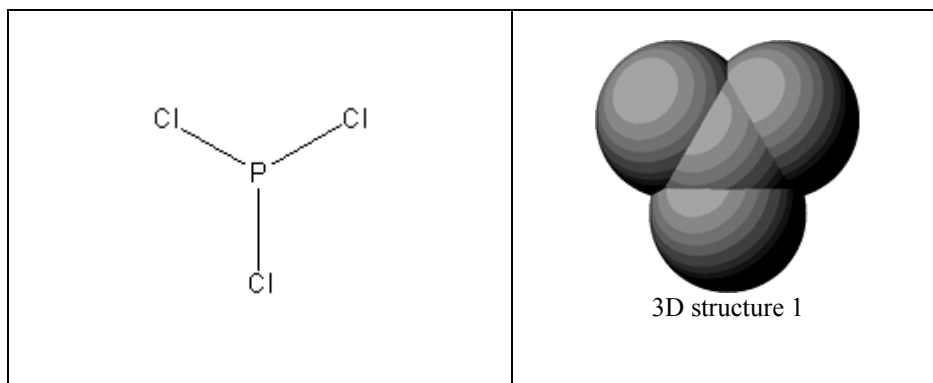
Procedure: Into an appropriate flask (as illustrated in the following illustration), place 300 milliliters (10 fluid oz.) of dry methylene chloride, or benzene, and then add in 24 grams (0.8 oz.) of powdered sulfur. Thereafter, place the flask into a cold-water bath, and then pass 26 grams (0.9 oz.) of dry chlorine gas into the mixture over a several hour period while rapidly stirring the reaction mixture. During the addition, keep the reaction mixture below 30 Celsius. After the addition of the chlorine gas, quickly filter the reaction mixture to remove any insoluble materials, and then place the filtered reaction mixture into a distillation apparatus (not illustrated below; the distillation apparatus can be any simple apparatus), and then boil-off the methylene chloride at 40 Celsius, or if using benzene, boil at 80 Celsius to drive-off the benzene. When no more methylene chloride or benzene distills over, remove the heat source, and then allow the remaining oily liquid of sulfur monochloride to cool to room temperature. Then pour the remaining oily liquid into any suitable dry amber glass bottle, and store in a cool dry place.

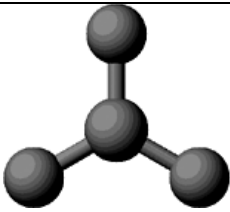
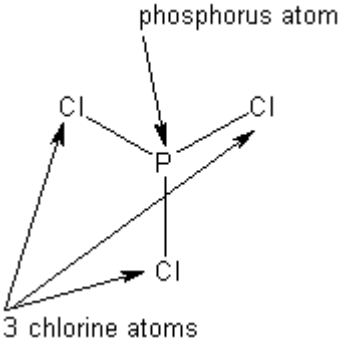


Suggestive apparatus for the preparation of sulfur monochloride from sulfur and chlorine.

Procedure 39: The Preparation of Phosphorus trichloride

Also known as: Chloride of phosphorus



	 <p>3D structure 2</p>
Chemical structure	3D Structure
 <p>phosphorus atom</p> <p>3 chlorine atoms</p>	PCl ₃
Structure make-up	Condensed chemical structure

Phosphorus trichloride

Phosphorus trichloride forms a colorless to slightly colored fuming liquid with a melting point of -112 Celsius, and a boiling point of 76 Celsius. The liquid is highly reactive and decomposes rapidly in the presence of water or alcohol. The liquid should be kept in airtight bottles and stored in a cool dry place. It is soluble in benzene, methylene chloride, ether, and hexane. Avoid inhalation of the fumes and skin contact.

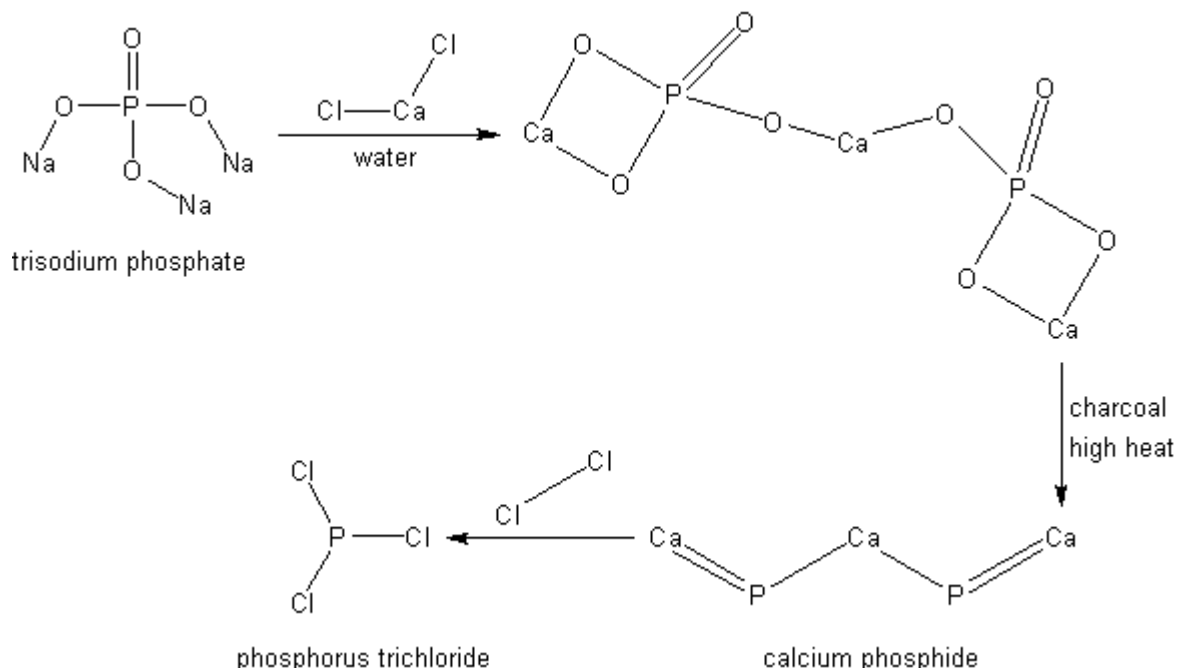
Method 1: Preparation of Phosphorus trichloride from trisodium phosphate, chlorine, calcium chloride, and charcoal

(By-products from reaction: calcium oxide, calcium chloride, tetrachlorethane, trichloroethane, chlorine, acetylene)

Materials:

1. 250 grams (8.8 grams) of technical grade (store bought) trisodium phosphate	6. 100 grams (3.5 oz.) of finely powdered wood charcoal
2. 750 milliliters (25.2 fluid oz.) of warm water	7. 250 milliliters (6.9 fluid oz.) of methylene chloride
3. 250 grams (8.8 oz.) of anhydrous calcium chloride	8. 200 grams (7 oz.) of extra dry chlorine gas
4. 750 milliliters (25.2 fluid oz.) of cold water	9. 100 grams (3.5 oz.) or so of aluminum oxide
5. 1500 milliliters (50.7 fluid oz.) of warm water	

Reaction summary: Phosphorus trichloride can be prepared in a three-step process starting with the formation of calcium phosphate. This crude technical grade of calcium phosphate is prepared by mixing aqueous solutions of trisodium phosphate and calcium chloride. The mixing causes the water insoluble calcium phosphate to precipitate. The precipitate is readily filtered-off, washed, and then dried in the usual manner. Thereafter, the crude calcium phosphate is then roasted at high temperature in the presence of carbon, whereby it gets reduced to calcium phosphide. Note: some elemental phosphorus may be liberated in the reaction, so use proper ventilation. The calcium phosphide crude mixture is then chlorinated to form a mixture of compounds, one of which being the desired phosphorus trichloride. After the chlorination process, the reaction mixture is filtered, and then distilled using a conventional distillation apparatus to remove the methylene chloride solvent. Finally, the remaining crude liquid left over after removal the methylene chloride, is then fractionally distilled to recover the liquid product of phosphorus trichloride. The distilled phosphorus trichloride can be re-distilled if desired.



Why does this reaction happen? In step 1, the reaction takes place because of a classic precipitation reaction. When two water soluble compounds like trisodium phosphate and calcium chloride are dissolved in water, they form two clear solutions. When these two solutions are mixed, the water insoluble calcium phosphate precipitates spontaneously. In step 2, the reaction takes place because charcoal contains active carbon, which is a strong reducing agent. This carbon in the charcoal reduces the calcium phosphate forming calcium phosphide. In step 3, the reaction takes place because the chlorine in the reaction wants to bond with the calcium forming calcium chloride. Excess chlorine then reacts with the free phosphorus forming phosphorus trichloride.

Hazards: Use good ventilation when working with chlorine gas, and avoid inhalation of the fumes. Use caution when heating the calcium phosphate as small amounts of white phosphorus may be evolved.

Procedure:

Step 1: Preparation of calcium phosphate

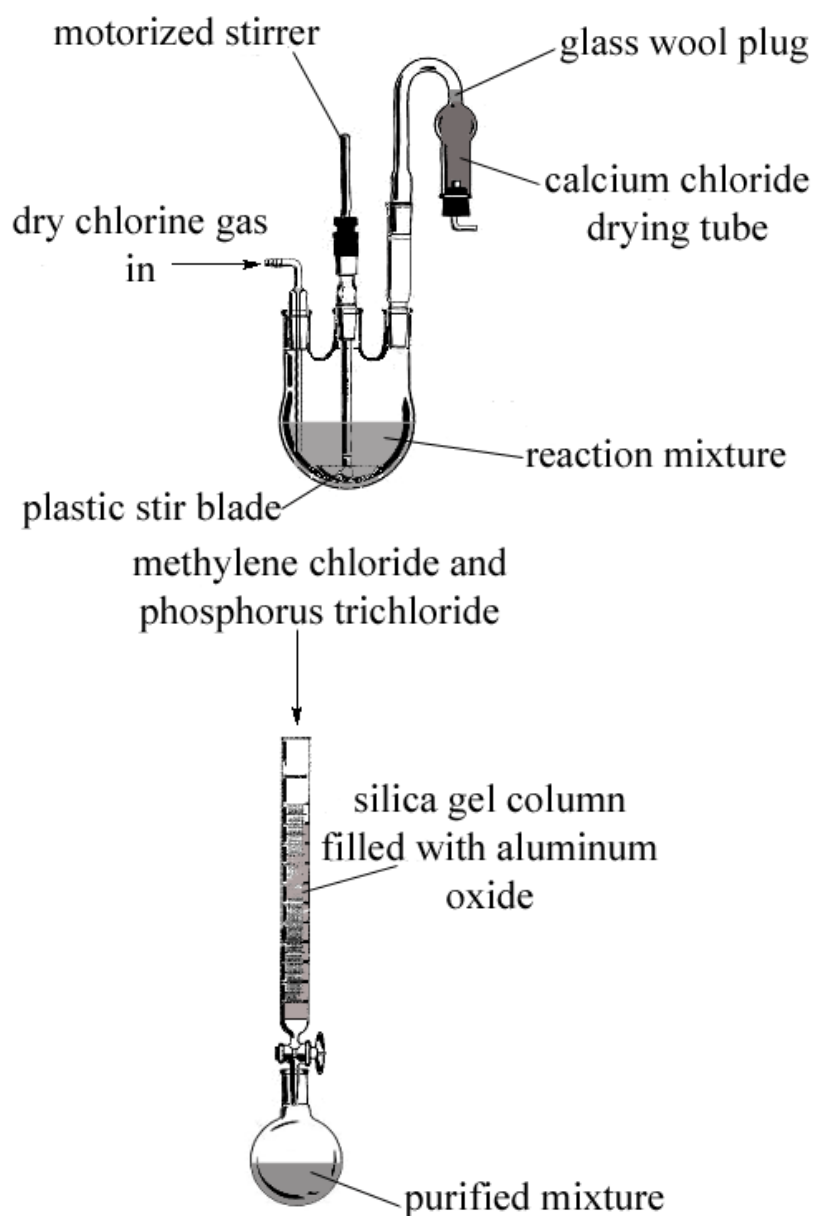
Into a suitable beaker or similar container, place 250 grams (8.8 grams) of technical grade (store bought) trisodium phosphate, and then add in 750 milliliters (25.2 fluid oz.) of warm water, and then stir the mixture to dissolve all of the trisodium phosphate (technical grade). Note: technical grade and store bought trisodium phosphate (TSP) contains sodium carbonate and maybe some sodium hydroxide to prevent the product from caking. This sodium carbonate and any other alkaline product needs to be neutralized before the initial reaction. To do this, first, place 250 grams (8.8 grams) of the dry TSP product from the packaging container into a suitable crucible or similar container, and then roast it at 150 to 250 Celsius for 1 hour to remove several moles of water of hydration. Thereafter, break-up the re-solidified TSP product from the crucible or similar container, and then dissolve it into 750 milliliters (25.2 fluid oz.) of water contained in a suitable beaker or container. Thereafter, drip hydrochloric acid, battery acid, or sulfuric acid into the trisodium phosphate solution until the evolution of carbon dioxide gas ceases. Once this point is achieved, the sodium carbonate has been neutralized. Now, to this trisodium phosphate solution, add in a calcium chloride solution prepared by adding and dissolving 250 grams (8.8 oz.) of anhydrous calcium chloride into 750 milliliters (25.2 fluid oz.) of cold water—heat will be evolved upon dissolving. Note: anhydrous calcium chloride is obtained by placing a sample of at least 300+ grams of technical grade calcium chloride, available in hardware stores and grocery stores sold as “ice melter”, into a crucible or similar container, and then roast the technical grade calcium chloride at 200+ Celsius to remove the water of hydration. Thereafter, the anhydrous calcium chloride can be dissolved into the 750 milliliters (25.2 fluid oz.) of water, once the anhydrous salt has cooled. Upon mixing the two solutions of trisodium phosphate and calcium chloride, a precipitate of crude calcium phosphate tribasic, mixed with a small amount of other calcium phosphates, will form a chunky and messy precipitate. Thereafter, filter-off this chunky precipitate using gravity filtration, or preferably vacuum filtration, and then wash this precipitate with three 500 milliliter (three 16.9 fluid oz. portions) portions of warm water. Thereafter, vacuum dry or air-dry the washed filtered-off precipitate. Finally, place the dried calcium phosphate into a crucible or similar container, and then roast it at 100 to 150 Celsius for an hour or so to remove any moisture or water of hydration. Thereafter, pulverize the heated calcium phosphate into a fine powder, once it has cooled.

Step 2: Preparation of calcium phosphide

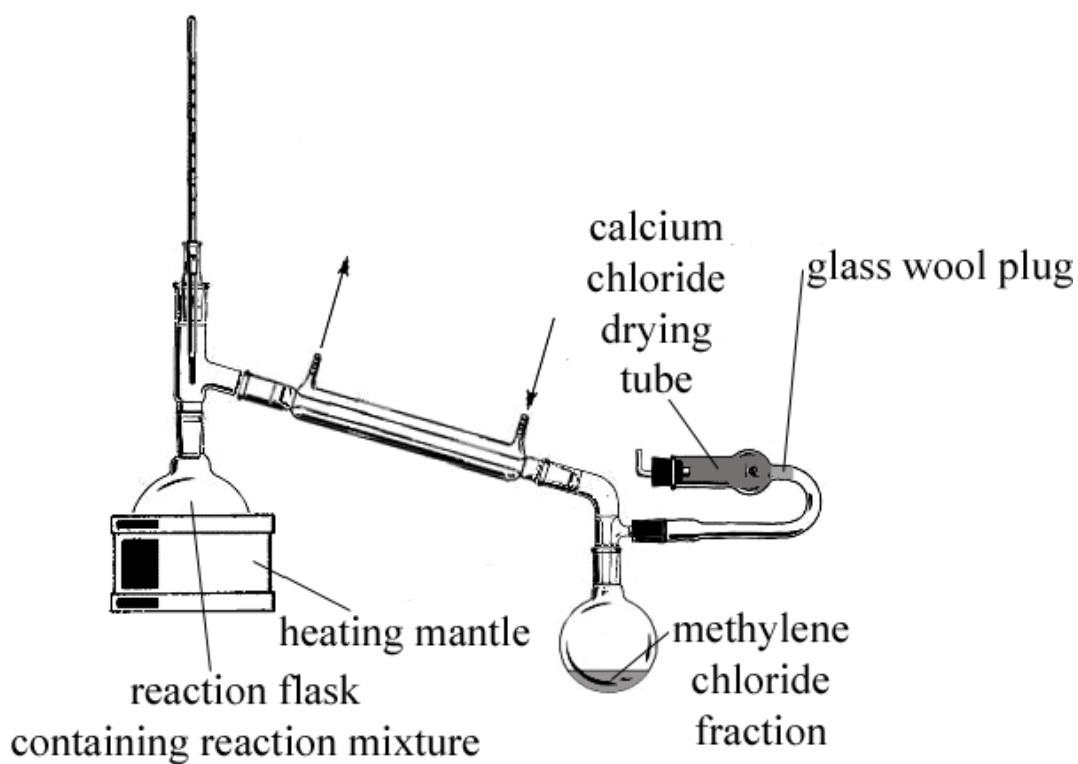
Now, into a zip lock bag, or similar plastic container, place the pulverized calcium phosphate (prepared in step 1), followed by 100 grams (3.5 oz.) of finely powdered wood charcoal (average charcoal used for cooking, with no quick-light garbage or other chemical additives), and then shake the bag thoroughly for about 10 to 15 minutes to thoroughly mix both solids. Thereafter, place this mixed powdered mixture into any high heat resistant crucible, such as stainless steel crucible, nickel, or quartz crucible, or equivalent, and then blast this mixture at 1000 Celsius using a high temperature Meeker Bunsen burner or equivalent for about 1 hour or so. If a high temperature Bunsen burner is unavailable, you can use a fire or some other means of heat. After the roasting process, remove the heat source, and allow the hot mixture to cool to room temperature. Now, at this point, the blackish-gray mixture will be composed of calcium phosphide, calcium oxide, a little calcium carbide, unreacted calcium phosphate, and un-reacted charcoal. Keep this mixture as is for use in step 3.

Step 3: Preparation of phosphorus trichloride

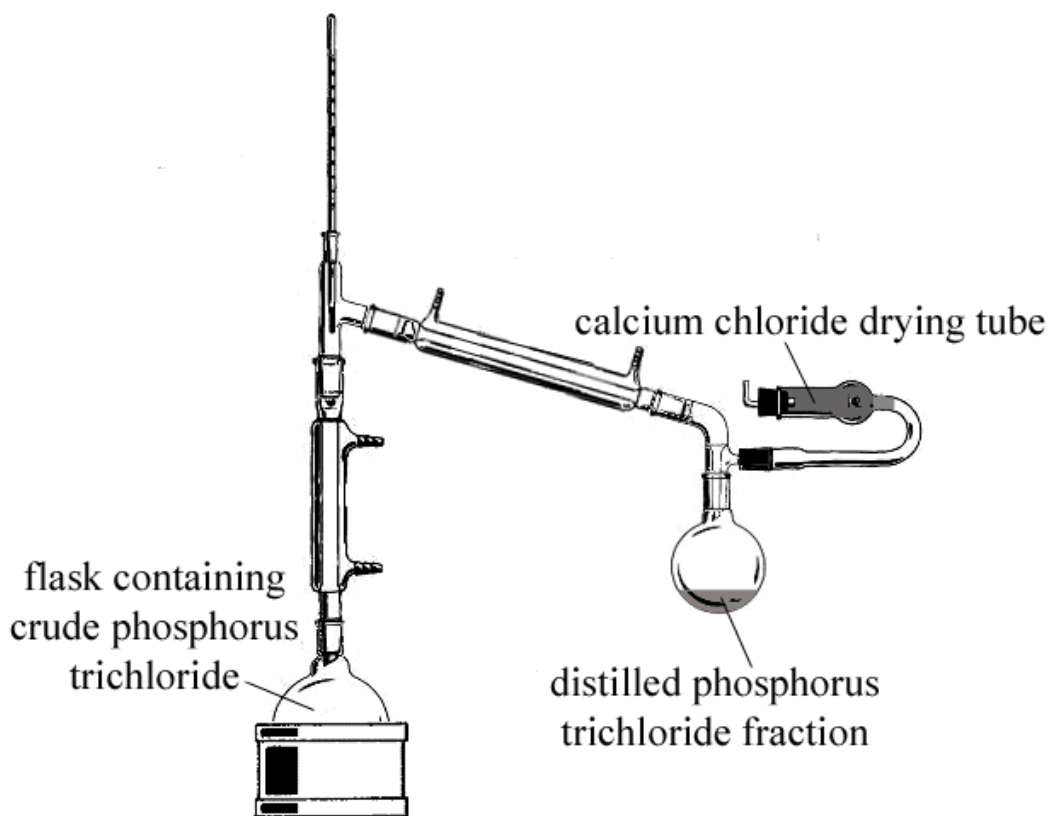
Into a suitable apparatus, as illustrated below, place the crude product obtained in step 2, and then add in 250 milliliters (6.9 fluid oz.) of methylene chloride. Then begin the motorized stirrer on moderate speed, and then bubble into the mixture, 200 grams (7 oz.) of extra dry chlorine gas over a period of 2 to 6 hours while stirring the reaction mixture on moderate speed. Note: dry chlorine can be obtained by passing the chlorine through multiple anhydrous calcium chloride drying tubes. During the addition of the chlorine gas, phosphorus trichloride will form, and will be taken-up into the methylene chloride. The by-products, will form insoluble precipitates along with the carbon. After the addition of the chlorine gas, the mixture needs to be filtered, to remove the insoluble materials, and then the resulting filtered mixture needs to be passed several times, through a silica gel column, filled with 100 grams (3.5 oz.) or so of aluminum oxide (see illustration). Thereafter, the mixture should be placed in a distillation apparatus, and first, distilled at 40 Celsius to remove the methylene chloride. Thereafter, place the remaining mixture into a fractional distillation apparatus, and fractionally distill the product mixture at 76 Celsius to collect the phosphorus trichloride. Note: during all distillations, keep a drying tube attached to the apparatus to keep moisture out.



*Top illustration: Apparatus for the addition of chlorine gas to a mixture of methylene chloride and calcium phosphide.
Bottom illustration: setup for the purification of the methylene chloride reaction mixture containing the phosphorus trichloride. The aluminum oxide helps remove impurities, and carbon.*



Distillation apparatus for removal of the methylene chloride.



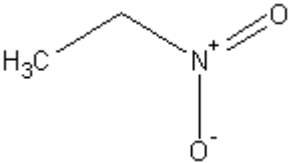

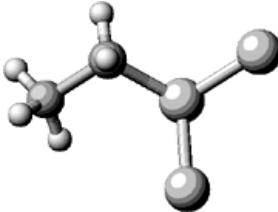
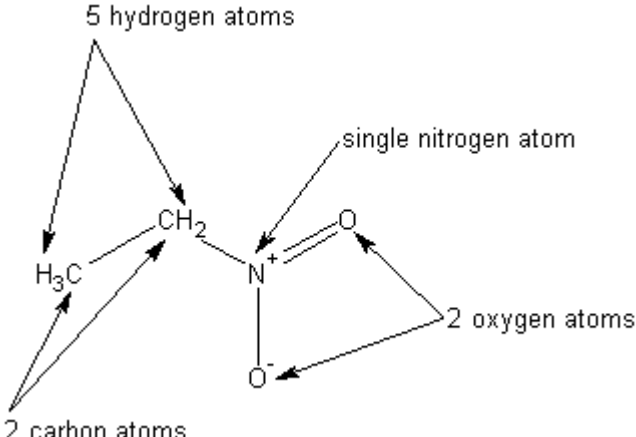
Fractional distillation apparatus for distillation of phosphorus trichloride.

Chapter 8: Advanced laboratory procedures

Advanced laboratory procedures compile the majority of laboratory chemistry. However, only a few examples of these types of processes will be included in this book due to their difficulty. Feel free to read thoroughly through each procedure to familiarize yourself with the true nature of chemistry, and keep in mind that there are over 21 million organic compounds known to man, so that's at least 21,000,000+ chemical reactions, could you possibly familiarize yourself with them all??

Procedure 40: The Preparation of Nitroethane

Also known as: Nitronium ethane

	 <p>3D structure 1</p>  <p>3D structure 2</p>
Chemical structure	3D Structure
	<p>$C_2H_5NO_2$</p>
Structure make-up	Condensed chemical structure

Nitroethane

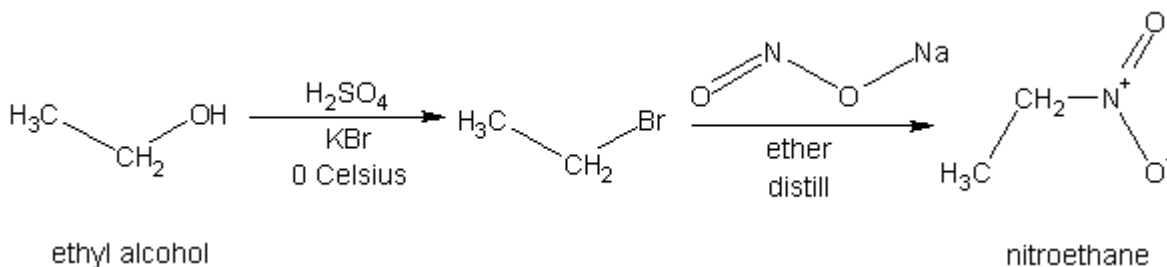
Nitroethane forms a colorless to oily liquid with a pleasant odor. It has a melting point of -50 Celsius, and a boiling point of 115 Celsius. Nitroethane is only very slightly soluble in water, but miscible in alcohol, and ether. It is also soluble in chloroform. Nitroethane forms explosive salts when treated with strong bases. It can be made by reacting ethyl bromide with sodium nitrite.

Method 1: Preparation of nitroethane from ethyl alcohol, sodium or potassium bromide, sulfuric acid, and sodium nitrite

Materials:

1. 45 milliliters (1.5 fluid oz.) of ice water	7. 30 grams (1 oz.) of sodium nitrite
2. 75 milliliters (4 fluid oz.) of 98% sulfuric acid	8. 1250 milliliters (42.2 fluid oz.) of ice water
3. 75 milliliters (4 fluid oz.) of 95% ethyl alcohol	9. Five 90-milliliter portions (five 3 fluid oz. portions) of diethyl ether
4. 60 grams (2.1 oz.) of potassium bromide or 52 grams (1.8 oz.) of sodium bromide	10. Three 75-milliliter portions (three 2.5 fluid oz. portions) of ice cold water
5. 10 grams (0.35 oz.) of anhydrous calcium chloride	11. 15 grams (0.52 oz.) of anhydrous magnesium sulfate
6. 500 milliliters (16.9 fluid oz.) of dimethylformamide (DMF)	

Reaction summary: Nitroethane is readily prepared by condensing ethyl bromide with sodium nitrite. The ethyl bromide can be made on site by the interaction of ethyl alcohol with potassium or sodium bromide in the presence of concentrated sulfuric acid. The ethyl bromide once prepared, can be purified by distillation. Once the reaction between the ethyl bromide and sodium nitrite is complete, the reaction mixture is extracted with ether, the ether removed, and the resulting remaining nitroethane then distilled to recover the purified product.

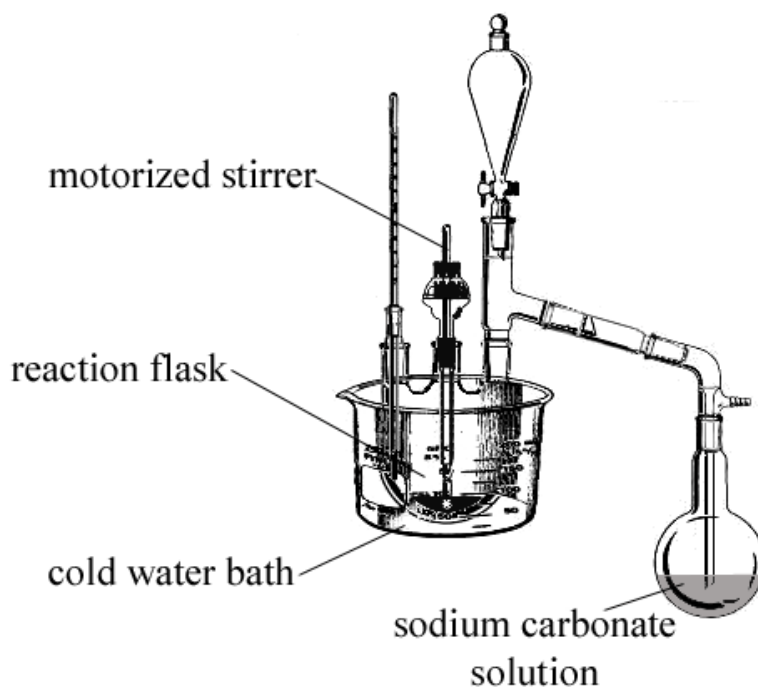


Hazards: Wear gloves when handling concentrated sulfuric acid, and use caution. Ethyl alcohol, dimethylformamide, and ether are highly flammable. Ether can form explosive mixtures with air, so use caution and extinguish all sources of ignition.

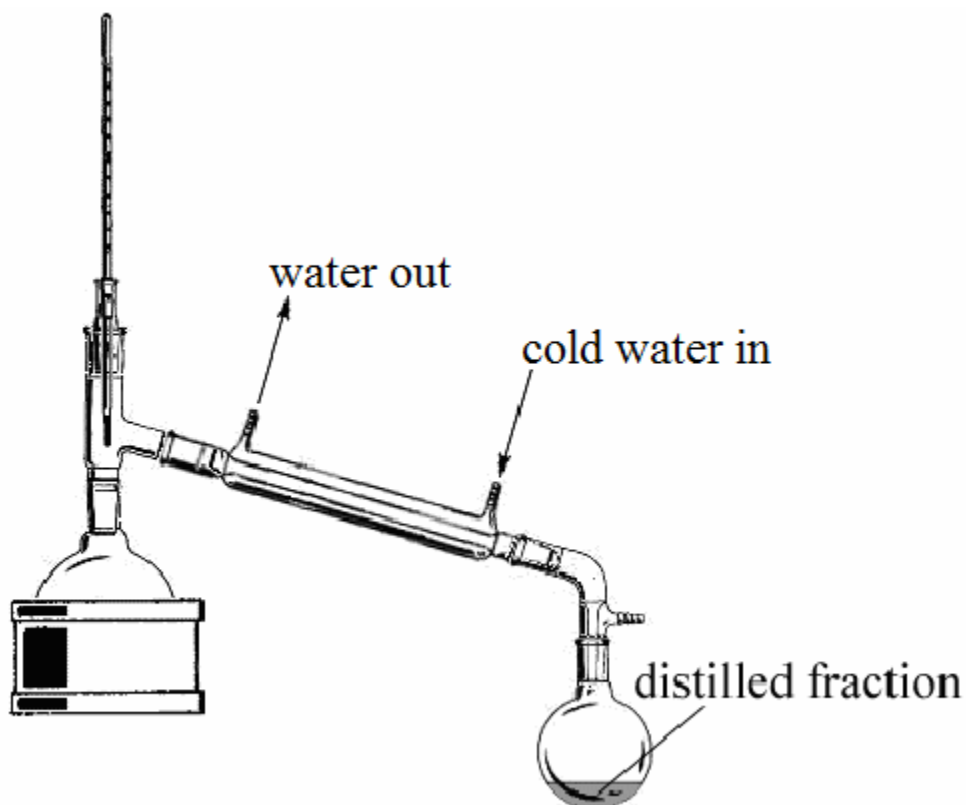
Procedure:

Step 1: Preparation of ethyl bromide

Into a standard flask (see illustration below), equipped with motorized stirrer or other stirring means, addition funnel, and thermometer, place 45 milliliters (1.5 fluid oz.) of ice water. Thereafter, slowly and carefully add in 75 milliliters (4 fluid oz.) of 98% sulfuric acid. Then place this acid mixture into an ice bath, and chill to about 0 Celsius. Afterwards, place 75 milliliters (4 fluid oz.) of 95% ethyl alcohol into the addition funnel, and then slowly add this ethyl alcohol, drop-wise, to the acid mixture. During the addition of the alcohol, stir the acid mixture and maintain its temperature around 0 Celsius at all times. After adding in the ethyl alcohol, slowly add in, 60 grams (2.1 oz.) of potassium bromide or 52 grams (1.8 oz.) of sodium bromide, in small portions at a time, over a period sufficient to keep the reaction mixture at 0 Celsius. After the addition of the bromide salt, continue to stir the entire reaction mixture for about 30 minutes, and thereafter, pour this entire reaction mixture into a distillation apparatus, and distill-off the ethyl bromide at 38 Celsius. When no more ethyl bromide passes over or is collected, stop the distillation process, and then recover the ethyl bromide from the receiver flask. Then add to this collected ethyl bromide, 10 grams (0.35 oz.) of anhydrous calcium chloride, and then stir the entire mixture for about 10 minutes—thereafter, filter-off the calcium chloride. Finally, re-distil this ethyl bromide using a fractional distillation apparatus at 38 Celsius. After the distillation process, collect the ethyl bromide and store it in an amber glass bottle in a refrigerator until use.



Advanced apparatus for the preparation of ethyl bromide.



Standard distillation apparatus for the recovery of ethyl bromide.

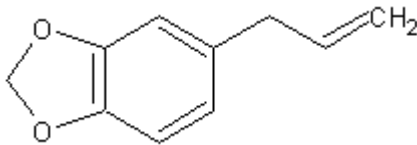
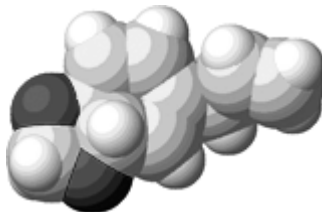
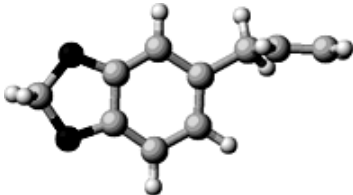
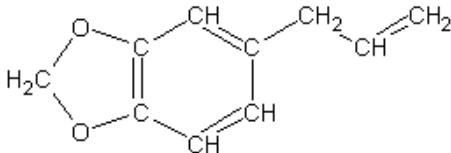
Step 2: Preparation of nitroethane

Into a standard flask, equipped with motorized stirrer or other stirring means, place 500 milliliters (16.9 fluid oz.) of dimethylformamide (DMF), followed by 30 grams (1 oz.) of sodium nitrite. Thereafter, stir this entire mixture to form a uniform mix, and then place this mixture into a cold-water bath. Afterwards, carefully and gradually add in, 27 grams (0.95 oz.) of ethyl bromide (prepared in step 1), over a period sufficient to keep the reaction mixtures temperature below 25 Celsius at all times. During the addition, rapidly stir the reaction mixture and maintain its temperature below 25 Celsius. After the

addition of the ethyl bromide, continue to rapidly stir the reaction mixture for about 6 hours at a temperature below 30 Celsius. After 6 hours, pour the entire reaction mixture into a suitable sized beaker, and then add in 1250 milliliters (42.2 fluid oz.) of ice water. Thereafter, extract this aqueous mixture with five 90-milliliter portions (five 3 fluid oz. portions) of diethyl ether, and after the extraction process, combine all ether portions, if not already done so, and then wash this combined ether portion with three 75-milliliter portions (three 2.5 fluid oz. portions) of ice cold water. Note: after each extraction and washing portion, the ether will be the upper layer each time. After the washing portions, dry the collected washed ether portion by adding to it, 15 grams (0.52 oz.) of anhydrous magnesium sulfate, and then stir the entire mixture for about 10 minutes—thereafter, filter-off the magnesium sulfate. Now, place this dried filtered ether mixture into a distillation apparatus, and distill-off the ether at 40 Celsius. When no more ether passes over or is collected, stop the distillation process, and then recover the left over remaining residue (after it has cooled). Finally, place this left over recovered residue into a distillation apparatus, and distill over the nitroethane at 115 Celsius. When no more nitroethane passes over or is collected, stop the distillation process, and recover the nitroethane (after it has cooled). Then place this nitroethane into an amber glass bottle and store it in a cool dry place until use.

Procedure 41: The Preparation of Safrole

Also known as: Allylcatechol methylene ether

	 3D structure 1  3D structure 2
Chemical structure	3D Structure
	$\text{C}_{10}\text{H}_{12}\text{O}_2$
Structure makeup	Condensed chemical structure

Safrole

Safrole forms a colorless to slightly yellow liquid with the odor of sassafras. The oil is insoluble in water, but very soluble in alcohol, and miscible with chloroform and ether. The oil has a boiling point of 232 Celsius, but can be distilled under high vacuum at 100 Celsius under 11 millimeters of mercury. Safrole is the main component of sassafras oil, from which it makes up 70 to 75% of the oil by weight. Safrole also exists in *Ocotea cymbarum* oil (Brazilian oil of sassafras), which it exists up to 90% by weight. The oil of massoria bark and *Cinnamomum massoia* contains about 14% safrole. Safrole can be extracted from sassafras oil, and it can be extracted from Massoria bark oil and *Cinnamomum massoia* by washing the corresponding oil with sodium hydroxide solution to remove the phenols, and then vacuum distilling to obtain the safrole boiling at about 100 Celsius under a vacuum of 11 millimeters of mercury, or by carefully fractionally distilling (two path distillation) the phenol free oil at 228 to 235 Celsius. Safrole can also be made synthetically from rather inexpensive reagents (see safrole). Sassafras oil can be obtained by steam distilling the ground up roots of the sassafras tree, which grows in the mid western United States. Other

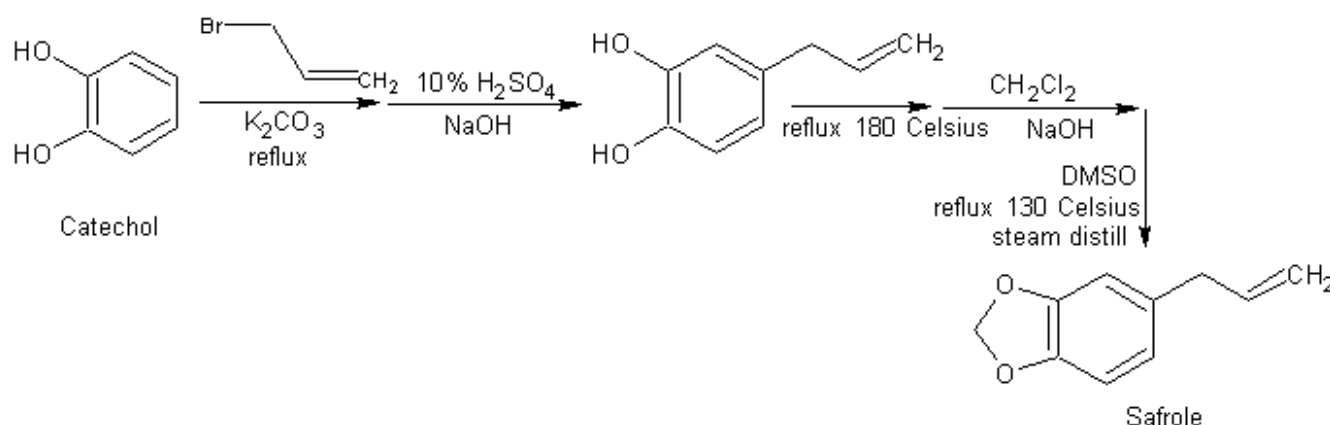
sassafras species of trees elsewhere in the world can also be used to obtain the safrole by steam distillation from the root. To identify a sassafras tree, consult a book that discusses the various types of trees and plants. The dried root bark of the sassafras tree contains about 10% safrole by weight, and the remainder of the root contains only about 1%. The dried root bark can be obtained from numerous sources, including herb stores, health food suppliers, and botanical suppliers. Sassafras oil can also be obtained from these aforementioned sources; if however your local suppliers do not offer the sale of sassafras oil, request them to order some for you, which they should have no problem doing. Safrole is also used in perfumes, so check out the types of perfumes, and their ingredients. Note: check out your local aromatherapy suppliers, as they are major consumers of oils, one of which may be sassafras oil. Sassafras oil may be used in adulterants in massage oils for use in aromatherapy. Ocotea cymbarum oil is obtained by steam distillation of the wood of the Ocotea pretiosa tree, which grows in South America. The wood contains about 1% oil by weight, which is easily collected by steam distillation of the wood chips, and the resulting steam distilled product contains about 90% safrole by weight. Distributors of perfume and flavoring compounds may contain this Ocotea cymbarum oil. Check the OPD directory for essential oils and botanical companies; also check out small herb shops nationwide.

Method 1: Preparation of Safrole

Materials:

1. 13 grams (0.45 oz.) of catechol	11. Three 50-milliliter portions (three 1.7 fluid oz. portions) of methylene chloride
2. 14 grams (0.49 oz.) of allyl bromide	12. 15 grams (0.52 oz.) of anhydrous magnesium sulfate
3. 22 milliliters (0.74 fluid oz.) of dry acetone	13. 50 milliliters (1.7 fluid oz.) of methylene chloride
4. 17 grams (0.59 oz.) of finely divided anhydrous potassium carbonate	14. 250 milliliters (8.5 fluid oz.) of dimethyl sulfoxide (DMSO).
5. 25 milliliters (0.84 fluid oz.) of cold water	15. 74 grams (2.6 oz.) of 4-allyl catechol
6. 100 milliliters (3.4 fluid oz.) of 10% sulfuric acid solution	16. 40 grams (1.4 oz.) of sodium hydroxide
7. 50-milliliter portion (1.7 fluid oz.) of diethyl ether	17. 10 milliliters (0.33 fluid oz.) of methylene chloride
8. 35 grams (1.2 oz.) of sodium hydroxide	18. 1.5 grams (0.05 oz.) of sodium hydroxide
9. 150 milliliters (5 fluid oz.) of water	19. 500 milliliters (17 fluid oz.) of water
10. 100 milliliters (3.4 fluid oz.) of 10% sulfuric acid	

Reaction summary: Safrole can be synthetically prepared by methylating 4-allyl catechol. This 4-allyl catechol is prepared by reacting catechol with allyl bromide, followed by an exhaustive extraction process, and then followed by a painstaking distillation process. Once the 4-allyl catechol has been successfully obtained, it is converted into safrole by refluxing it with methylene chloride and dimethyl sulfoxide in the presence of sodium hydroxide. The resulting reaction mixture is then distilled to remove any unreacted methylene chloride, and the resulting mixture is then steam distilled to collect the oily safrole product, which is then collected via a separatory funnel.



Hazards: Extinguish all flames before using diethyl ether, which is highly flammable, and can form explosive mixtures with air. Use care when handling acetone, which is also highly flammable. Use maximum ventilation when handling allyl bromide, which is very irritating to the eyes, nose, and throat.

Procedure:

Step 1: Preparation of 4-allyl catechol

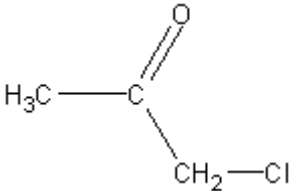

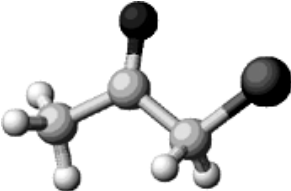
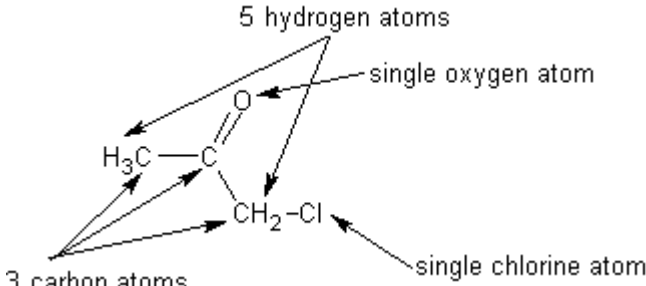
Into a suitable reflux apparatus, place 13 grams (0.45 oz.) of catechol, followed by 14 grams (0.49 oz.) of allyl bromide, and then add in 22 milliliters (0.74 fluid oz.) of dry acetone. Then stir the entire mixture to form a uniform mixture. Immediately thereafter, gradually add in 17 grams (0.59 oz.) of finely divided anhydrous potassium carbonate, and stir the mixture while adding this potassium carbonate. After the addition of the potassium carbonate, reflux the entire reaction mixture at 60 Celsius for about 3 hours. Note: fit a calcium chloride drying tube to the top of the reflux condenser to keep moisture out from the apparatus. After refluxing for about 3 hours, quickly remove the reflux condenser, and replace it with a conventional cold water condenser, fitted with a receiver flask, and then distill-off the acetone until no more acetone passes over into the receiver flask. When this point is reached, stop the distillation process, and allow the reaction mixture to cool to room temperature. Thereafter, pour the distilled reaction mixture left over, into a clean beaker, and then add in 25 milliliters (0.84 fluid oz.) of cold water, followed by 100 milliliters (3.4 fluid oz.) of 10% sulfuric acid solution. Then stir the entire acidic reaction mixture for about 10 minutes. Thereafter, extract the entire reaction mixture with one 50-milliliter portion (1.7 fluid oz.) of diethyl ether. After the extraction process, wash the ether portion by adding to it, a sodium hydroxide solution prepared by adding and dissolving 35 grams (1.2 oz.) of sodium hydroxide into 150 milliliters (5 fluid oz.) of water. Note: the addition of sodium hydroxide to water generates much heat, so allow the mixture to cool to room temperature before using. Thereafter, remove the upper ether layer by using a separatory funnel, or by decantation, and then discard or recycle this upper ether layer (will contain diallyl ether). Now to the lower water layer, add in 100 milliliters (3.4 fluid oz.) of 10% sulfuric acid, and upon the acid addition, some oil should separate. After the addition of the sulfuric acid, extract the entire acidic mixture (including any separated oil) with three 50-milliliter portions (three 1.7 fluid oz. portions) of methylene chloride. Note: after each extraction, the methylene chloride will be the upper layer. After the extraction process, combine all methylene chloride extracts, if not already done so, and then dry this combined methylene chloride mixture by adding to it, 15 grams (0.52 oz.) of anhydrous magnesium sulfate—thereafter, stir the whole mixture for about 10 minutes, and then filter-off the magnesium sulfate. Thereafter, place the filtered methylene chloride mixture into a distillation apparatus or rotary evaporator, and remove the methylene chloride. When no more methylene chloride is collected, recover the left over remaining oil. Now, to this oil, place it into a reflux apparatus, and heat it to 180 Celsius. Note: during the heating process, the oil will self heat raising the temperature to about 260 Celsius. When this temperature change results, stop the heating process, and then place the oil (which will now be red in color) into a vacuum distillation apparatus, and distill the oil at 158 Celsius under a vacuum of 16 millimeters of mercury. When no more oil is obtained at this temperature and vacuum, stop the distillation process, and then remove the left over remaining residue, and discard it. To the collected fraction, re-vacuum distill it using the same apparatus (after it has been cooled, and cleaned), and re-vacuum distill the oil at 158 Celsius, under a vacuum of 16 millimeters of mercury to obtain a refined 4-allyl catechol product.

Step 2: Preparation of safrole by methylating the 4-allyl catechol

Into a suitable 3-neck flask fitted with motorized stirrer, reflux condenser, thermometer, and a mercury bubbler fitted into the left or right female joint of the 3-neck flask to form an apparatus that is excluded from the atmosphere. Note: the top of the reflux condenser should be fitted with a calcium chloride drying tube, and to the other end of the drying tube, a gas inlet tube connected to a cylinder of dry nitrogen should be in place. Before assembling the calcium chloride drying tube with nitrogen purge adapter, charge the 3-neck flask with 50 milliliters (1.7 fluid oz.) of methylene chloride, followed by 250 milliliters (8.5 fluid oz.) of dimethyl sulfoxide (DMSO). Thereafter, begin the nitrogen purge and allow the entire apparatus to be flushed with nitrogen to exclude air and moisture—the mercury bubbler is to allow the nitrogen gas to run through the apparatus, and then escape into the atmosphere without exposing the apparatus to the atmosphere. Once the apparatus has been purged with nitrogen, reflux the contents in the 3-neck flask using a heating mantle to 130 Celsius, and when the temperature of these contents reaches the desired 130 Celsius mark, add in 3.7 grams (0.13 oz.) of 4-allyl catechol (obtained in step 1), followed immediately by 2 grams (0.07 oz.) of sodium hydroxide. Note: the 4-allyl catechol and sodium hydroxide can be quickly added through the top of the reflux condenser by quickly and temporarily removing the calcium chloride drying tube briefly, and then reattaching it after the addition. This process of addition should then be repeated 19 times (3.7 grams of 4-allyl catechol and 2 grams of sodium hydroxide each time for a total of 74 grams (2.6 oz.) of 4-allyl catechol and 40 grams (1.4 oz.) of sodium hydroxide. During each addition maintain the reflux at 130 Celsius. After the last addition, reflux the entire reaction mixture for an additional 10 minutes, and thereafter, add in 10 milliliters (0.33 fluid oz.) of methylene chloride followed by 1.5 grams (0.05 oz.) of sodium hydroxide. Thereafter, continue to reflux the reaction mixture at 130 Celsius for 35 minutes. Finally, after refluxing for the final 35 minute time period, stop the heating and reflux, and then allow the entire reaction mixture to cool to room temperature. Then pour the entire reaction mixture into a distillation apparatus, and distil-off any unreacted methylene chloride. When no more methylene chloride distills over, allow the left over remaining contents to cool to room temperature before removing from the apparatus, and then place these contents into a steam distillation apparatus, and then add in 500 milliliters (17 fluid oz.) of water, and then steam distill the safrole from the mixture to obtain safrole and water in the receiver flask. The oily safrole layer can then be recovered by using a separatory funnel in the usual manner. The safrole can be vacuum distilled at 100 Celsius under a vacuum of 11 millimeters of mercury to obtain a refined product if desired.

Procedure 42: The Preparation of Chloroacetone

Also known as: 1-Chloro-2-propanone; monochloro acetone

	 3D structure 1  3D structure 2
Chemical structure	3D Structure
	C_3H_5OCl Condensed chemical structure
Structure makeup	Condensed chemical structure

Chloroacetone

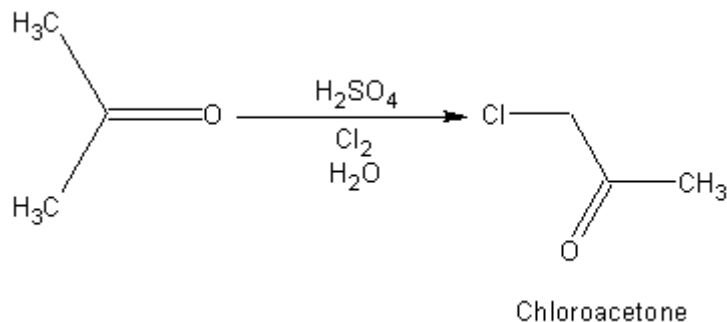
Chloroacetone forms a colorless slightly oily liquid with a pungent and irritating odor. It has a melting point of -45 Celsius, and a boiling point of 120 Celsius. It is volatile with steam, and hence, can be steam distilled. Chloroacetone is soluble in water to about 10% by weight, but it is miscible with alcohol, ether, and chloroform. Pure chloroacetone tends to darken on exposure to light due to polymerization, and it can be stabilized by the addition of a small amount of water, or calcium carbonate. Chloroacetone is a potent eye irritant. Eye exposure to this agent produces severe eye pain and irritation; effects of which last for hours.

Method 1: Preparation of Chloroacetone from acetone, chlorine gas, and sulfuric acid (as seen in A Laboratory History of Chemical Warfare Agents, by Jared Ledgard)

Materials:

1. 30 grams of acetone	4. 250 milliliters of methylene chloride or chloroform
2. 5 small drops of 98% sulfuric acid	5. 10 grams of anhydrous calcium chloride
3. 36.6 grams of chlorine gas	

Reaction summary: Chloroacetone is readily prepared by reacting chlorine gas with an acetone solution in the presence of sulfuric acid. The reaction proceeds smoothly, without any excessive heat buildup. After the reaction, the resulting reaction mixture can then be used directly in riot control operations, or it can be purified by extraction with methylene chloride or chloroform. The resulting solvent extractions are then combined, and then evaporated to remove the solvent and leave behind the chloroacetone.



Special Hazards regarding this procedure (read thoroughly):

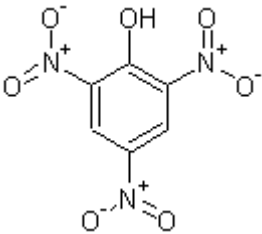
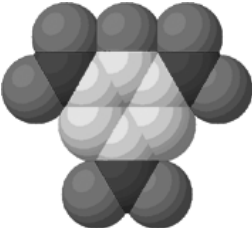
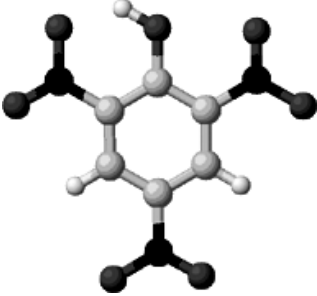
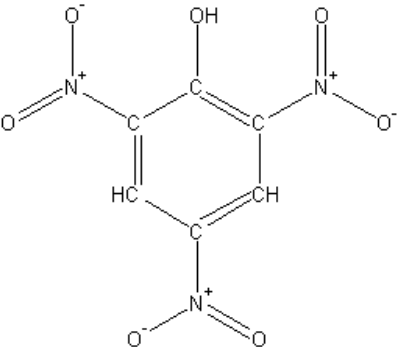


Do not attempt in anyway to prepare chloroacetone using the following procedure unless proper safety precautions are taken. 1) Perform all operations with good ventilation, maintain proper eye protection using eye wear that completely covers the eyes and forms a good seal on the face, and wear nitrile gloves. 2) After each procedure, all glassware and non-electric equipment should be soaked in a hot sodium hydroxide solution before removing from the clean box, and/or before rinsing and storing. Any electrical equipment that may be contaminated (even if suspected), such as hot plates and stirring equipment should be carefully wiped down with a rag soaked in a hot sodium hydroxide solution followed by wiping down with a rag soaked in hot water. 3) The desired chloroacetone product should be stored in amber bottles, preferably non-breakable containers, and stored in a cool dry place away from sunlight. The bottles should also be placed inside an airtight sealed plastic bag, such as a 'ziplock' bag. 4) Storage of any and all irritant agents should be in cabinets, drawers, or the like, and said storage spaces should be equipped with proper ventilation. Acetone is highly flammable, extinguish all flames before using. Use caution when handling chlorine gas, which is very toxic and irritating.

Procedure: Into a suitable flask, place 30 grams of acetone, 550 milliliters of water, and then 5 small drops of 98% sulfuric acid. Thereafter, place the mixture into a cold-water bath, and chill to about 15 to 20 Celsius. Then bubble 36.6 grams of chlorine gas into the mixture over a period ranging from 2 to 6 hours. Note: see Chlorine for its preparation and use. During the addition of the chlorine, continuously stir the acetone mixture. After the addition of the chlorine, stir the reaction mixture for 30 minutes, and then remove the cold-water bath. The resulting reaction mixture will contain the dissolved chloroacetone. Now, to collect the chloroacetone, extract the reaction mixture with five 50-milliliter portions of methylene chloride (or chloroform), and after each extraction, combine all extraction portions (if not already done so), and then dry the combined solvent extracts by adding 10 grams of anhydrous calcium chloride, and then stirring the mixture for several minutes; followed by filtration to remove the calcium chloride. Afterwards, place the filtered mixture into a rotary evaporator, or vacuum distillation apparatus and evaporate-off the solvent. Evaporation of the solvent will leave the chloroacetone behind, which it can then be recovered. The resulting chloroacetone should then be stabilized by the addition of 1 milliliter of regular tap water. The resulting chloroacetone should then be stored in an amber glass bottle in a refrigerator until use to prevent any possible polymerization.

Procedure 43: Picric Acid. 2,4,6-trinitrophenol

Also known as: picronitric acid; carbazotic acid

	 <p>3D structure 1</p>  <p>3D structure 2</p>
Chemical structure	3D Structure
	$\text{C}_6\text{H}_3\text{N}_3\text{O}_7$
Structure makeup	Condensed chemical structure

Picric acid

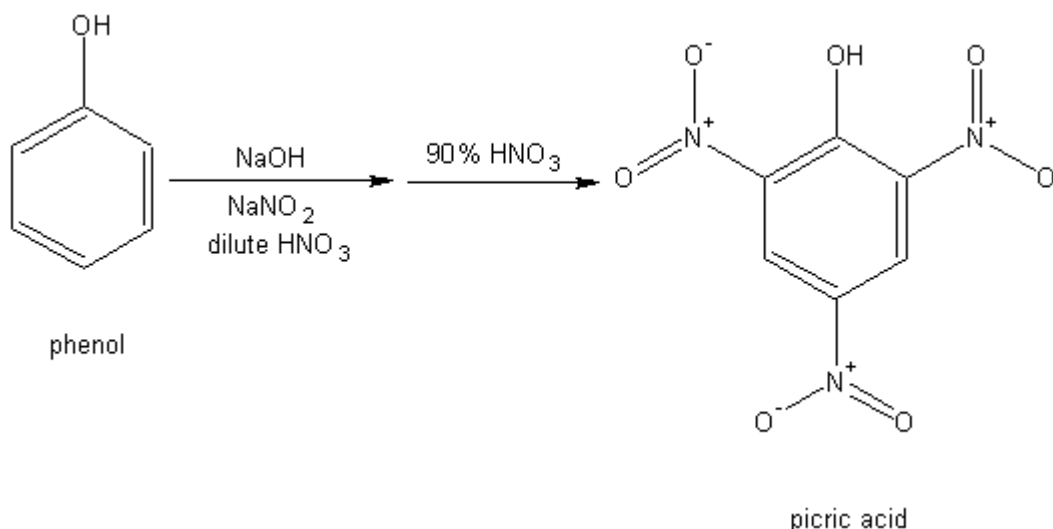
Picric acid is a pale yellow, odorless solid with a melting point of 123 Celsius. It explodes when heated to 300 Celsius. It is relatively insoluble in water, but soluble in alcohol, and benzene. Picric acid is toxic and can be absorbed through the skin with effects similar to DNP. Picric acid should be stored wet with 10% water, and kept in a cool place and away from fire. Picric acid will detonate if rapidly heated, or on percussion—the percussion is much higher than for most primary explosives. Picric acid was largely used during WWI, but its use has decreased. However, picric acid still demonstrates outstanding properties for use in explosives charges. It can be alloyed with TNT, RDX, HMX, and many other secondary explosives for use in blasting compositions, warheads, grenades, and land mines. Picric acid is also used in high performance gun propellants with ammonium picrate, nitrocellulose, and sodium or potassium nitrate. Picric acid readily forms primary explosives when treated with metal hydroxides, or carbonates.

Method 1: Preparation of Picric Acid from phenol, lye, sodium nitrite, and fuming nitric acid (as seen in *The Preparatory Manual of Explosives*, by Jared Ledgard)

Materials:

	1. 94 grams phenol
	2. 42 grams sodium hydroxide
	3. 86 grams sodium nitrite
	4. 800 milliliters 26% nitric acid
	5. 250 milliliters of 90% fuming nitric acid

Reaction summary: Picric acid is prepared from phenol by nitration of the nitrosophenol intermediate. The nitrosophenol intermediate is prepared by treating phenol with sodium hydroxide and sodium nitrite. The resulting nitrosophenol intermediate is then nitrated with fuming nitric acid.



Hazards: Use gloves when handling phenol. Phenol is very toxic and is rapidly absorbed by the skin. Both phenol and sodium hydroxide are very corrosive. Wear gloves and use proper ventilation when handling 90% nitric acid. The acid is very toxic and emits poisonous fumes of nitrogen oxides.

Procedure: Note: This procedure uses an excessive amount of 26% and 90% nitric acid to properly carryout the reaction. This excessive amount of acid can be recovered by treating the reaction mixture (after complete removal of the product) with methylene chloride to recover the nitric acid. Prior to this action, a small amount of sulfuric acid should be added to the reaction mixture. Afterwards, the reaction mixture should be extracted with several volumes of methylene chloride. These combined volumes can then be carefully distilled to recover the nitric acid of 99% purity (see nitric acid, 70%, 90% and 99% respectively).

Prepare a solution by dissolving 42 grams of sodium hydroxide, and 86 grams sodium nitrite into 1000 milliliters of water. Afterwards, place this solution into a cold-water bath, and then add 94 grams of phenol while stirring the solution and maintaining its temperature below 20 Celsius by means of the water bath. After the addition of the phenol, place the reaction mixture into an ice/salt bath to lower its temperature to -5 Celsius. After which, slowly add 800 milliliters of 26% nitric acid while stirring the reaction mixture and maintaining its temperature at -5 Celsius. After the addition of the 26% nitric acid, keep the temperature of the reaction mixture at -5 Celsius for 1 hour, and continue stirring to form a slurry. After 1 hour, add 250 milliliters of 90% nitric acid to a 3-liter flask, and then heat to 50 Celsius. When the 90% nitric acid reaches 50 Celsius, slowly transfer the reaction mixture slurry into the hot 90% nitric acid while stirring the 90% nitric acid and maintaining its temperature at 50 Celsius (during the addition, if the 90% nitric acid's temperature rises above 50 Celsius, temporarily remove the heat source). After the addition of the reaction mixture slurry into the hot 90% nitric acid, raise the temperature of the reaction mixture to 96 Celsius, and keep it at this temperature while stirring for 3 hours. After 3 hours, remove the heat source and allow the reaction mixture to cool to room temperature. Afterwards, mix the entire reaction mixture with 1500 milliliters of cold water, and then filter-off the picric acid precipitate. Then wash the picric acid with 500 milliliters of cold water, and then vacuum dry, or air-dry.

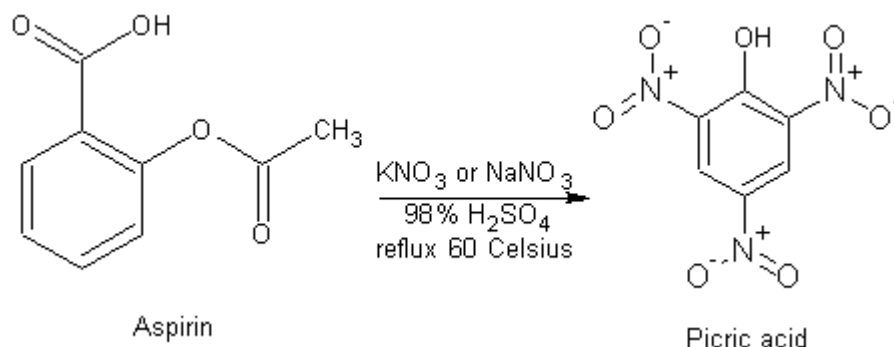
Note: Keep this substance away from heavy metals and their salts to prevent potential formation of shock sensitive materials, which easily detonate by heat, shock, sparks, or friction.

***Method 2: Preparation of Picric Acid from Aspirin, and potassium or sodium nitrate with sulfuric acid
(as seen in The Preparatory Manual of Explosives, by Jared Ledgard)***

Materials:

	1. 50 grams of aspirin
	2. 350 milliliters of 95% ethyl alcohol
	3. 350 grams of 98% sulfuric acid
	4. 115 grams of potassium nitrate or 95 grams of sodium nitrate

Reaction summary: In this procedure, picric acid is prepared by the reaction of common aspirin (acetylsalicylic acid) with potassium or sodium nitrate in the presence of excess concentrated sulfuric acid. After the reaction, the entire mixture is then drowned into an excessive amount of ice water, whereby the picric acid precipitates, and is then collected by filtration, washed, and then dried.

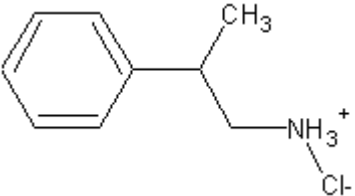
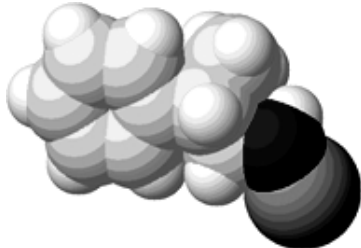
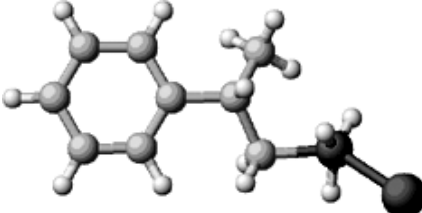
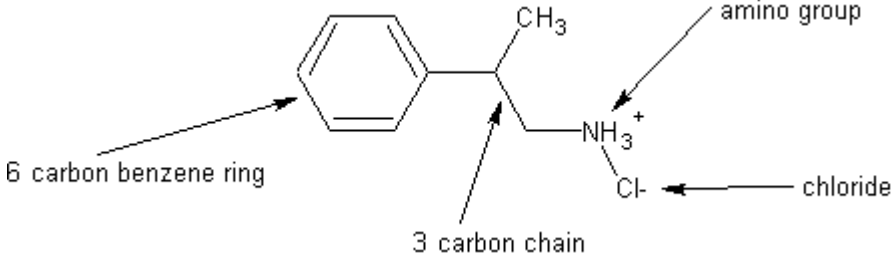


Hazards: Use caution when handling 98% sulfuric acid, which is highly corrosive and chars many substances. Extinguish all flames before using 95% ethyl alcohol, which is flammable.

Procedure: Place 50 grams of aspirin into a beaker, and then add 350 milliliters of 95% ethyl alcohol (note: about 50 grams of aspirin can be obtained by crushing up 100, 500-milligram tablets of store bought aspirin tablets—these crushed up tablets can be added directly to the 350 milliliters of 95% ethyl alcohol). Thereafter, stir the mixture to fully dissolve the aspirin, and then filter-off any insoluble impurities—such as starch, and other fillers (if using medical aspirin tablets), and then recrystallize the aspirin from this 95% ethyl alcohol solution. Thereafter, vacuum dry or air-dry the collected aspirin crystals. Now, into a clean flask equipped with thermometer, motorized stirrer and powder funnel, place 350 grams of 98% sulfuric acid, and then place this flask into an ice bath, and chill to 0 Celsius. Thereafter, slowly add in small portions, 115 grams of potassium nitrate, or 95 grams of sodium nitrate to the sulfuric acid over a period of about 1 hour, while rapidly stirring the sulfuric acid, and maintaining its temperature below 5 Celsius. After the addition of the sodium or potassium nitrate, slowly add the dry recrystallized aspirin, in small portions, over a period of about 1 hour while rapidly stirring the sulfuric acid/nitrate mixture, and keeping it temperate below 5 Celsius. After the addition of the aspirin, replace the powder funnel with a condenser, and reflux the mixture for 2 hours at 60 Celsius with rapid stirring. After 2 hours, remove the heat source, and allow the reaction mixture to cool to room temperature. Then gradually add the reaction mixture to 1500 milliliters of ice water, and then allow the entire mixture to stand for 3 hours. Thereafter, filter-off the precipitated picric acid, wash excessively with 10, 250-milliliter portions of ice cold water. Note: washing with base to remove traces of acid should be avoided as formation of the corresponding picrate salts may develop. After the washing process, vacuum dry or air-dry the solid product.

Procedure 44: The reparation of beta-Methylphenylethylamine hydrochloride. 2-phenylpropan-1-amine hydrochloride

Also known as: N/A

	 <p>3D structure 1</p>  <p>3D structure 2</p>
Chemical structure	3D Structure
	<p>$C_9H_{14}NCl$</p>
Structure makeup	Condensed chemical structure

beta-Methylphenylethylamine hydrochloride forms colorless to light brownish crystals with a melting point ranging from 180 to 195 Celsius (depending on purity). The crystals are moderately soluble in water, and relatively insoluble in ether.

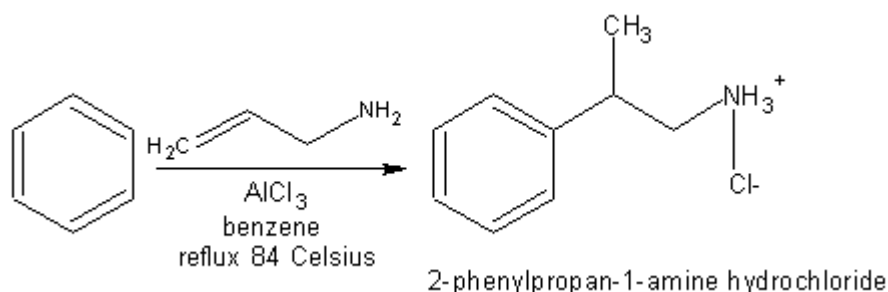
Method 1: Preparation of beta-Methylphenylamine hydrochloride (as seen in A Laboratory History of Narcotics, vol.1, by Jared Ledgard)

Materials:

1. 40 grams of anhydrous aluminum chloride	5. 305 milliliters of diethyl ether
2. 39 grams of dry benzene	6. 100 milliliters of a 30% sodium hydroxide solution
3. 5.7 grams of allylamine	7. 3.3 grams of dry hydrogen chloride gas
4. 10 milliliters of dry benzene	

Reaction summary: In this interesting procedure, beta-Methylphenylamine hydrochloride is prepared by reacting allylamine with benzene in the presence of anhydrous aluminum chloride as catalyst. The reaction is generally simple, and the product is recovered by several separations, extractions with ether, and treatment with sodium hydroxide to make the product mixture alkaline in order to remove any hydrochloride that may have formed. The product layer is then separated, and then carefully treated with dry hydrogen chloride gas to form the corresponding hydrochloride salt, which is recovered in the usual manner.

Note: for related information, see serial number 609,028, August 4th, 1945 by Chester M. Suter, of Albany N.Y., and Arthur W. Weston, of Waukegan Ill., assigned by Sharp & Dohme, Inc.



Hazards: Use caution when handling diethyl ether, which is highly flammable and can form explosive mixtures with air. Use proper ventilation when handling allylamine, which is an irritant and inhalation may cause nausea, vomiting, or headache. Use proper ventilation when handling benzene, and avoid inhalation, as it is a suspected carcinogen.

Procedure:

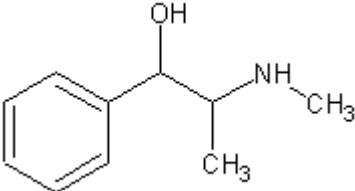
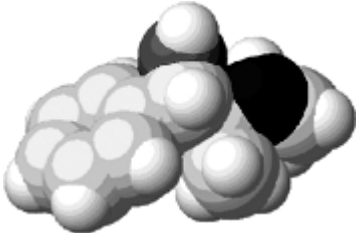
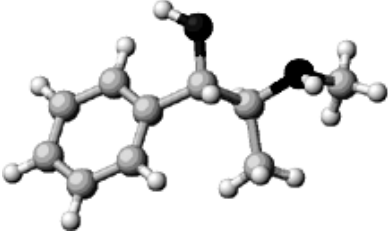
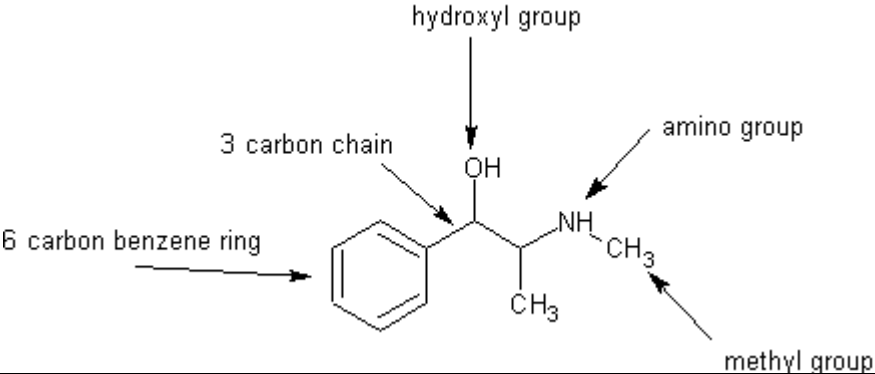
Into a suitable 3-neck flask equipped with a motorized stirrer, thermometer, and addition funnel, place 40 grams of anhydrous aluminum chloride, followed by 39 grams of dry benzene. Then place this flask into an ice bath and chill to about 5 Celsius. Thereafter, prepare a solution by adding and dissolving 5.7 grams of allylamine into 10 milliliters of dry benzene, and then place this solution into the addition funnel. Then slowly add the allylamine/benzene solution to the aluminum chloride/benzene mixture over a period sufficient to keep the reaction mixture below 10 Celsius. During the addition, constantly stir the reaction mixture. After the addition of the allylamine/benzene solution, remove the ice bath, and allow the reaction mixture to warm (do not exceed 50 Celsius). During this warming period, stir the reaction mixture constantly for about 30 minutes. Thereafter, replace the addition funnel with a reflux condenser, and then reflux the reaction mixture at 84 Celsius for about 3 hours. After 3 hours, remove the heat source, and allow the reaction mixture to cool to room temperature over night (whereby a brownish coloration will take place). The following day, pour the reaction mixture into a suitable sized beaker, and then add in 100 grams of crushed ice. After the ice has melted, place the entire mixture into a suitable separatory funnel, and then remove the upper benzene layer (after removing the lower water layer first). The benzene layer can be discarded or recycled. Now, quickly mix 25 milliliters of diethyl ether with the lower water layer and swirl the total contents for about 1 minute (this is to remove the brownish color imparted to the lower water layer). Then place the total mixture into a clean separatory funnel, and remove the upper ether layer (after removing the lower water layer first). The ether layer can be discarded or recycled. Now, place the lower water layer (which should be much clearer now), into a suitable sized beaker, and then slowly add in an excessive amount (100 milliliters of a 30% sodium hydroxide solution), to this water layer, and stir the mixture during the addition and after the addition for 10 minutes. During the addition of the sodium hydroxide solution add in small portions (no more than 100 grams) of crushed ice to the water layer to keep its temperature cool. After the addition of the sodium hydroxide solution, chill the entire water mixture (which should now be two layers) to about 10 Celsius. Note: a freezer works best for this chilling process. Afterwards, place the entire water mixture into a separatory funnel, and then remove the upper product layer, and then place this upper product layer into a clean beaker for the meantime. Note: in some cases, the product layer will be the lower layer depending on how much sodium hydroxide you add. After removing the product layer, extract the water layer with three 50 milliliter portions of diethyl ether, and then combine all three layers afterwards (if not already done so), and then evaporate-off the ether (using a distillation apparatus or rotary evaporator) to obtain a small amount of additional product. Now add this additional product to the main product layer and then dissolve the total volume into 100 milliliters of diethyl ether. Thereafter, place this ether solution of the product into an ice bath, and then slowly pass into the mixture, 3.3 grams of dry hydrogen chloride. During the hydrogen chloride addition, keep the temperature of the reaction mixture below 15 Celsius, and stir. After the addition of the hydrogen chloride, continue to stir the reaction mixture for 30 minutes at a temperature below 10 Celsius. Thereafter, filter-off any precipitated product, carefully wash this filtered-off product with 20 milliliters of diethyl ether (several times using the same 20 milliliter portion), and then vacuum dry or air dry the product. Then evaporate-off the diethyl ether from the remaining filtered mixture using a distillation apparatus or rotary evaporator to recover any dissolved product, and then wash this additional product with 10 milliliters of ether (several time using the same washing portion), and then vacuum dry or air dry the additional product.

Note: Other salts can be formed by treating beta-methylphenylethylamine freebase with the corresponding acid in an ether/ethyl alcohol solution. For example, the sulfate or tartaric acid salts can be prepared by dissolving the freebase into an ether/ethyl alcohol solution (1 gram of freebase into 10 parts ether/5 parts 95% ethyl alcohol, and then adding the desired acid (2 moles of freebase product with 1 mole sulfuric or tartaric acids). For the citric acid or phosphoric acid salts, prepare the freebase solution in ether/ethyl alcohol as previously described, but use 3 moles of freebase per 1

mole of phosphoric acid or citric acid. For each case, the ether/alcohol mixture should then be evaporated, but only evaporated to the point where 80% of the total volume is reduced. The resulting ether/alcohol concentrate can then be filtered to recover the precipitated product. All the salts are very powerful stimulants and are preferred over the freebase compound.

Procedure 45: The Preparation of Ephedrine

Also known as: 2-(methylamino)-1-phenylpropan-1-ol

	 3D structure 1  3D structure 2
Chemical structure	3D Structure
	$C_{10}H_{15}NO$
Structure makeup	Condensed chemical structure

Ephedrine

Ephedrine is an interesting compound that has several forms. The DL-form forms colorless to whitish crystals with a melting point of 79 Celsius. The L-form forms colorless to white crystals with a melting point of 34 Celsius, and the crystals may contain up to $\frac{1}{2}$ molecule of water of hydration. Ephedrine also forms a pseudoephedrine with a melting point of 119 Celsius. Ephedrine is naturally occurring, and exists in the aforementioned forms from Chinese Ma Huang herb (*Ephedra vulgaris*, *E. sinica* Stapf., *E. equisetina* Bunge, Gnetaceae), from which it can be extracted with several solvents. Ephedrine is widely used in cold and nasal products and for use as a bronchodilator (the L-form).

Method 1: Preparation of ephedrine, DL and L forms (as seen in A Laboratory History of Narcotics, vol.1, by Jared Ledgard)

Materials:

1. 53 grams of benzaldehyde	7. 7 grams of a 37% formaldehyde solution
2. 37 grams of nitroethane	8. 6 grams of finely divided zinc
3. 30 milliliters of a 30% potassium carbonate solution	9. 6 grams of glacial acetic acid
4. 650 milliliters of diethyl ether	10. 6 grams of hydrogen sulfide gas
5. 90 milliliters of a 10% sodium bisulfite solution	11. 10 milliliters of 35 to 38% hydrochloric acid
6. 30 grams of anhydrous magnesium sulfate	12. 6 grams of anhydrous sodium carbonate

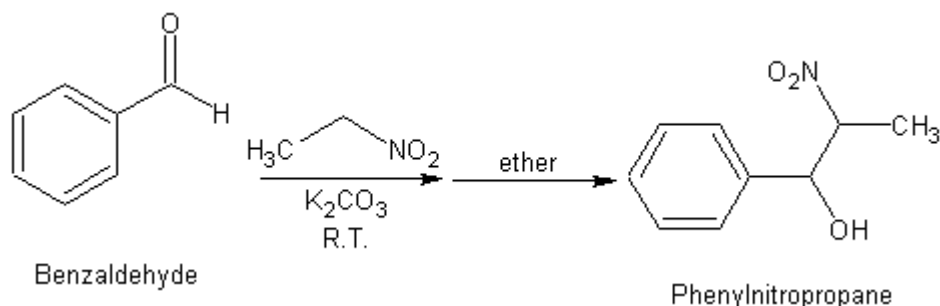
Reaction summary: Ephedrine is a major starting material for the preparation of methamphetamine and other stimulants. In this process, ephedrine is prepared by the condensation of benzaldehyde with nitroethane in the presence of potassium carbonate. The reaction is rather general, and afterwards, the mixture is treated with diethyl ether, and then a solution of sodium bisulfite, which removes any unreacted benzaldehyde. Thereafter, the ether layer is recovered, washed, and then dried, and the resulting ether mixture then evaporated to recover the intermediate product, phenylnitropropanol. This phenylnitropropanol is then reduced by reacting it with formaldehyde in the presence of acetic acid and zinc dust. After the reduction, the reaction mixture is treated with hydrogen sulfide (to precipitate zinc), and the resulting mixture is then filtered, and then treated with ether followed by hydrochloric acid. The ether layer is then removed, and the lower water layer is then treated with sodium carbonate to liberate the freebase of ephedrine. The freebase is then extracted into ether, the ether layer is then recovered, and then evaporated to recover the desired ephedrine. For related information, see Serial number, 88,224, April 1st, 1916 by Wilhelm Nagajoshi, of Tokyo Japan, to M. Dick Bunnell, of San Francisco, CA; also see Serial number, 433,816, March 6th, 1930, by Chogi Nagai of Tokyo, Japan, to Alexander Nagai, of Berlin Germany.

Hazards: Use care when handling nitroethane, and diethyl ether, both of which form explosive mixtures with air and are highly flammable. Use caution when handling hydrogen sulfide gas, which is very toxic and flammable. Use maximum ventilation when handling concentrated formaldehyde solution, and avoid inhalation. Wear gloves when handling concentrated acetic acid, and hydrochloric acid, as they are both corrosive and can cause irritation.

Procedure:

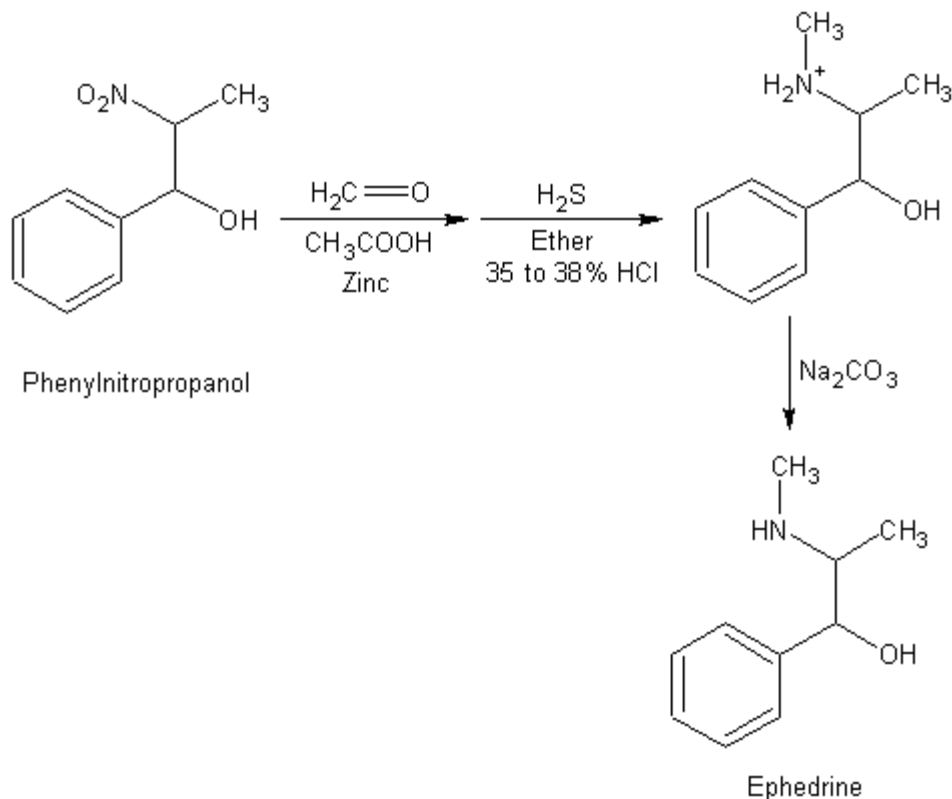
Step 1: Preparation of phenylnitropropanol

Into a suitable beaker or flask, place 53 grams of benzaldehyde, followed by 37 grams of nitroethane. Immediately thereafter, add in 30 milliliters of a 30% potassium carbonate solution, and rapidly stir the entire mixture at room temperature for 2 hours. Note: A cold-water bath may or may not be needed to keep the reaction mixture at ambient temperature (room temperature). Do not allow the reaction mixture to get above 25 Celsius. After stirring for 2 hours, add to the reaction mixture 200 milliliters of diethyl ether, and shortly thereafter, add in 90 milliliters of a 10% sodium bisulfite solution, and then moderately stir the entire reaction mixture for 30 minutes. Afterwards, place the entire reaction mixture into a separatory funnel, and then remove the upper ether layer (after removing the lower aqueous layer). Thereafter, wash this upper ether layer with three 75-milliliter portions of cold water. Note: after each washing portion, use a separatory funnel to recover the ether layer, which will be the upper layer each time. After the washing portion, add to the ether layer, 15 grams of anhydrous magnesium sulfate (to absorb water), and then stir the entire mixture for 10 minutes. Then filter-off the magnesium sulfate. Then, place the filtered ether mixture into a distillation apparatus, or rotary evaporator, and remove the ether. When no more ether passes over or is collected, remove the remaining oily residue (after allowing it to cool to room temperature), and then place aside for step 2. This oily residue will consist of the desired phenylnitropropanol.



Step 2: Preparation of Ephedrine

Into a suitable flask equipped with motorized stirrer, and gas inlet tube, place 15 grams of the oily residue obtained in step 1, and then place this flask into an ice bath, and chill to 0 Celsius. When the oily residue from step 1 reaches a temperature of about 0 Celsius, add in 7 grams of a 37% formaldehyde solution. Thereafter, begin to stir these contents vigorously, and then carefully add in an acetic acid solution (prepared by adding and dissolving 6 grams of glacial acetic acid into 14 milliliters of ice cold water), and then carefully add in 6 grams of finely divide zinc (preferable zinc dust). During the addition of the acetic acid solution and zinc, vigorously stir the reaction mixture, and maintain its temperature below 5 Celsius at all times. After the addition of the zinc, continue to stir the reaction mixture below 5 Celsius for about 90 minutes to complete the reaction. After 90 minutes, stop stirring, and then filter-off any insoluble zinc or other materials. Thereafter, place the filtered reaction mixture back into the ice bath, and then bubble into this filtered reaction mixture, 6 grams of hydrogen sulfide gas. Note: during the addition, vigorously stir the reaction mixture. Note: the addition of the hydrogen sulfide will precipitate any dissolved zinc. After the addition of the hydrogen sulfide, filter-off any insoluble materials, and then add to this filtered mixture, 250 milliliters of cold water, followed by 150 milliliters of diethyl ether, followed by 10 milliliters of a 35 to 38% hydrochloric acid solution. Then vigorously stir this entire mixture for about 1 hour. After 1 hour, place the entire reaction mixture into a separatory funnel, and remove the lower water layer (which will contain the desired ephedrine product as the hydrochloride). Note: the upper ether layer can be discarded or recycled if desired. Now, to the recovered lower water layer, add in a sodium carbonate solution prepared by adding and dissolving 6 grams of anhydrous sodium carbonate into 15 milliliters of cold water, and then stir the whole mixture for about 15 minutes. Finally, extract this entire mixture with three 100-milliliter portions of diethyl ether, and after the extraction process (after each extraction, the ether will be the upper layer), combine all ether portions, if not already done so, and then dry this combined ether portion by adding in 15 grams of anhydrous magnesium sulfate. Then stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Thereafter, place this filtered ether mixture into a distillation apparatus or rotary evaporator, and remove the ether. When no more ether is recovered, recover the left over remaining residue (after it has cooled to room temperature), and then store in an appropriate amber glass bottle until use.

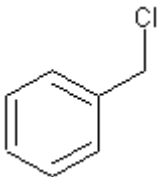
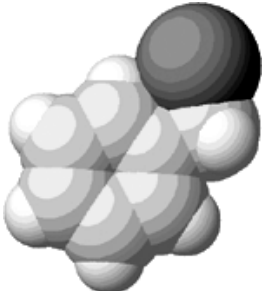
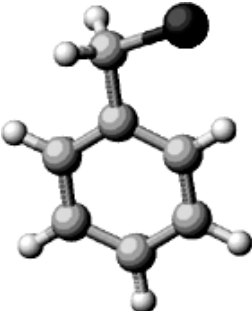
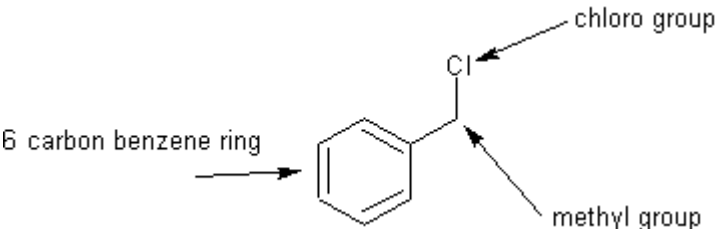


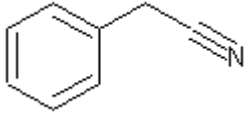

Note: Other salts of the freebase ephedrine such as the sulfate, tartrate, citrate, and phosphate can be prepared by adding the corresponding acid to the freebase compound obtained at the end of step 2 (the freebase should be dissolved into ether in the ratio of 1 grams of freebase to 15 grams of diethyl ether). For the hydrochloride, 1 mole of acid gas should be bubbled into the ether mixture for every 1 mole of freebase ephedrine (not for the total weight of the ether mixture). For sulfuric acid or tartaric acid, 1 mole of sulfuric acid or d-tartaric acid should be added for 2 moles of the freebase ephedrine (not for the total weight of the ether mixture); and for citric acid or phosphoric acid, 1 mole of the citric acid or phosphoric acid should be added to 3 moles of the freebase ephedrine (not for the total weight of the ether mixture). The ether mixture in each of these cases can be

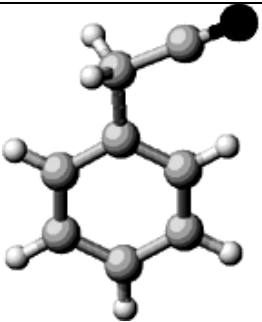
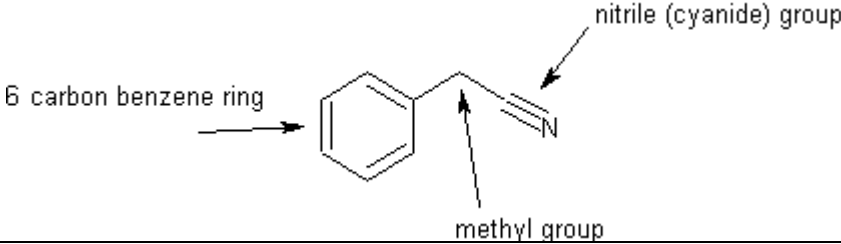
evaporated using a distillation apparatus, or rotary evaporator only to the point where 80% of the total volume is reduced. The resulting ether concentrate can then be filtered to recover the product, which can then be vacuum dried or air-dried.

Procedure 46: The Preparation of Benzyl Cyanide and Benzyl Chloride

Also known as: Benzeneacetonitrile and (chloromethyl)benzene

 <p>Benzyl Chloride</p>	 <p>3D structure 1</p>  <p>3D structure 2</p>
Chemical structure	3D Structure
	C_7H_7Cl
Structure makeup	Condensed chemical structure

 <p>Benzyl Cyanide</p>	 <p>3D structure 1</p>
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	 <p>3D structure 2</p>
Chemical structure	3D Structure
 <p>6 carbon benzene ring</p> <p>nitrile (cyanide) group</p> <p>methyl group</p>	C_7H_7CN
Structure makeup	Condensed chemical structure

Benzyl Cyanide

Benzyl cyanide forms an oily liquid with an aromatic odor. The liquid has a melting point of -24 Celsius, and a boiling point of 234 Celsius at 760 mm. The oily liquid is insoluble in water, but miscible with alcohol and ether. Benzyl cyanide is toxic, so where nitrile gloves while handling.

Benzyl Chloride

Benzyl chloride forms a liquid with an irritating odor. The liquid boils at 179 Celsius, with a melting point of -43 Celsius. The liquid is miscible in alcohol, chloroform, and ether. The liquid decomposes vigorously in the presence of iron. Keep benzyl chloride in a tightly sealed amber glass bottle in a cool place.

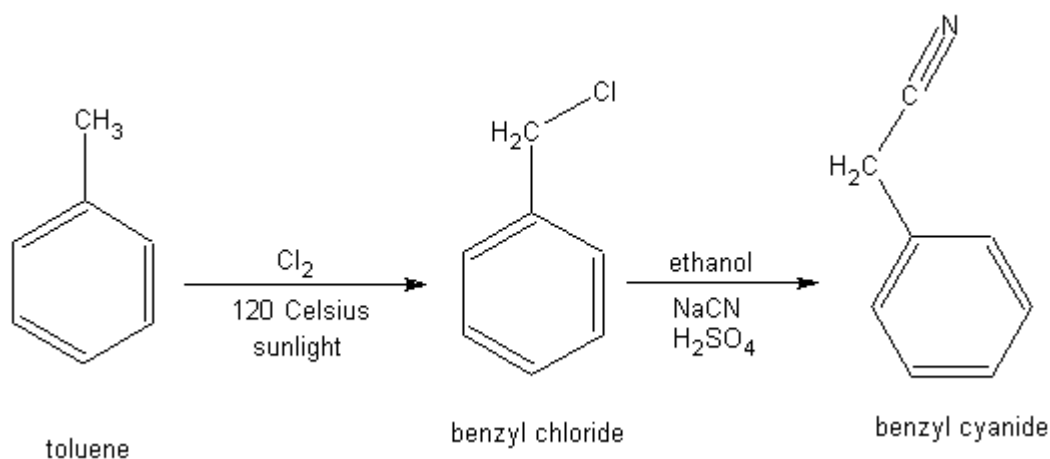
Method 1: Preparation of Benzyl Cyanide and Benzyl Chloride (as seen in A Laboratory History of Narcotics, vol.1, by Jared Ledgard)

Materials:

1. 150 milliliters of dry toluene	2. 30 grams of chlorine gas total
3. 5 grams of finely divided sodium cyanide	4. 10 milliliters of warm water
5. 15 grams of 95% ethyl alcohol	6. Two 25-milliter portions of 95% ethyl alcohol
7. 3 milliliters of 98% sulfuric acid	8. 5 milliliters of water
9. 25 milliliters of a 5% sodium bicarbonate solution	9. 20 milliliter portion of water

Reaction summary: This interesting procedure begins by synthesizing benzyl chloride from toluene. The first step involves the formation of benzyl chloride, which is prepared by the photo chlorination of toluene at 120 Celsius. After the reaction, the desired benzyl chloride can be obtained by distillation using a multiple path distillation apparatus. Multiple path distillation apparatus takes advantage of density, and each fraction will carry over into its own receiver flask based on its density. Because benzyl chloride is more dense than toluene, but less dense than benzylidichloride, it collects in the second receiver flask. After the distillation process, the benzyl chloride can be purified by fractional distillation. Note: benzotrichloride will also be a by-product, and it will remain in the distillation flask. Once the benzyl chloride has been prepared, and has been collected, the rest of the process is relatively down hill. The second step involves the formation of benzyl cyanide, which is conveniently prepared from the benzyl chloride by reaction with sodium cyanide in the presence of alcohol. The reaction is rather general, and after refluxing the reaction mixture for a short amount of time, the reaction mixture is filtered, and then distilled to remove water and solvent. The left over liquid is then treated with a semi-concentrated sulfuric acid solution to break down impurities.

Afterwards, the benzyl cyanide layer is removed from the aqueous acid by use of a separatory funnel, and then distilled using a two-path distillation apparatus—whereby the desired benzyl cyanide collects in the upper receiver flask.

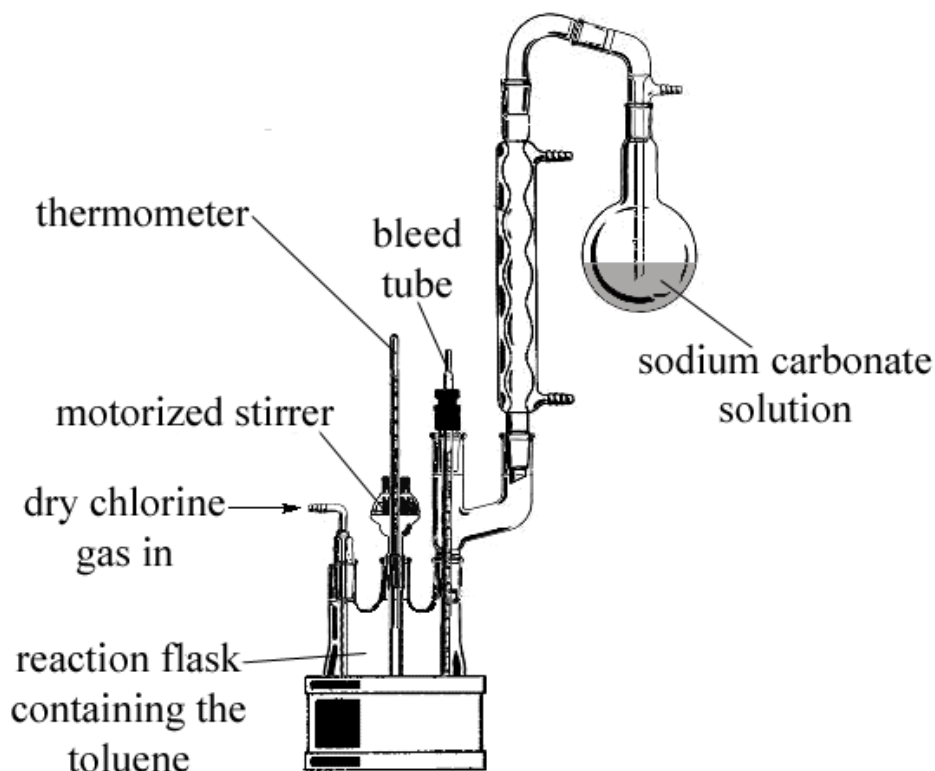


Hazards: Use proper ventilation when using diethyl ether and tetrahydrofuran, as they are both highly flammable and capable of forming explosive mixtures with air. Wear gloves when handling sodium cyanide, and avoid contact with the skin. Keep sodium cyanide away from acids at all times. Wear gloves when handling sodium hydroxide, sulfuric acid, and hydrochloric acid, as they are all capable of producing mild irritation. Magnesium turnings are flammable, so keep them away from strong sources of ignition. Methyl alcohol burns with a colorless flame, so burning methyl alcohol can be hard to see; keep methyl alcohol away from fire and other sources of ignition. Wear gloves when handling sodium borohydride, and keep it out of contact with water. Use proper ventilation when handling toluene, and avoid inhalation of the vapors.

Procedure:

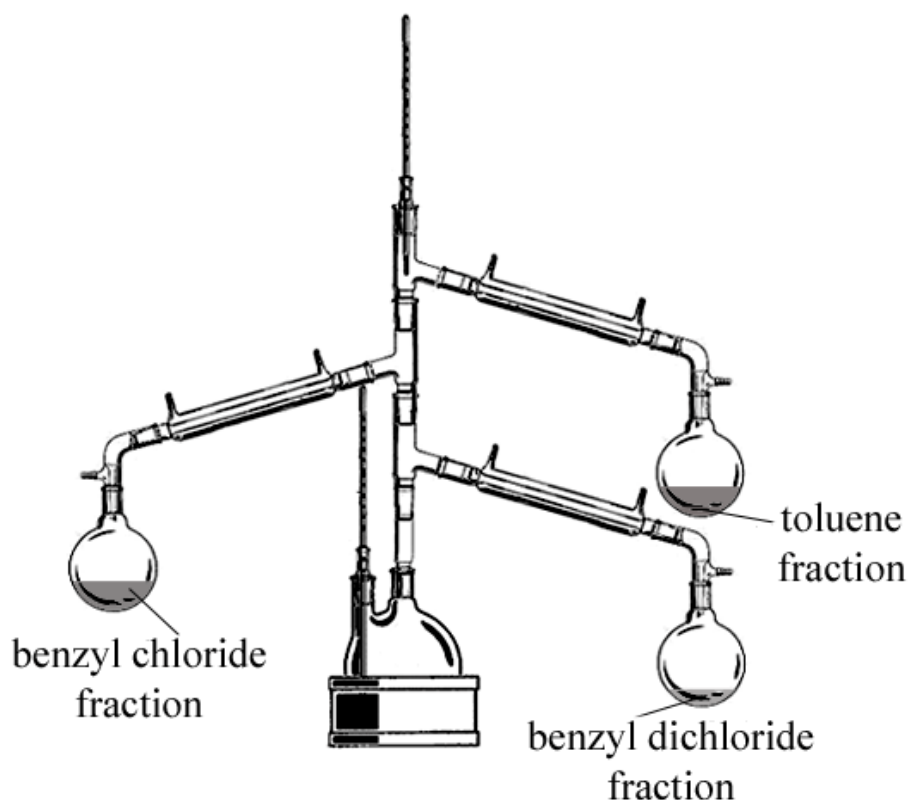
Step 1: Preparation of benzyl chloride through chlorination of toluene

Into the apparatus as illustrated below, place 150 milliliters of dry toluene, followed by a few glass beads (about 2 to 4 millimeters in diameter each glass bead). Thereafter, reflux the toluene at about 110 to 120 Celsius. Note: the temperature should be slowly brought up at the start of the reflux—no rapid heating. When the toluene reaches a temperature of about 90 to 100 Celsius, start bubbling in the chlorine gas (about 30 grams of chlorine gas total; an excess), over a period of about several hours—the chlorine gas addition should not be too fast, and only a nice slow steady stream of gas is desired. Note: the chlorine gas should also be as dry as possible. During the chlorine gas addition, monitor the temperature of the toluene, and do not let it rise above 140 to 150 Celsius. Also, during the addition of the chlorine gas, rapidly stir the toluene. **Note: this entire reaction should be carried out by exposing the reaction flask of the apparatus to direct sunlight, UV light from any suitable UV light bulb, mercury vapor lamp, or halogen lamp. To do this, simply expose the apparatus (preferably only the reaction flask containing the toluene) to direct sunlight (by placing it outside on a sunny day, placing it on a window ledge with the window open on a sunny day, etc., etc.), or simply place the corresponding light bulb 12 to 18 inches away from the reaction flask containing the toluene. A powerful regular light bulb, say a 180 to 220 watt light bulb can also be used to speed up the reaction, but only by 25 to 30% of the ability of sunlight or other UV light sources. To use a regular 180 to 220 watt light bulb, simply place the light bulb 1 to 2 inches away from the reaction flask containing the toluene.** If not using direct sunlight or any other UV light source, the reaction will take much longer, so continue to reflux the toluene at about 110 to 120 Celsius for about 8 to 10 hours (even if the chlorine addition has been completed for some time). If using direct sunlight, or another UV light source, continue to reflux the reaction mixture at about 110 to 120 Celsius for about 3 hours (even if the chlorine addition has been completed for some time). Note: if using a standard light bulb (as of 180 to 220 watt), continue to reflux the toluene at 110 to 120 Celsius for about 5 to 6 hours (even if the chlorine addition has been completed for some time). After the necessary amount of time for refluxing the toluene mixture as ended, stop the reflux process, and allow the toluene reaction mixture to cool to room temperature.



Set-up for the preparation of benzyl chloride. Employed here is a 4-neck reaction flask (the fourth neck is concealed—set behind the center neck). The bleed tube should be submerged just below the surface of the boiling toluene, and is designed to act as relief for any back-pressure in the apparatus. The sodium carbonate solution will neutralize any hydrogen chloride gas formed as a byproduct during the reaction. The chlorine gas inlet tube should be submerged below the surface of the boiling toluene.

After the toluene reaction mixture has cooled to room temperature, pour it into a multiple path distillation apparatus (as illustrated below). Then distill this toluene reaction mixture at about 180 Celsius. Note: if a heating mantle or hot plate capable of achieving such a high temperature is unavailable, use a Bunsen burner—but monitor the flame, and the temperature of the toluene reaction mixture carefully—do not exceed 200 Celsius. Note: obviously, vacuum distillation would be more convenient, and is recommend if such technology is available.



Multiple path distillation apparatus for the distillation of benzyl chloride. Note: the benzyl chloride fraction should then be re-distilled at 179 Celsius using a fractional distillation apparatus.

Step 2: Preparation of benzyl cyanide

Into a suitable sized reflux apparatus, equipped with addition funnel, thermometer, and motorized stirrer or other stirring means, place 5 grams of finely divided sodium cyanide, followed by 10 milliliters of warm water. Thereafter, briefly stir the entire mixture to form a uniform mix. Then prepare a solution by adding and dissolving 10 grams of benzyl chloride (prepared in step 1) into 15 grams of 95% ethyl alcohol. Thereafter, place this benzyl chloride mixture into the addition funnel, and then slowly add it, drop-wise to the sodium cyanide mixture over a period of time sufficient to keep the sodium cyanide mixture below 40 Celsius. During the addition of the benzyl chloride mixture, rapidly stir the sodium cyanide mixture. Note: the addition should take no longer than 10 to 15 minutes. After the addition of the benzyl chloride/alcohol mixture, reflux the entire reaction mixture at 79 Celsius for about 1 hour with rapid stirring. After refluxing the entire reaction mixture for about 1 hour, stop the reflux process, and then allow the reaction mixture to cool to room temperature. Thereafter, filter the reaction mixture to remove insoluble sodium chloride, and then wash this filtered-off sodium chloride with two 25-milliliter portions of 95% ethyl alcohol. Then combine both of these washing portions with the filtered reaction mixture, and then place this combined filtered reaction mixture into a distillation apparatus (equipped with motorized stirrer or other stirring means). Now, before distilling the mixture, add in 20 milliliters of water, and then distill the entire reaction mixture at 100 Celsius to remove water and alcohol. Note: during the distillation process, rapidly stir the entire reaction mixture. When no more water or alcohol passes over or is collected, stop the distillation process, and then allow the remaining left over contents to cool to room temperature. Thereafter, filter this left over remaining liquid (to remove any impurities; if any). Now, place this filtered left over liquid into a clean flask or beaker, and then add in a warm sulfuric acid solution prepared by adding and mixing 3 milliliters of 98% sulfuric acid into 5 milliliters of water. Note: sulfuric acid generates excessive heat when dissolved in water, so allow the acid solution to cool to about 50 to 60 Celsius before using. After adding in the warm acid solution, rapidly stir the entire mixture for about 30 minutes. After 30 minutes, place the entire mixture into a separatory funnel, and then remove the upper benzyl cyanide layer. Then place this upper benzyl cyanide layer into a clean beaker, and then add in, 25 milliliters of a 5% sodium bicarbonate solution, and then stir the entire mixture for about 10 minutes. Thereafter, place this entire two-phase mixture into a separatory funnel, and remove the lower benzyl cyanide layer. Note: in some cases, the benzyl cyanide might be the upper layer. Finally, place this benzyl cyanide layer into a two-path distillation apparatus (similar to the multiple path distillation apparatus used in step 1, except with only two paths, not three), and then distill the benzyl cyanide at 234 Celsius. Note: if a high temperature heating mantle or hot plate is un-available, a Bunsen burner can be used. If using a Bunsen burner, monitor the flame and temperature of the benzyl cyanide closely—do not exceed 250 Celsius. Obviously, vacuum distillation works best. Second note: the benzyl cyanide fraction will collect in the highest receiver flask (the second path). After the

distillation process, collect the benzyl cyanide fraction, and then add to it, a small amount of anhydrous sodium sulfate and then stir the entire mixture for about 5 minutes—thereafter, filter-off the sodium sulfate.

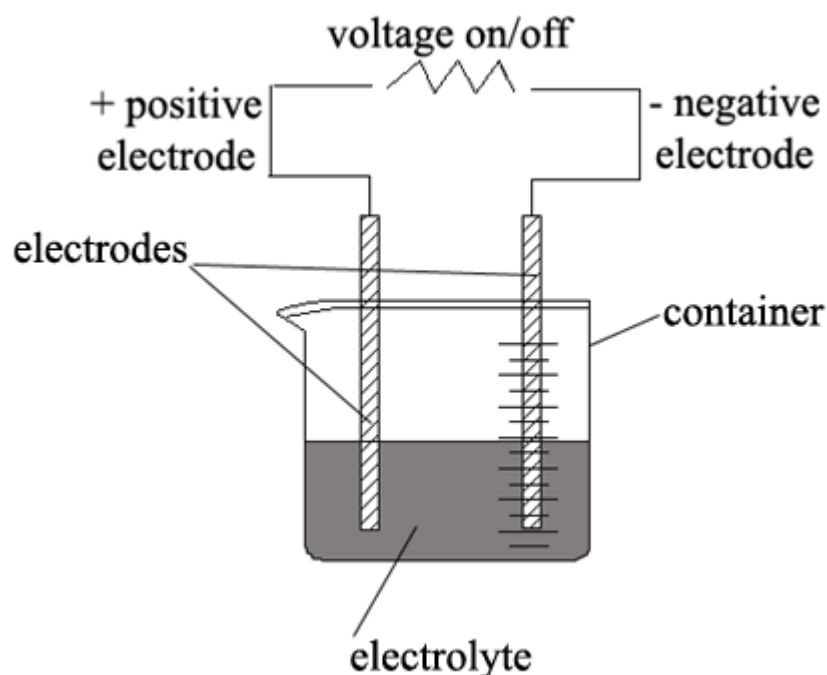
Chapter 9: Electrochemical processes in general chemistry Utilizing “Open Cells”

Introduction to electrochemical methods

Electrochemical processes involve specially designed cells for use in carrying out unique and un-common chemical reactions that would otherwise be impossible. For most people, electrochemistry will be unfamiliar, and strange; however, electrochemistry is by far the most interesting of the laboratory techniques, and can be learned by anyone with ease. You don't have to be a chemist to carryout your own electrochemical processes, and the cell design, function, and operation is very easy to learn and operate. Electrochemistry is a fun aspect of laboratory procedures, and can be used to produce a variety of interesting substances. In general, electrochemical methods can be broken down into two different categories, a) open cells, and b) divided cells.

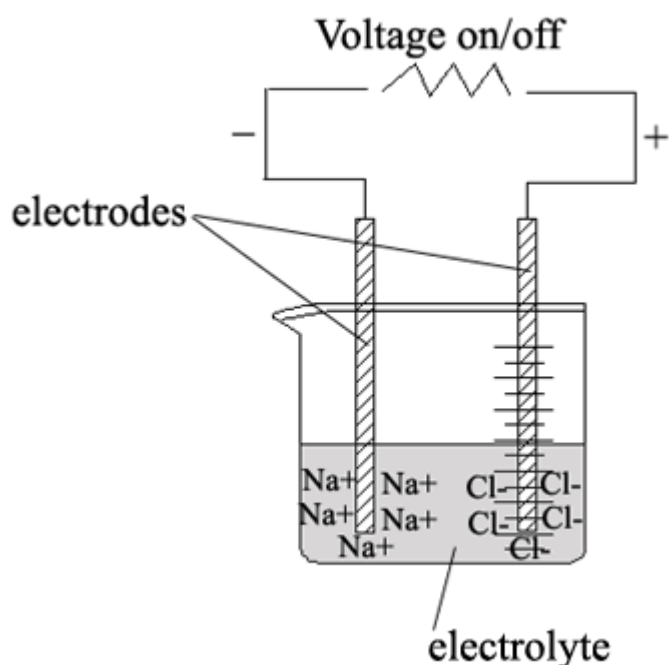
9a. Standard “Open cell” designs and processes

Open cells in electrochemical methods are quite common for use in electroplating, and carrying out general reactions. In an open cell process, the electrodes are adjacent to each other, and not separated by any means such as a salt bridge or porous membrane.



In open cells, a single container is used to house the cell, and at least two electrodes are suspended into the container, which contains an electrolyte. The two electrodes must protrude into the electrolyte making contact with it in order for the cell to work. Also, the electrolyte must be of suitable capacitance in order to generate electricity. The electrolyte generates electricity because it allows the flow of electrons to migrate from negative to positive utilizing the negative and positive ions of the electrolyte, which is how the electricity flows in the electrolyte. The electrodes like the electrolyte must be able to support the flow of electricity, meaning the electrodes must be conductive. Most electrodes are made of metal, but non-metal electrodes commonly found are graphite, and conductive polymers.

In an open cell, at least two electrodes protrude into an electrolyte. The electrolyte is chosen from a series of water-soluble ionic compounds, mainly of the sodium chloride or sulfuric acid type. Some common electrolytes include, table salt, or sodium chloride, sulfuric acid, sodium bromide, sodium carbonate, sodium hydroxide, sodium sulfate, and/or ionic organic compounds such as acetic acid. The electrolyte must be soluble in water forming a solution. This solution is what the electrolyte is.



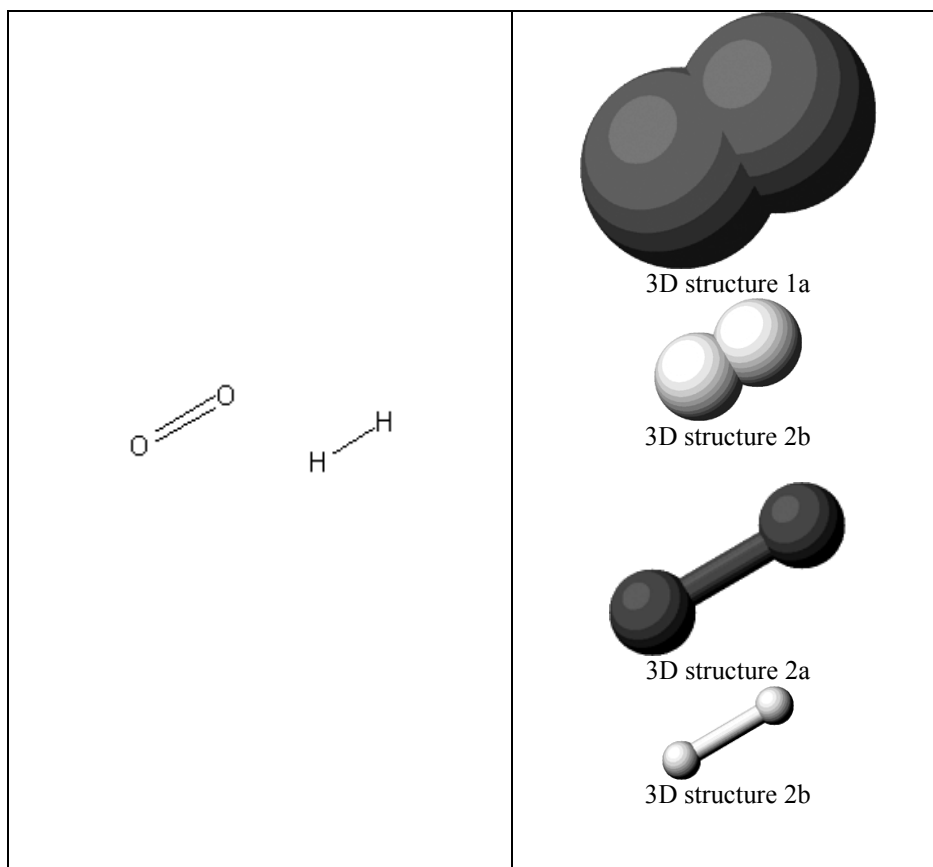
When electrodes are placed into the electrolyte, and a current from a battery or power supply is applied to the negative electrode, the ions that make up the electrolyte migrate to their respective electrodes. For example, when sodium chloride is

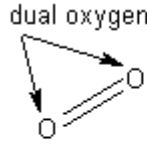
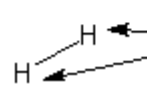
dissolved in water to form a brine electrolyte, and a current is applied to the negative electrode of the system, the positively charged sodium ions migrate to the negative electrode, and the negatively charged chlorine ions migrate to the positive electrode (as such i.e., positive to negative, and negative to positive). The negative electrode is referred to as the cathode, and the positive electrode is referred to as the anode. Now, in order for an electrochemical reaction to happen, you must have an electrolyte, and at least two electrodes (one positive and one negative) protruding into the electrolyte.

What happens when electric current is applied to the system can be summed in the following example: when current is applied to the negative electrode in a system composed of sodium chloride as the electrolyte and two iron rods are used as the electrodes, some of the positively charged sodium ions migrate to the negative electrode, and gain a single electron. At the same time, some of the negatively charged chlorine ions migrate to the positive electrode and give up an electron. This process of gaining and giving up electrons is then repeated millions of times per minute, and is ultimately how the electricity flows through the electrolyte from the negative electrode to the positive electrode. Now, because we used two iron electrodes, when some of the positively charged sodium ions migrate to the negative electrode, and gain a single electron per sodium ion, the sodium ions change from ions to base metal, meaning the sodium ions become free sodium atoms, which however, spontaneously react with the water present forming sodium hydroxide and liberating hydrogen gas. The hydrogen gas bubbles to the surface of the liquid, and at the same time, some of the negatively charged chlorine ions migrate to the positive electrode and give up one electron per chlorine ion. When this happens, the chlorine ions become chlorine atoms in the free state; however, some of the chlorine atoms spontaneously react with the positive iron electrode forming ferrous chloride. However, because sodium hydroxide is being formed at the same time, the sodium hydroxide bonds with the ferrous chloride forming a tacky messy mass of ferrous hydroxide, which precipitates due to its lack of solubility, and sodium chloride is re-formed, which gets recycled back into the electrolyte. This process of reaction is repeated continuously as long as electricity is being applied to the system. Over time, the tacky messy mass of ferrous chloride increases, and at the same time, the positive iron electrode becomes corroded and slowly disappears during the process. Note: the negative electrode of iron is not corroded due to the fact that it's inert towards the sodium hydroxide being formed; however for note only, if the negative iron electrode were replaced with aluminum, the aluminum would be corroded due to its reactivity with sodium hydroxide.

Procedure 47: Electro preparation 1: The Preparation of Oxygen and Hydrogen gases (electrolysis of water)

Also known as: Diatomic oxygen and diatomic hydrogen



Chemical structure	3D Structure
 	O_2 H_2
Structure make-up	Condensed chemical structure

Oxygen gas

Oxygen is a colorless, tasteless, and odorless gas with boiling point of -183 Celsius, and a melting point of -218 Celsius. Oxygen comprises the air we breathe and occupies about 19% by weight of the earth's atmosphere. Pure oxygen is commercially available as a gas compressed into cylinders. Oxygen definitely supports combustion, and excess oxygen can cause ordinary flames to flare up. Oxygen is very reactive and combines with most elements under heat and pressure.

Hydrogen gas

Hydrogen forms a colorless, odorless, and tasteless gas. The gas has a boiling point of an amazing -253 Celsius, with an astronomical melting point of -259 Celsius. The gas is highly flammable and forms explosive mixtures with air. Hydrogen gas is the most abundant element in the universe.

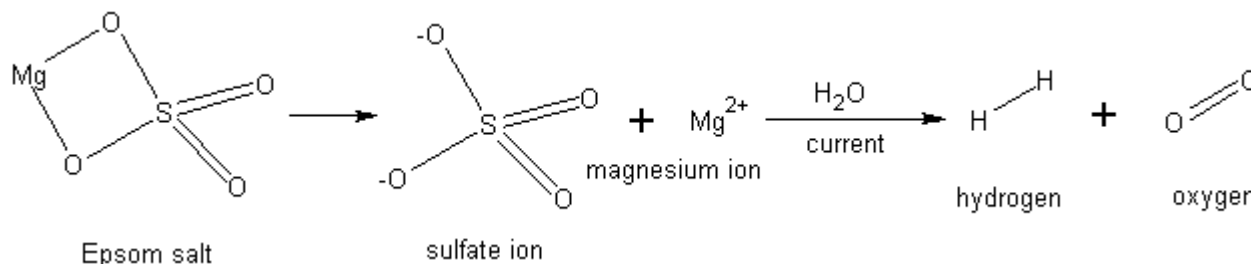
Method 1: Preparation of oxygen and hydrogen gases from Epsom salt and a lead anode utilizing an open cell (electrolysis of water)

(By-products from reaction: a little lead dioxide and a little hydrogen peroxide)

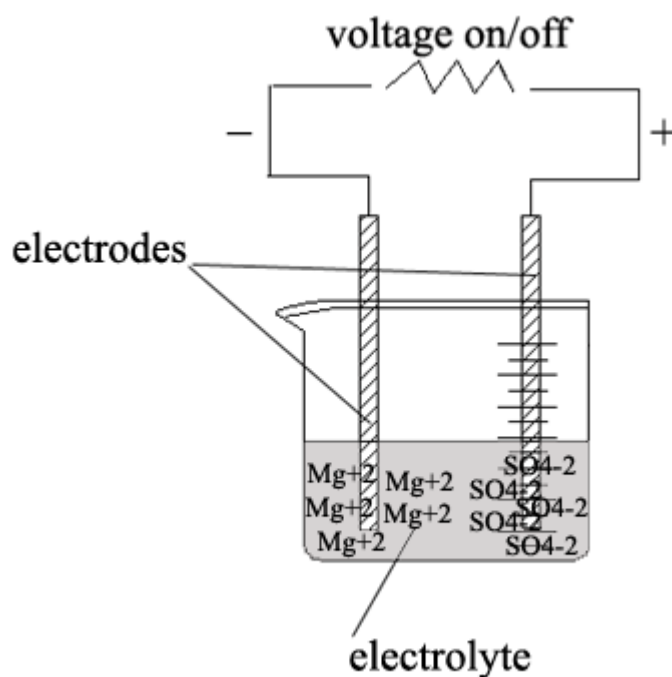
Materials:

1. 150 milliliters (5 fluid oz.) of hot tap water	2. 50 grams (1.7 oz.) of Epsom salt
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Reaction summary: Oxygen and Hydrogen gases are readily prepared by electrolyzing a solution of Epsom salt utilizing a lead anode in an open cell. The reaction is continuous and will proceed so long as there is water and electricity.

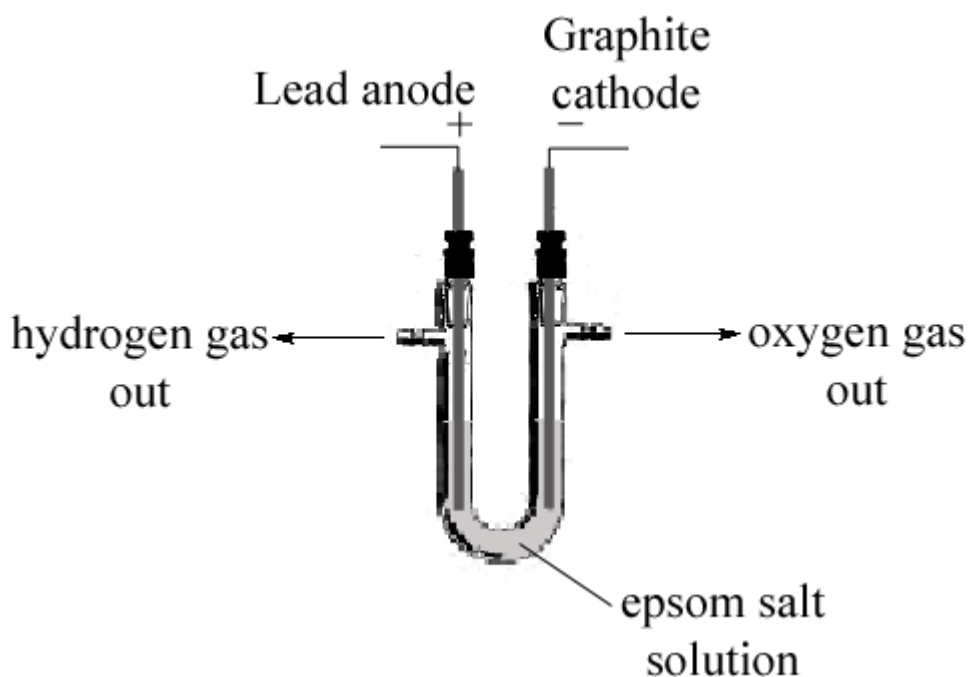


Why does this reaction happen? Epsom salt is the electrolyte, and when it is used as such the following reaction takes place: When current is passed into a solution of Epsom salt, the positively charged magnesium ions migrate to the negative electrode gaining two electrons, and thereby being reduced to elemental magnesium. Because this form of magnesium is very reactive, it reacts with the water forming magnesium hydroxide and liberating hydrogen gas. At the same time, the negatively charged sulfate ions migrate to the positive electrode giving-up 2 electrons and reacting with the water forming sulfuric acid, and liberating oxygen gas. The sulfuric acid then spontaneously reacts with the magnesium hydroxide forming the magnesium sulfate all over again, which gets recycled back into the electrolyte.



Hazards: Extinguish all flames before performing this procedure as hydrogen and oxygen gases are liberated. If these gases are exposed to an open flame an explosion will result.

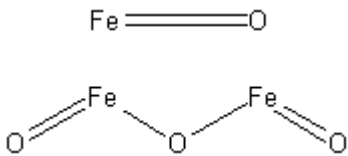
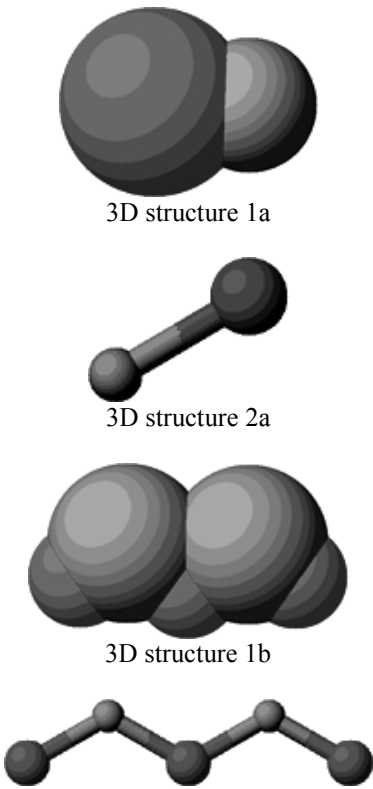
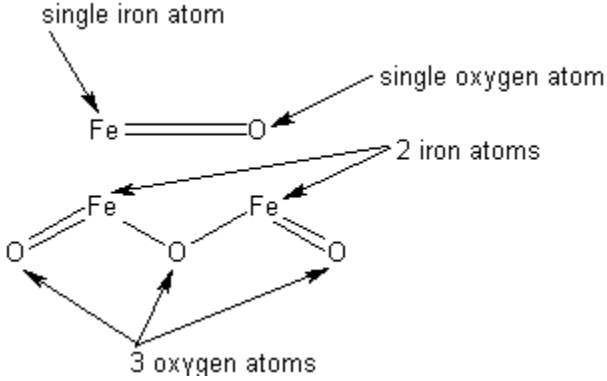
Procedure: Setup a cell as illustrated below, and then into a suitable beaker, place 150 milliliters (5 fluid oz.) of hot tap water. Thereafter, add and dissolve 50 grams (1.7 oz.) of Epsom salt (magnesium sulfate heptahydrate). Then, pour the Epsom salt solution into the apparatus as illustrated below, and then electrolyze the mixture at 2 to 6 amp by 6 to 12 volt DC for as long as period as desired. During the electrolysis process, oxygen and hydrogen gases will be steadily evolved. Also, during the initial electrolysis, a small amount of a brownish solid will form. This brownish solid will be composed of lead dioxide, which will coat the lead anode.



Apparatus for the preparation of oxygen and hydrogen gases.

Procedure 48: Electro preparation 2: The Preparation of Iron oxide(s)

Also known as: Oxides of iron

	 <p>3D structure 1a</p> <p>3D structure 2a</p> <p>3D structure 1b</p> <p>3D structure 2b</p>
<p>Chemical structure Top: Iron-II-oxide Bottom: Iron-III-oxide</p>	<p>3D Structure Top: Iron-II-oxide Bottom: Iron-III-oxide</p>
	<p>FeO</p> <p>Fe₂O₃</p>
<p>Structure make-up</p>	<p>Condensed chemical structure</p>

Iron-II-oxide

Ferrous oxide forms a black powder with a melting point of 1360 Celsius. The powder readily oxidizes when heated in air forming ferric oxide. The powder can absorb carbon dioxide from the air, and is a strong base. It is insoluble in water and the usual solvents.

Iron-III-oxide (ferric oxide)

Iron-III-oxide forms a red to reddish to blood red powder with varying chemical composition. It is insoluble in water, and the usual solvents, and iron-III-oxide that has been roasted for several hours is almost insoluble in acids. Iron-III-oxide is a major ingredient in thermite and other incendiary agents.

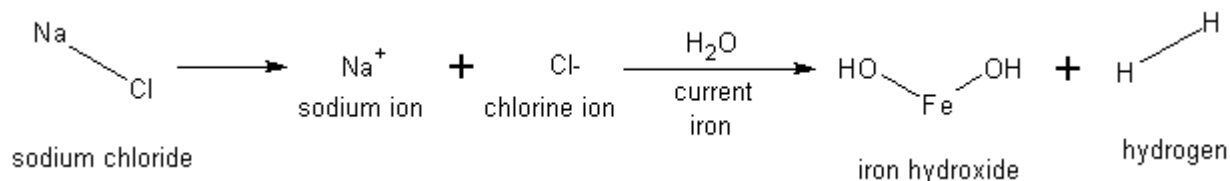
Method 1: Preparation of Iron-II-oxide and Iron-III-oxide from iron nails or rods using an open cell and pickling salt

(By-products from reaction: Hydrogen gas)

Materials:

1. 100 grams (3.5 oz.) of pickling salt	3. Two iron nails, or rods ranging 5 to 12 millimeters in diameter (0.19 inch to 0.47 inch) by 6 to 9 inches in length
2. 400 milliliters (13.5 fluid oz.) of water	4. 750 milliliters of warm water (25.3 fluid oz.)

Reaction summary: Iron-II-oxide can be readily prepared by first, electrolyzing a solution of pickling salt using iron electrodes. During the electrolysis process, a messy precipitate of mixed hydrated iron oxides is formed. Thereafter, this precipitate is collected by filtration, and then dried. The dried messy mass is then dried in a desiccator under mild heat for 12 to 24 hours to facilitate formation of the iron-II-oxide. For the preparation of iron-III-oxide, the same electrolysis process is used to form the initial messy mass, and this mass is then collected by filtration and dried in the usual manner. However, instead of drying this mass in a desiccator, it is roasted at high temperature for several hours to facilitate formation of iron-III-oxide, which is formed by the oxidization of the iron-II-oxides.



Why does this reaction happen? In this procedure, sodium chloride is used as the electrolyte. When current is applied to the cell, some of the positively charged sodium ions migrate to the negative electrode and gain one electron per sodium ion. When this happens the sodium ions are converted into the free state. Because sodium in the free state is so reactive, the free sodium atoms react spontaneously with the water forming sodium hydroxide and liberating hydrogen gas, which bubbles to the surface. At the same time on the other end, some of the negatively charged chlorine ions migrate to the positive electrode giving up a single electron per chlorine. When this happens the chlorine goes from ion to free state, and because chlorine is so reactive the free chlorine atoms react spontaneously with the iron electrode forming ferrous chloride. Because sodium hydroxide is present, the ferrous chloride reacts spontaneously with the sodium hydroxide forming a messy mass of ferrous hydroxide, which precipitates immediately due to its lack of solubility. When the sodium hydroxide and ferrous chloride react with each other, sodium chloride is formed all over again, which gets recycled back into the electrolyte. The reaction will continue as is, until the iron electrode is completely corroded, or the electric current is removed.

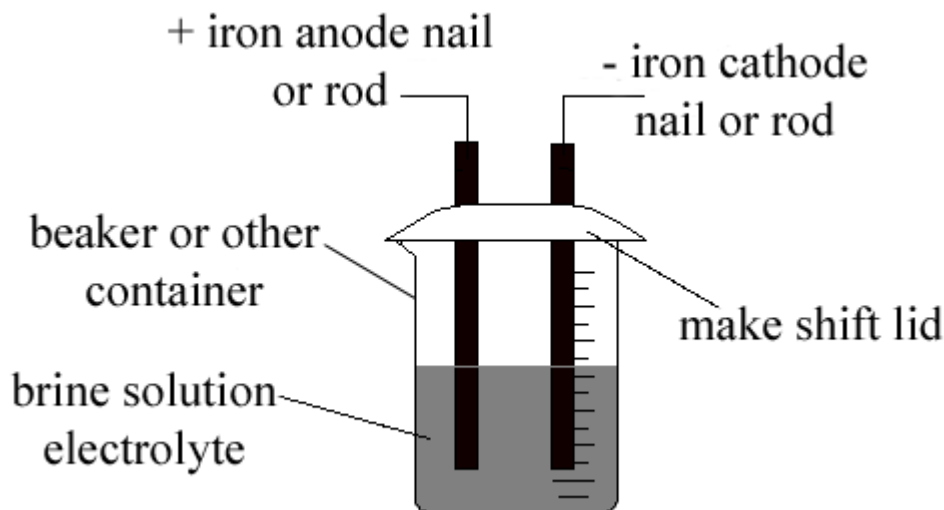
Hazards: use caution when using electricity, and do not touch both electrodes at the same time while the electricity is on.

Procedure:

Step 1: Formation of mixed hydrated iron oxide slag using an open electrochemical cell

Set-up the apparatus illustrated below, and then add and dissolve 100 grams (3.5 oz.) of pickling salt into 400 milliliters (13.5 fluid oz.) of water, and then put in place two iron nails, or rods ranging 5 to 12 millimeters in diameter (0.19 inch to 0.47 inch) by 6 to 9 inches in length, and then place the positive and negative clamps of your power supply onto the corresponding nails, and then begin the electrolysis. Use a current of 6 to 12 volt at 2 to 6 amp. Note: a simple house hold car battery charger is well suitable as a power supply. During the electrolysis, a multi colored precipitate of mixed hydrated iron oxides will precipitate. The color will vary from green to orange-like. Hydrogen gas will be steadily evolved from the cathode so use proper ventilation and do not allow the gas to accumulate. The iron anode will slowly corrode and disappear. The electrolysis process can be slow, and can take any where from 12 to 16 hours to complete, depending on the thickness of the iron nails or rods. Thick nails or rods will take much longer to consume as the electro-chemical reaction proceeds. Thin iron nails or rods should be avoided as they will corrode much faster. After the electrochemical reaction, or after the desired amount of time has passed,

remove the power source, and then filter-off the precipitated messy mass, using preferably vacuum filtration. After the filtration process, wash the filtered-off mass with three 250-milliliter portions (three 8.4 fluid oz. portions) of warm water, and then vacuum dry or air-dry the filtered-off mass. Note: the iron nails or rods should be as pure as possible, and steel, galvanized, and plated iron nails or rods should be avoided to avoid the formation of other metal impurities.



Set-up for the preparation of mixed hydrated iron oxides.

Step 2A: Preparation of iron-II-oxide

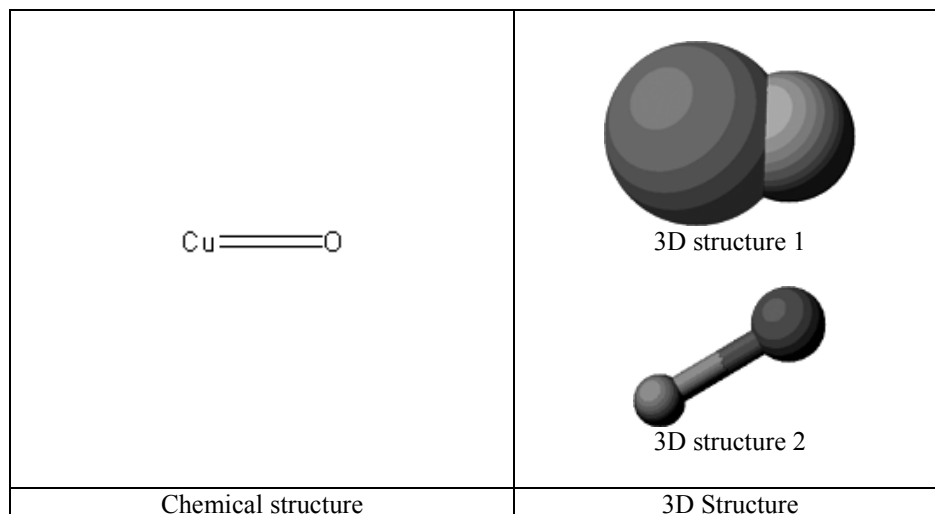
To prepare basic iron-II-oxide, the dried mixed hydrated iron oxide mass (prepared in step 1), needs to be dehydrated under suitable conditions. To do this, place the dried mass into a desiccator, filled with concentrated sulfuric acid, and then gently heat the dried mass to about 50 to 60 Celsius for 12 to 24 hours. After the heating process, the final product should be a jet-black fine mass, which can be ground into a powder if desired. The final dried product should be kept in airtight containers.

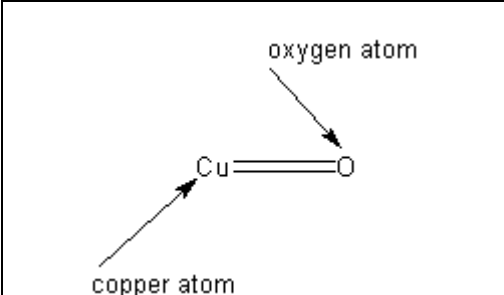
Step 2B: Preparation of iron-III-oxide

To prepare iron-III-oxide, all you need to do is place the dried mass of hydrated iron oxides (prepared in step 1) into a crucible and then heat at 600 to 800 Celsius using a typical Bunsen burner for about 3 to 4 hours. During the heating process, water is volatilized and removed, and the iron oxides are oxidized to iron-III-oxide forming a red powder. After the roasting process, the iron-III-oxide is cooled, and then stored in any suitable container. This iron-III-oxide can be powdered and used in thermite compositions, or pyrotechnics, or used as a catalyst for the oxidation of various gases.

Procedure 49: Electro preparation 3: The Preparation of Copper-II-oxide

Also known as: Cupric oxide



	<p style="text-align: center;">CuO</p>
Structure make-up	Condensed chemical structure

Copper-II-oxide

Copper-II-oxide forms a black to dark brownish-black amorphous or crystalline powder. The powder is insoluble in water and most organic solvents, but soluble in ammonia solution, acids and alkalis. The powder, after being roasted at high temperature for sometime, may become resistant to acids. Copper-II-oxide is widely used in the manufacture of fireworks and other pyrotechnic compositions for imparting a blue flame.

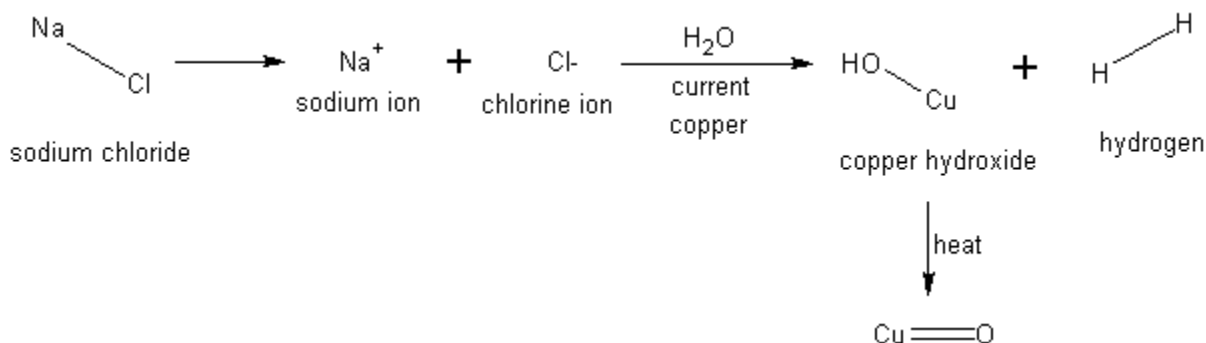
Method 1: Preparation of copper-II-oxide from copper pipes utilizing an open cell and pickling salt

(By-products from reaction: Hydrogen gas)

Materials:

1. 100 grams (3.5 oz.) of pickling salt	3. Two copper pipes, or rods ranging 5 to 18 millimeters in diameter (0.19 inch to 0.70 inch) by 6 to 9 inches in length
2. 400 milliliters (13.5 fluid oz.) of water	4. 750 milliliters of water (25.3 fluid oz.)

Reaction summary: Copper-II-oxide is formed in a similar manner as for iron-II-oxide. It is prepared, first, by electrolyzing a solution of pickling salt using copper electrodes. During the electrolysis process, a messy precipitate of mixed hydrated copper hydroxides are formed. Thereafter, this precipitate is collected by filtration, and then dried. The dry mass is then roasted at high temperature for several hours to facilitate formation of copper-II-oxide, which is formed by the dehydration and oxidization of the hydrated copper hydroxides.



Why does this reaction happen? Similar to nature as in the previous procedure.

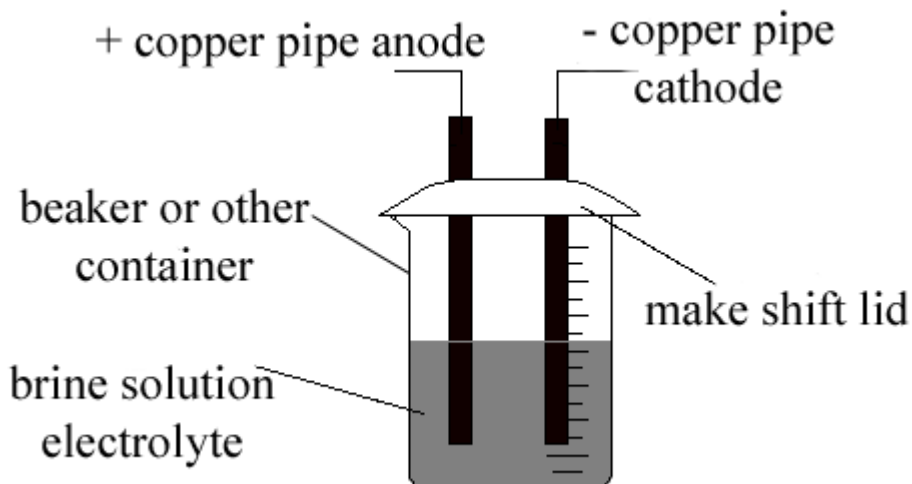
Hazards: use caution when using electricity, and do not touch both electrodes at the same time while the electricity is on.

Procedure:

Step 1: Formation of mixed hydrated copper hydroxides using an open electrochemical cell.

Set-up the apparatus illustrated below, and then add and dissolve 100 grams (3.5 oz.) of pickling salt into 400 milliliters (13.5 fluid oz.) of water, and then put in place two copper pipes, or rods ranging 5 to 18 millimeters in diameter (0.19 inch to 0.70 inch) by 6 to 9 inches in length, and then place the positive and negative clamps of your power supply onto the corresponding pipes, and then begin the electrolysis. Use a current of 6 to 12 volt at 2 to 6 amp. Note: a simple house hold car battery charger is well suitable as a power supply. During the electrolysis, a multi colored precipitate of mixed hydrated copper hydroxides

will precipitate. The color will vary from yellowish to yellowish-orange. Hydrogen gas will be steadily evolved from the cathode so use proper ventilation and do not allow the gas to accumulate. The copper pipe anode will slowly corrode and disappear. The electrolysis process can be slow, and can take anywhere from 12 to 16 hours to complete, depending on the thickness of the copper pipes or rods, and depending on how much of the copper oxide you want. After the electrochemical reaction, or after the desired amount of time has passed, remove the power source, and then filter-off the precipitated messy mass, using preferably vacuum filtration. After the filtration process, wash the filtered-off mass with three 250-milliliter portions (three 8.4 fluid oz. portions) of warm water, and then vacuum dry or air-dry the filtered-off mass.



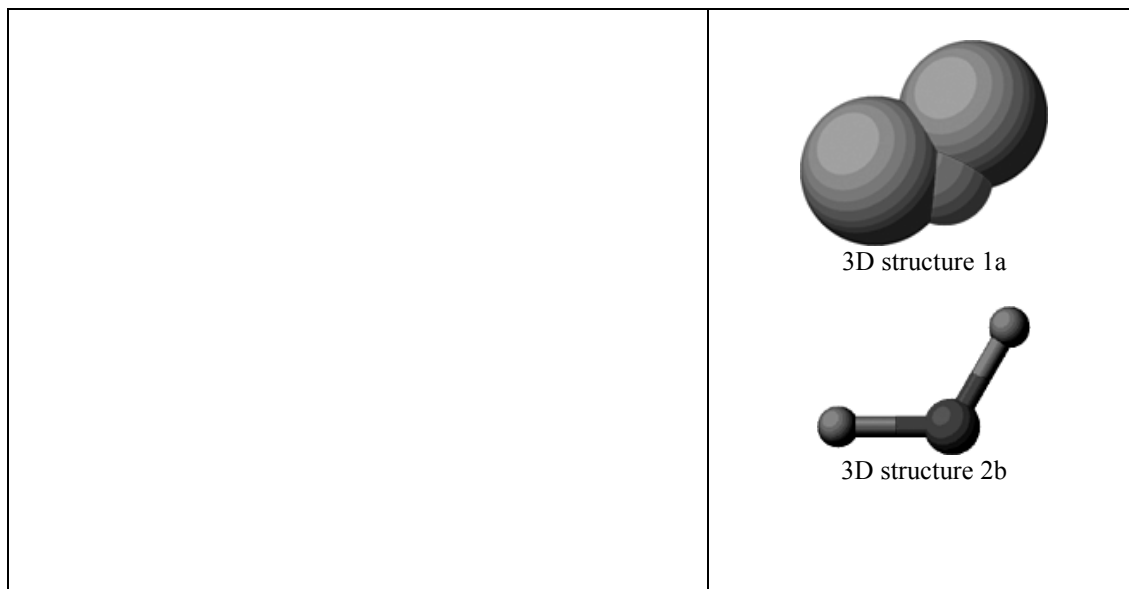
Set-up for the preparation of mixed hydrated copper hydroxides (practically identical to the iron oxide preparations).

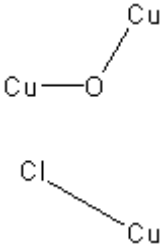
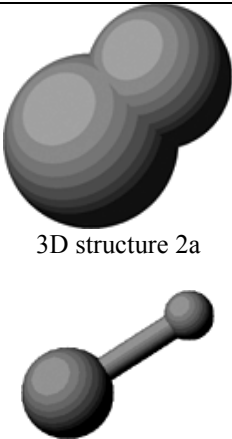
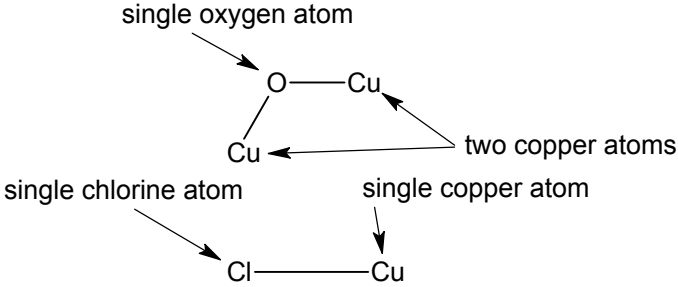
Step 2: Preparation of copper-II-oxide

To prepare copper-II-oxide, all you need to do is place the dried mass of hydrated copper hydroxides (prepared in step 1) into a crucible and then heat at 600 to 800 Celsius using a typical Bunsen burner for about 3 to 4 hours. During the heating process, water is volatilized and removed, and the copper hydroxides are oxidized to copper-II-oxide forming a black powder. After the roasting process, the copper-II-oxide is cooled, and then stored in any suitable container. This copper-II-oxide can be used in pyrotechnic compositions, or used as a catalyst for the oxidation of various gases, such as the oxidation of methanol to formaldehyde.

Procedure 50: Electro preparation 4: The Preparation of Copper-I-oxide and Copper-I-chloride

Also known as: Cuprous oxide, and cuprous chloride



	 <p>3D structure 2a</p> <p>3D structure 2b</p>
<p>Chemical structure Top: Copper-I-oxide Bottom: Copper-I-chloride</p>	<p>3D Structure Top: Copper-I-oxide Bottom: Copper-I-chloride</p>
	<p>Cu_2O</p> <p>CuCl</p>
Structure make-up	Condensed chemical structure

Copper-I-oxide

Copper-I-oxide forms a brick red crystalline powder, which may be yellow, red, or brown depending on method of preparation. The powder is insoluble in water, and the usual solvents, but is readily soluble in acids, and ammonia solutions. The melting point of the powder is about 1232 Celsius, and the dry compound slowly oxidizes to copper-II-oxide on standing. Copper-I-oxide should be used within 1 month of its preparation.

Copper-I-chloride

Copper-I-chloride forms a white crystalline powder, which is stable in dry air, but tends to turn yellow, green, blue, or brown on exposure to moisture, air, and light. Store dry in airtight amber glass bottles. The dry crystals have a melting point of 430 Celsius. The crystals are relatively insoluble in water, with partial decomposition, but are soluble in concentrated hydrochloric acid, and ammonia solutions. Copper-I-chloride can be used in pyrotechnic compositions, for imparting cool effects to the flames. Copper-I-chloride is a very useful catalyst for use in chemistry.

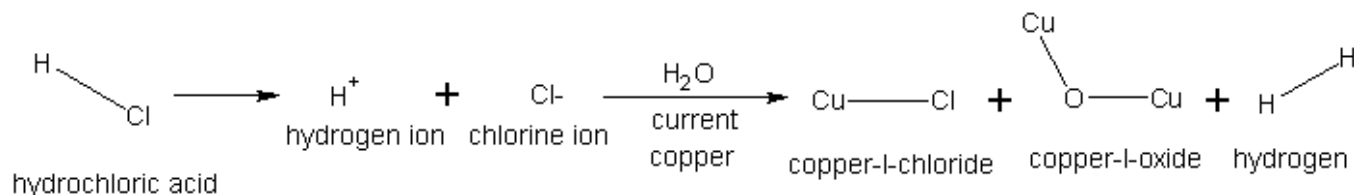
Method 1: Preparation of copper-I-oxide and copper-I-chloride from copper pipes utilizing an open cell and dilute hydrochloric acid

(By-products from reaction: Hydrogen gas)

Materials:

1. 150 milliliters (5 fluid oz.) of concentrated hydrochloric acid or Muriatic acid	3. Two copper pipes, or rods ranging 5 to 18 millimeters in diameter (0.19 inch to 0.70 inch) by 6 to 9 inches in length
2. 750 milliliters (25.3 fluid oz.) of cold water	4. 150 milliliters of alcohol (5 fluid oz.)

Reaction summary: This procedure is rather interesting in the regards that replacing the pickling salt with dilute hydrochloric acid causes something entirely different to happen. In this reaction, the hydrochloric acid reacts with the copper forming copper-II-chloride, which then gets reduced at the cathode forming a brick-red layer of copper-I-oxide, and a small amount of granulated metallic copper. Over at the copper anode, some copper-I-chloride forms, which then falls to the bottom of the cell. This white flaky precipitate of copper-I-chloride can be collected by submerging a small beaker or similar container into the cell underneath the copper anode. The cell has to be stopped multiple times every 5 to 10 minutes in order to scrape-off the copper-I-oxide coating that forms on the copper cathode. If this layer of copper-I-oxide is not scraped-off periodically, the electrochemical reaction will cease, as the circuit is disrupted. The collected precipitate of copper-I-oxide is then washed, and then dried. The copper-I-chloride can be recovered and dried as well. Note: eventually all the chlorine ions will react with the copper of the anode forming copper-I-chloride. When this happens, the current flow will cease, and hence the reaction will stop, or if all the copper anode has been consumed.



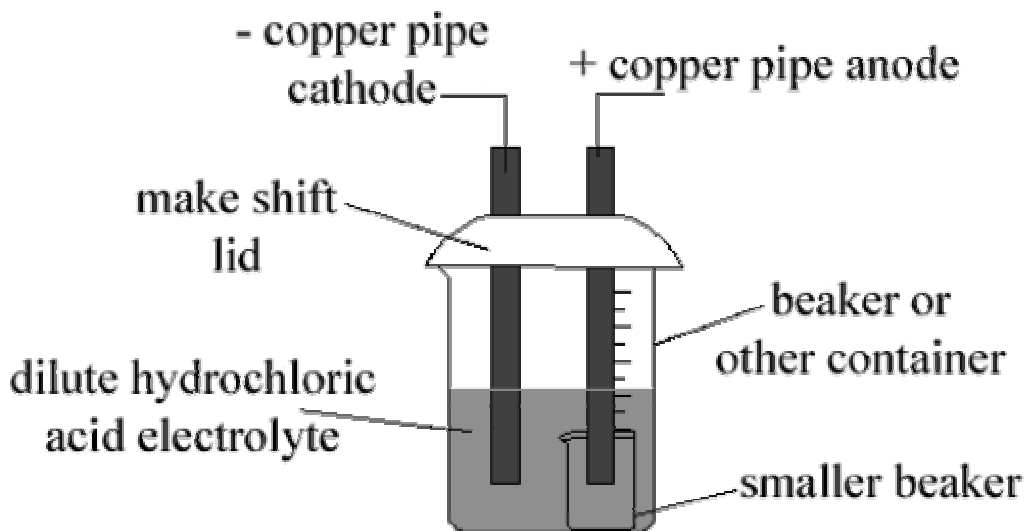
Why does this reaction happen? In this particular reaction, hydrochloric acid is used as the electrolyte. When current is applied to the system, the positively charged hydrogen ions migrate to the negative electrode gaining an electron, and thereby forming hydrogen gas, which bubbles to the surface. At the same time, some of the negatively charged chlorine ions migrate to the positive electrode giving up an electron and forming free chlorine. Because free chlorine is so reactive, it spontaneously reacts with the copper of the electrode forming copper-II-chloride. Now, this copper-II-chloride remains dissolved in the solution and then embarks on a secondary reaction, which works like the following: the copper ions of the copper-II-chloride migrate to the negative cathode where it gains two electrons, and forms the free state of copper, some of which sticks to the cathode as finely divided metallic copper of extra high purity. The rest of the copper ions are reduced at this cathode, reacting with the water and forming copper-I-oxide, which also sticks to the copper cathode. At the same time, the chlorine ions of the copper-II-chloride migrate to the positively charged copper electrode giving up a single electron per chlorine and reacting with the copper forming copper-I-chloride, which precipitates simultaneously due to its lack of solubility in the water.

Hazards: Use caution when handling hydrochloric acid. Use proper ventilation as hydrogen gas is evolved and can accumulate in enclosed areas.

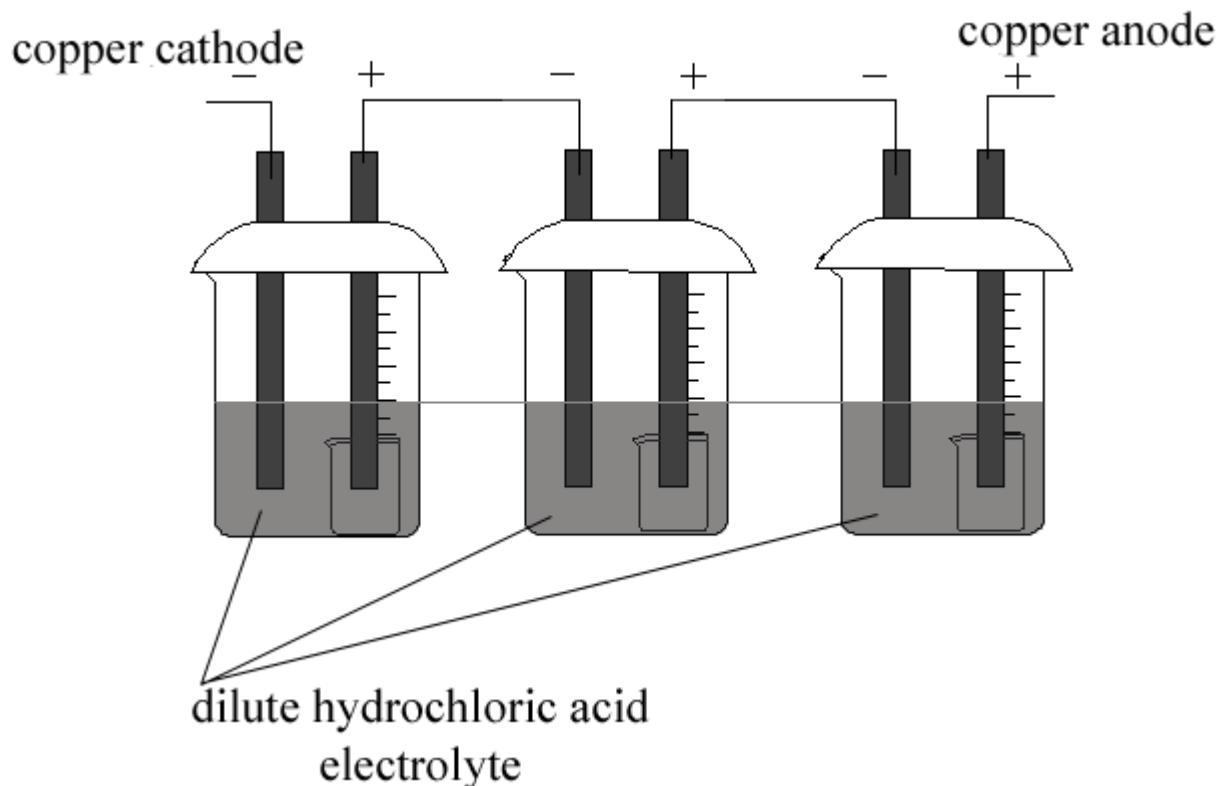
Procedure: As in the previous two procedures, set-up the apparatus illustrated below, and then add and dissolve 150 milliliters (5 fluid oz.) of concentrated hydrochloric acid or Muriatic acid into 750 milliliters (25.3 fluid oz.) of cold water, and then put in place two copper pipes, or rods ranging 5 to 18 millimeters in diameter (0.19 inch to 0.70 inch) by 6 to 9 inches in length, and then place the positive and negative clamps of your power supply onto the corresponding pipes, and then begin the electrolysis. Use a current of 6 to 12 volt at 2 to 6 amp. Note: a simple house hold car battery charger is well suitable as a power supply. Note: A large volume of hydrogen gas will be steadily evolved from the electrodes so use proper ventilation and do not allow the gas to accumulate. The copper pipe anode will corrode and disappear at a steady rate. The electrolysis process will be quite fast, much faster then the previous two procedures, and the power supply will have to be turned-off once every 5 to 10 minutes, and the positive copper cathode will have to be quickly and temporally removed, and then scraped-clean using a spatula or any suitable utensil to recover a layer of copper-I-oxide that forms. After each scraping, simply scrape or place the brick red copper-I-oxide precipitate into any beaker or similar container, and store submerged under water temporarily, until all the desired amount of copper-I-oxide is collected. Note: Over time, the electrolyte will be consumed, as the hydrochloric acid is precipitated in the form of copper-I-chloride. To collect the copper-I-chloride precipitate, simply place a small beaker or similar container directly under the copper anode, submerged, so the white flaky precipitate of copper-I-chloride can be collected as it forms. As the electrochemical reaction proceeds, the acid in the electrolyte is consumed by the formation of the copper-I-chloride. Even though the acid gets consumed, the electrochemical reaction can continue as long as necessary by simply adding in a little more concentrated hydrochloric acid or Muriatic acid. The amount of acid added, should not exceed 150 milliliters so as to prevent your power supply from being damaged due to the high conductivity of the cell. However, to maximize power input, you can hook several cells in series to maximize electrical current consumption.

After the electrochemical reaction, or after the desired amount of time has passed, remove the power source, and then filter-off the precipitated brick-red copper-I-oxide (contained submerged under water in a beaker), and then wash the filtered-off mass with three 250-milliliter portions (three 8.4 fluid oz. portions) of warm water, and then vacuum dry or air-dry the washed solids. Note: do not use heat to dry the copper-I-oxide as it is easily oxidized to copper-II-oxide. Now, for the copper-I-chloride, carefully extract the small submerged beaker (placed under the copper anode to collect the flaky precipitate of copper-

I-chloride), using a suitable utensil or equivalent, and then carefully filter-off the white mass, using preferably vacuum filtration. After the filtration process, wash the filtered-off copper-I-chloride with three 50-milliliter portions (three 1.6 fluid oz. portions) of cold alcohol, and then vacuum dry or air-dry the washed copper-I-chloride. Note: copper-I-chloride is rather unstable and tends to change colors when exposed to air and moisture.



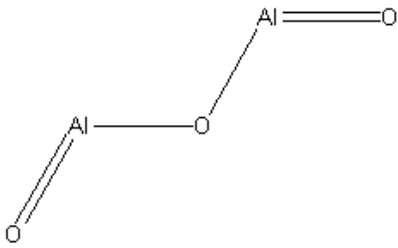
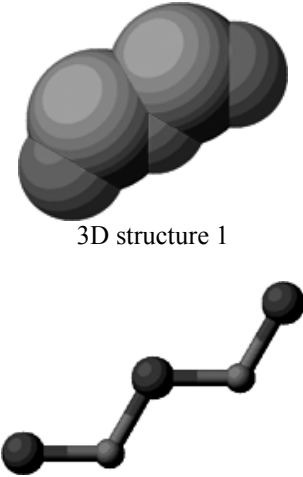
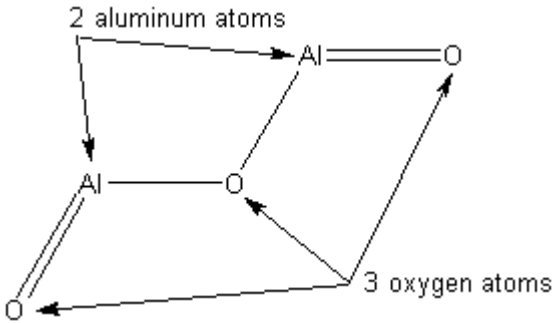
Apparatus for the preparation of copper-I-oxide and copper-I-chloride. The copper-I-oxide forms on the cathode, and then copper-I-chloride collects in the small beaker as a white flaky precipitate. The lid should be non airtight, and is sole used to secure the copper pipes and keep them aligned in a good vertical position without falling over.



Apparatus for running more then 1 cell on the same power supply to maximum current efficiency. This “in series” type operation can be used for any electrolysis procedures discussed in this book, even if using a diaphragm cell, and is not restricted sole to copper-I-oxide and copper-I-chloride as in this procedure.

Procedure 51: Electro preparation 5: The Preparation of Aluminum oxide

Also known as: Oxide of aluminum

	 <p>3D structure 1</p> <p>3D structure 2</p>
Chemical structure	3D Structure
	Al_2O_3
Structure make-up	Condensed chemical structure

Aluminum oxide

Aluminum oxide forms a white powder, granules, or crystalline material. The compound is highly resistant to heat and chemical attack. Aluminum oxide becomes resistant to acids when roasted at 800 Celsius for an hour or so. It is widely used in organic chemistry as a catalyst, filtering aids, chromatography, and purification of organic liquids and solvents.

Method 1: Preparation of aluminum oxide from aluminum, an open cell, and pickling salt

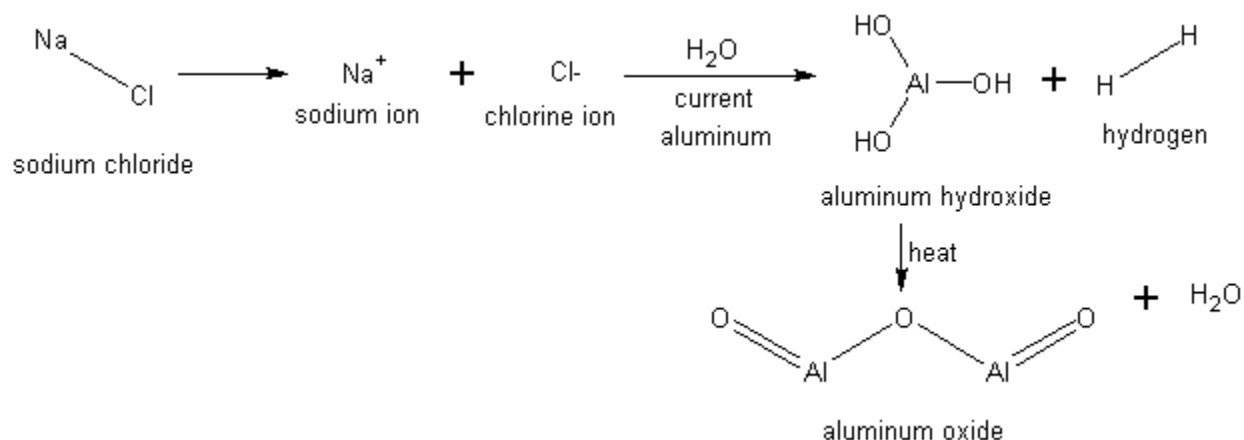
(By-products from reaction: Hydrogen gas)

Materials:

1. 100 grams (3.5 oz.) of pickling salt	3. Two aluminum rods, or nails, ranging from 5 to 12 millimeters in diameter (0.19 inch to 0.47 inch) by 6 to 9 inches in length
2. 400 milliliters (13.5 fluid oz.) of water	4. 750 milliliters of water (25.3 fluid oz.)

Reaction summary: Aluminum oxide can be readily prepared in a similar manner as for iron-III-oxide utilizing an open cell in an identical manner. To prepare aluminum oxide, a solution of pickling salt is electrolyzed using aluminum electrodes. During the electrolysis process, a fine white precipitate of mixed hydrated aluminum hydroxides is formed. Thereafter, this precipitate is collected by filtration, and then dried in the usual manner. The dried mass is then roasted at high temperature for several

hours to facilitate formation of aluminum oxide, which is formed by the dehydration of the mixed hydrated aluminum hydroxides.



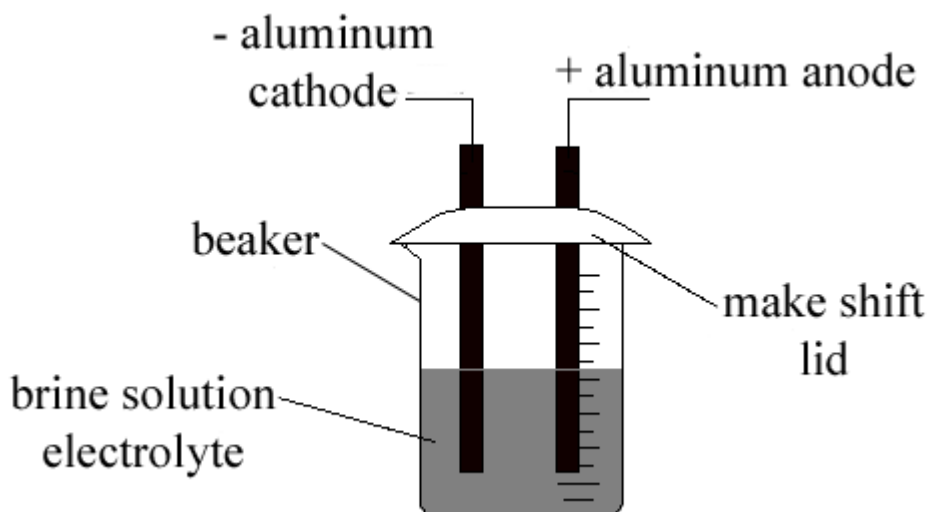
Why does this reaction happen? Similar to the preparation of iron oxide and copper oxide, except the electrode is made of aluminum rather than of iron or copper.

Hazards: use caution when using electricity, and do not touch both electrodes at the same time while the electricity is on.

Procedure:

Step 1: Formation of mixed hydrated aluminum hydroxides.

Set-up the apparatus as illustrated below, and then add and dissolve 100 grams (3.5 oz.) of pickling salt into 400 milliliters (13.5 fluid oz.) of water, and then put in place two aluminum rods, or nails, ranging from 5 to 12 millimeters in diameter (0.19 inch to 0.47 inch) by 6 to 9 inches in length, and then place the positive and negative clamps of your power supply onto the corresponding rods or nails, and then begin the electrolysis. Note: do not use aluminum cans or foil, as impurities will contaminate the end product. Use a current of 6 to 12 volt at 2 to 6 amp. Note: a simple house hold car battery charger is well suitable as a power supply. During the electrolysis, a fine white flaky precipitate of mixed hydrated aluminum hydroxides will precipitate. Hydrogen gas will be steadily evolved from the cathode so use proper ventilation and do not allow the gas to accumulate. As in the usual manner, the aluminum anode will slowly corrode and disappear. The electrolysis process can be slow, and can take anywhere from 12 to 16 hours to complete, depending on the current density and amount of time. Very thin aluminum nails or rods should be avoided as they will corrode much faster. After the electrochemical reaction, or after the desired amount of time has passed, remove the power source, and then filter-off the precipitated flaky mass, using preferably vacuum filtration. After the filtration process, wash the filtered-off mass with three 250-milliliter portions (three 8.4 fluid oz. portions) of warm water, in the usual manner, and then vacuum dry or air-dry the filtered-off mass.



Set-up for the preparation of mixed hydrated aluminum hydroxides. Note: the cathode can be composed of aluminum or graphite.

Step 2: Preparation of aluminum oxide

As in the previous procedures, to prepare aluminum oxide, all you need to do is place the dried mass of hydrated aluminum hydroxides (prepared in step 1) into a crucible and then heat at 600 to 800 Celsius using a typical Bunsen burner for about 3 to 4 hours. During the heating process, water is volatilized and removed, and the hydrate aluminum hydroxides are dehydrated forming a white powder. After the roasting process, the aluminum oxide is cooled, and then stored in any suitable container. This aluminum oxide is well suitable for use as a filtering aid, and for use in silica gel columns for filtration purification.

Procedure 52: Electro preparation 6: The Preparation of highly purified metals and methods for electroplating

In this cool procedure, you can learn how to make highly purified metals, and the foundations of electroplating.

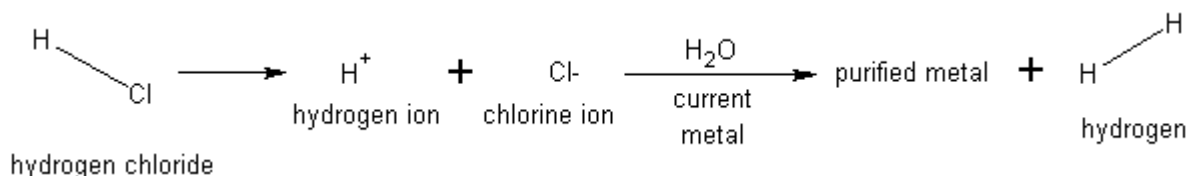
Method 1: Preparation of flaked highly purified iron, zinc, nickel, gold, chromium, silver, cadmium, titanium, cobalt, or zirconium using an open cell, and dilute hydrochloric acid

(By-products from reaction: Hydrogen gas)

Materials:

1. 450 milliliters (15.2 fluid oz.) of cold water	3. 1 iron, zinc, nickel, gold, chromium, silver, cadmium, titanium, cobalt, or zirconium nail, rod, wire or similar objects ranging from 5 to 15 millimeters (0.19 inch to 0.59 inches) in diameter by 6 to 9 inches in length
2. 25 milliliters (0.84 fluid oz.) of concentrated hydrochloric acid	4. 1 graphite rod of 5 to 10 millimeters (0.19 inch to 0.39 inch) in diameter, or graphite rods of 1/4 th inch diameter by 6 to 12 inches in length

Reaction summary: Highly purified flaked metals can be prepared by electrolyzing a dilute solution of hydrochloric acid utilizing a corresponding metal anode and a graphite cathode. The electrolysis is generally simple, and produces beautiful flakes of the desired metal.



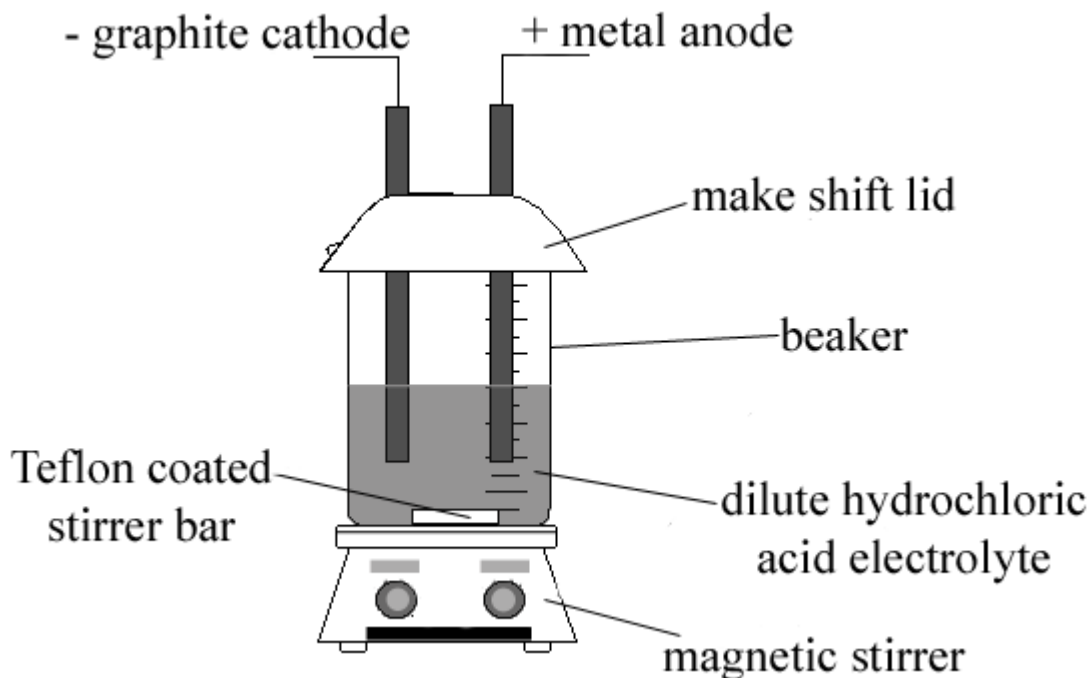
Why does this reaction happen? Hydrochloric acid is used as the electrolyte and as in previous procedures when current is passed into the system, the hydrogen ions migrate to the negative cathode, and the chlorine ions migrate to the positive anode. The hydrogen ions gain an electron forming free hydrogen gas, which bubbles to the surface. At the same time the chlorine ion gives up a single electron per chlorine forming free chlorine, which simultaneously reacts with the metal anode forming the corresponding metal chloride. Thereafter, a secondary reaction takes place with the metal chloride. The metal ion of the metal chloride migrates to the negative cathode whereby the metal gains electrons forming the free metal. When the metal is iron, zinc, gold, silver, chromium, or cobalt, the free metal precipitates out of solution forming a flaky or thin coating on the cathode.

Hazards: Use caution when handling hydrochloric acid, and avoid inhalation of the fumes. Extinguish all flames before carrying out the procedure as hydrogen gas is evolved, and use proper ventilation.

Procedure:

Forming highly purified flaked metals

Set-up the apparatus as illustrated below, and then fill the cell with 450 milliliters (15.2 fluid oz.) of cold water, and then add in 25 milliliters (0.84 fluid oz.) of concentrated hydrochloric acid. Thereafter, turn on the magnetic stirrer, and then stir the mixture and then attach the power supply clamps to the corresponding electrodes, and electrolysis the mixture at 6 to 12 volt at 2 to 6 amp direct current. During the electrolysis, the hydrochloric acid will be consumed forming the corresponding water-soluble metal chloride. Within about 15 to 30 minutes of starting the electrolysis, highly purified corresponding metal begins to form around the cathode. At the same time, the corresponding metal anode will slowly corrode and disappear. Even though the hydrochloric acid gets consumed, the electrolyte does not need to be replaced over time, and the electrochemical reaction can go on as long as there is a corresponding metal anode. In this procedure, the cathode should be composed of a graphite rod of 5 to 10 millimeters (0.19 inch to 0.39 inch) in diameter, and graphite rods of 1/4th inch diameter by 6 to 12 inches in length are preferred. Continue the electrolysis as long as necessary, and afterwards, the highly purified corresponding metal can be filtered-off, washed, and then dried in the usual manner. The metal anode can be composed of iron, zinc, nickel, gold, chromium, silver, cadmium, titanium, cobalt, or zirconium. Note: The metal anodes should be rods, nails or similar objects ranging from 5 to 15 millimeters (0.19 inch to 0.59 inches) in diameter by 6 to 9 inches in length.



Set-up for the preparation of highly pure flaked metals.

Method 2: Preparation of highly purified metal crystals of iron, zinc, nickel, gold, chromium, silver, cadmium, titanium, cobalt, or zirconium using an open cell, and dilute hydrochloric acid

(By-products from reaction: Hydrogen gas)

Materials:

1. 450 milliliters (15.2 fluid oz.) of cold water	3. 1 iron, zinc, nickel, gold, chromium, silver, cadmium, titanium, cobalt, or zirconium rod, nail or similar objects ranging from 5 to 15 millimeters (0.19 inch to 0.59 inches) in diameter by 6 to 9 inches in length
2. 25 milliliters (0.84 fluid oz.) of concentrated hydrochloric acid	4. 1 graphite rod of 5 to 10 millimeters (0.19 inch to 0.39 inch) in diameter, or graphite rod of 1/4 th inch diameter by 6 to 12 inches in length

Reaction summary: Highly purified metal growth crystals can be prepared by electrolyzing a dilute solution of hydrochloric acid in an identical manner as in method 1, utilizing a corresponding metal anode and a graphite cathode. The electrolysis is generally simple, and produces beautiful growth crystals of the corresponding metal forming various elongated shapes.

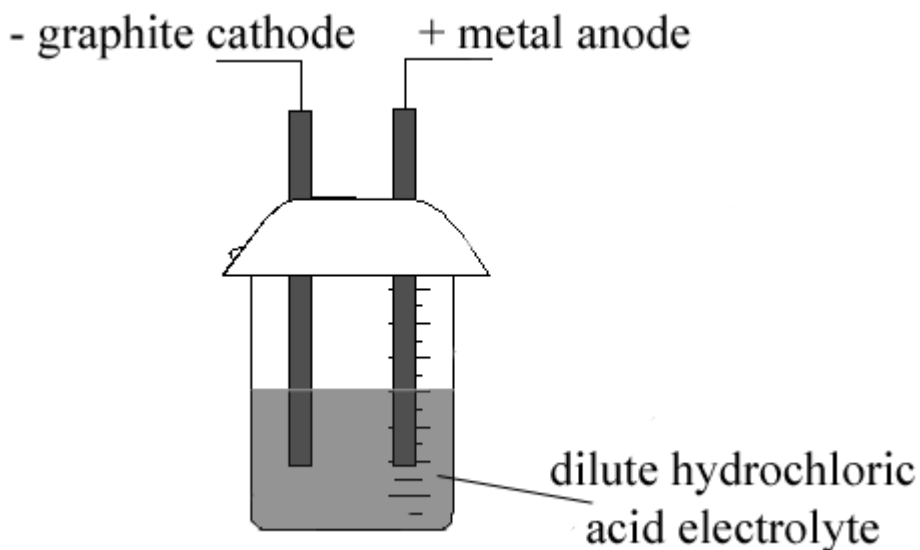
Why does this reaction happen? Same as in method 1.

Hazards: Use caution when handling hydrochloric acid, and avoid inhalation of the fumes. Extinguish all flames before carrying out the procedure as hydrogen gas is evolved, and use proper ventilation.

Procedure:

Growing crystals of highly purified metals

Set-up the apparatus as illustrated below, and then fill the cell with 450 milliliters (15.2 fluid oz.) of cold water, and then add in 25 milliliters (0.84 fluid oz.) of concentrated hydrochloric acid. Thereafter attach the clamps of the power supply to the corresponding electrodes, and electrolysis the mixture at 6 to 12 volt at 2 to 6 amp direct current. During the electrolysis, the hydrochloric acid will be consumed forming the corresponding water-soluble metal chloride. Within about 15 to 30 minutes of starting the electrolysis, highly purified corresponding metal will begin to plate, and then form on the cathode. Over time, the metal will “grow” on the cathode forming beautiful elongated crystals reaching towards the anode. At the same time, the corresponding metal anode will slowly corrode and disappear. Even though the hydrochloric acid gets consumed, the electrolyte does not need to be replaced over time, but the electrochemical reaction will have to be stopped periodically to remove metal growth as it will grow to touch the anode, thereby closing the circuit. In this procedure, as in the above procedure, the cathode should be composed of a graphite rod of 5 to 10 millimeters (0.19 inch to 0.39 inch) in diameter, and graphite rods of 1/4th inch diameter by 6 to 12 inches in length are preferred. Continue the electrolysis as long as necessary. The metal anode can be composed of iron, zinc, nickel, gold, chromium, silver, cadmium, titanium, cobalt, or zirconium. Note: The metal anodes should be rods, nails or similar objects ranging from 5 to 15 millimeters (0.19 inch to 0.59 inches) in diameter by 6 to 9 inches in length.



Set-up for growing highly pure crystals of metals.

Method 3: Electroplating techniques using iron, zinc, nickel, gold, chromium, silver, cadmium, titanium, cobalt, or zirconium using an open cell, and dilute hydrochloric acid or water soluble corresponding salts

(By-products from reaction: Hydrogen gas)

Materials:

1. 1000 milliliters (33.9 fluid oz.) of cold water	4. 50 to 60 grams (1.7 to 2.1 oz.) of zinc chloride, ferrous chloride hexahydrate, hydrated nickel chloride, cobalt-II-chloride, or chromium-II-chloride
2. 25 milliliters (0.84 fluid oz.) of concentrated hydrochloric acid	5. 1000 milliliters (33.9 fluid oz.) of water
3. 10 grams (0.35 oz.) of zinc chloride, ferrous chloride hexahydrate, hydrated nickel chloride, cobalt-II-chloride, or chromium-II-chloride	6. 1 gold, silver, cadmium, titanium, zirconium, iron, zinc, chromium, or cobalt rod, wire, or nails or similar objects ranging from 1 to 10 millimeters (0.03 inch to 0.39 inches) in diameter by 6 to 9 inches in length

Reaction summary: Electroplating is carried out by utilizing identical electrolysis techniques as seen in methods 1 and 2, but instead of forming metal flakes or crystals, the cathode is composed of a desired metal object to be plated. To plate metal objects, the metal object is placed as the cathode, and a solution of either dilute hydrochloric acid and corresponding metal anode, or a solution of a water soluble metal salt utilizing a metal anode or inert anode, is electrolyzed using varying current to plate the desired metal object. The electrolysis process is rapid and can be carried out with success in minimal amounts of time.

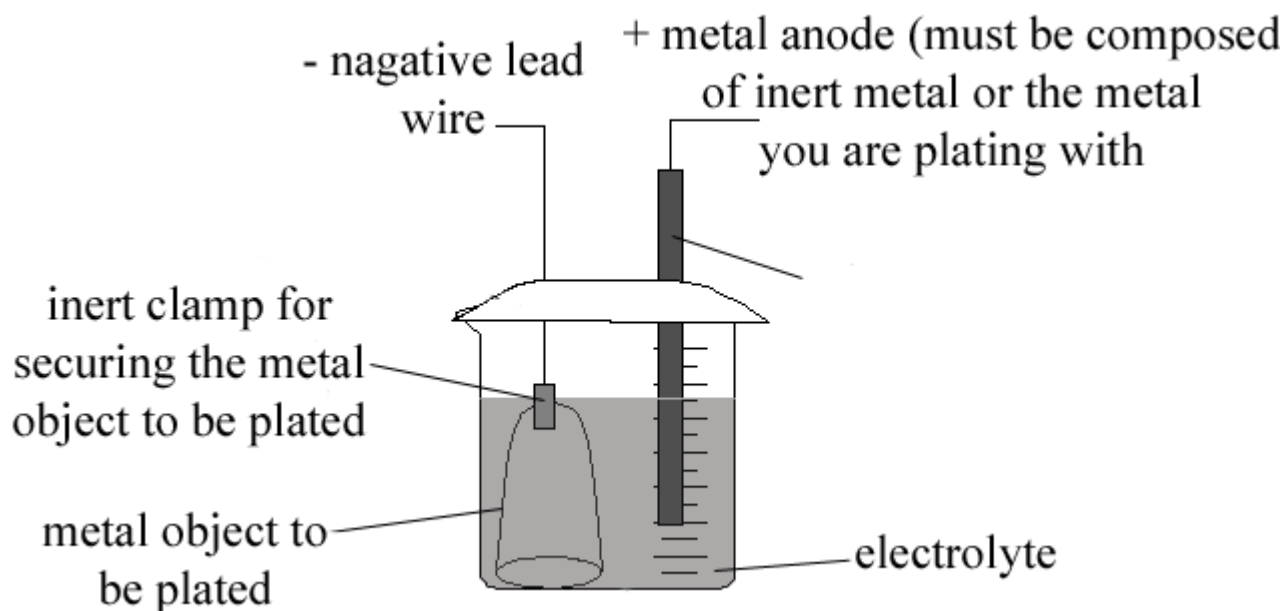
Why does this reaction happen? In electroplating, the electrolyte is composed of a dissolved salt that contains the desired metal to be plated. For example, if we wanted to silver plate a cup or container, we would use silver nitrate as the electrolyte, and we would use the cup or container to be plated as the cathode, and then we would use a silver anode. When current is applied to this type of system, the positively charged silver ions will migrate to the cathode, and gain electrons forming the free metal. The free metal then attaches itself or coats the cathode made up of the cup or container to be plated. At the same time, the negatively charged nitrate ions migrate to the positive anode, give-up electrons, forming the free acid, which then reacts with the silver anode forming silver nitrate all over again. The reaction continues until the silver anode is totally corroded, or the current is removed. In electroplating, the current is only applied for a few minutes to form a thin coating of metal.

Hazards: Use caution when handling hydrochloric acid, and avoid inhalation of the fumes. Extinguish all flames before carrying out the procedure as hydrogen gas is evolved, and use proper ventilation.

Procedure:

Electroplating

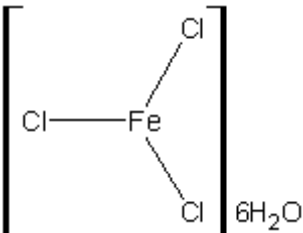
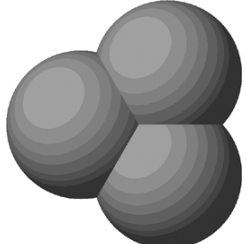
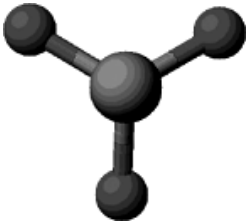
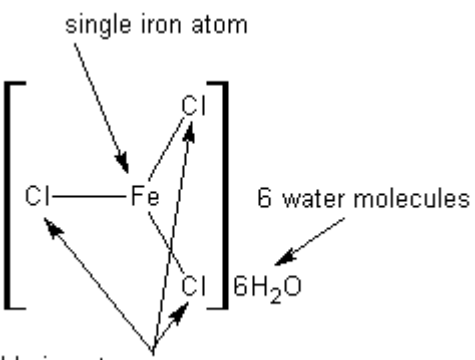
Set-up the apparatus as illustrated below, and then fill the cell with 1000 milliliters (33.9 fluid oz.) of cold water. Now, depending on what you want to plate and with what metal you wish to plate the desired object with, add 25 milliliters (0.84 fluid oz.) of concentrated hydrochloric acid into the cell if you wish to plate gold, silver, cadmium, titanium, zirconium, iron, zinc, chromium, or cobalt and use a metal anode of the metal you wish to plate. For plating iron, zinc, nickel, cobalt, or chromium without the use of hydrochloric acid, dissolve 10 grams (0.35 oz.) of zinc chloride, ferrous chloride hexahydrate, hydrated nickel chloride, cobalt-II-chloride, or chromium-II-chloride into 1000 milliliters (33.9 fluid oz.) of cold water. Note: You must use the exact metal your plating as the anode of the cell if using only 10 grams of dissolved salt. For example, if using 10 grams of nickel-II-chloride, to plate nickel onto an object, you must use a nickel anode, depending on how many objects you wish to plate (as ten grams of dissolved salt will not produce enough metal to plate very many objects; however, if you are using an inert anode (composed of some other material than the metal your plating, you should dissolve 50 to 60 grams (1.7 to 2.1 oz.) of zinc chloride, ferrous chloride hexahydrate, hydrated nickel chloride, cobalt-II-chloride, or chromium-II-chloride (just so there's enough metal to plate the desired number of objects, etc., etc.), into 1000 milliliters (33.9 fluid oz.) of cold water. Also note: if using hydrochloric acid, and if plating gold, silver, cadmium, titanium, or zirconium, it is preferred you use the corresponding metal anode, for example, if you wish to plate gold, it is preferred you use a gold anode; however, this is not necessary as any water soluble salt of gold, silver, cadmium, titanium, zirconium, etc., etc., can be used. In this regards, simply dissolve 10 to 50 grams of any water soluble salt of gold, silver, cadmium, titanium, or zirconium (depending on how many objects you want plated) into 1000 milliliters (33.9 fluid oz.) of water. Thereafter attach the clamps of the power supply to the corresponding metal anode or inert anode, and then secure the negative clamp of the power supply on the metal item you wish to plate. Now, if you're plating gold, silver, cadmium, titanium, zirconium, iron, zinc, chromium, or cobalt, and your using dilute hydrochloric acid, turn on the power supply, and electrolysis the mixture at 6 to 12 volt at 2 to 6 amp direct current but only until the hydrochloric acid is consumed. This point should take anywhere from 10 to 15 minutes, and needs to be monitored carefully, because once the hydrochloric acid has been consumed, the plating of the dissolved metal will being immediately and rapidly. Thereafter, immediately reduce the current to 0.10 to 0.8 volt at 0.002 to 0.10 amps, and then continue to electrolysis the mixture on short 5 to 6 second bursts to form a uniform metal coating on the desired object. Now, if your plating gold, silver, cadmium, titanium, zirconium, iron, zinc, chromium, or cobalt, and your using the corresponding dissolved salt, briefly electrolysis the salt solution at 0.10 to 0.8 volt at 0.002 to 0.10 amp for brief 5 to 6 second bursts for a couple of minutes or so, depending on the desired thickness of the metal plate, and the number of objects to be plated. Note: The metal anodes should be rods, wire, or nails or similar objects ranging from 1 to 10 millimeters (0.03 inch to 0.39 inches) in diameter by 6 to 9 inches in length. Metal foils should be avoided.



Set-up for electroplating. Multiple set-ups can be used, and you should use a design that best suites you.

Procedure 53: Electro preparation 7: The Preparation of Ferric chloride hexahydrate

Also known as: Ferric trichloride 6 hydrate; trichloride of iron with 6 hydrate

	 <p>3D structure 1</p>  <p>3D structure 2</p>
Chemical structure	3D Structure
	<p>FeCl₃·6H₂O</p>
Structure make-up	Condensed chemical structure

Ferric chloride hexahydrate

Ferric chloride hexahydrate forms brownish black crystals. The crystals are readily soluble in water or alcohol, and have a melting point 37 Celsius. Aqueous solutions of ferric chloride hexahydrate are readily decomposed by boiling, forming red ferric oxide, and hydrogen chloride.

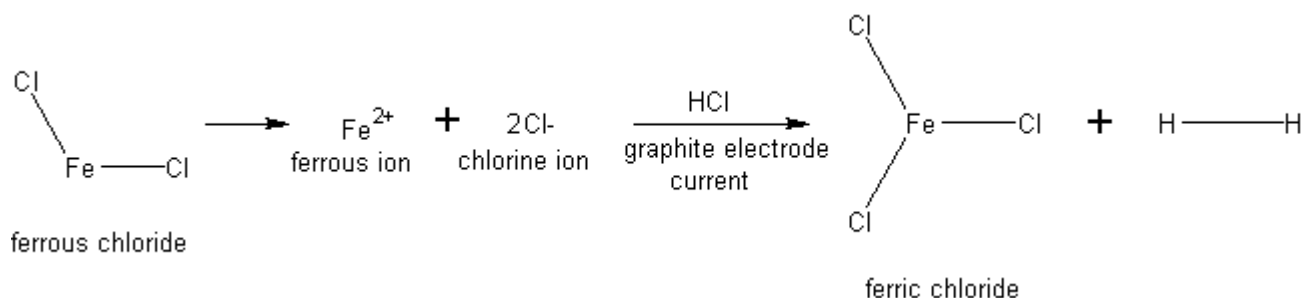
Method 1: Preparation of ferric chloride hexahydrate from ferrous chloride and hydrochloric acid utilizing an open cell and graphite electrodes

(By-products from reaction: hydrogen gas)

Materials:

1. 300 milliliters (10.1 fluid oz.) of cold tap water	3. 59 milliliters (2 fluid oz.) of concentrated hydrochloric acid
2. 100 grams (3.5 oz.) of ferrous chloride tetrahydrate or 100 grams (3.4 oz.) of ferrous chloride dihydrate	4. 72 milliliters (2.4 fluid oz.) of concentrated hydrochloric acid

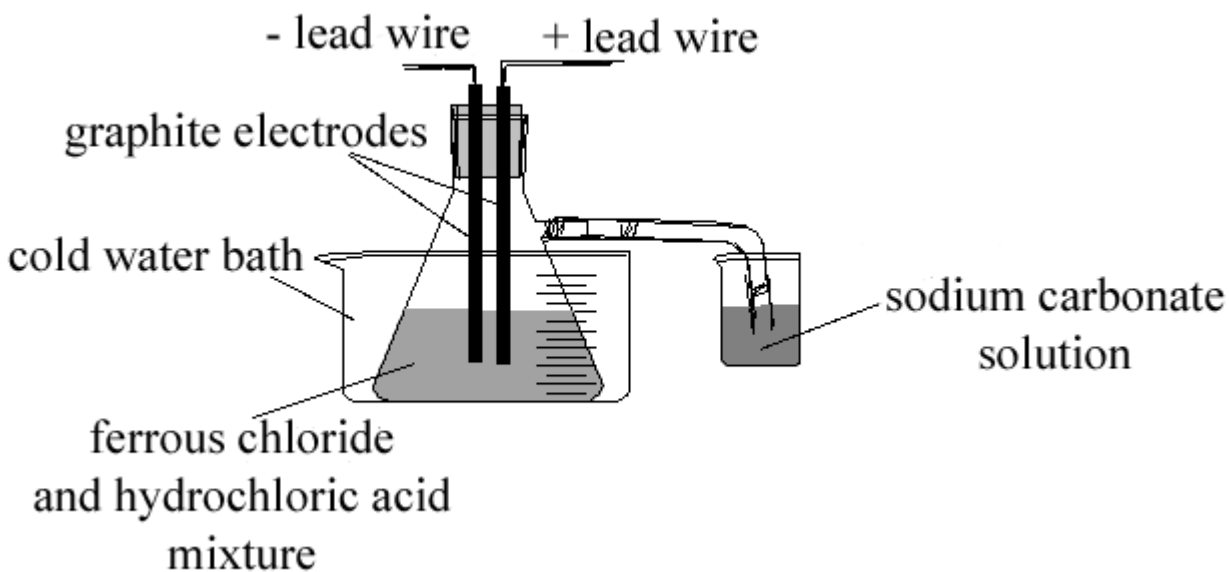
Reaction summary: Ferric chloride hexahydrate can be prepared by electrolyzing a solution of ferrous chloride utilizing an open cell containing hydrochloric acid utilizing graphite electrodes.



Why does this reaction happen? In this particular reaction, we use ferrous chloride and hydrogen chloride both as electrolytes. We also use graphite electrodes because graphite is a non-metal, and is non reactive towards the electrolytes, meaning the graphite does not react with, nor gets corroded by the electrolytes. Note: graphite is an example of an inert electrode, and when these inert electrodes are used, something different happens, as explained in the following: when current is applied to this type of system, the hydrogen ions of the hydrochloric acid migrate to the cathode, gaining an electron and forming free gas, which bubbles to the surface. At the same time, the negatively charged chlorine ions migrate to the anode giving up an electron and forming free chlorine. Now, because the anode electrode is composed of inert graphite, the free chlorine remains free. However, instead of the free chlorine gas bubbling to the surface, which would normally happen, the free chlorine spontaneously reacts with the ferrous chloride oxidizing the ferrous chloride to ferric chloride. The reaction proceeds until all the ferrous chloride is oxidized to ferric chloride.

Hazards: Perform the operation in a well-ventilated area, as hydrogen gas is evolved in large quantities.

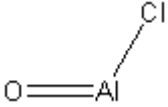
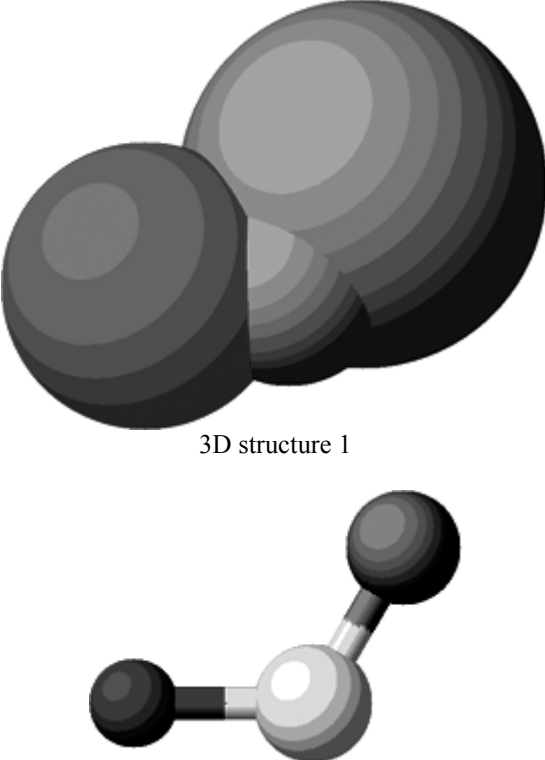
Procedure: Into a suitable flask, as illustrated below, place 300 milliliters (10.1 fluid oz.) of cold tap water, and then add and dissolve 100 grams (3.5 oz.) of ferrous chloride tetrahydrate, or 100 grams (3.4 oz.) of ferrous chloride dihydrate. Thereafter, pour in 59 milliliters (2 fluid oz.) of concentrated hydrochloric acid (31 to 35% by weight) if you used ferrous chloride tetrahydrate, or pour in 72 milliliters (2.4 fluid oz.) of concentrated hydrochloric acid (31 to 35% by weight) if you used ferrous chloride dihydrate. Thereafter, briefly stir the mixture for a few moments, and then assemble the apparatus as illustrated below, and then turn on your power supply, and electrolyze the mixture for about 3 to 4 hours at 6 to 12 volt at 2-amp direct current. Note: during the electrolysis the mixture will slowly turn a brownish black color due to the formation of ferric chloride hexahydrate. After the electrolysis is complete, turn off your power supply, and then quickly filter the mixture to remove any potential insoluble materials. At this point, the ferric chloride solution can be used for etching copper, or the mixture can be placed onto a shallow tray or pan and allowed to evaporate to form crystals of the ferric chloride hexahydrate. Note: do not boil the solution to remove water, as this will cause decomposition of the ferric chloride.



Apparatus for the electrolysis of ferrous chloride and hydrochloric acid. Remember that when hydrochloric acid is electrolyzed, chlorine gas is evolved. In this case, as soon as the chlorine gas is evolved, it spontaneously reacts with the ferrous chloride, oxidizing it to ferric chloride. Note: the sodium carbonate solution removes any corrosive vapors that may come over.

Procedure 54: Electro preparation 8: The Preparation of Aluminum oxy chloride

Also known as: oxygenated aluminum chloride

	 <p>3D structure 1</p> <p>3D structure 2</p>
Chemical structure	3D Structure

	AlOCl
Structure make-up	Condensed chemical structure

Aluminum oxychloride

Aluminum oxychloride forms a colorless glassy solid, which is readily soluble in water. In some cases, aluminum oxychloride is sometimes called aluminum hydroxy chloride with a condensed formula of $\text{Al}_2(\text{OH})_5\text{Cl} \cdot 2\text{H}_2\text{O}$.

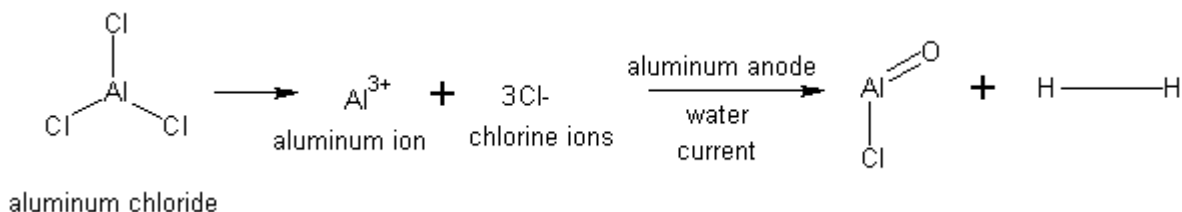
Method 1: Preparation of Aluminum oxychloride from aluminum chloride hexahydrate using an open eletro cell utilizing an aluminum anode

(By-products from reaction: Hydrogen gas)

Materials:

1. 300 milliliters (10.1 fluid oz.) of cold tap water	2. 100 grams (3.5 oz.) of aluminum chloride hexahydrate
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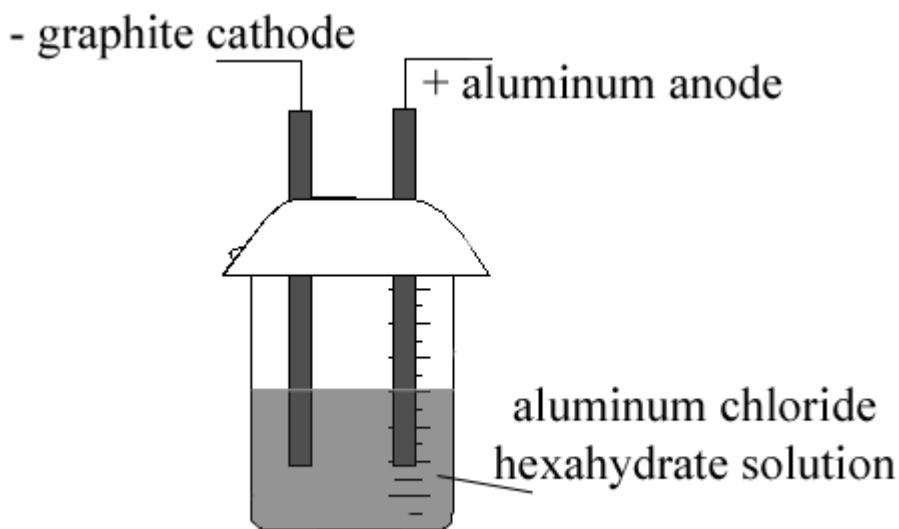
Reaction summary: aluminum oxychloride is readily prepared by electrolyzing a solution of aluminum chloride hexahydrate using an aluminum anode.



Why does this reaction happen? When current is applied to this system, the positively charged aluminum ions migrate to the cathode gaining electrons, and forming free aluminum. This free aluminum then spontaneously reacts with the water present forming aluminum hydroxide and liberating hydrogen gas. At the same time, the chlorine ions migrate to the anode, giving-up electrons, and forming free chlorine. Because the free chlorine is so reactive, it reacts with the aluminum anode forming aluminum chloride. This aluminum chloride then spontaneously reacts with the formed aluminum hydroxide forming a hydrated aluminum chloride complex, which gets reduced to the aluminum oxychloride by the cathode. The exact reaction is bit more complicated, and will not be discussed at this time.

Hazards: None.

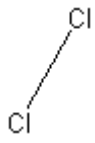
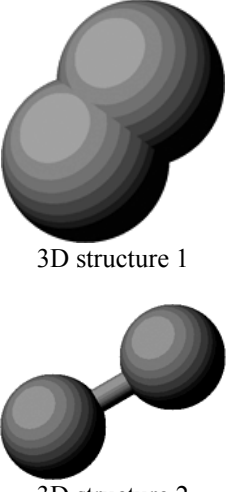
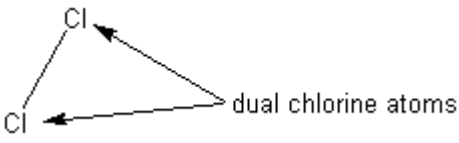
Procedure: Assemble an electro-chemical cell as illustrated below, and then add in 300 milliliters (10.1 fluid oz.) of cold tap water. Thereafter, add and dissolve 100 grams (3.5 oz.) of aluminum chloride hexahydrate. Thereafter, electrolyze the mixture for about 6 to 8 hours using an aluminum anode. During the reaction, the aluminum anode will slowly corrode away, forming aluminum oxy chloride, which remains dissolved in solution. After 6 to 8 hours, remove the power source, and then pour the entire mixture onto a shallow pan or tray, and allow the mixture to evaporate. Once the water has evaporated, a glassy solid of aluminum oxychloride will remain.



Electro-setup for preparing aluminum oxychloride by electrolyzing a solution of aluminum chloride hexahydrate. A current of 6 to 12 volt at 2 to 6 amp DC should be used.

Procedure 55: Electro preparation 9: The Preparation of Chlorine gas

Also known as: Diatomic chlorine

	
Chemical structure	3D Structure
	Cl ₂
Structure make-up	Condensed chemical structure

Chlorine

Chlorine forms a greenish yellow gas with a choking odor. The gas has a melting point of -101 Celsius, and a boiling point of -34 Celsius. The gas when heated, is easily condensed into a liquid by passing it through a glass coil surrounded by ice and salt. Chlorine gas is a major chemical produced industrially from common salt. Chlorine is a strong oxidizer and is capable of oxidizing a great many substances. Chlorine is poisonous, so avoid inhalation of the fumes.

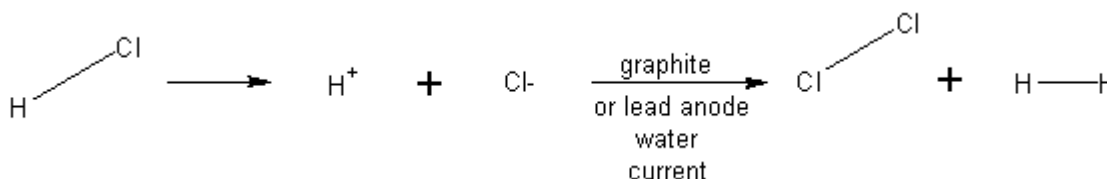
Method 1: Preparation of chlorine gas from dilute hydrochloric acid utilizing an open cell

(By-products from reaction: Hydrogen gas and oxygen gas)

Materials:

1. 400 milliliters (13.5 fluid oz.) of cold water	2. 100 milliliters (3.3 fluid oz.) of concentrated hydrochloric acid or concentrated Muriatic acid
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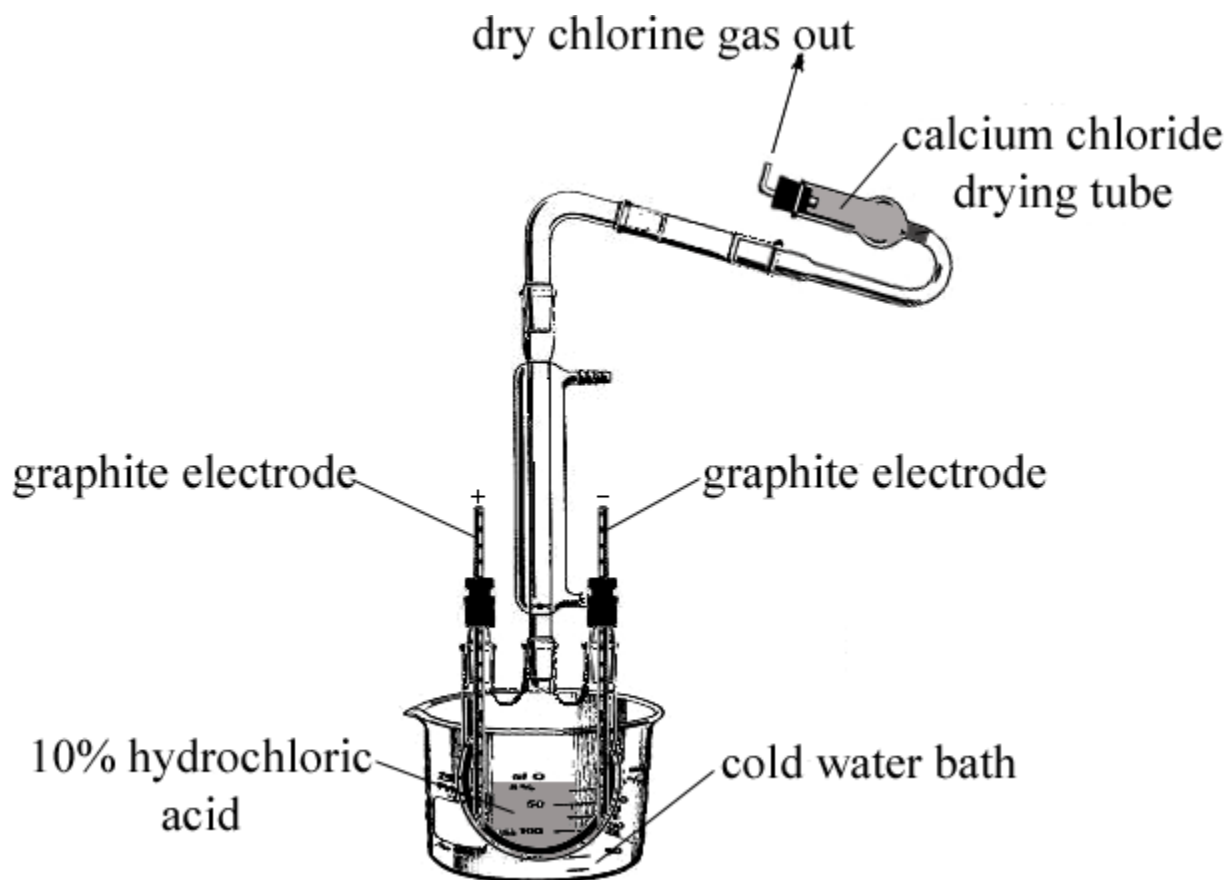
Reaction summary: Chlorine gas is readily prepared by the electrolysis of hydrochloric acid. The reaction generates heat, so the flask or container should be submerged in a cold-water bath.



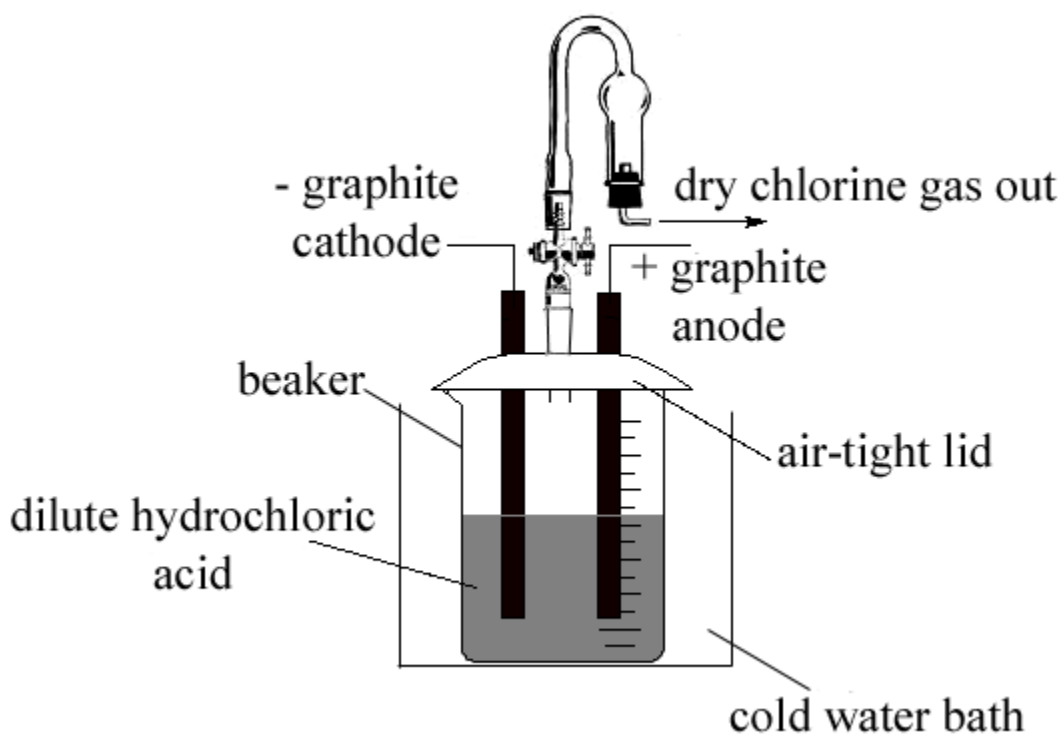
Why does this reaction happen? In this classic electrolysis procedure, when electric current is applied to the system, the hydrogen ions of the hydrochloric acid migrate to the cathode, gain an electron, forming free gas, which bubbles to the surface. Now, at the same time, the chlorine ions migrate to the anode, giving up an electron, and forming free chlorine gas. Because the graphite is inert, and because there are no other substances in the mixture for the chlorine to react with, the free chlorine gas also bubbles to the surface. The reaction continues as long as there is acid present and current is applied. During the electrolysis, a steady stream of hydrogen and chlorine gases will be steadily evolved.

Hazards: Use proper ventilation when handling chlorine gas, and avoid contact with direct sunlight.

Procedure: Into either of the apparatus listed below, place 400 milliliters (13.5 fluid oz.) of cold water, followed by 100 milliliters (3.3 fluid oz.) of concentrated hydrochloric acid or concentrated Muriatic acid. Thereafter, turn on the power supply and run the cell for about 6 to 7 hours at 2 to 12 volt at 2 to 6 amp. The amperage determines how long the process will take. Higher amperage is preferred. Note: This process will produce about 31 grams of chlorine gas. Note: the graphite electrodes can be replaced with lead if desired. **NOTE: This operation should be carried out in a room or shed free of windows, and/or direct contact to sunlight. If direct sunlight, magnesium light, or light from a UV lamp reaches the apparatus, the flask or container will explode! Please be advised that the electrolysis of hydrochloric acid utilizing an open cell is perfectly safe, and can be carried out without worries or hazards, as long as the operation is carried out in the absence of direct sunlight.**



Apparatus for the electrolysis of hydrochloric acid. Warning: If the apparatus is exposed to direct sunlight, an explosion will occur—as hydrogen gas is also liberated (hydrogen gas at the negative electrode, and the chlorine gas at the positive anode). Chlorine explodes when mixed with hydrogen and then exposed to sunlight (only when exposed to sunlight). The detonation propagates downwards, so there is no immediate danger from fragments. As a reminder, the electrolysis of hydrochloric acid is perfectly safe as long as the apparatus is protected from direct sunlight, magnesium light, halogen lamps, or UV lamps (cover all windows, ect.). Note: the black clamp of your battery charge goes on the (-) electrode, and the red clamp of the battery charger goes on the (+) electrode. Use a current of 6 volt 2 amp or what ever works best for you. If using 6 volt 2 amp, it should take about 2 hours for the chlorine evolution to cease.



Apparatus for the preparation of chlorine gas. Keep the apparatus away from direct sunlight, magnesium light, or UV light. Note: the entire apparatus should be airtight, 100%

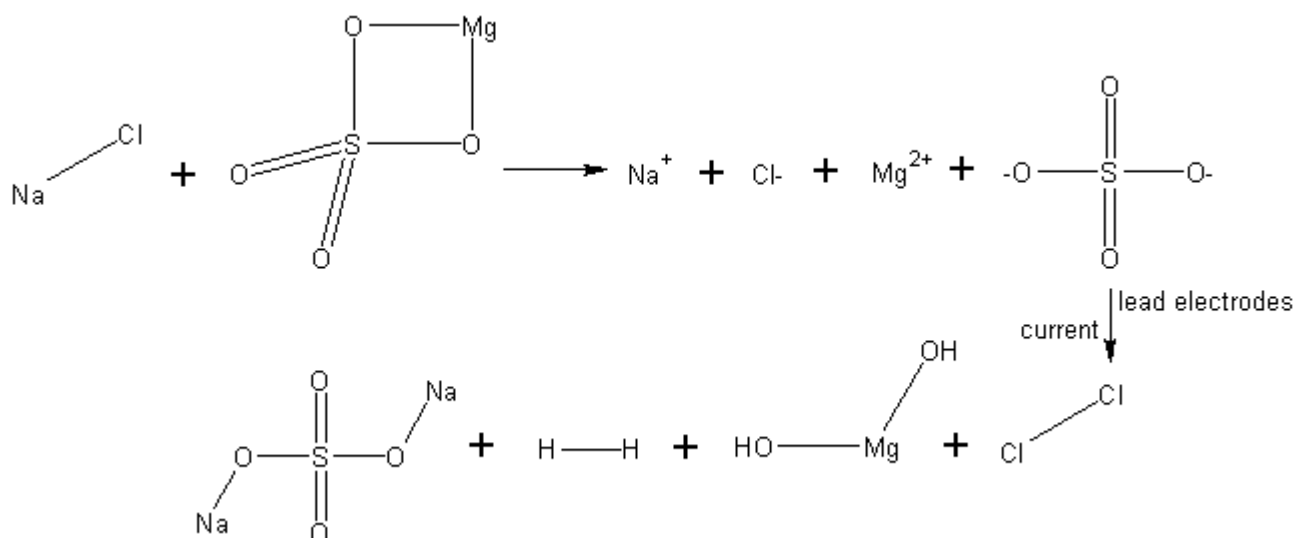
Method 2: Preparation of chlorine gas from Epsom salt, and pickling salt utilizing an open cell

(By-products from reaction: Hydrogen gas, oxygen gas, sodium sulfate, and magnesium hydroxide)

Materials:

1. 200 grams (7 oz.) of Epsom salt	3. 95 grams (3.4 oz.) of pickling salt
2. 1000 milliliters (33.8 fluid oz.) of cold water	

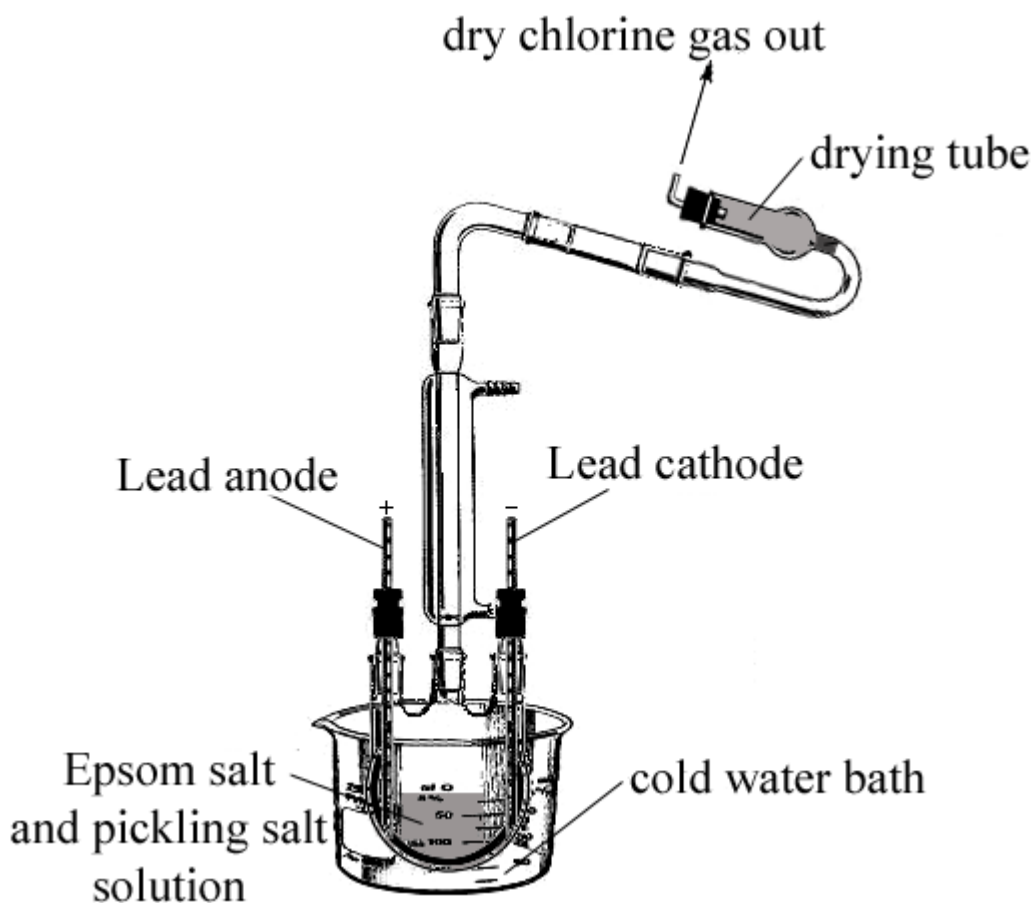
Reaction summary: Chlorine gas can also be generated by electrolysis of a mixture of Epsom salt and pickling salt. The electrodes used should be made of lead to prevent excessive corrosion. During the reaction, water insoluble magnesium hydroxide will precipitate, and a steady stream of chlorine gas will be evolved over time. This procedure is useful in generating moderate amounts of chlorine gas while using simple and readily available materials.



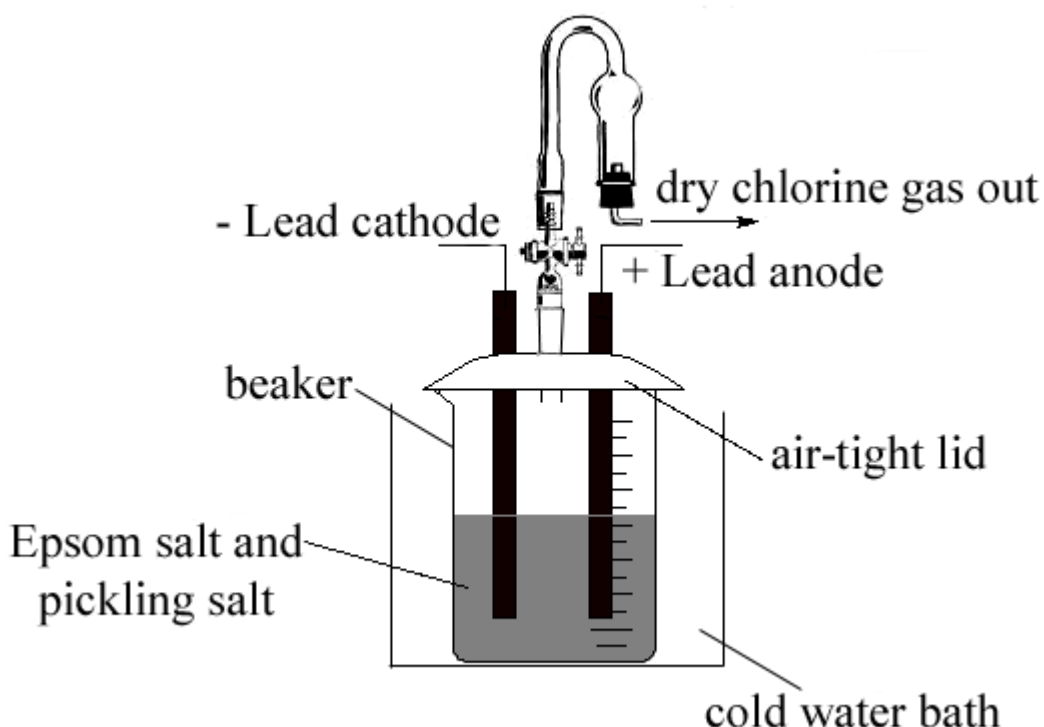
Why does this reaction happen? In this example, sodium chloride and magnesium sulfate (Epsom salt) are used as electrolytes. When current is applied to the system, the magnesium ions migrate to the cathode gaining electrons, and forming magnesium hydroxide, which precipitates spontaneously due to its lack of solubility. At the same time, the sulfate ions migrate to the anode giving up electrons and forming sulfuric acid. Now, thereafter, the sulfuric acid formed reacts with the sodium chloride forming sodium sulfate and hydrogen chloride. Thereafter, the hydrogen chloride that has formed undergoes the same process as in the previous procedure whereby the hydrogen ions migrate to the cathode, giving off free gas, which bubbles to the surface, and the chlorine ions migrate to the anode forming free chlorine gas, which also bubbles to the surface. The reaction continues until all the magnesium is converted into magnesium hydroxide.

Hazards: Use proper ventilation when handling chlorine gas, and avoid contact with direct sunlight.

Procedure: Dissolve 200 grams (7 oz.) of Epsom salt into 1000 milliliters (3.8 fluid oz.) of cold water, and thereafter, add and dissolve 95 grams (3.4 oz.) of pickling salt. Now, pour this salt solution into an apparatus as illustrated below, and then electrolysis the mixture at 6 to 12 volt at 6 to 12 amp for a period ranging from 12 to 18 hours. During the electrolysis process, a moderate, yet steady stream of chlorine gas will be steadily evolved and carried over. **NOTE: This operation should be carried out in a room or shed free of windows, and/or direct contact to sunlight. If direct sunlight, magnesium light, or light from a UV lamp reaches the apparatus, the flask or container will explode! Please be advised that this electrolysis is perfectly safe, and can be carried out without worries or hazards, as long as the operation is carried out in the absence of direct sunlight.**



Setup for the generation of chlorine gas from readily available materials. As with the electrolysis of hydrochloric acid, keep the apparatus away from direct sunlight, magnesium light, or UV light.



Setup for the preparation of chlorine. As with the electrolysis of hydrochloric acid, keep the apparatus away from direct sunlight, magnesium light, or UV light.

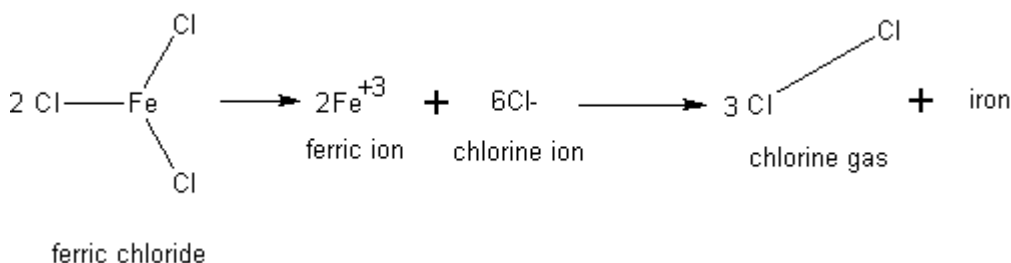
Method 3: Preparation of hydrogen and oxygen free chlorine gas from ferric chloride utilizing an open cell

(By-products from reaction: metallic iron)

Materials:

1. 400 milliliters (13.5 fluid oz.) of warm water	2. 200 grams (7 oz.) of ferric chloride hexahydrate
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Reaction summary: A oxygen and hydrogen gas free chlorine can be prepared by electrolyzing a solution of ferric chloride using an open cell. During the process, metallic iron will plate out on the cathode, and hence, can be collected for recycle.

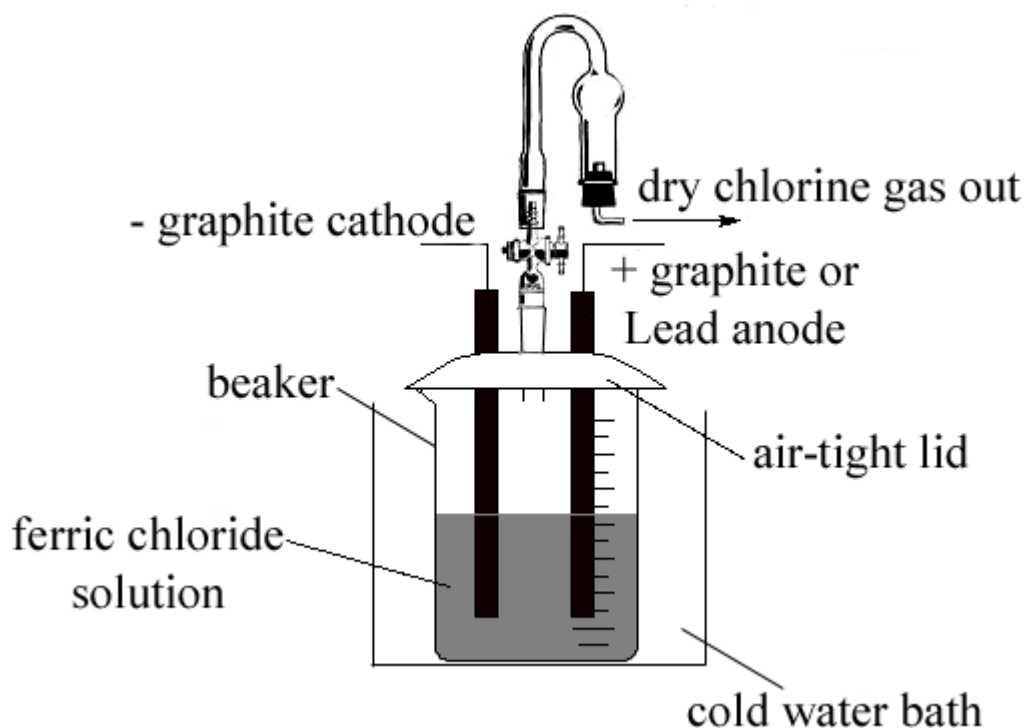


Why does this reaction happen? In this system, when current is applied, the positively charged ferric ions migrate to the negative cathode gaining electrons, and forming free iron, which coats the cathode. At the same time, the negatively charged chlorine ions migrate to the positive anode giving up electrons, and forming free chlorine. Because the positive anode is made of an inert material, the free chlorine gas bubbles to surface. The reaction proceeds as long as ferric chloride is present, and during the reaction, a steady stream of hydrogen and oxygen free chlorine gas is steadily evolved.

Hazards: Use proper ventilation when handling chlorine gas, and avoid contact with direct sunlight.

Procedure: Into an open cell as illustrated below, place 400 milliliters (13.5 fluid oz.) of warm water, and then add and dissolve 200 grams (7 oz.) of ferric chloride hexahydrate. Thereafter, electrolysis the solution from 6 to 12 volt, by 2 to 6 amp direct current for about 8 to 12 hours. During the electrolysis, a steady stream of hydrogen and oxygen free chlorine gas will be steadily evolved, and metallic iron will slowly plate the cathode. Note: The fresh metallic iron, can be recycled by using it as

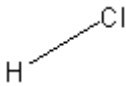
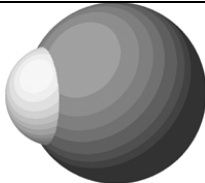

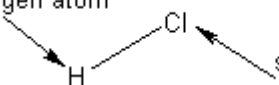
the anode for another electrolysis process using a diaphragm cell or divided cell, or it can be dissolved into hydrochloric acid directly if desired to prepare more ferrous chloride.



Setup for the preparation of hydrogen and oxygen free chlorine gas. During the electrolysis, metallic iron will slowly coat the iron cathode. Note: your apparatus can be sealed airtight by the use of plumber's putty, if your lid does not fit airtight, or there are gaps between the holes and your protruding electrodes.

Procedure 56: Electro preparation 10: The Preparation of Hydrogen chloride gas

Also known as: Anhydrous hydrochloric acid

	 <p>3D structure 1</p>  <p>3D structure 2</p>
Chemical structure	3D Structure
<p>single hydrogen atom</p>  <p>single chlorine atom</p>	<p>HCl</p>
Structure make-up	Condensed chemical structure

Hydrogen chloride

Hydrogen chloride gas forms a colorless to white fuming vapor, with a highly corrosive and choking odor. The gas has a melting point of -114 Celsius, and a boiling point of -85 Celsius. The gas is readily soluble in water forming hydrochloric acid. The gas is also highly soluble in alcohols, and other solvents. Hydrogen chloride is highly corrosive and reacts with almost any metal.

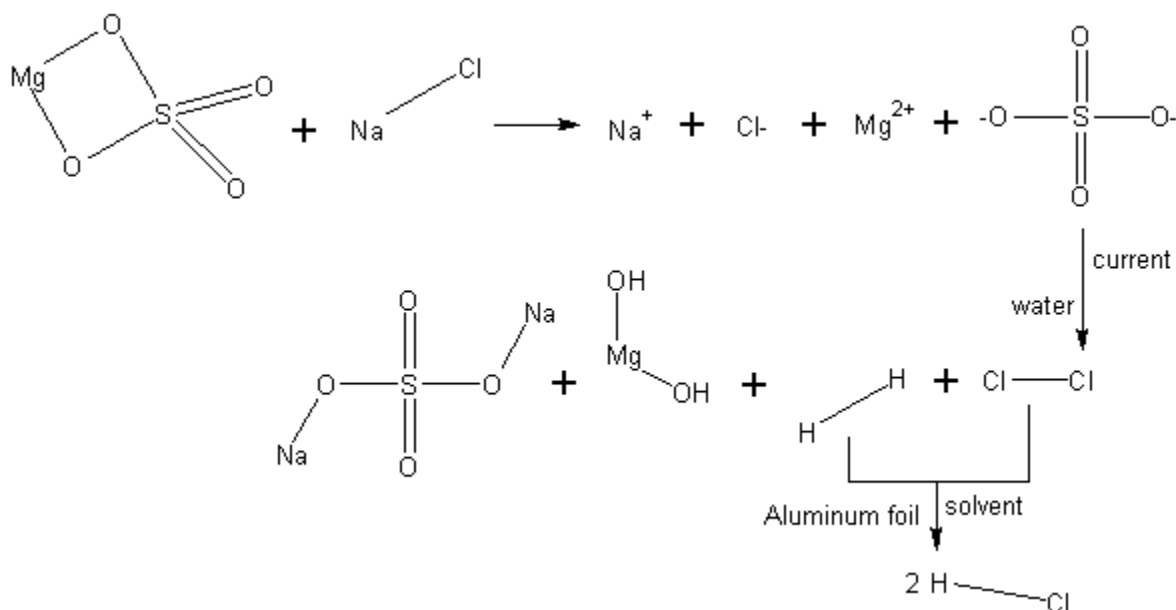
Method 1: Preparation of hydrogen chloride from Epsom salt, pickling salt, and using an open cell

(By-products from reaction: oxygen gas)

Materials:

1. 300 milliliters (10.1 fluid oz.) of dry hexane	4. 1000 milliliters (3.8 fluid oz.) of cold water
2. 20 grams (0.70 oz.) of aluminum foil pieces	5. 95 grams (3.4 oz.) of pickling salt
3. 200 grams (7 oz.) of Epsom salt	

Reaction summary: Hydrogen chloride can be prepared using electrochemical methods. In this regards, hydrogen chloride can be prepared by first, electrolyzing a mixture of Epsom salt and pickling salt, and then passing the gas mixture produced, into a mixture of hexane containing aluminum foil pieces. The aluminum foil acts as a catalyst, causing hydrogen gas to combine with chlorine gas, forming hydrogen chloride.

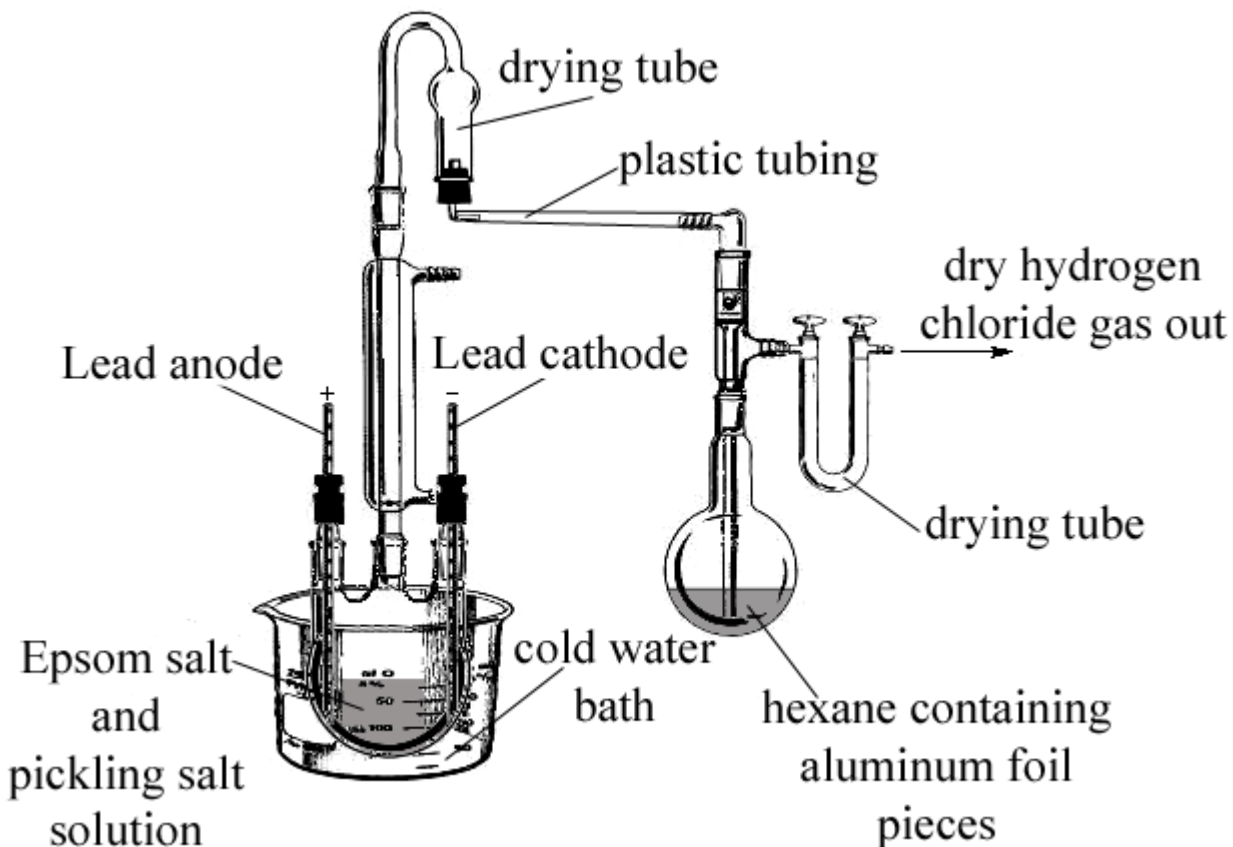


Why does this reaction happen? In this example, sodium chloride and magnesium sulfate (Epsom salt) are used as electrolytes. When current is applied to the system, the magnesium ions migrate to the cathode gaining electrons, and forming magnesium hydroxide, which precipitates spontaneously due to its lack of solubility. At the same time, the sulfate ions migrate to the anode giving up electrons and forming sulfuric acid. Now, thereafter, the sulfuric acid formed reacts with the sodium chloride forming sodium sulfate and hydrogen chloride. Thereafter, the hydrogen chloride that has formed undergoes the following: the hydrogen ions migrate to the cathode, giving off free gas, which bubbles to the surface, and the chlorine ions migrate to the anode forming free chlorine gas, which also bubbles to the surface. The reaction continues until all the magnesium is converted into magnesium hydroxide. Now, the evolved chlorine and hydrogen gases are then condensed using a non-electro chemical reaction, whereby the chlorine and hydrogen gases react with aluminum foil in an inert solvent such as hexane forming hydrogen chloride gas.

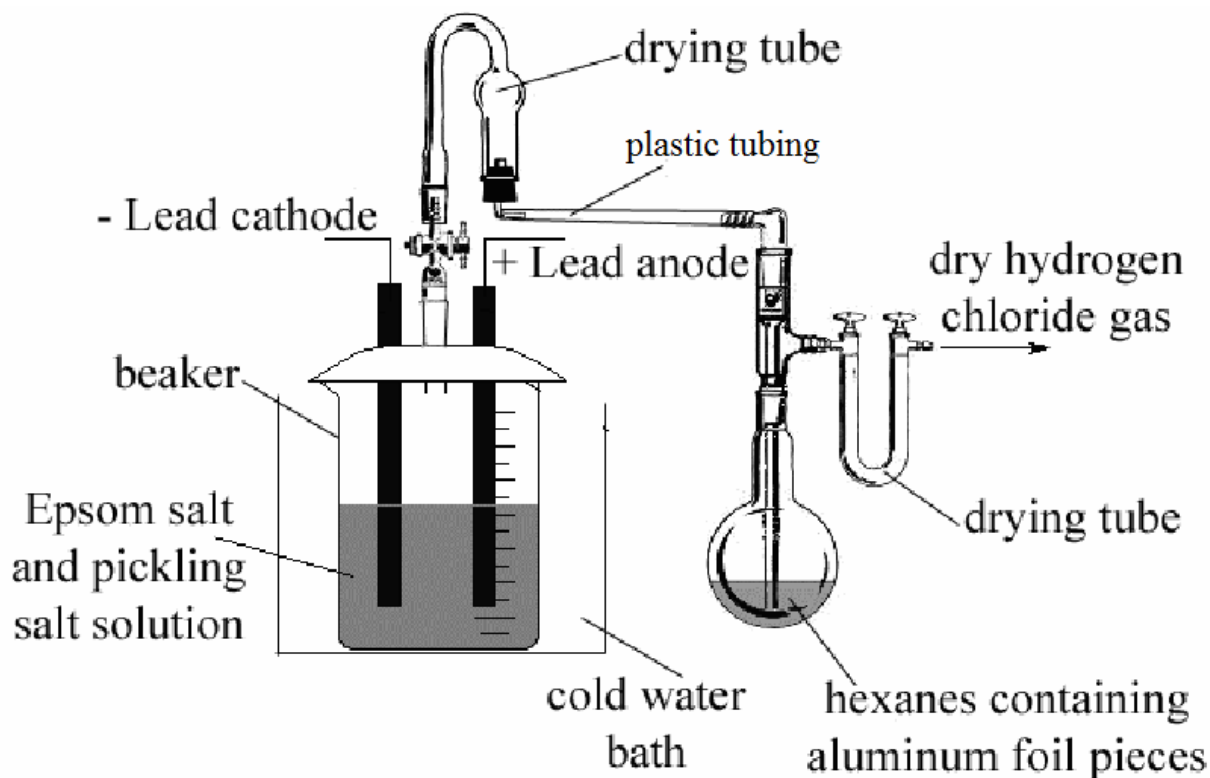
Hazards: Use caution when handling chlorine gas, which is toxic—avoid inhalation of the fumes. Extinguish all flames before using hexane, which is highly flammable.

Procedure: Setup the apparatus as illustrated below (place 300 milliliters (10.1 fluid oz.) of dry hexane and 20 grams (0.70 oz.) of aluminum foil pieces into the appropriate round bottom flask as indicated), and then dissolve 200 grams (7 oz.) of Epsom salt into 1000 milliliters (3.8 fluid oz.) of cold water, and thereafter, add and dissolve 95 grams (3.4 oz.) of pickling

salt. Now, pour this salt solution into an apparatus as illustrated below, and then electrolysis the mixture at 6 to 12 volt at 6 to 12 amp for a period ranging from 12 to 18 hours. During the electrolysis process, a moderate, yet steady stream of chlorine gas (mixed with hydrogen and oxygen) will be steadily evolved and carried over. **NOTE: This operation should be carried out in a room or shed free of windows, and/or direct contact to sunlight. If direct sunlight, magnesium light, or light from a UV lamp reaches the apparatus, the flask or container will explode! Please be advised that this electrolysis process is perfectly safe, and can be carried out without worries or hazards, as long as the operation is carried out in the absence of direct sunlight.**



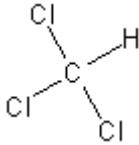
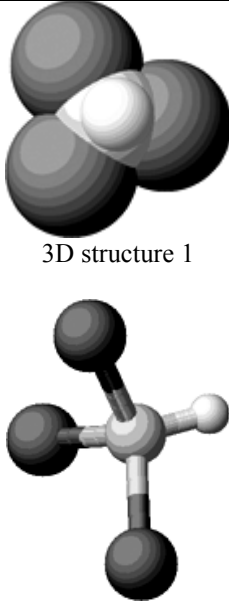
Advanced setup for the preparation of hydrogen chloride. As a gas mixture of hydrogen, oxygen, and chlorine is evolved from the electrolysis process, the gas mixture is bubbled into a mixture of hexane containing aluminum foil pieces. The aluminum foil acts as a catalyst, causing the hydrogen to combine with the chlorine without violence, forming a steady stream of hydrogen chloride. This hydrogen chloride can be used directly or dissolved in water forming hydrochloric acid. As with the electrolysis of hydrochloric acid, keep the apparatus away from direct sunlight, magnesium light, or UV light.

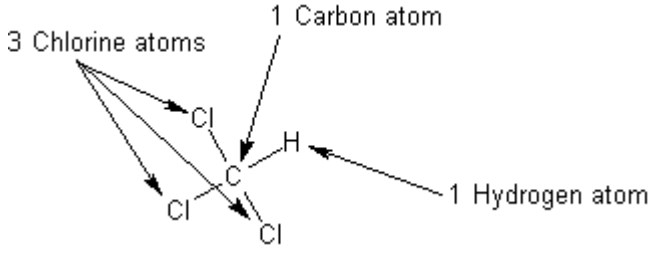


Another setup for the preparation of hydrogen chloride. Note: the lid on the cell should be air-tight. As with the electrolysis of hydrochloric acid, keep the apparatus away from direct sunlight, magnesium light, or UV light.

Procedure 57: Electro preparation 11: The Preparation of Chloroform

Also known as: Trichloromethane

	 <p>3D structure 1</p> <p>3D structure 2</p>
Chemical structure	3D Structure

	CHCl ₃
Structure make-up	Condensed chemical structure

Chloroform

Chloroform is a highly refractive, nonflammable, heavy, very volatile, and sweet-tasting liquid with a peculiar odor. It has a boiling point of 62 Celsius, and a melting point of -64 Celsius. Chloroform forms a constant boiling mixture with alcohol containing 7% alcohol, and boiling at 59 Celsius. Commercial chloroform contains a very small amount of ethanol as stabilizer. It is insoluble in water, but miscible with alcohol, benzene, ether, petroleum ether, and carbon disulfide. Pure chloroform is light sensitive, so store in amber glass bottles in a cool place. Chloroform is a suspected light carcinogen, so use proper ventilation when handling. Over exposure to chloroform vapors causes dizziness, and headache. **Note:** Distilling mixtures containing chloroform mixed with one or more strong base (lithium, sodium, or potassium hydroxide) can result in explosion or violent reaction. Always neutralize any base, or extract the chloroform before distilling.

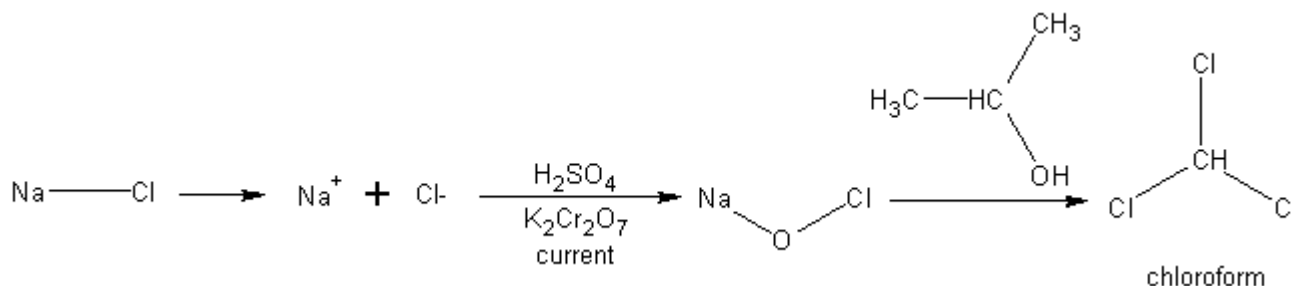
Method 1: Preparation of chloroform from Rubbing alcohol, potassium dichromate, pickling salt, and dilute sulfuric acid using an electrochemical cell

(By-products from reaction: sodium acetate, sodium sulfate, and sodium hydroxide)

Materials:

1. 40 grams (1.4 oz.) of 70% rubbing alcohol (just regular rubbing alcohol, no fancy colored stuff or fancy additives—just plain old rubbing alcohol)	5. 375 milliliters (12.6 fluid oz) of warm tap water
2. 148 grams (5.2 oz.) of pickling salt	6. 225 milliliters (7.6 fluid oz.) of benzene, toluene, or xylene
3. 1 gram of potassium dichromate (available on-line)	7. 5 milliliters of concentrated sulfuric acid (1 teaspoon) (plumbers grade or better)
4. 1 milliliter of 95% ethyl alcohol	8. 15 grams of anhydrous magnesium sulfate

Reaction summary: Chloroform can be prepared in an unusual manner by electrolyzing a solution of pickling salt and isopropyl alcohol in the presence of a small amount of sulfuric acid and potassium dichromate using graphite or lead electrodes. The process develops low yields of chloroform, and takes considerable amounts of time, but is effective at producing small amounts of chloroform. After the initial reaction, the mixture is extracted with benzene, toluene, or xylene, and the resulting solvent extracts are then combined, and then distilled to recover the chloroform.



Why does this reaction happen? In this reaction we have a pair of reactions that take place. Here we see sodium chloride as the electrolyte, and we see the sulfuric acid, isopropyl alcohol, and potassium dichromate as additives, but not electrolytes. When current is applied to the system, the sodium ions migrate to the negative cathode, gaining an electron and forming sodium hydroxide while liberating hydrogen gas. At the same time on the other end the chlorine ions migrate to the anode giving up an electron and forming free chlorine. Now, because of the presence of potassium dichromate and sulfuric acid, the

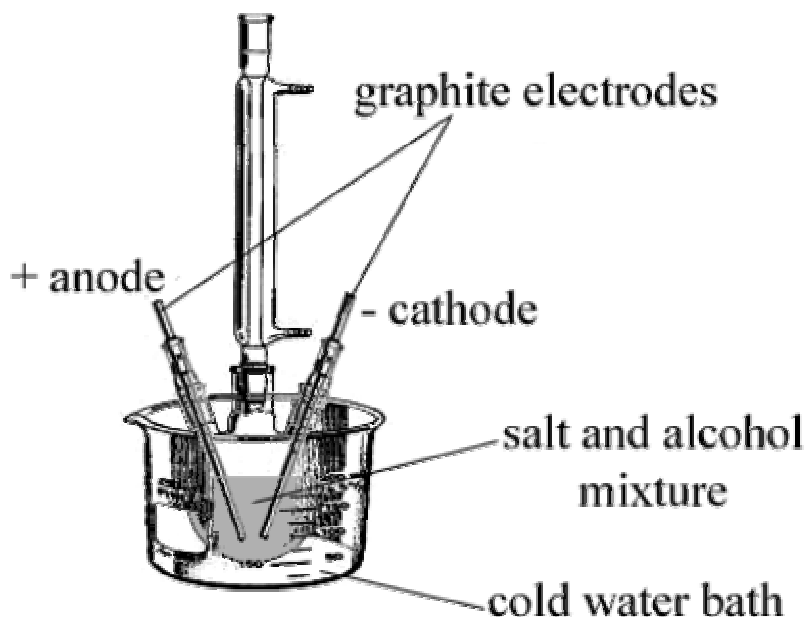
free chlorine that is generated reacts with the sodium hydroxide forming sodium hypochlorite. This sodium hypochlorite then spontaneously reacts with the isopropyl alcohol forming chloroform and sodium acetate. The chloroform remains dissolved in the solution and is later recovered using listed techniques such as extraction.

Hazards: Avoid breathing in the fumes of the reaction mixture. Use proper ventilation when carrying out this procedure.

Procedure: Into a suitable separatory funnel, place 40 grams (1.4 oz.) of 70% rubbing alcohol (just regular rubbing alcohol, no fancy colored stuff or fancy additives—just plain old rubbing alcohol), followed by 20 to 30 grams (0.7 to 1 oz.) of pickling salt. Note: make sure the stopcock on the separatory funnel is closed before starting. Thereafter, stopper the separatory funnel, and then shake the entire funnel vigorously for about 5 minutes. Then allow the separatory funnel to stand (upright) for about 30 minutes. Thereafter, drain-off the bottom brine layer (and any excess salt), and thereafter, recover the upper isopropyl alcohol layer. Now, pour this isopropyl alcohol into a suitable container (see the following illustration for electrochemical cell make-up), and then add in 118 grams (4.1 oz.) of pickling salt, followed by 375 milliliters (12.6 fluid oz) of warm tap water, followed by 1 gram of potassium dichromate, and then followed by 5 milliliters of concentrated sulfuric acid. Thereafter, vigorously stir the entire mixture for about 30 minutes to dissolve all solids. At this point, you will end up with an orangey colored solution. Now, setup your electro chemical cell, and then electrolyze this mixture for 15 hours at 12 volt 6 amp current. Use a battery charger with this setting—these battery chargers are very common and availed in many stores. After 15 hours, unplug the battery charger, and then pour the entire mixture into a beaker, and then extract this entire mixture with three 75-milliliter portions (2.5 fluid oz. portions) of benzene, toluene, or xylene, and after the extraction process, combine all solvent portions, if not already done so. Thereafter, dry this combined solvent portion by adding to it, 15 grams of anhydrous magnesium sulfate (to absorb water). After adding in the magnesium sulfate, stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Then place this filtered dried solvent mixture into a distillation apparatus, and distill over the chloroform at 65 Celsius until no more chloroform passes into the receiver flask. When no more chloroform passes into the receiver flask, stop the distillation, and then remove the receiver flask from the distillation apparatus. Then place this distilled crude chloroform into a clean fractional distillation apparatus and distill at 62 Celsius until no more chloroform passes over into the receiver flask. When no more chloroform passes into the receiver flask, stop the distillation, and then remove the chloroform from the receiver flask and then add to it, 1 milliliter of 95% ethyl alcohol. Then store this chloroform in an amber glass bottle in a cool dry place.

Note: the benzene, toluene, or xylene used in the extraction, can be recovered after the first distillation process.

The electrolysis process



Electro chemical cell for the preparation of chloroform. This apparatus is a laboratory apparatus, and can be replaced with similar apparatus designs. The 3-neck flask shown in the illustration can be replaced with a large canning jar, by cutting three holes in the lid, or something of that nature. Other containers can be used, and you should use your imagination. Note: the red clamp of your battery charger goes on the positive anode, and the black clamp goes on the negative cathode.

Graphite rods of various lengths and diameters are available on line for reasonable prices.

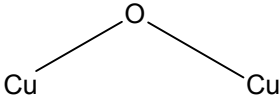
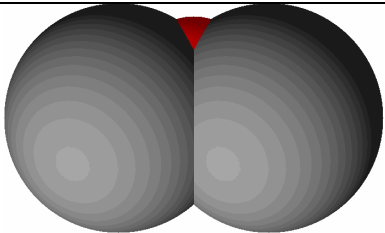
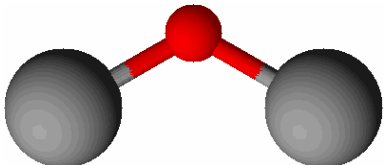
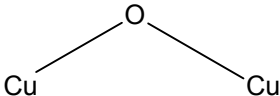
Final note for method 3

The by-products of sodium acetate, and sodium sulfate can be recovered as follows:

1. Filter the extracted reaction mixture (after the extraction process), to filter-off any insoluble materials (graphite and/or lead sludge).
2. Recover acetate by adding in dilute sulfuric acid or hydrochloric acid, and then distill the resulting acidic mixture at 110 Celsius to recover the acetic acid. After the distillation process, evaporate the left over reaction mixture to dryness after the distillation (to remove acetic acid), so as to recover the sodium chloride or sulfate (depending on which acid you used). This sodium chloride can be recycled if desired for a new run. Second note: acetic acid is a useful by-product and can be used in a variety of applications.

Procedure 58: Electro preparation 12: The Preparation of Copper-I-oxide, and chlorine gas

Also known as: N/A

	 3D structure 1  3D structure 2
Chemical structure	3D Structure
 2 copper atoms 1 oxygen atom	Cu_2O
Structure make-up	Condensed chemical structure

Copper-I-oxide

Copper-I-oxide forms a brick red crystalline powder, which may be yellow, red, or brown depending on method of preparation. The powder is insoluble in water, and the usual solvents, but is readily soluble in acids, and ammonia solutions. The melting point of the powder is about 1232 Celsius, and the dry compound slowly oxidizes to copper-II-oxide on standing. Copper-I-oxide should be used within 1 month of its preparation.

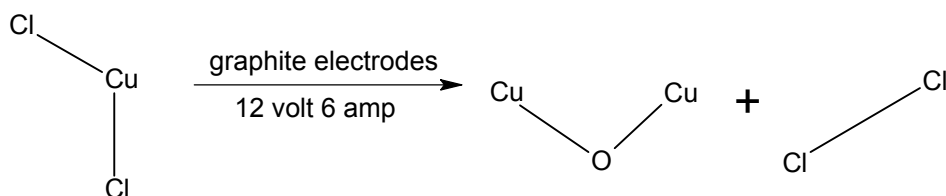
Method 1: Preparation of Copper-I-oxide, and chlorine from copper-II-chloride utilizing an open cell

(By-products from reaction: Hydrogen and oxygen gases)

Materials:

1. 50 grams of copper-II-chloride	2. 400 milliliters of tap water
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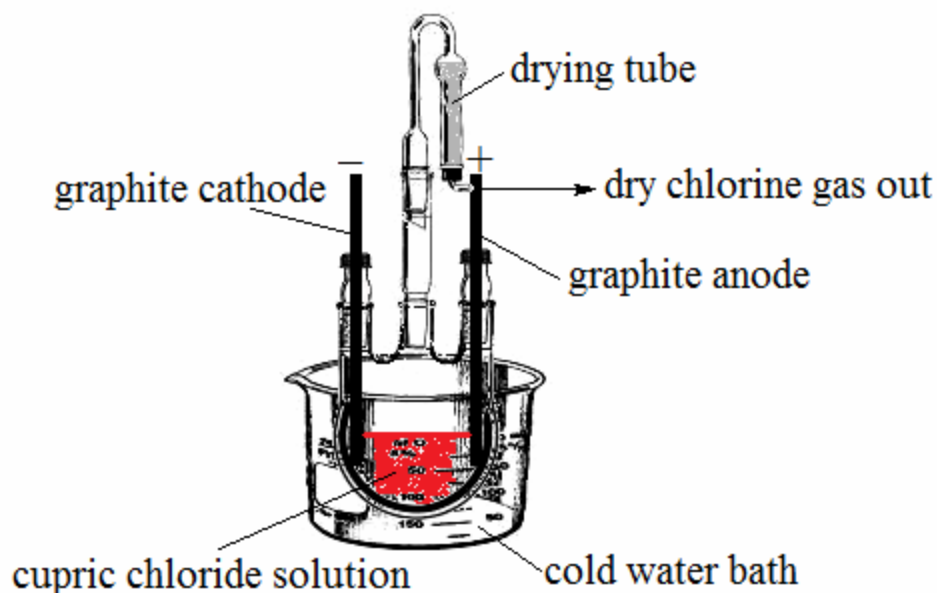
Reaction summary: Copper-I-oxide is readily prepared by electrolyzing a solution of copper-II-chloride utilizing an open cell. In an open cell, the copper-II-chloride is reduced at the cathode forming copper-I-oxide. A little Copper-I-chloride forms at the anode and settles out. The insoluble copper-I-oxide separates out and floats on the surface. Some metallic copper is deposited on the cathode as well. Chlorine gas is steadily evolved at the anode.



Why does this reaction happen? When copper-II-chloride is dissolved in water, it forms positive copper ions, and negative chlorine atoms, which remain encircled by water molecules forming a solution. When an inert anode made of graphite is inserted into the solution, and current passed there into, the positively charged copper ions migrate to a negative cathode, also introduced into the solution, whereby they gain two electrons forming free copper, some of which reacts with the water forming copper-I-oxide, and plating the negative cathode with copper. At the same time, the negatively charged chlorine atoms migrate to the positive anode, and give up two electrons (one electron per chlorine x two), forming free chlorine, some of which, bubbles to the surface, and some of which reacts with the copper ions forming a little copper-I-chloride, which settles out, but tends to decompose.

Hazards: Use a proper apparatus or carryout the reaction in a well-ventilated area as chlorine gas is evolved.

Procedure: Simply dissolve 50 grams of cupric chloride into 400 milliliters of tap water, and then assemble the apparatus as illustrated below. Then electrolyze the cell at 12 volt 6 amp for about 6 hours, or until no more chlorine is evolved. After six hours or so, disassemble the apparatus and then collect the brick red copper-I-oxide by filtration.



Apparatus for preparing copper-I-oxide and chlorine gas. As with the electrolysis of hydrochloric acid, keep the apparatus away from direct sunlight, magnesium light, or UV light.

Chapter 10: Electrochemical processes, Electro chemical methods in general chemistry Utilizing “divided Cells”

10a. Standard Diaphragm “Salt Bridge” Divided Cells

Introduction

In this section we will discuss the use of divided cells. Divided cells are composed of two separate compartments separated by a barrier. The barrier can be a porous membrane, or salt bridge. Unlike “open” cells, as seen in the previous section, “divided” cells are used to carryout a whole new set of reactions that would be impossible in an open cell, and the chemical reactions themselves would be other wise impossible overall. Even though there are numerous designs and systems for utilizing and forming divided cells, we will discuss the most basic of the divided cells by using and starting with a common “salt bridge” cell using simple store bought materials.

Divided cells see the formation of different chemical reactions because the anode and cathode compartments are separated by the barrier. Because the anode and cathode compartments are separated by a barrier, the nature of the chemical reactions are much different then in open cells. In open cells, the products that the positive and negative ions form at their corresponding electrodes when current is applied, tend to react with each other because there is no barrier to separate them; however in divided cells, the products that the positive and negative ions form at their corresponding electrodes when current is applied, are separated by the barrier and hence, produce different effects and resulting products. Note: Open cells favor reduction, and divided cells favor oxidation.

A standard diaphragm “salt bridge” cell can be made from simple and inexpensive store bought materials. Anyone can easily assemble their own cell, and carryout interesting and useful procedures to make a variety of interesting products. First of all, you need to learn how to assemble your own homemade cell. The first thing you will need to do is go to the store and purchase the following materials:

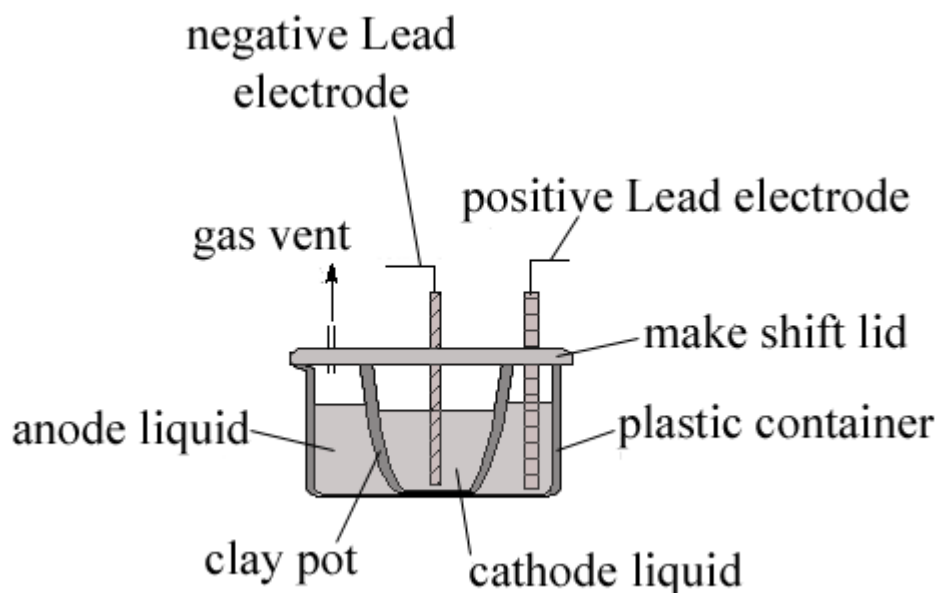
1. 1 box of Epsom salt (available in any grocery store)
2. 1 standard clay pot (just under 1 quart size; 500 milliliters; try to find one that does not have a hole in the bottom)
3. 1 plastic Tupperware container w/lid (just under ½ gallon size; about 1800 milliliters)
4. 2 lead electrodes (about 6 inches long each).
5. 1 car battery charger; with power output with at least 12 volt 6 amp (available in most stores for \$20.00 to \$40.00)

Note: The lead electrodes should be rectangular with dimensions of 5 to 6 inches long, by $\frac{1}{2}$ inch to $\frac{1}{4}$ inch in diameter (they don't have to be perfectly round, and can be square or rectangle). The lead electrodes may be circular if desired with dimensions of 6 inches long by 0.30 to 0.50 inch radius (9 millimeter to 12.9 millimeter). The lead electrodes can be made by melting pieces of lead into a make shift mold, made by forming a rectangular shaped mold using aluminum foil. After shaping the mold with the aluminum foil, place the pieces of lead there into, and then use a standard propane torch to melt the lead. After allowing the lead to cool, remove the aluminum foil. Lead can be found in car batteries, lead solder for welding copper and in electronics. Lead air gun pellets (are the best source of lead from store bought materials), and lead sheet squares used in dental x-rays.

Note: The clay pot is a standard clay flowerpot, which can be found at any hardware store or pottery store. The clay pot should not have a hole in the bottom, as most of them do. ***If a clay pot with no hole in the bottom is not available, use plumbers putty to seal the hole on both sides, or use a small rubber or cork stopper to plug the hole.***

10b. Assemble your cell

Now that you have your materials, assemble your cell. Use the following illustration to aid you. Note: For the electrodes, suitable sized holes should be cut into the plastic lid. One hole on the left or right side of the plastic lid, and the other directly in the center of the plastic lid. The holes if properly cut, can secure the electrodes firmly (with a tight fit). The clay pot is then placed into the plastic Tupperware container as shown in the illustration. Note: The plastic lid should be able to fit securely onto the plastic Tupperware container without disrupting the clay pot. A small hole should also be cut into the plastic lid to allow for gas venting. Note: This cell is not airtight. To make the cell airtight, the holes in which the electrodes protrude need to be sealed airtight. To do this, a glue gun may be used, or plumbers putty. The plastic lid should also be sealed gas tight. To do this, wrap Teflon tape (available at any hardware store) around the outer lip of the plastic Tupperware container many times, so that when the lid is attached, it forms a firm seal. Note: Other methods may be used to create an airtight cell. Use your own imagination, and see what you can come up with. An airtight seal of your cell is not necessary for most procedures. An airtight seal is only desired if a producing gas is the desired product, as in the production of chlorine gas.

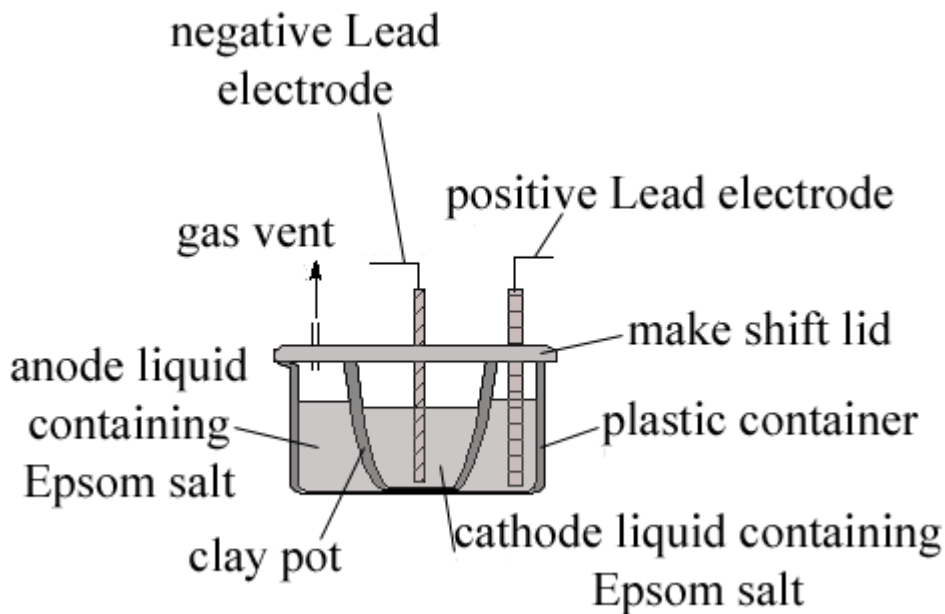


Setup for the assembly of the cell. Note: The lead electrodes can be replaced with graphite, but lead is better to reduce corrosion of the electrodes.

10c. Charge your cell (charge your clay pot)

Now that you have setup your cell, the next task is to "charge the cell". What does this mean? Well, to put it into simple terms, the clay pot acts as a salt bridge. In order for this salt bridge to work properly, it must be "Charged" with ions, so that during a particular process, the electrochemical reaction works properly. To charge the cell, carryout the following:

1. Into a beaker or other container, place 1500 milliliters (50.7 fluid oz.) of water. Then add 100 grams (3.6 oz.) of Epsom salt, and stir the water to completely dissolve the Epsom salt.
2. Place 500 milliliters (17 fluid oz.) of this Epsom salt solution into the clay pot. This will be called the "cathode" liquid, designating the "negative" side.
3. Place the remaining Epsom salt solution into the plastic Tupperware container (the outer compartment). This will be called the "anode" liquid, designating the "positive" side.
4. Then secure the plastic lid to the plastic Tupperware container, and then secure both electrodes.
5. Then attach each electrode to their respective electric wire from the battery charger. Note: The battery charger comes with a black clamp, and a red clamp. The black clamp is the "negative", and the red clamp is the "positive". Note: The battery charger only works when plugged in! Before plugging the battery charger in, secure both clamps to the cell. The black clamp goes on the negative (-) lead electrode, and the red clamp goes on the positive (+) lead electrode.
6. When everything is in place, plug the battery charger in, and allow it to run for about 6 hours. Note: During the process, hydrogen gas will be steadily evolved. Carryout the process in a ventilated area, such as a garage, bathroom, or shed.
7. After 6 hours, unplug the battery charger, and remove the clamps from each electrode. Then open the plastic lid, and pullout the clay pot. Note: The clay pot will be filled with a white precipitate. This white precipitate is *magnesium hydroxide*. The cathode liquid will contain a *dilute sulfuric acid solution*. This dilute sulfuric acid solution can be saved, if desired, or mixed with the contents in the clay pot. Mixing both solutions will reform magnesium sulfate (Epsom salt). If you wish to recycle the Epsom salt for future cell charge, mix both liquids. **Note:** To verify, for your own amusement, drop in a little baking soda to the anode liquid.
8. After you have decided what you want to do with both liquids, rinse out the clay pot with tap water, and do the same for the plastic Tupperware container. **Your clay pot is now charged.**



Charging the cell.

10d. Carryout your first procedure (The Preparation of ferrous chloride dihydrate)

Now your ready to carryout your first process. The following process will help you familiarize yourself with electrochemical methods, and the endless processes that can be carried out. So lets get started!

With your "charged" clay pot (just established), reassemble the apparatus as illustrated above. This time, instead of using Epsom salt, we are going to use another common salt, called "pickling salt", which is sodium chloride. Also, the contents in the clay pot will be considered the anode liquid, and the contents in the Tupperware container the cathode liquid. Under most conditions, sodium chloride is relatively inert. Although, when we place this salt into a cell, like the one we have, strange things begin to happen! Now is the time for you to find out what! Go to the store and purchase a box of "pickling" salt. Make sure its just pure salt with nothing added.

1. Assemble the cell as illustrated above. Then dissolve 50 grams (1.8 oz.) of sodium chloride into 300 milliliters (10 fluid oz.) of water, and then dissolve another 50 grams (1.8 oz.) of salt into 1 liter (33.8 fluid oz.) of water. Thereafter, pour the 300

milliliters (10 fluid oz.) of salt solution into the clay pot (anode liquid). Then pour the 1-liter (33.8 fluid oz.) of salt solution into the outer plastic container (cathode liquid).

2. Now secure the plastic lid to the Tupperware container, and then slid in 1 lead or graphite electrode into the cathode side (outer side). The same lead electrode or graphite electrode you used during the charging process may be used. Now, instead of using a lead electrode or graphite electrode in the anode compartment, we are going to use an iron one. Go to the hardware store, and purchase an iron rod (about $\frac{1}{4}$ inches in diameter, and about 12 inches long, or $\frac{1}{2}$ inch in diameter by 12 inches long). 3 Foot long iron rods are usually sold in hardware stores for welding purpose. This rod can be cut into segments of 1 foot each, or simply purchase an iron nail, bolt, or equivalent.

3. Using the same plastic lid as before, or using a different one (of the same size with a custom hole designed just for your iron rod), slid in the iron electrode.

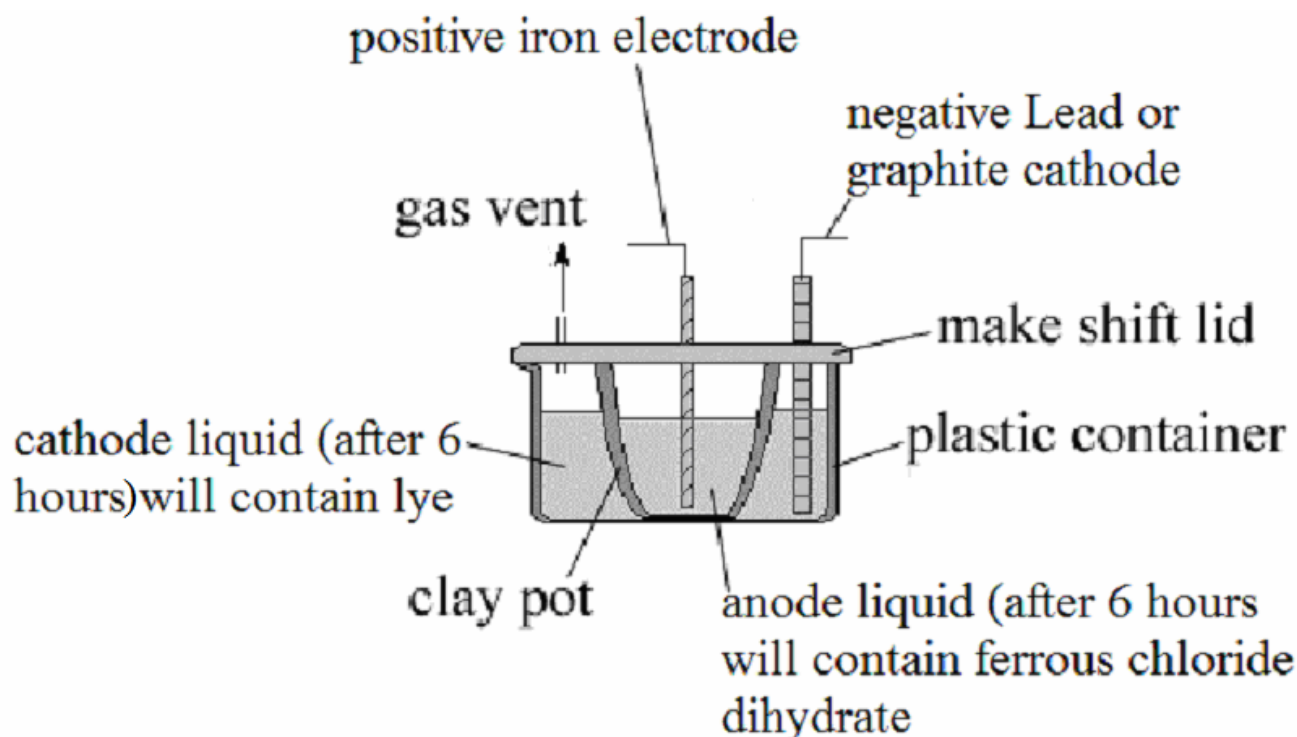
4. Then hook up the battery charger clamps to their respective electrodes (black for negative, red for positive; positive for iron, negative for the lead or graphite electrode), and then plug in the battery charger.

5. Allow the battery charger to run for about 6 hours. Note: hydrogen gas will be steadily evolved during the process. Use a well-ventilated area as before.

6. After 6 hours, unplug the battery charger, and then open the lid to the plastic container. Then carefully remove the clay pot, and then filter the contents of the clay pot. The contents of the clay pot will contain ferrous chloride. By this time you will notice that part of your iron electrode has disappeared! Never fear, it's now in the form of ferrous chloride dihydrate, which is dissolved in the anode liquid. After filtering said anode liquid, you will notice that it has a light greenish color to it. This greenish color is caused by ferrous chloride dihydrate.

7. Filter-off the cathode liquid, if desired, and then perform a little experiment Add a few drops of the anode liquid to a small sample of your cathode liquid. Wham! What you get is a gelatinous precipitate of ferrous hydroxide hydrate. *The cathode liquid will contain sodium hydroxide, commonly called lye.* Use this solution to unclog your sink, or simply discard it. If you want, you can play around with it a little. Take some aluminum foil, and drop it into your cathode liquid. Watch as the sodium hydroxide in the cathode liquid consumes the aluminum foil, producing hydrogen gas.

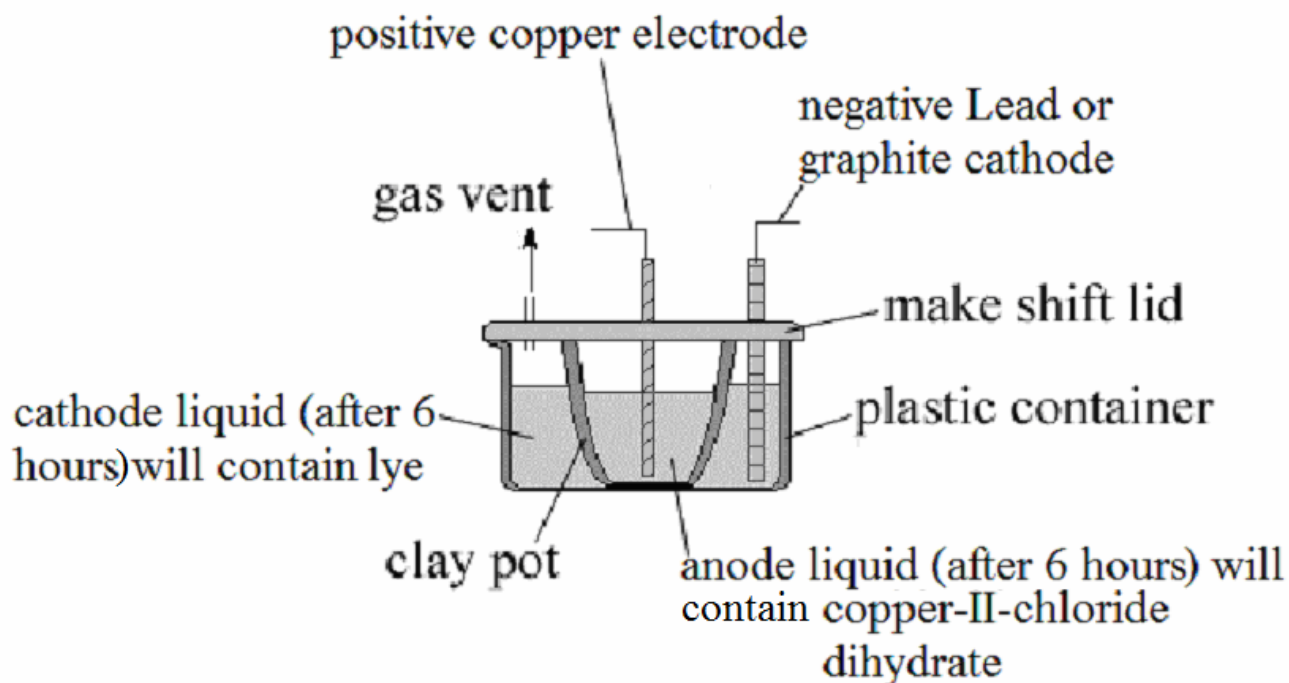
8. *The anode liquid can be evaporated (by simply boiling-off the water), to yield crystals of ferrous chloride.* Before hand, if you like, add a few drops of your anode liquid to a small sample of Clorox bleach or hydrogen peroxide. Wham! What happened? It turned black! The ferrous chloride was oxidized by the Clorox bleach (or hydrogen peroxide) to ferric chloride. Ferric chloride is commonly used by electricians to etch printed circuit boards!



Preparation of ferrous chloride dihydrate.

10e. Now that you have learned to make ferrous chloride, you can apply this new skill to making other metal chlorides. Let's try one more using a different metal anode, and then we will move onto something more advanced.

1. Assemble the cell as previously described, using a clean, freshly charged clay pot. Note: In this procedure, a brand new clay pot should be charged using the aforementioned process for cell charging. If we wanted to carryout another process using another iron anode, we could use the same cell we just finished with (a freshly charged cell can be used for the SAME procedure multiple times without having to recharge said cell). Although, since we are going to use a copper anode in this procedure, we should either recharge the cell we just used (for the ferrous chloride preparation), or we should use a clean, freshly charged clay pot.
2. Then dissolve 50 grams (1.8 oz.) of sodium chloride into 300 milliliters (10 fluid oz.) of water, and then add this solution to the clay pot (anode liquid). Then dissolve 50 grams (1.8 oz.) of sodium chloride into 1 liter (33.8 fluid oz.) of water, and then pour this solution into the plastic Tupperware container (cathode liquid).
3. Then attach the plastic lid, and then insert your electrodes. 1 Lead of graphite electrode for the negative cathode, and a copper electrode for the positive anode. Note: Use copper pipes about 10 to 12 inches long, by ½ inch in diameter for the electrode. If desired, you can use copper wire of up to 20 gauge, wound around a pencil to form a coil, as the anode.
4. Then attach the battery charger clamps to their respective electrodes (red for copper, black for lead or graphite).
5. Then plug in the battery charger, and allow it to run for 6 hours. After 6 hours, unplug the battery charger, and then open the cell. Remove the clay pot from the plastic Tupperware container, and then pour the contents into a clean glass, or beaker. You can discard the cathode liquid if desired, as it will contain sodium hydroxide (lye). Allow the anode liquid to stand for 24 hours, and then carefully decant (pour off), the liquid above the bluish precipitate. The bluish precipitate will be very finely divided cupric hydroxide, which is very difficult to filter-off. Use centrifuge apparatus if you have one. The anode liquid will have an emerald greenish-blue color to it, and will contain cupric chloride dihydrate. If you want, you can add a few drops of this cupric chloride dihydrate to a small sample of your cathode liquid, to obtain an interesting precipitate of cupric hydroxide hydrate. *You can evaporate the anode liquid to dryness (by simply boiling off the water) to obtain brownish black crystals of semi anhydrous cupric chloride.*



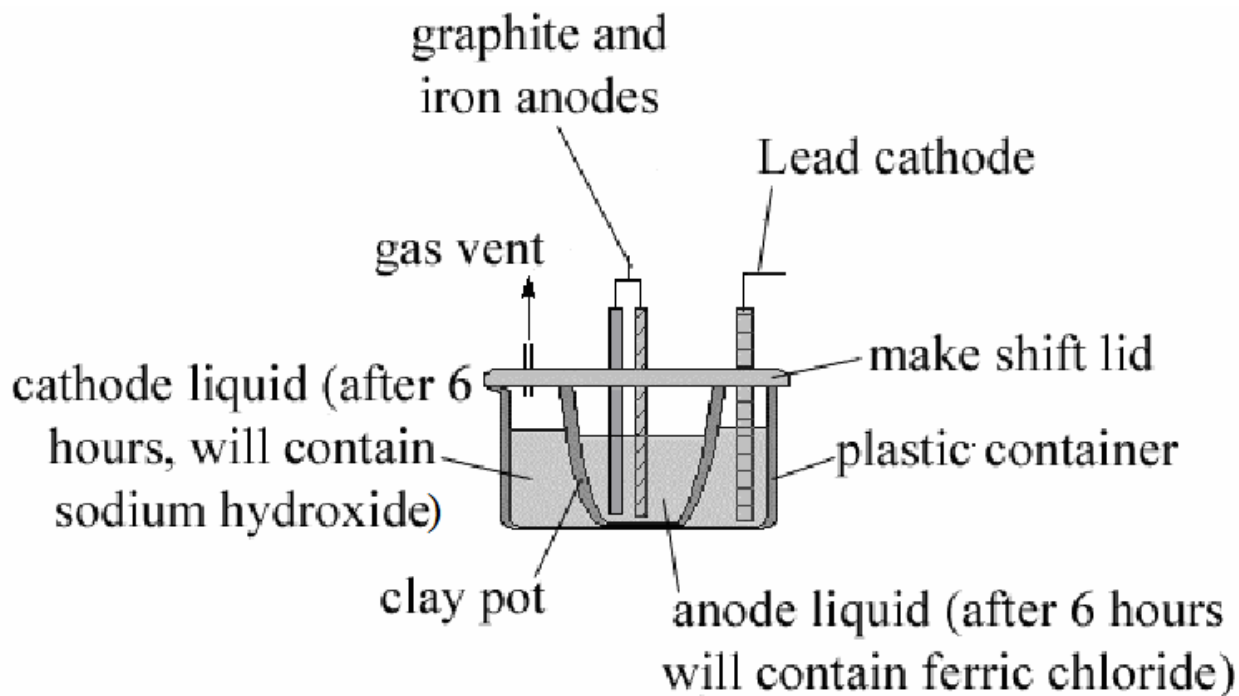
Preparation of cupric chloride dihydrate.

Now that we have run through the two simple processes. Its time to move onto something a little more advanced. To review what we have learned so far, look at the two similarities between the two procedures we have done. In the first procedure we made ferrous chloride by electrolyzing a salt solution using an iron anode. In the second procedure we used a copper anode, and got cupric chloride. Now, you should remember that if we replaced the copper anode with zinc for example, we would get zinc chloride. Note: Any metal can be used with the exception of lead, platinum, and a few others we need not discuss at this point. If you want, try it with aluminum, zinc, nickel, chromium, or magnesium to get the respective chlorides. Note: The sodium chloride can be replaced with sodium bromide, or sodium iodide to make the corresponding bromides and iodides.

10f. Now lets try something a little bit more interesting

This time, we will use more then one anode, to carryout a double reaction. In this example, we will make ferric chloride hydrate by using an iron anode, and a graphite anode. The graphite anode should be 8 to 12 inches long, by at least ¼ inch in diameter. Note: these graphite rods can be obtained from numerous arts and craft stores. Look for arts and crafts stores that sell drawing freehand accessories. Graphite is a form of carbon, and is commonly called "pencil lead".

1. Assemble the cell as previously described, using a clean, freshly charged clay pot. Note: In this procedure, a brand new clay pot should be charged using the aforementioned process for cell charging. If we wanted to carryout another process using another copper anode, we could use the same cell we just finished with (a freshly charged cell can be used for the SAME procedure multiple times without having to recharge said cell). Although, since we are going to use two different anodes in this procedure, we should either recharge the cell we just used (for the cupric chloride preparation), or we should use a clean, freshly charged clay pot.
2. Then dissolve 50 grams (1.8 oz.) of sodium chloride into 300 milliliters (10 fluid oz.) of water, and then add this solution to the clay pot (anode liquid). Then dissolve 50 grams (1.8 oz.) of sodium chloride into 1 liter (33.8 fluid oz.) of water, and then pour this solution into the plastic Tupperware container (cathode liquid).
3. Then attach the plastic lid, and then insert your electrodes. 1 Lead electrode for the negative cathode, a graphite electrode and a iron electrode for the positive anode.
4. Then attach the battery charger clamps to their respective electrodes; Red for graphite and iron (to connect a single battery charger clamp to both electrodes, wrap a copper wire around both the graphite and iron electrodes), and black for lead.
5. Then plug in the battery charger, and allow it to run for 6 hours. After 6 hours, unplug the battery charger, and then open the cell. Remove the clay pot from the plastic Tupperware container, and then pour the contents into a clean glass, or beaker. You can discard the cathode liquid if desired, as it will contain sodium hydroxide (lye). Filter the anode liquid (using a standard plastic funnel, and coffee filters). The anode liquid will have a dark brown to black color to it. This color is caused by ferric chloride hydrate. If you want, you can add a few drops of this ferric chloride hydrate to a small sample of your cathode liquid, to obtain an interesting precipitate of ferric hydroxide hydrate. You can evaporate the anode liquid to dryness to obtain brownish black crystals of ferric chloride hydrate. Note: Do not boil the anode liquid to drive off the water. Doing so will produce highly corrosive hydrochloric acid vapors. If you want, you can boil the anode liquid under appropriate conditions to collect, and then condense the hydrochloric acid for later use.

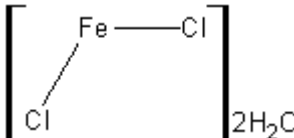
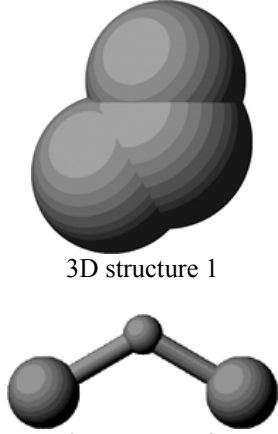
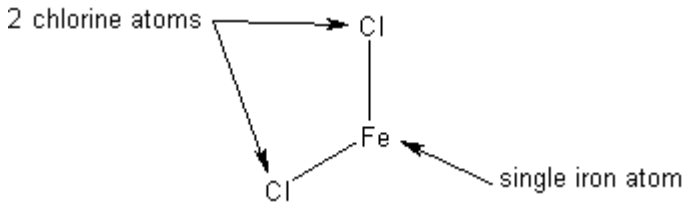


Preparation of ferric chloride.

Now you can try different combinations if desired, by using different electrodes, and/or using multiple electrodes.

Procedure 59: Electro Preparation 13: The Preparation of Ferrous chloride dihydrate (review process)

Also known as: Iron dichloride 2 hydrate

	
Chemical structure	3D Structure
	$\text{FeCl}_2 - 2\text{H}_2\text{O}$ <p>(Mixed hydrates, but mostly the dihydrate)</p>
Structure make-up	Short hand chemical structure

Ferrous chloride dihydrate

Ferrous chloride dihydrate forms white monoclinic crystals with a slight greenish color (causes solutions to have a slight greenish tint). The crystals form the monohydrate when heated to 120 Celsius, but some decomposition may result from oxidation. The crystals are readily soluble in water or alcohol.

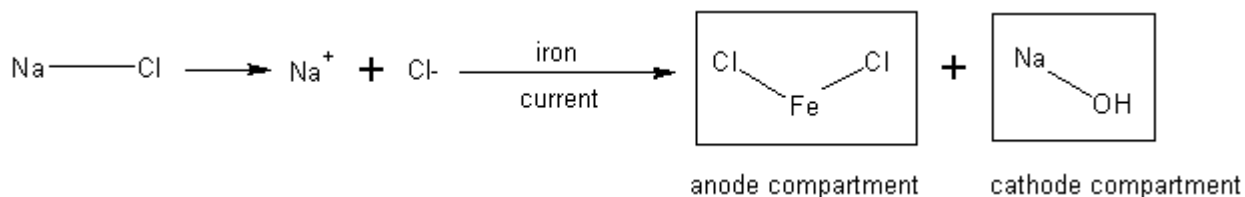
Method 1: Preparation of ferrous chloride dihydrate from pickling salt and an iron electrode

(By-products from reaction: sodium hydroxide and hydrogen gas)

Materials:

1. 250 milliliters (8.5 fluid oz.) of warm water	3. 500 to 750 milliliters (17 to 25.4 fluid oz.) of warm water
2. 10 grams (0.35 oz.) of pickling salt	4. 50 grams (1.8 oz.) of pickling salt

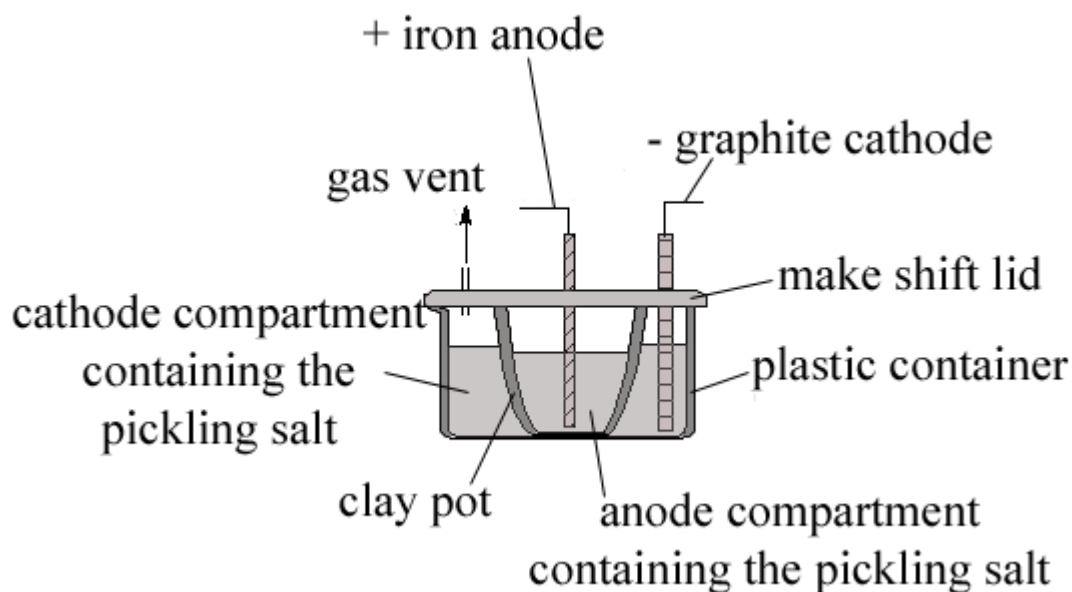
Reaction summary: Ferrous chloride dihydrate can be prepared by electrolyzing a solution of pickling salt using an iron anode in a diaphragm “salt bridge” cell.



Why does this reaction happen? In a divided cell, when current is applied to the system, the positively charged sodium ions migrate to the negative cathode. At the same time, the negatively charged chlorine ions migrate to the positive anode. The sodium gains an electron forming free sodium, which then spontaneously reacts with the water forming sodium hydroxide and liberating hydrogen gas. At the same time, the chlorine ions give up an electron at anode forming free chlorine, which then reacts with the iron electrode forming ferrous chloride. Because there is a barrier the sodium hydroxide and ferrous chloride cannot react with one another, so both compounds remain dissolved in solution.

Hazards: Use caution as sodium hydroxide is formed in the cathode compartment. Sodium hydroxide, otherwise known as lye, is very corrosive to skin and clothes

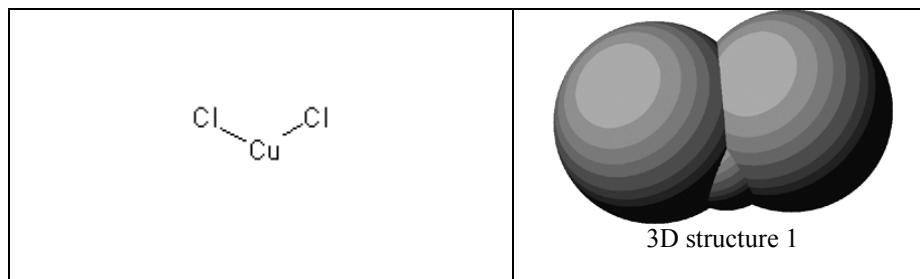
Procedure: In review, assemble the cell as illustrated below, using a clean, freshly charged clay pot. Thereafter, into the freshly charged cell, place 250 milliliters (8.5 fluid oz.) of warm water into the clay pot (anode compartment), and then add and dissolve, 10 grams (0.35 oz.) of pickling salt there into. Then place 500 to 750 milliliters (17 to 25.4 fluid oz.) of warm water into the cathode compartment, followed by adding and dissolving 50 grams (1.8 oz.) of pickling salt. Thereafter, using the appropriate electrodes, attach the clamps of your power supply to the proper electrode, and then electrolysis the cell at 6 to 12 volt, by 2 to 6 amp direct current for about 6 to 12 hours (depending on how much ferrous chloride you want). Thereafter, remove the power source, and then open the cell. Then pour the contents of the anode compartment into a clean beaker. The contents of the cathode compartment, which will contain sodium hydroxide, can be discarded, or the sodium hydroxide can be recovered by boiling-off the water. Now, filter the anode liquid to remove any insoluble impurities, and then place the filtered anode liquid aside temporarily.

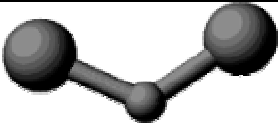
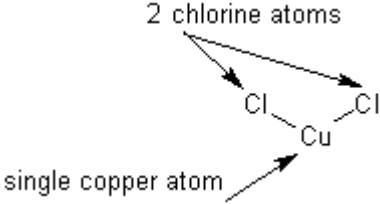


Setup for the preparation of ferrous chloride.

Procedure 60: Electro Preparation 14: The Preparation of Cupric chloride dihydrate (review process)

Also known as: Copper-II chloride 2 hydrate



	 3D structure 2
Chemical structure	3D Structure
	CuCl ₂
Structure make-up	Short hand chemical structure

Cupric chloride Dihydrate

Cupric chloride dihydrate forms beautiful bluish green crystals or blue powder. The crystals are freely soluble in water and alcohol, and only moderate soluble in acetone. The crystals have a crude melting point of about 100 Celsius, but water loss begins when the crystals are heated to 70 Celsius.

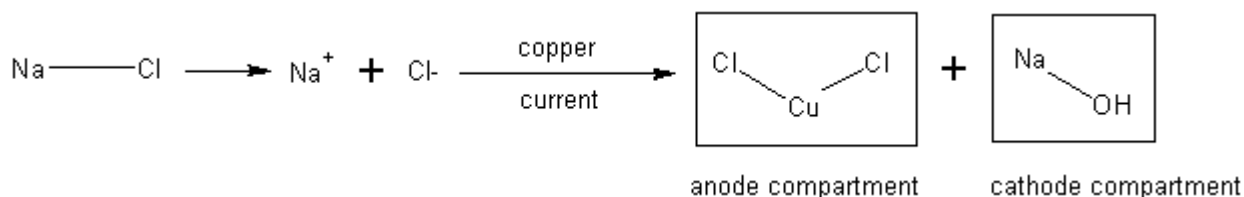
Method 1: Preparation of cupric chloride dihydrate from pickling salt and an iron electrode

(By-products from reaction: sodium hydroxide)

Materials:

1. 250 milliliters (8.5 fluid oz.) of warm tap water	2. 10 grams (0.35 oz.) of pickling salt
3. 500 to 750 milliliters (17 to 25.4 fluid oz.) of warm tap water	4. 50 grams (1.8 oz.) of pickling salt

Reaction summary: Cupric chloride dihydrate can be prepared by electrolyzing a solution of pickling salt using a copper anode in a diaphragm “salt bridge” cell.



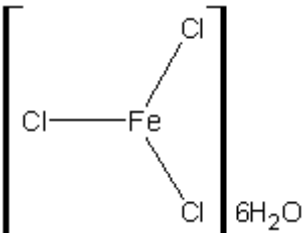
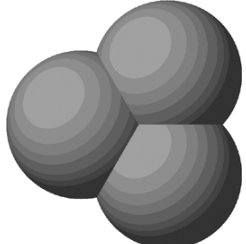
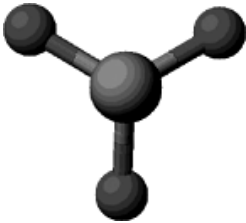
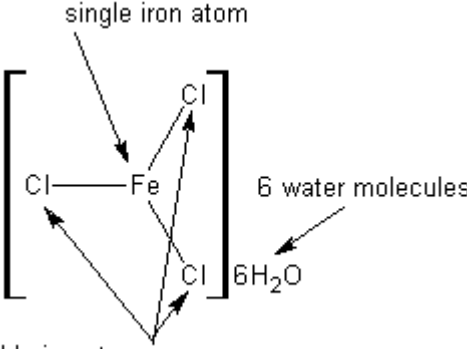
Why does this reaction happen? Same as in the previous procedure, but the iron anode is replaced with a copper one.

Hazards: Use caution as sodium hydroxide is formed in the cathode compartment. Sodium hydroxide, otherwise known as lye, is very corrosive to skin and clothes

Procedure: In review, assemble the cell as illustrated below, using a clean, freshly charged clay pot. Thereafter, into the freshly charged cell, place 250 milliliters (8.5 fluid oz.) of warm tap water into the clay pot (anode compartment), and then add and dissolve, 10 grams (0.35 oz.) of pickling salt there into. Then place 500 to 750 milliliters (17 to 25.4 fluid oz.) of warm tap water into the cathode compartment, followed by adding and dissolving 50 grams (1.8 oz.) of pickling salt. Thereafter, using the appropriate electrodes, attach the clamps of your power supply to the proper electrode, and then electrolysis the cell at 6 to 12 volt, by 2 to 6 amp direct current for about 6 to 12 hours (depending on how much cupric chloride you want). Thereafter, remove the power source, and then open the cell. Then pour the contents of the anode compartment into a clean beaker. The contents of the cathode compartment, which will contain sodium hydroxide, can be discarded, or the sodium hydroxide can be recovered by boiling-off the water. Now, filter the anode liquid to remove any insoluble impurities, and then place the filtered anode liquid aside temporarily.

Procedure 61: Electro Preparation 15: The Preparation of Ferric chloride hexahydrate (review process)

Also known as: Trichloride of iron 6 hydrate

	 3D structure 1  3D structure 2
Chemical structure	3D Structure
	$\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$
Structure make-up	Condensed chemical structure

Ferric chloride hexahydrate

Ferric chloride hexahydrate forms brownish black crystals. The crystals are readily soluble in water or alcohol, and have a melting point 37 Celsius. Aqueous solutions of ferric chloride hexahydrate are readily decomposed by boiling, forming red ferric oxide, and hydrogen chloride.

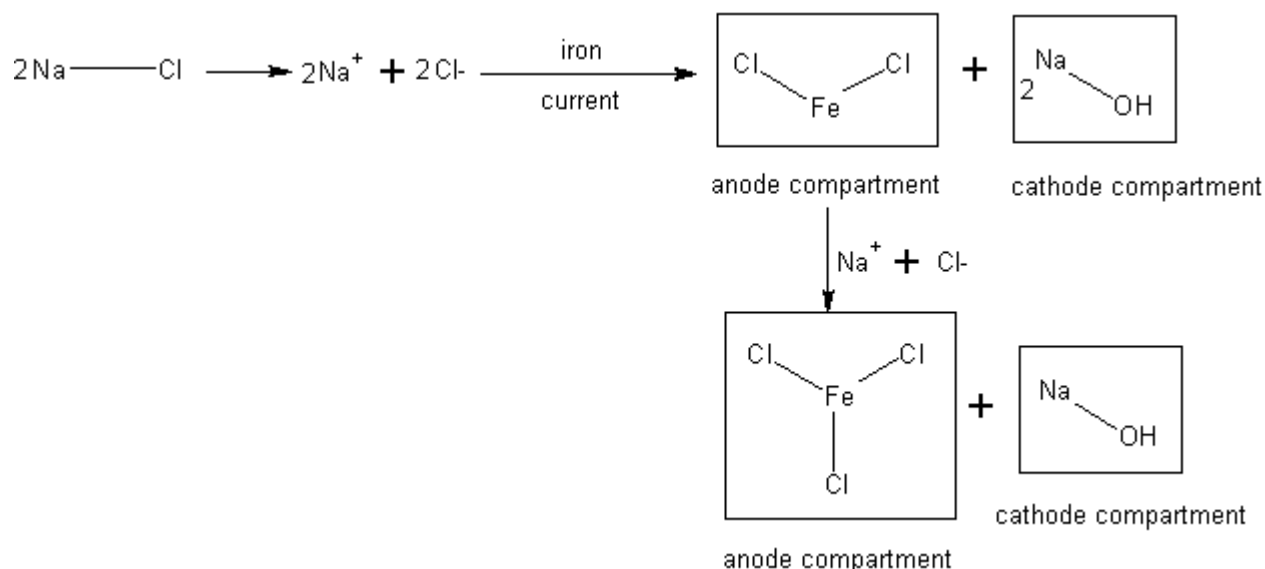
Method 1: Preparation of ferric chloride hexahydrate from pickling salt and graphite and iron electrodes (1 pot method)

(By-products from reaction: sodium hydroxide and hydrogen gas)

Materials:

1. 50 grams (1.8 oz.) of sodium chloride	2. 300 milliliters (10 fluid oz.) of tap water
2. 50 grams (1.8 oz.) of sodium chloride	3. 1 liter (33.8 fluid oz.) of tap water

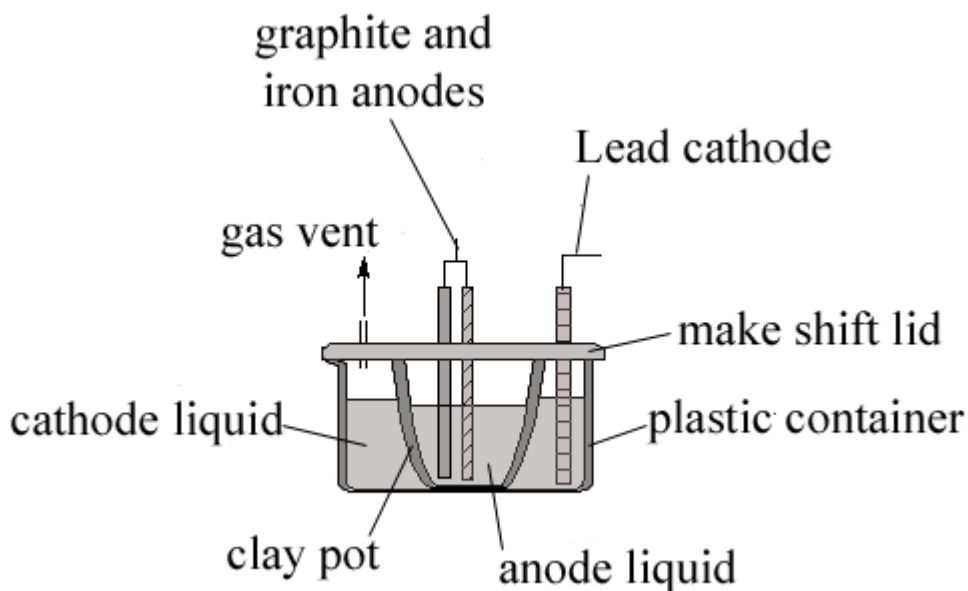
Reaction summary: Ferric chloride hexahydrate can be prepared by electrolyzing a solution of pickling salt using a graphite and iron anodes in a salt bridge cell.



Why does this reaction happen? In a divided cell, when current is applied to the system, the positively charged sodium ions migrate to the negative cathode, gaining an electron, and forming free sodium. Because free sodium is so reactive, it reacts spontaneously with the water forming sodium hydroxide and liberating hydrogen gas. At the same time, the negatively charged chlorine ions migrate to the positive anode giving up an electron and forming free chlorine, which then reacts with the iron electrode forming ferrous chloride. Because there is a barrier the sodium hydroxide and ferrous chloride cannot react with one another, so both compounds remain dissolved in solution. However, because there is an inert electrode of graphite in this procedure, excess chlorine ions migrate to the graphite anode forming free chlorine, which evolves as free chlorine. This free chlorine then immediately oxidizes the ferrous chloride to ferric chloride. As before, because there is a barrier, the anode products cannot mix with the cathode products, and hence, the ferric chloride and sodium hydroxide remain dissolved in solution in their respective compartments.

Hazards: Use caution as sodium hydroxide is formed in the cathode compartment. Sodium hydroxide, otherwise known as lye, is very corrosive to skin and clothes.

Procedure: In review, assemble the cell as illustrated below, using a clean, freshly charged clay pot. Thereafter, add and dissolve 50 grams (1.8 oz.) of sodium chloride into 300 milliliters (10 fluid oz.) of tap water, and then add this solution to the clay pot (anode liquid). Then dissolve 50 grams (1.8 oz.) of sodium chloride into 1 liter (33.8 fluid oz.) of tap water, and then pour this solution into the plastic Tupperware container (cathode liquid). Thereafter, attach the plastic lid, and then insert your electrodes. 1 Lead electrode for the negative cathode, and a graphite electrode and an iron electrode for the positive anodes. Then attach the power supply clamps to their respective electrodes, and then turn on your power supply and allow it to run for 6 to 8 hours at 6 to 12 volt at 2 to 6 amp direct current. After such time, unplug your power supply, and then open the cell. Remove the clay pot from the plastic Tupperware container, and then pour the contents of the anode liquid into a clean glass, or beaker. You can discard the cathode liquid if desired, as it will contain sodium hydroxide (lye). Filter the anode liquid to remove any insoluble materials. Note: The anode liquid will have a dark brown to black color to it. At this stage, you should allow the filtered anode liquid to stand in the open to allow the water to evaporate, or you can place the liquid into shallow bowls, and place these bowls into a food dehydrator machine (used to dry foods). In any case, you want to evaporate the water below a temperature of 70 Celsius to prevent decomposition of the ferric chloride. Note: Do not boil the anode liquid to drive off the water. Doing so will produce highly corrosive hydrochloric acid vapors. If you want, you can boil the anode liquid under appropriate conditions to collect, and then condense the hydrochloric acid for later use.



Setup for preparing ferric chloride.

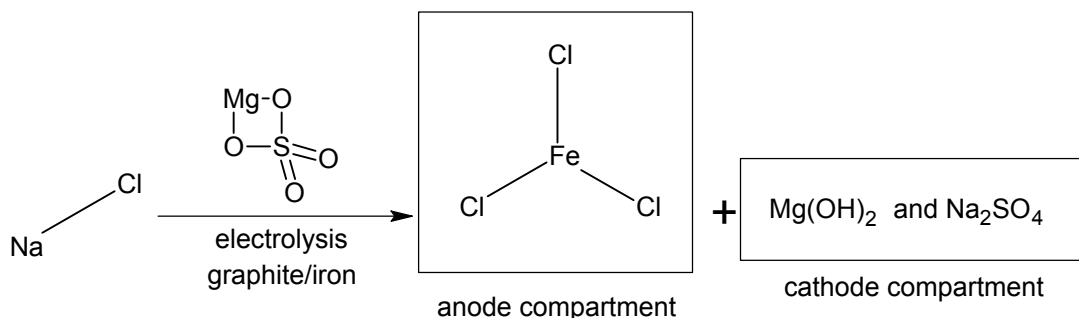
Method 2: Preparation of ferric chloride from pickling salt, Epsom salt, and iron/graphite electrodes (Preferred method)

(By-products from reaction: magnesium hydroxide, sodium sulfate and hydrogen gas)

Materials:

1. 120 grams (4.2 oz.) of sodium chloride	3. 251 grams (8.8 oz.) of Epsom salt
2. 1100 milliliters (37 fluid oz.) of tap water	

Reaction summary: Ferric chloride can be prepared by electrolyzing a solution of pickling salt using a graphite and iron anodes in a salt bridge cell.

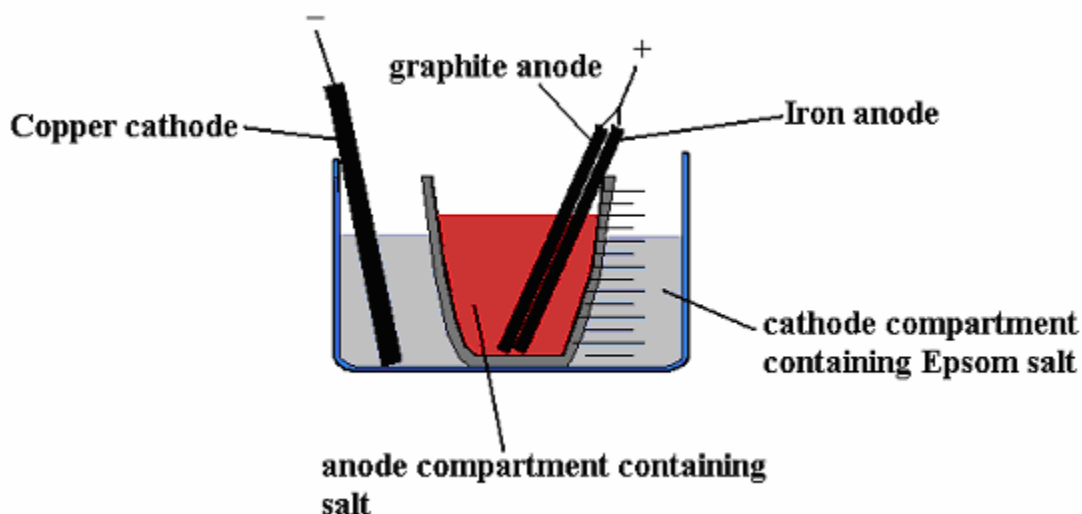


Why does this reaction happen? When a clay pot is used in a two-compartment cell, a special reaction occurs. In this process, the dissolved sodium ions in the anode liquid migrate through the clay pot and bond with the sulfate ions dissolved in the cathode liquid, forming sodium sulfate, which remains dissolved in the cathode compartment. At the same time the chlorine ions dissolved in the anode liquid, react with the iron anode forming ferrous chloride, which remains dissolved in the anode liquid; at the same time, additional chlorine ions dissolved in the anode liquid gains an electron from the anodes forming free chlorine, which spontaneously oxidizes the ferrous chloride to ferric chloride, which dissolves and remains dissolved in the anode liquid. Because the magnesium has no place to go it precipitates as magnesium hydroxide in the cathode liquid.

Hazards: Use proper ventilation as hydrogen gas is evolved and accumulate in enclosed areas forming explosive mixtures.

Procedure: Dissolve 120 grams of sodium chloride into 400 milliliters of tap water. Thereafter, pour this solution into the anode compartment. Thereafter, dissolve 251 grams of Epsom salt into 700 milliliters of tap water, and then pour this solution into the cathode compartment. Then electrolyze the apparatus at 12 volt 6 amp for 12 hours or more, using a iron/graphite anode, and copper cathode. After about 12 hours, remove the electrical source, and then filter the anode liquid to remove any

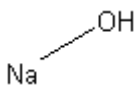


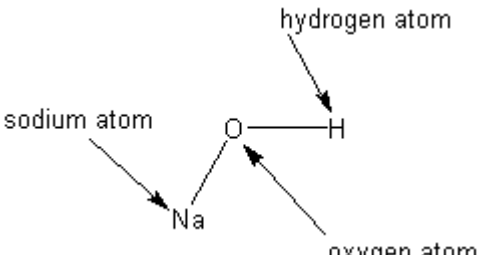
insoluble materials. Then, place the filtered anode liquid into a crystallizing dish and allow it to evaporate to form brilliant golden yellow crystals. The cathode liquid can also be filtered to recover the insoluble magnesium hydroxide. If desired, the filtered cathode liquid can be evaporated to recover colorless crystals of sodium sulfate decahydrate.



Apparatus for producing ferric chloride in the anode compartment.

Procedure 62: Electro preparation 16: The Preparation of Sodium Hydroxide (Lye)

Also known as: Caustic soda, Caustic alkaline, sodium hydroxy oxide

	 <p>3D structure 1</p>  <p>3D structure 2</p>
Chemical structure	3D Structure
	<p>NaOH</p>
Structure make-up	Condensed chemical structure

Sodium hydroxide

Sodium hydroxide forms fused solid pieces, granules, rods, or powder. It rapidly absorbs moisture and carbon dioxide from the air. Solutions of sodium hydroxide are very corrosive to animal tissue, and aluminum. It has a melting point of 318 Celsius. Sodium hydroxide is very soluble in water and alcohol. It generates large amounts of heat when dissolving in water, or when mixed with acid. Sodium hydroxide is toxic. Handle sodium hydroxide with care. Sodium hydroxide is a widely available commercial chemical, which is sold under a variety of names such as “Lye”. Sodium hydroxide is prepared on an industrial scale in a procedure called the “chloro-alkali” process. In the chloro-alkali process, a sodium chloride solution is electrolyzed in a special cell composed of two compartments separated by a porous membrane. Chlorine gas is produced at the positive anode, and sodium hydroxide forms at cathode.

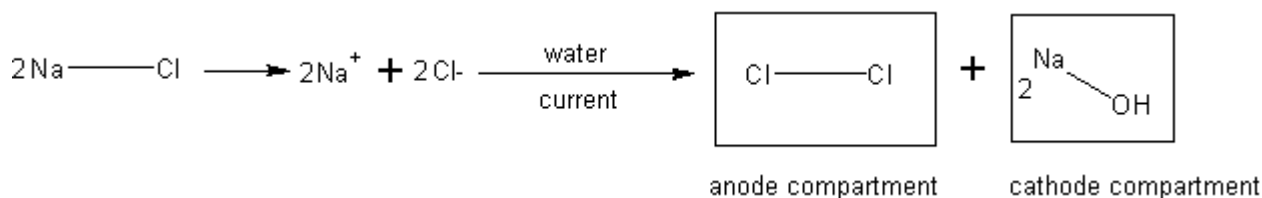
Method 1: Preparation of Sodium Hydroxide from pickling salt (sodium chloride) using a special membrane “divided cell” cell

(By-products from reaction: chlorine gas and hydrogen gas)

Materials:

1. 500 grams (1.1 lbs.) of table salt (sodium chloride preferable sold under the name “pickling salt”)	3. 1000 milliliters (2.1 pints) of tap water
2. 1500 milliliters (3.1 pints) of tap water	

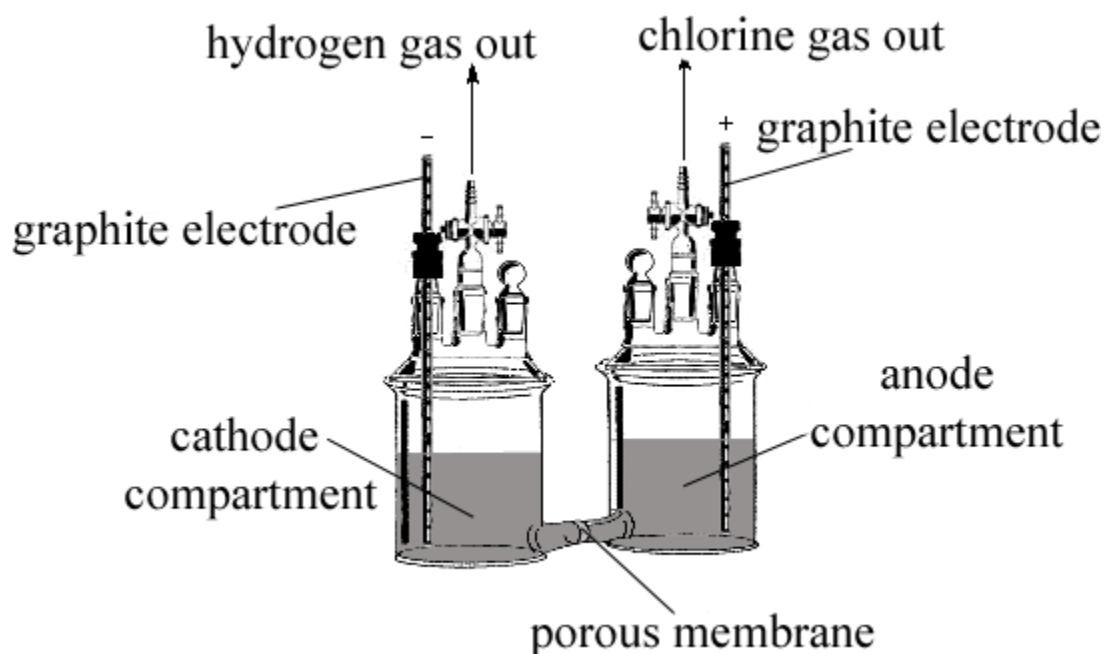
Reaction summary: Sodium hydroxide can be prepared by electrolyzing a sodium chloride solution in a two-compartment cell separated by a porous membrane. Chlorine gas is liberated at the positive anode and hydrogen and sodium hydroxide are liberated at the cathode. *Use proper ventilation when running the electrolysis cell because of chlorine and hydrogen gas evolution. Run the cell in an area that is away from direct sunlight.*



Why does this reaction happen? As in previous reactions, when current is applied to this system, the sodium ions migrate to the cathode forming sodium hydroxide and hydrogen gas. At the same time, the chlorine ions migrate to the anode forming free chlorine because an inert anode electrode of graphite is used. Because there is a porous membrane barrier, the anode products cannot react with the cathode products; therefore, the chlorine gas bubbles to the surface and the sodium hydroxide remains dissolved in the cathode liquid.

Hazards: Chlorine gas is produced in this procedure; either properly vent the gas, or neutralize it by bubbling it through a sodium hydroxide or sodium carbonate solution. Carryout this procedure away from direct sun-light, and keep any source of ignition away— hydrogen gas is very flammable and explosive.

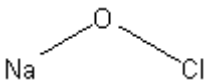


Procedure: Prepare the cell shown in the following illustration, and then add 500 grams (1.1 lbs.) of table salt (sodium chloride preferable sold under the name “pickling salt”) to a beaker and then add 1500 milliliters (3.1 pints) of tap water. Then stir the mixture to dissolve the table salt. After which, pour 1000 milliliters (2.1 pints) of tap water into the apparatus cathode compartment. Then pour about 100 milliliters (3.4 fluid oz.) of the sodium chloride solution into the cathode compartment to bring its total volume to about 1100 milliliters. Afterwards, pour the rest of the sodium chloride solution into the apparatus anode compartment. Then put the graphite electrodes in place and electrolysis at 12-amp/12-volt until no more chlorine gas is evolved. When no more chlorine is evolved, stop the electrolysis. Then pour the cathode liquid into a beaker, and then filter to remove any insoluble materials. After filtering, pour the sodium hydroxide solution into a clean stainless steel beaker and then boil-off the water until dry sodium hydroxide solid remains. Do not use glass when boiling-off the water because the sodium hydroxide will corrode the glass and cause it to break.



Professional all glass apparatus for the production of sodium hydroxide. This process is used in the “chloro alkali process” as seen on an industrial scale for the preparation of chlorine gas, lye, and many other chemicals. Chlorine gas is liberated at the positive anode electrode, and hydrogen gas is liberated at the negative cathode electrode. The sodium hydroxide is formed at the negative cathode electrode and remains dissolved in water. Note: The porous membrane is made-up of a special organic-fluoro polymers.

Procedure 63: Electro preparation 17: The Preparation of Bleach (sodium hypochlorite)

Also known as: Chloro sodium oxide

	 <p>3D structure 1</p>  <p>3D structure 2</p>
Chemical structure	3D Structure

	NaOCl
Structure make-up	Condensed chemical structure

Sodium hypochlorite solution

Sodium hypochlorite solution, commonly called bleach, is a light yellowish liquid with a characteristic chlorine-like odor. It is a powerful oxidizing agent, and is used extensively in disinfections and decontamination procedures. It is quite stable at room temperature, but decomposes when heated forming sodium chlorate and salt. It can be easily prepared using a diaphragm cell, or by passing chlorine gas into a cold dilute sodium hydroxide solution.

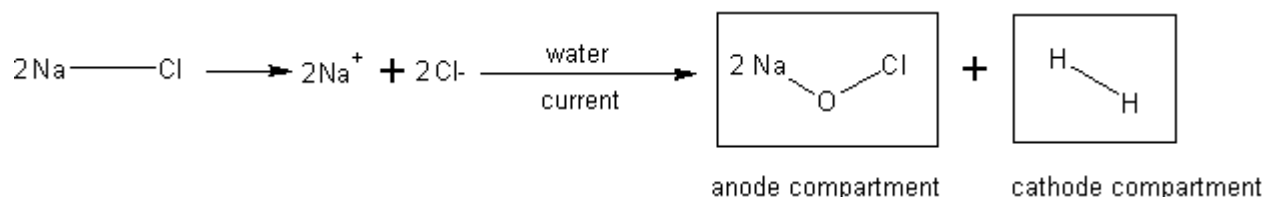
Method 1: Preparation of Sodium Hypochlorite solution from pickling salt (sodium chloride)

(By-products from reaction: some chlorine gas, sodium hydroxide, and hydrogen gas)

Materials:

1. 100 grams (3.5 oz.) of pickling salt	3. 25 grams (1.0 oz.) of pickling salt
2. 350 milliliters (11.8 fluid oz.) of cold tap water	4. 350 milliliters (11.8 fluid oz.) of cold tap water

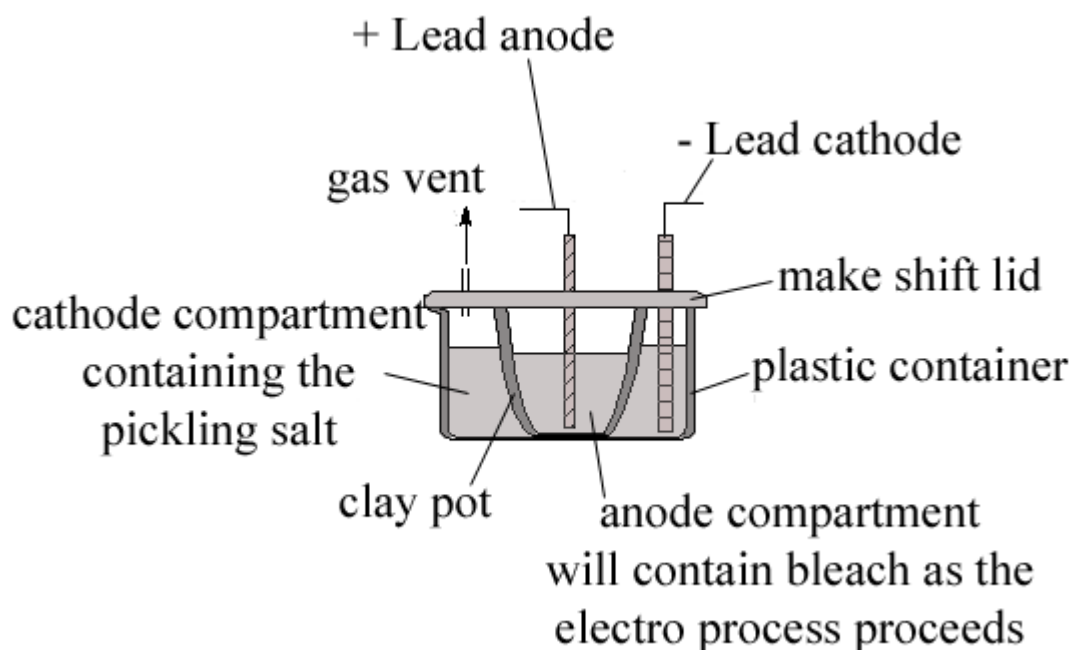
Reaction summary: Bleach can be made using an electrochemical process whereby pickling salt is electrolyzed. During the process, chlorine gas is evolved at the anode, and sodium hydroxide is liberated at the cathode. As the process proceeds, the chlorine reacts with the sodium hydroxide forming sodium hypochlorite (bleach). Some chlorine gas does escape, so use proper ventilation when carrying out this operation.



Why does this reaction happen? As in previous reactions, when current is applied to the system, the sodium ions migrate to the cathode forming sodium hydroxide and hydrogen gas. At the same time, the chlorine ions migrate to the anode forming chlorine gas. Now, when using a standard salt bridge cell, there is a secondary reaction that takes place. The sodium hydroxide that forms in the cathode compartment is attracted to the free chlorine that forms in the anode compartment. This attraction causes the free chlorine to react with the sodium hydroxide forming sodium hypochlorite. Because there is a barrier, the sodium hypochlorite remains dissolved in the anode compartment. Note: this reaction would be impossible in an open cell because the sodium hypochlorite would get reduced back to sodium chloride by the cathode.

Hazards: Perform this operation in a well-ventilated area as chlorine gas is evolved. However, if you have an airtight system, the operation can be performed within enclosed areas as long as you neutralize the chlorine gas by bubbling it into a baking soda slurry or lye solution.

Procedure: Assemble the cell as illustrated below, using a clean brand-new clay pot. Note: unlike previous procedures, this procedure requires a clean brand new clay pot, rather than a freshly charged clay pot as seen earlier. Thereafter, add and dissolve 100 grams (3.5 oz.) of pickling salt into 350 milliliters (11.8 fluid oz.) of cold tap water. Thereafter, place this salt solution into the cathode compartment (-). Thereafter, dissolve 25 grams (1.0 oz.) of pickling salt into 350 milliliters (11.8 fluid oz.) of cold tap water, and then place this salt solution into the anode compartment (+). Now, assemble the cell as illustrated below, using two lead electrodes, and then begin the operation by turning on your power supply, or by plugging in your battery charger. Allow the cell to electrolysis for about 15 hours at 6 Volt/6 Amp or equivalent—be sure to monitor the temperature so as it does not exceed 40 Celsius.



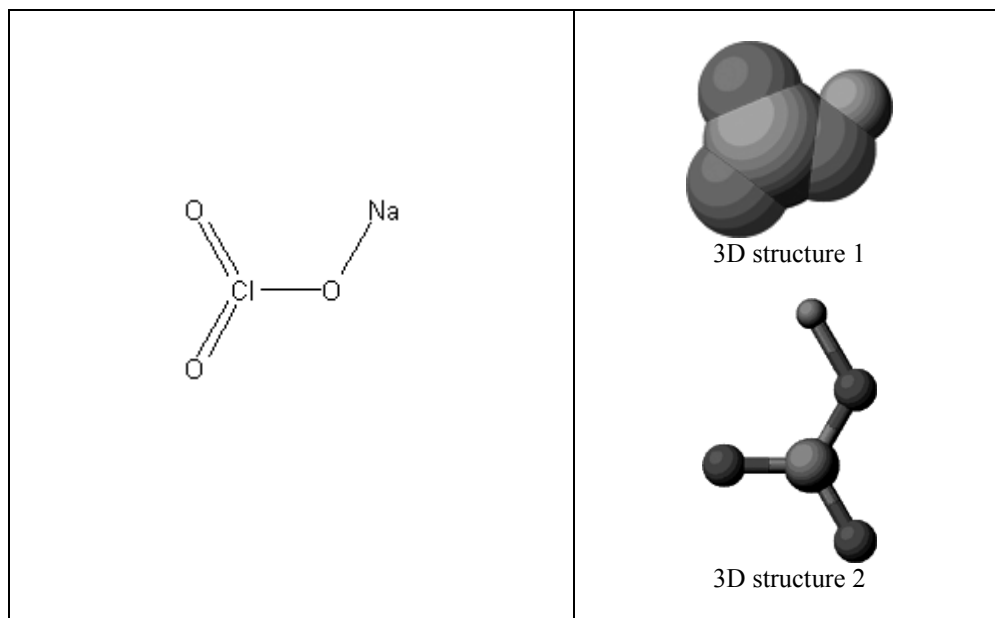
Set-up for the preparation of bleach from pickling salt. The lead electrodes can be replaced with titanium, chromium, or graphite.

After the 15-hour electrolysis process, unplug your power supply, and then open the cell. Carefully remove the clay pot, and then dump its contents into a clean beaker or similar glass container—the anode compartment will contain the bleach. The cathode compartment will contain some sodium hydroxide. You can discard the cathode liquid if desired, or you can use it for other applications. Your bleach should then be quickly filtered, to remove any insoluble impurities, and then stored in any suitable containers. The bleach solution will be about 20 to 25% concentration. Note: the cathode liquid will probably also contain some bleach admixed with the sodium hydroxide, and un-reacted salt.

Note: Bleach can also be made by bubbling chlorine gas into a solution of lye.

Procedure 64: Electro preparation 18: The Preparation of Sodium Chlorate

Also known as: Atlacide; Defol



Chemical structure	3D Structure
	NaClO_3
Structure make-up	Short hand chemical structure

Sodium chlorate

Sodium chlorate forms colorless, odorless crystals or white granules. The melting point of the crystals is 248 Celsius, but it begins to decompose into oxygen and sodium perchlorate when heated to 300 Celsius. The crystals are highly soluble in water, but relatively insoluble in alcohol, and most common organic solvents. Sodium chlorate is widely used in pyrotechnic compositions, and in the preparation of ammonium chlorate, and perchlorate, which are used in powerful solid rocket fuels.

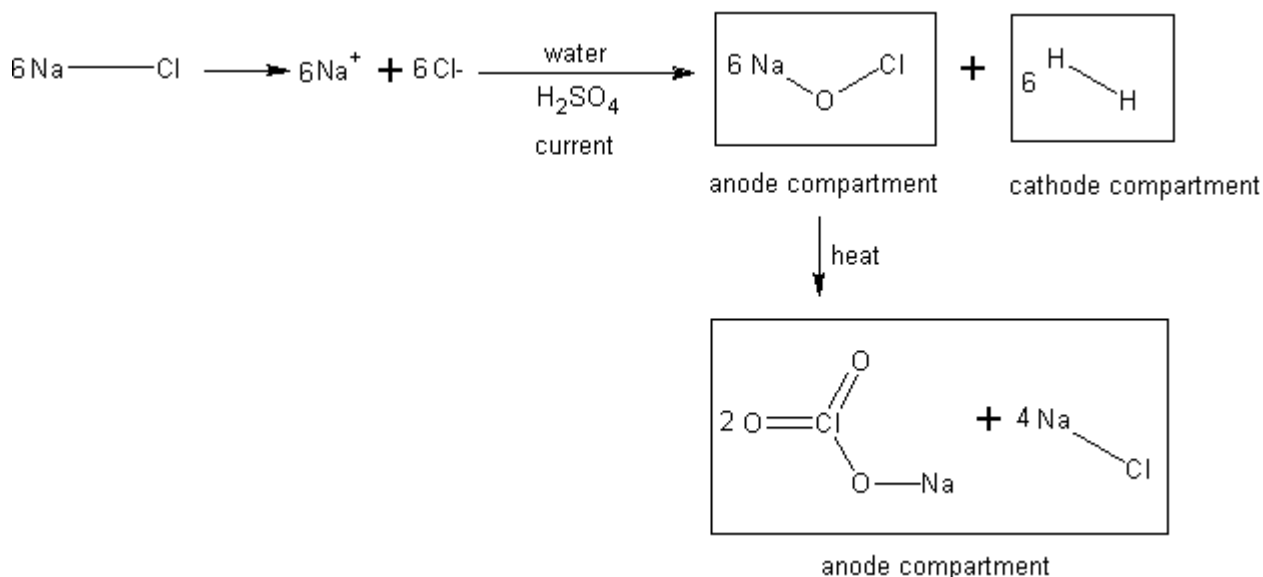
Method 1: Preparation of Sodium chlorate from pickling salt using a diaphragm “divided cell”

(By-products from reaction: sodium hypochlorite, chlorine gas, sodium hydroxide, and hydrogen gas)

Materials:

1. 100 grams (3.5 oz.) of pickling salt	5. 5 drops of concentrated sulfuric acid
2. 350 milliliters (11.8 fluid oz.) of hot tap water	6. 100 milliliters (3.4 fluid oz.) of rubbing alcohol
3. 25 grams (1.0 oz.) of pickling salt	7. 150 milliliters (5 fluid oz.) of warm tap water
4. 350 milliliters (11.8 fluid oz.) of hot tap water	

Reaction summary: Sodium chlorate can be made using an electrochemical process whereby pickling salt is electrolyzed under slightly different conditions than as in the preparation of bleach. During the process, some chlorine gas is evolved at the anode, and sodium hydroxide is liberated at the cathode. As the process proceeds, the chlorine reacts with the sodium hydroxide forming sodium hypochlorite (bleach); this bleach then spontaneously oxidizes under heat to form sodium chlorate and sodium chloride. Some chlorine gas does escape, so use proper ventilation when carrying out this operation.



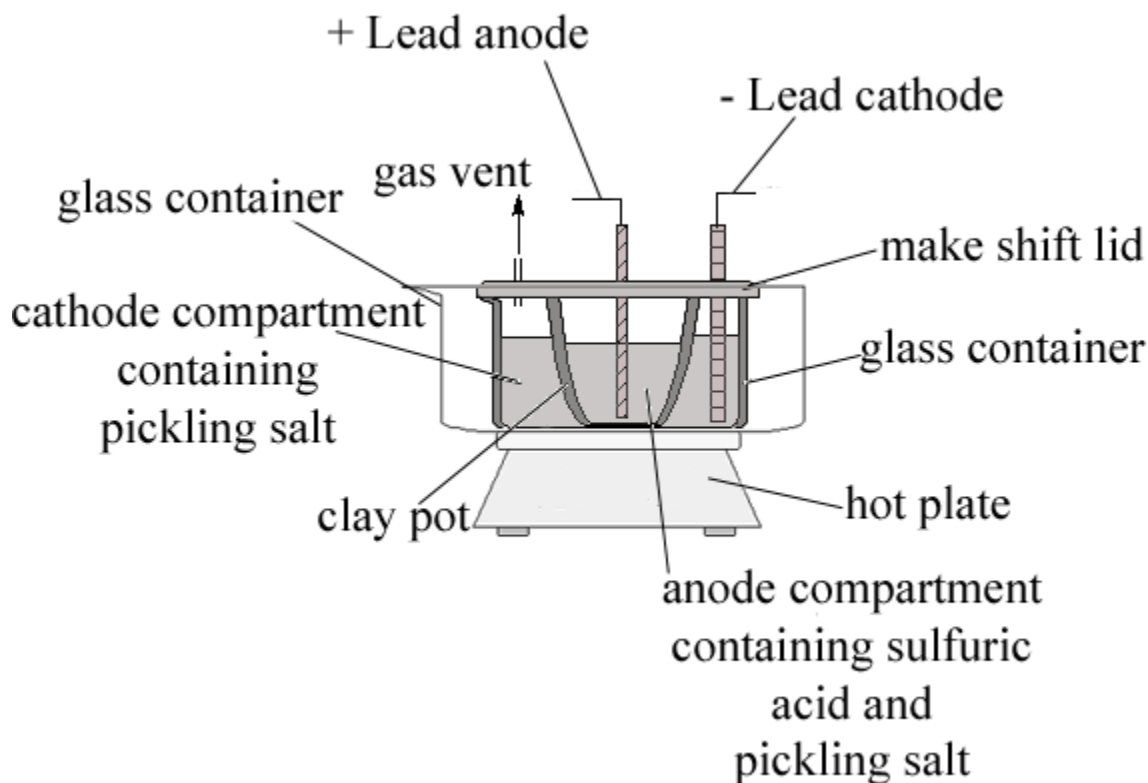
Why does this reaction happen? As in previous reactions, when current is applied to the system, the sodium ions migrate to the cathode forming sodium hydroxide and hydrogen gas. At the same time, the chlorine ions migrate to the anode forming chlorine gas. Now, when using a standard salt bridge cell, there is a secondary reaction that takes place. The sodium hydroxide that forms in the cathode compartment is attracted to the free chlorine that forms in the anode compartment. This attraction causes the free chlorine to react with the sodium hydroxide forming sodium hypochlorite. Because there is a barrier, the sodium hypochlorite remains dissolved in the anode compartment. Now, in this procedure there is a third reaction that takes place. Because the cell temperature is around 70 to 80 Celsius, the sodium hypochlorite self oxidizes forming sodium chlorate and sodium chloride. The sodium chloride that forms is merely recycled back into the system. As before, because there is a barrier, the sodium chlorate remains dissolved in the anode liquid. Note: this reaction would be impossible using an open cell, because the sodium chlorate that forms would be simultaneously reduced to salt by the cathode.

Hazards: Perform this operation in a well-ventilated area as chlorine gas is evolved. However, if you have an airtight system, the operation can be performed within enclosed areas as long as you neutralize the chlorine gas by bubbling it into a baking soda slurry or lye solution.

Procedure:

Step 1: Carryout the electrolysis process.

Assemble the cell as illustrated below, using a clean brand-new clay pot. Note: unlike previous procedures, this procedure requires a clean brand new clay pot, rather than a freshly charged clay pot as seen earlier. Then dissolve 100 grams (3.5 oz.) of pickling salt into 350 milliliters (11.8 fluid oz.) of hot tap water. Note: the hot tap water should be at least 50 Celsius. Thereafter, place this salt solution into the cathode compartment (-). Thereafter, dissolve 25 grams (1.0 oz.) of pickling salt into 350 milliliters (11.8 fluid oz.) of hot tap water of at least 50 Celsius, and then add in 5 drops of concentrated sulfuric acid. Then place this acidified salt solution into the anode compartment (+). Thereafter, begin the operation by turning on your power supply, or by plugging in your battery charger, and allow the cell to electrolysis for about 15 hours at 6 Volt/6 Amp or equivalent—be sure to monitor the temperature so as it does not fall below 50 Celsius. The optimal temperature for maximizing chlorate formation in the cell is around 80 to 90 Celsius.



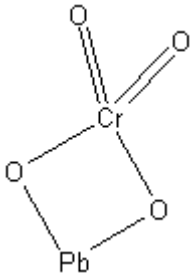

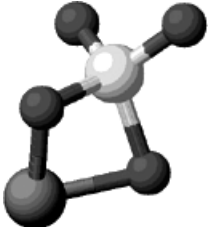
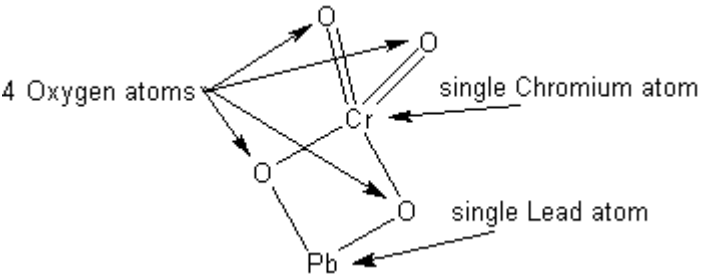
Set-up for the preparation of sodium chlorate from pickling salt. The lead electrodes can be replaced with titanium, chromium, or graphite, but should be of lead due its anti-corrosion ability. A hot plate should be used to maintain an internal cell temperature of at least 70 to 80 Celsius. Note: do not use plastic containers, as they will melt. You don't have to use a plastic lid if desired, and water can be replaced due to evaporation upon demand.

Step 2: recrystallization of the desired product.

After the 15-hour electrolysis process, unplug your power supply, and then open the cell. Carefully remove the clay pot, and then dump its contents into a clean beaker or similar glass container—the anode compartment will contain the bulk of the sodium chlorate, as well as some bleach, and small amounts of sodium perchlorate and sodium hydroxide. The cathode compartment will contain sodium chloride, and some sodium hydroxide, bleach, and sodium chlorate. You can discard the cathode liquid if desired, or you can recycle it for another crop of chlorate—to do this, simply fortify it with about 50 to 60 additional grams of salt, and then place it back into the cathode compartment. Your sodium chlorate solution (anode liquid) should be quickly filtered, to remove any insoluble materials, and then gently heated to a boil for about 10 minutes to drive-off any dissolved gasses, and to break down any bleach. Thereafter, for best results, pour the entire anode liquid onto a large shallow pan, and then allow to air dry. Blowing air over the surface of the pan using a cooling fan can help speed-up the process. Once all the water has evaporated, there will be left behind crystals of sodium chlorate, sodium hydroxide, and small amounts of sodium chloride, and sodium perchlorate. Now, scrape-up all the crystals, and then place them into a beaker, and then add to this beaker, 250 milliliters (8.5 fluid oz.) of a rubbing alcohol solution prepared by adding and mixing 100 milliliters (3.4 fluid oz.) of rubbing alcohol into 150 milliliters (5 fluid oz.) of warm tap water. Then gently swirl the beaker or similar container for several minutes, and then filter-off the insoluble crystals of the sodium chlorate. Finally, recrystallize these filtered-off crystals from 150 milliliters of hot tap water (see laboratory techniques guide on how to carryout a recrystallization). The final product after recrystallization will be about 95 to 96% sodium chlorate, well suitable for use in making fireworks, or pyrotechnic compositions.

Procedure 65: Electro preparation 19: The Preparation of Lead-VI-Chromate

Also known as: Chromic acid lead-II-salt

	 <p>3D structure 1</p>  <p>3D structure 2</p>
Chemical structure	3D Structure
	<p>PbCrO₄</p>
Structure make-up	Condensed chemical structure

Lead-VI-Chromate

Lead-VI-chromate forms yellow to orange-yellow crystals, with a melting point of 844 Celsius. The chromate is highly insoluble in water, and all solvents; however, it is soluble in sodium hydroxide or potassium hydroxide solutions, and in dilute nitric acid. Lead chromate is widely used in pigments, and in paint compositions.

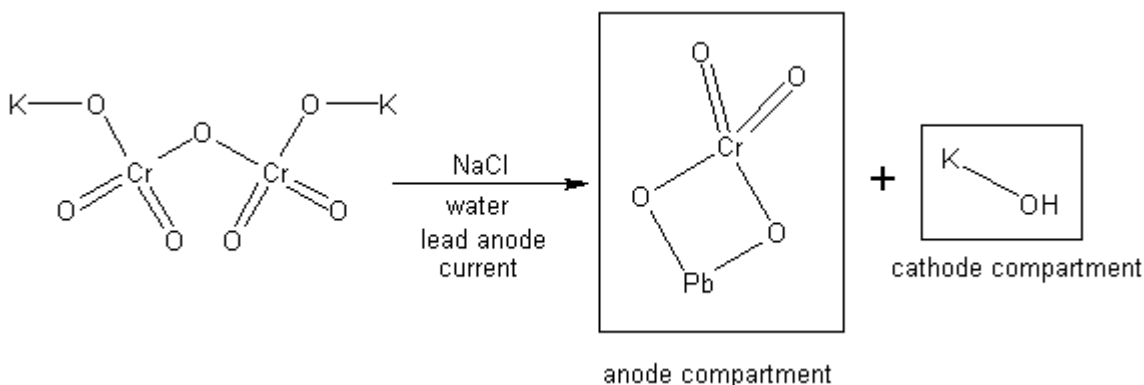
Method 1: Preparation of Lead-VI-Chromate from Potassium dichromate

(By-products from reaction: Potassium hydroxide and hydrogen gas)

Materials:

1. 50 grams (1.8 oz.) of pickling salt	4. 350 milliliters (11.8 fluid oz.) of cold tap water
2. 350 milliliters (11.8 fluid oz.) of cold tap water	5. 75 grams (2.6 grams) of potassium dichromate
3. 15 grams (1/2 oz.) of potassium dichromate	

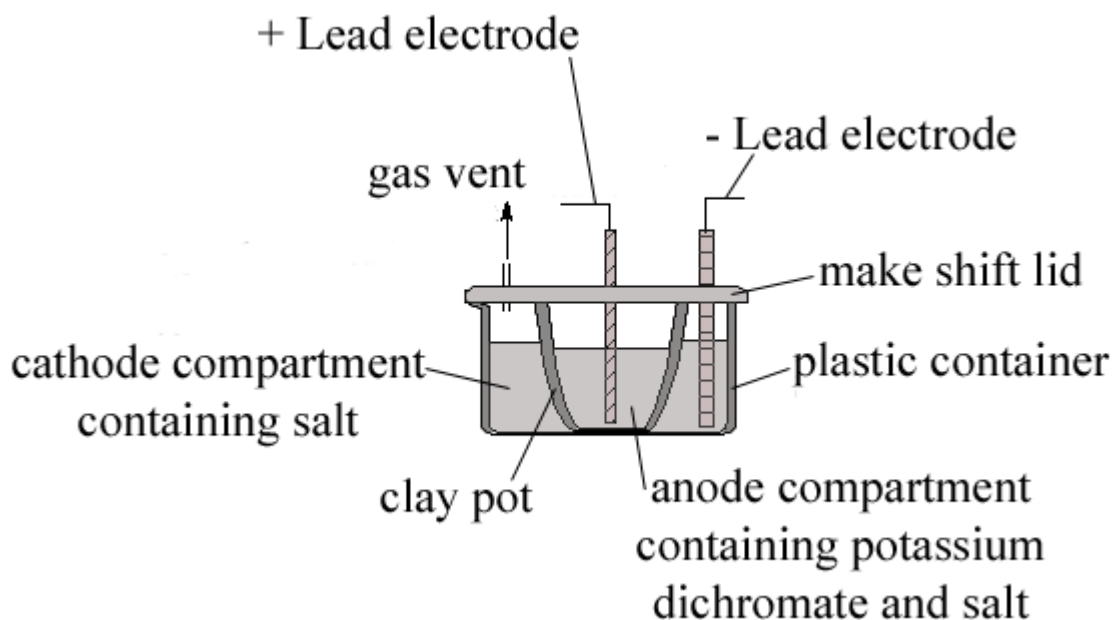
Reaction summary: Lead-VI-chromate can be made by electrolyzing a solution of potassium dichromate and sodium chloride using a lead anode. During the process, lead-VI-chromate is formed on the lead anode, from where it flakes off. Potassium hydroxide is liberated at the cathode, along with hydrogen gas.



Why does this reaction happen? In this procedure when current is applied, the potassium ions of the potassium dichromate migrate to the cathode forming potassium hydroxide, and liberating hydrogen gas. At the same time, the dichromate ions migrate to the anode forming dichromic acid, which then reacts with the lead anode forming lead chromate, which precipitates immediately due to its lack of solubility.

Hazards: Perform this operation in a well-ventilated area as some chlorine gas may be evolved.

Procedure: Assemble the cell as illustrated below, using a clean, freshly charged clay pot. Thereafter, add and dissolve 50 grams (1.8 oz.) of pickling salt into 350 milliliters (11.8 fluid oz.) of cold tap water, followed by 15 grams (1/2 oz.) of potassium dichromate. Thereafter, place this salt solution into the cathode compartment (-). Thereafter, dissolve 15 grams (1/2 oz.) of pickling salt into 350 milliliters (11.8 fluid oz.) of cold tap water, followed by 75 grams (2.6 grams) of potassium dichromate. Then place this salt solution into the anode compartment (+). Now, assemble the cell as illustrated below, using two lead electrodes, and then begin the operation by turning on your power supply, or by plugging in your battery charger. Allow the cell to electrolysis for about 15 hours at 6 Volt/6 Amp or equivalent—be sure to monitor the temperature so as it does not rise above 50 Celsius.



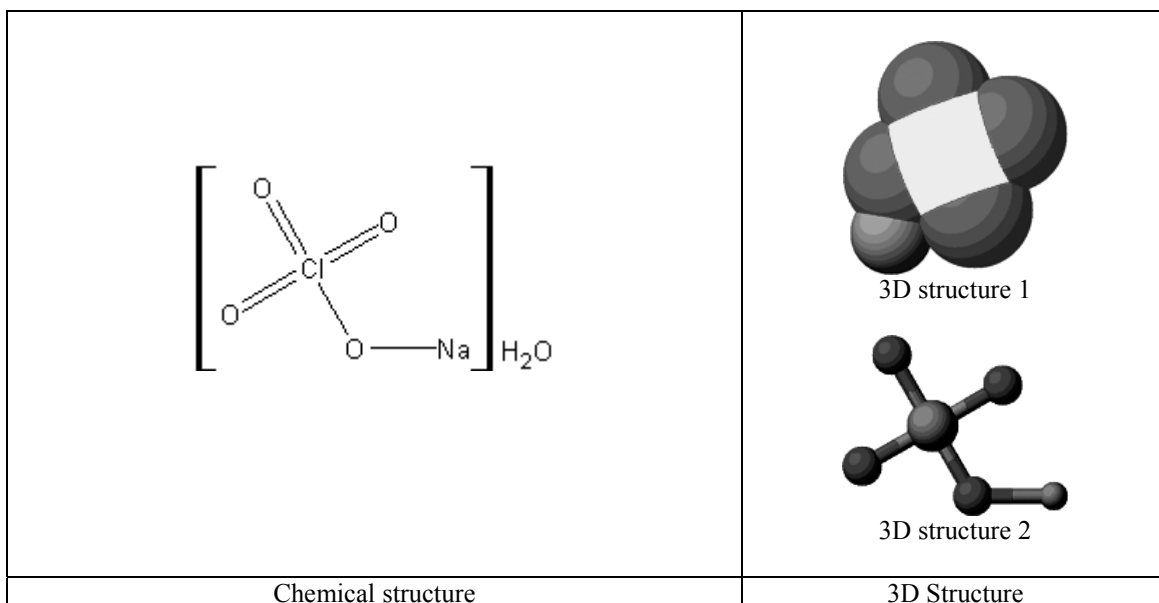
Set-up for the preparation of lead-VI-chromate from potassium dichromate. The positive lead electrode will slowly be corroded in this operation. The Lead-VI-Chromate will form a flaky precipitate that will collect in the anode compartment. The negative lead electrode can be replaced with titanium, chromium, or graphite.

After the 15-hour electrolysis process, unplug your power supply, and then open the cell. Carefully remove the clay pot, and then dump its contents into a clean beaker or similar glass container—the anode compartment will contain the insoluble lead-VI-chromate, and some dissolved sodium hypochlorite, and some sodium chloride and potassium hydroxide. The cathode compartment will contain potassium hydroxide, sodium chloride, sodium hydroxide, and some bleach. You can discard the cathode liquid if desired. The lead-VI-chromate can be easily collected by filtering the anode liquid. The collected lead salt should then be air-dried, and then stored in a suitable container.

Note: Many other metals can be used to replace the lead anode electrode. Such metals include, aluminum, zinc, iron, nickel, copper, and various other metals forming the corresponding metal chromates.

Procedure 66: Electro preparation 20: The Preparation of Sodium perchlorate monohydrate

Also known as: Irenat



	NaClO_4
Structure make-up	Condensed chemical structure

Sodium perchlorate

Sodium perchlorate forms whitish to colorless deliquescent monohydrated crystals with a decomposition temperature of 130 Celsius. The crystals are very soluble in water, but have limited solubility in the usual solvents.

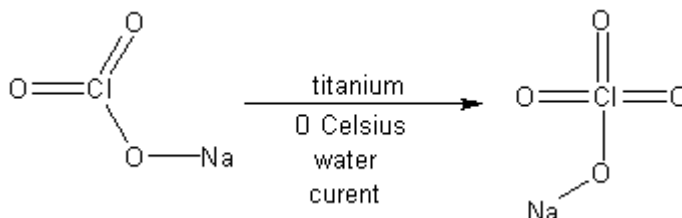
Method 1: Preparation of Sodium perchlorate monohydrate from sodium chlorate

(By-products from reaction: Hydrogen and oxygen gases)

Materials:

1. 300 milliliters (10.1 fluid oz.) of ice-cold tap water	3. 500 milliliters (17 fluid oz.) of ice-cold tap water
2. 50 grams (1.8 oz.) of sodium chlorate	4. 50 grams (1.8 oz.) of sodium chlorate

Reaction summary: Sodium perchlorate monohydrate can be prepared by electrolyzing a solution of sodium chlorate using a typical diaphragm salt bridge cell utilizing titanium anodes at a temperature of about 0 Celsius. The exact dimensions of the electrodes, the specific distance between the electrodes and the current density can vary widely and plays a role in the yield of sodium perchlorate. Many cell designs and operations exist, but many are sketchy and leave various details subdued; however, the overall operation consists of electrolyzing a solution of sodium chlorate using inert electrodes in a separated cell (diaphragm cell) at ice-cold temperatures.

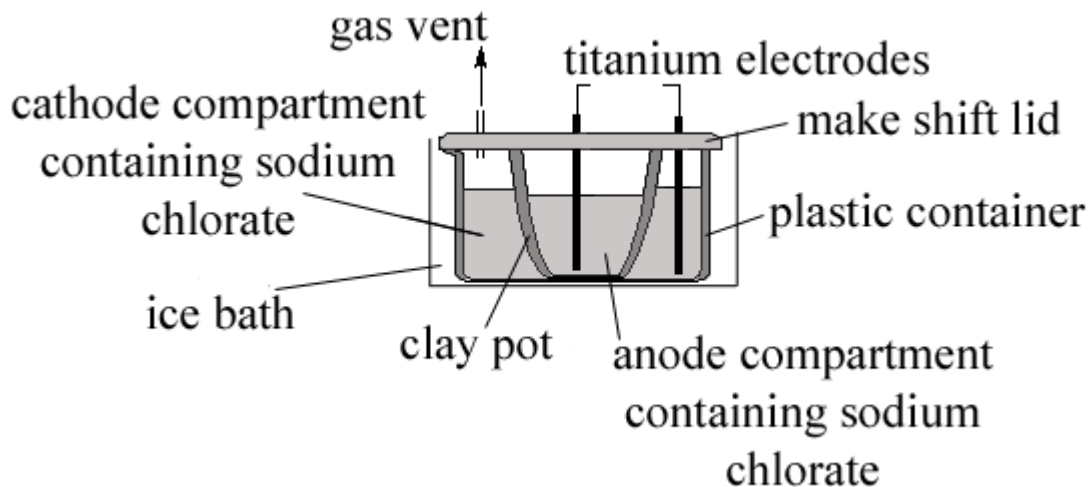


Why does this reaction happen? In this procedure, when current is applied, the sodium ions migrate to the cathode forming sodium hydroxide. At the same time, the chlorate ions migrate to the anode forming chloric acid, which gets oxidized to perchloric acid. Even though there is a barrier, the sodium hydroxide has a strong attraction to the perchloric acid, so both compounds react forming sodium perchlorate. Because there is a barrier, the sodium perchlorate remains dissolved in solution in the anode compartment.

Hazards: Perform this operation in a well-ventilated area as some chlorine gas may be evolved, and avoid contact with flames as hydrogen gas is evolved.

Procedure: Into a freshly charged clay pot, place 300 milliliters (10.1 fluid oz.) of ice-cold tap water into the anode compartment (clay pot), and then add and dissolve 50 grams (1.8 oz.) of sodium chlorate into this water. Thereafter, place 500 milliliters (17 fluid oz.) of ice-cold tap water into the cathode compartment, and then add and dissolve 50 grams (1.8 oz.) of sodium chlorate there into. Thereafter assemble the cell as illustrated below, and then place the cell into an ice bath, and then begin the electrolysis process. The desired dimensions of the titanium electrodes may vary, but it is recommended to use rectangular bars of 10 to 15 millimeters in width of surface area (0.39 inches by 0.59 inches), and the electrodes should be placed about 127 millimeters apart (5 inches). The current should be 1.5 to 2.7 volts DC current at about 50 amps. Over voltage should be minimum, and the cell temperature should be kept below 5 Celsius at all times. Electrolyze the solution for about 18 to 24 hours.

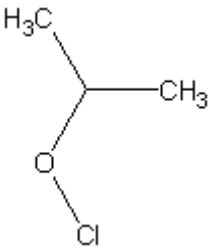
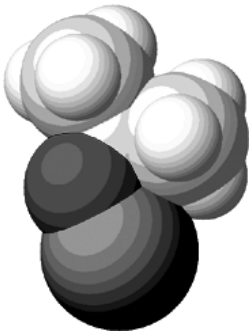
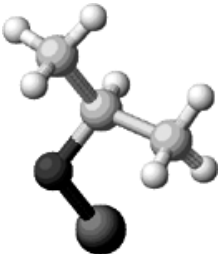
After the electrolysis process, open the cell and then pour the anode liquid into a clean beaker or similar container, and then recrystallize the sodium perchlorate monohydrate from the solution using the normal techniques. The sodium perchlorate can be recrystallized from 150 milliliters of boiling water in the usual manner if desired.

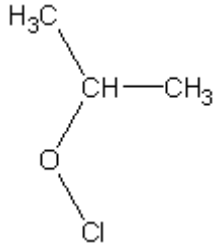


Set-up for the preparation of sodium perchlorate.

Procedure 67: Electro preparation 21: The Preparation of isopropyl hypochlorite

Also known as: N/A

 <chem>CC(C)OC(=O)Cl</chem>	 <p>3D structure 1</p>  <p>3D structure 2</p>
Chemical structure	3D Structure

 <p>3 Carbon atoms 7 Hydrogen atoms 1 Oxygen atom 1 Chlorine atom</p>	C_3H_7OCl
Structure make-up	Condensed chemical structure

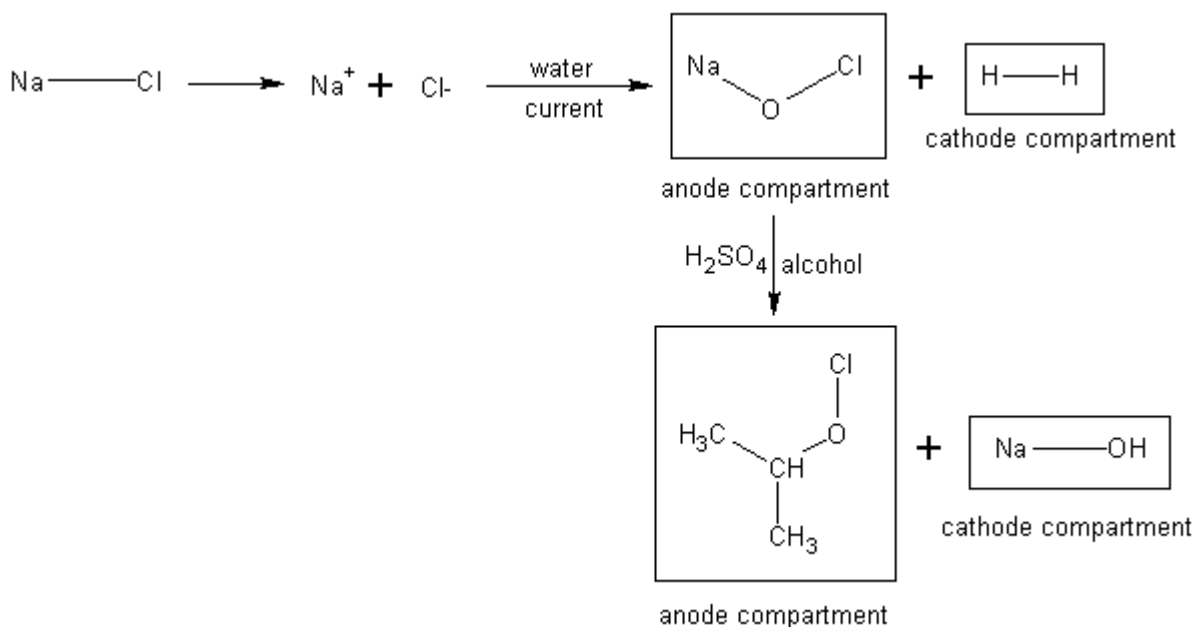
Method 1: Preparation of isopropyl hypochlorite from rubbing alcohol, pickling salt, and sulfuric acid using a diaphragm “salt bridge” cell

(By-products from reaction: Hydrogen gas, and oxygen gas)

Materials:

1. 100 milliliters (3.4 fluid oz.) of rubbing alcohol containing 70% isopropyl alcohol	6. 500 to 750 milliliters (17 to 25.4 fluid oz.) of water
2. 50 grams (1.8 oz.) of pickling salt	7. 5 drops of concentrated sulfuric acid
3. 10 grams (0.35 oz.) of anhydrous sodium sulfate, or magnesium sulfate	8. Three 50-milliliter portions (three 1.7 fluid oz. portions) of methylene chloride
4. 250 milliliters (8.5 fluid oz.) of cold water	9. 10 grams (0.35 oz.) of anhydrous sodium or magnesium sulfate
5. 50 grams (1.8 oz.) of pickling salt	

Reaction summary: Isopropyl hypochlorite is readily prepared by electrolyzing a solution of isopropyl alcohol and pickling salt in the presence of sulfuric acid in a diaphragm salt bridge cell. The electrolysis takes considerable amount time, but produces no significant heat build-up. After the electrolysis process, the anode liquid is filtered, and then extracted with a suitable, low boiling solvent. Afterwards, the solvent is removed via evaporation, and then the desired isopropyl hypochlorite is recovered.



Why does this reaction happen? As in previous reactions, when current is applied to the system, the sodium ions migrate to the cathode forming sodium hydroxide and hydrogen gas. At the same time, the chlorine ions migrate to the anode forming chlorine gas. Now, when using a standard salt bridge cell, there is a secondary reaction that takes place. The sodium hydroxide

that forms in the cathode compartment is attracted to the free chlorine that forms in the anode compartment. This attraction causes the free chlorine to react with the sodium hydroxide forming sodium hypochlorite. Because there is a barrier, the sodium hypochlorite remains dissolved in the anode compartment. Now, in this procedure there is a third reaction that takes place. Because isopropyl alcohol is present, the sodium hypochlorite reacts with the alcohol forming isopropyl hypochlorite. Because of the barrier, the isopropyl hypochlorite remains dissolved in solution in the anode liquid.

Hazards: Use caution when handling concentrated sulfuric acid. Although this procedure is safe, and produces little or no noxious fumes, take good caution when opening the cell after the reaction. Isopropyl hypochlorite is a highly irritating liquid. Use caution.

Procedure:

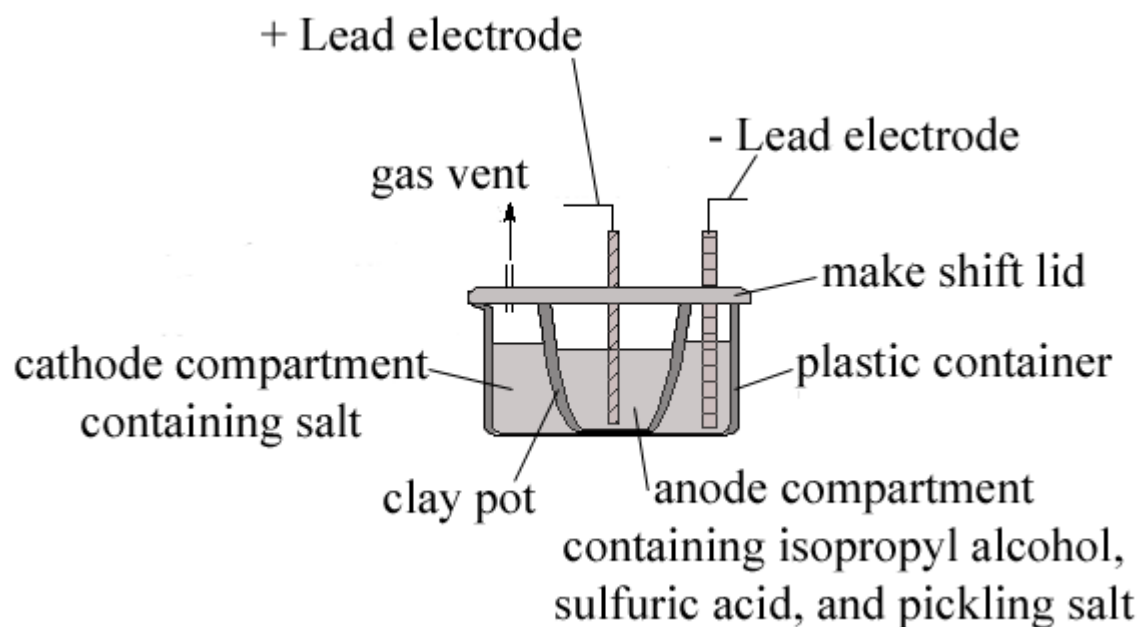
Step 1: Salting out rubbing alcohol

First, you will need to “salt out” isopropyl alcohol from rubbing alcohol. To do so, place 100 milliliters (3.4 fluid oz.) of rubbing alcohol containing 70% isopropyl alcohol (avoid colored products), into a separatory funnel, and then add in 50 grams (1.8 oz.) of pickling salt. Then vigorously shake the funnel for about 5 minutes. Thereafter, allow the funnel to stand for about 5 minutes, whereby a two phase mixture will result. Thereafter, drain-off the bottom water layer, and then carefully pour-off or drain-off the upper isopropyl alcohol layer. Finally, place this alcohol layer into a clean beaker or similar container, and then add in 10 grams (0.35 oz.) of anhydrous sodium sulfate, or magnesium sulfate, and then blend the mixture for about 10 minutes. Thereafter, filter-off the sulfate compound.

Step 2: Carrying out the electrolysis

Assemble the cell as illustrated below, using a freshly charged clay pot, place into the anode compartment, 250 milliliters (8.5 fluid oz.) of cold water, followed by 50 grams (1.8 oz.) of the dried “Salted out” isopropyl alcohol (obtained above), followed by 10 (0.35 oz.) grams of pickling salt. Then briefly stir the mixture (or before placing into the cell) to dissolve the salt. Then dissolve 40 grams (1.4 oz.) of pickling salt into 500 to 750 milliliters (17 to 25.4 fluid oz.) of water (depending on how big the outer Tupperware container is), and then place this salt solution into the cathode compartment. Thereafter, add 5 drops of concentrated sulfuric acid to the anode liquid. Thereafter, attach the lid, and then begin the electrolysis. Electrolysis at 6 to 12 volt by 2 to 6 amp direct current for about 12 to 18 hours. After the electrolysis process, carefully open the cell, and then pour the contents of the anode compartment into a clean beaker, and then immediately cover the beaker with plastic wrap. The contents of the cathode compartment can be discarded or the sodium hydroxide dissolved there in can be recovered for other use. **Note: Use caution as the anode liquid will be highly irritating to the eyes, nose, and throat. Use caution, and avoid inhalation of the vapors.**

Now, quickly filter the anode liquid to remove insoluble impurities, and then extract the filtered anode liquid, with three 50-milliliter portions (three 1.7 fluid oz. portions) of methylene chloride. After the extraction, combine all methylene chloride extract portions, if not already done so, and then dry this combined methylene chloride portion by adding to it, 10 grams (0.35 oz.) of anhydrous sodium or magnesium sulfate, and then stir the mixture for several minutes. Thereafter, filter-off the sodium or magnesium sulfate, and then place the dried filtered methylene chloride mixture into a distillation apparatus, and distill-off the methylene chloride at 40 Celsius until no more methylene chloride passes over. Thereafter, remove the heat source, and allow the remaining liquid to cool to room temperature. Thereafter, store the remaining liquid of isopropyl hypochlorite in an amber glass bottle in a cool place and away from sunlight.

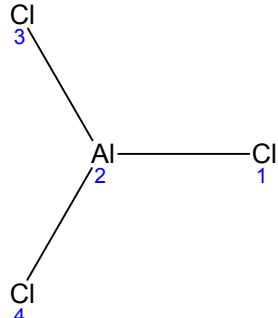


Common set-up for the preparation of isopropyl hypochlorite.

Procedure 68: Electro preparation 22: The Preparation of Aluminum chloride hexahydrate, Magnesium hydroxide, and sodium sulfate

Also known as: Dichlorohexachloride, Aluwets, Anhydrol, Driclor

	<p>3D structure 1</p> <p>3D structure 2</p>
Chemical structure	3D Structure

 <p>1 Aluminum atom 3 chlorine atoms</p>	AlCl_3
Structure make-up	Condensed chemical structure

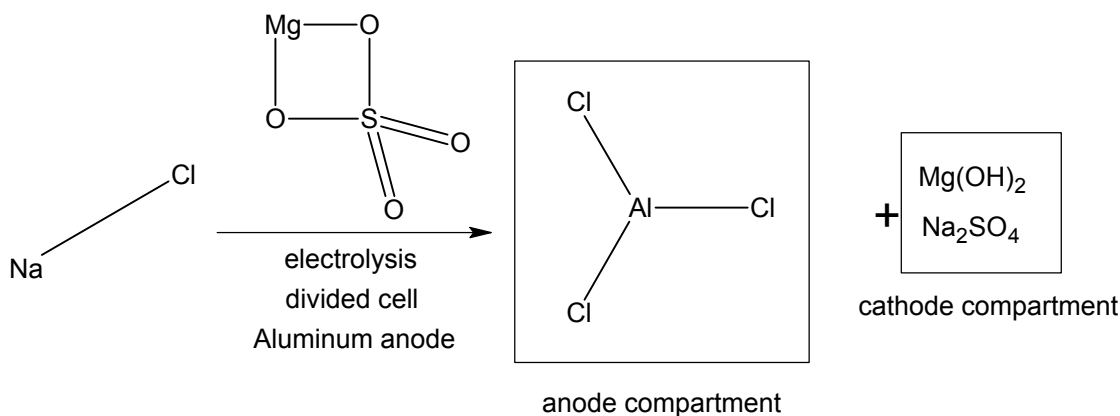
Method 1: Preparation of aluminum chloride hexahydrate, magnesium hydroxide, and sodium sulfate decahydrate from pickling salt, Epsom salt, aluminum foil, and copper tubing

(By-products from reaction: magnesium hydroxide (insoluble), and sodium sulfate (soluble in the cathode liquid))

Materials:

1. 50 grams (1.76 oz.) of sodium chloride (pickling salt)	4. 1100 milliliters (37 fluid oz.) of tap water
2. 105 grams (3.7 oz.) of Epsom salt	5. Copper electrode (1/4 inch diameter by six inches, copper tubing)
3. Three feet of aluminum foil	

Reaction summary: Aluminum chloride hexahydrate is readily prepared by electrolyzing a solution of pickling salt utilizing a divided clay pot cell and Epsom salt. The pickling salt is placed in the anode compartment (as illustrated below) and the Epsom salt is placed in the cathode compartment (as illustrated below). An aluminum anode is used as the electrode for the anode compartment, and a copper electrode is used as the cathode for the cathode compartment.



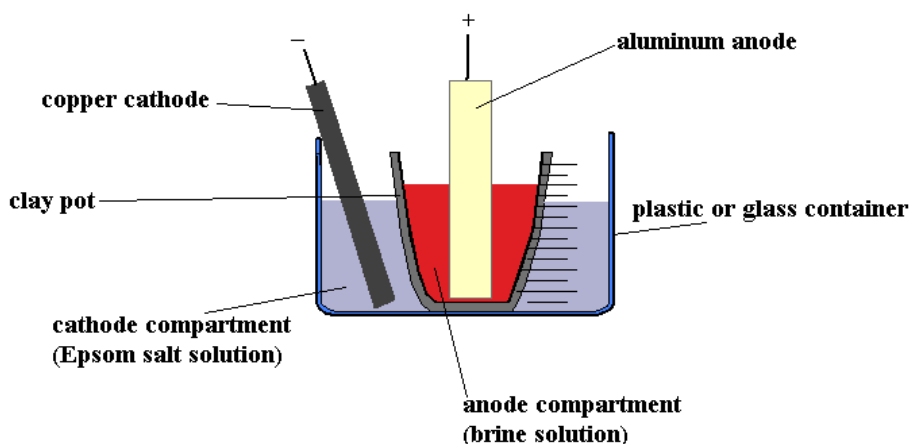
Why does this reaction happen? When a clay pot is used as a “dividing” wall, separating two different compartments, the clay pot acts as a salt bridge allowing water and certain ions to migrate through it. In this reaction, the sodium ions dissolved in the anode liquid, migrate through the walls of the clay pot, and end up in the cathode compartment, gaining an electron from the copper cathode. The sodium then bonds with the sulfate ions dissolved in the cathode liquid forming sodium sulfate, and precipitating magnesium hydroxide because the magnesium ions dissolved in the cathode liquid have a higher affinity for oxygen than sulfate. The chlorine ions dissolved in the anode compartment, give up an electron to the aluminum anode, and then spontaneously reacts with the aluminum forming aluminum chloride, which remains dissolved in the anode liquid. Note: this process is not the best method of preparing aluminum chloride hexahydrate as secondary reactions take place forming complex hydrated aluminum oxy chlorides.

Hazards: Carryout this process in a ventilated area as hydrogen gas is formed, and can accumulate in enclosed areas forming an explosive mixture.

Procedure:

Step 1: Prepare the aluminum anode by taking three feet of aluminum foil, folding it in half, folding in half, and folding in half until a 1 ½ inch width by six inches in length is obtained (use your own discretion).

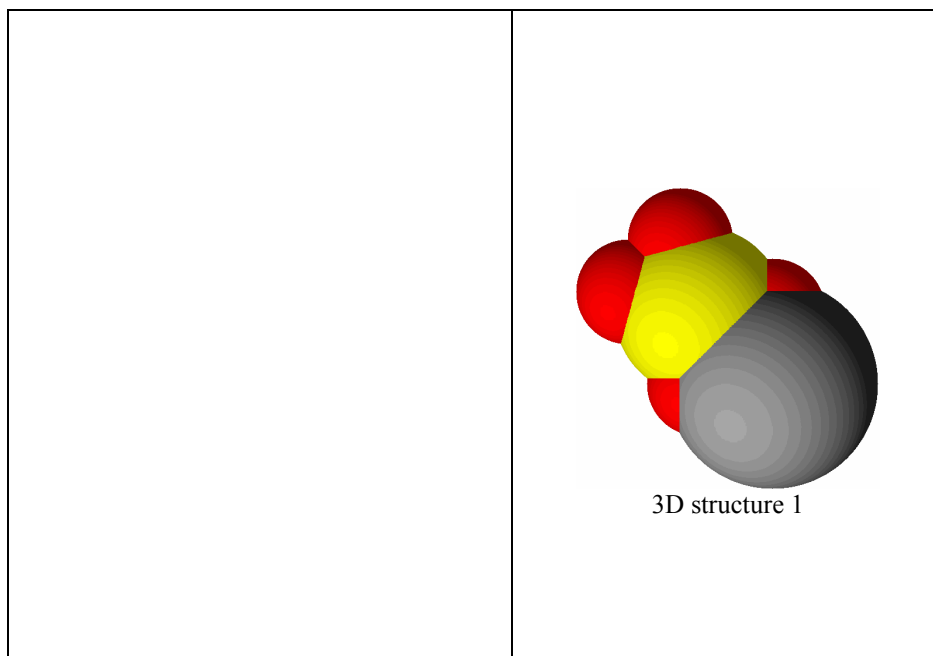
Step 2: Dissolve 50 grams of pickling salt into 400 milliliters of tap water, and then pour this solution into the anode compartment. Then, dissolve 105 grams of Epsom salt into 700 milliliters of water, and then pour this solution into the cathode compartment. Thereafter, place the aluminum electrode into the respective anode liquid, and then place the copper electrode into the cathode compartment and then attach the negative and positive clamps from the power supply (battery charger), and then electrolyze the apparatus at 12 to 50 volt, at 2 to 6 amp for 8 to 10 hours. After 8 to 10 hours, remove the power source, and then filter the anode liquid to remove any insoluble impurities. Thereafter, filter the cathode liquid to recover the insoluble magnesium hydroxide, and then place both the filtered anode liquid and filtered cathode liquids into separate crystallizing bowls, and allow them to evaporate. Once evaporated the anode liquid will give up colorless crystals of aluminum chloride hexahydrate, and the cathode liquid will give up colorless crystals of sodium sulfate decahydrate.

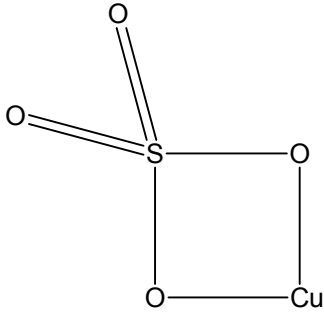
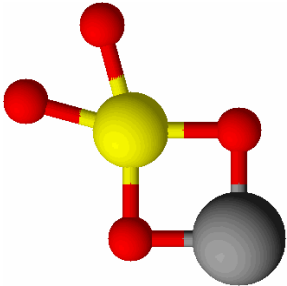
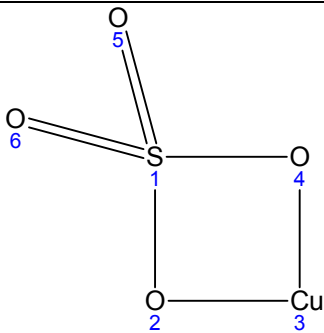


Note: The copper tubing cathode electrode can be replaced with graphite, lead, zinc, iron, or magnesium. Second note: Do not use aluminum as a cathode as it will be heavily corroded forming aluminate ion.

Procedure 69: Electro preparation 23: The Preparation of Cupric sulfate pentahydrate

Also known as: Bluestone, Blue vitriol



	 <p>3D structure 2</p>
Chemical structure	3D Structure
	$\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$
Structure make-up	Condensed chemical structure

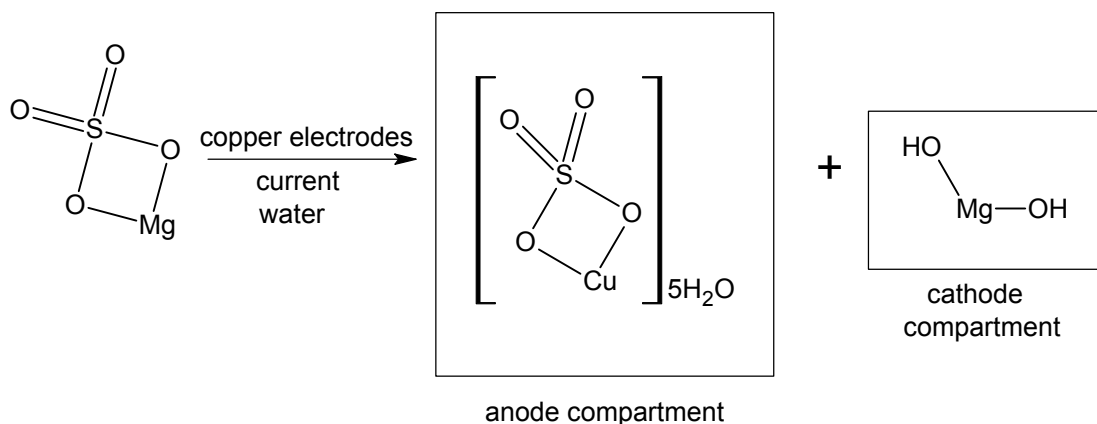
Method 1: Preparation of cupric sulfate pentahydrate from Epsom salt and copper tubing

(By-products from reaction: magnesium hydroxide)

Materials:

1. 300 grams (10.5 oz.) of Epsom salt	5. Copper electrode (1/2 inch diameter by six inches, copper tubing)
2. 1900 milliliters (64.2 fluid oz.) of tap water	

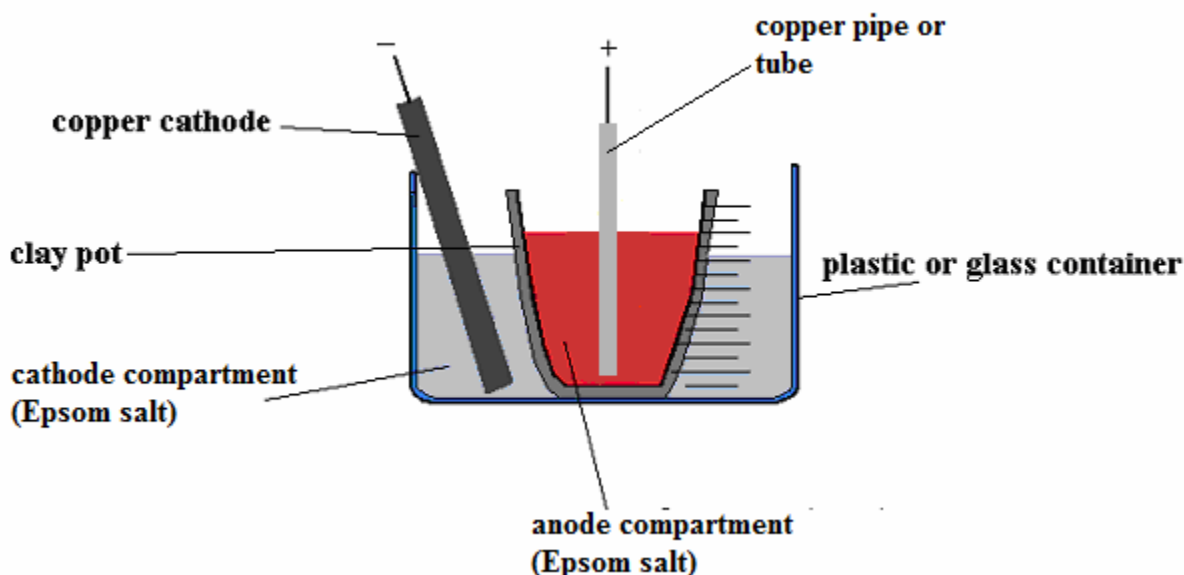
Reaction summary: Cupric sulfate pentahydrate is readily prepared by electrolyzing a solution of Epsom salt utilizing a divided clay pot cell. The Epsom salt is placed in both the anode compartment (as illustrated below), and the cathode compartment (as illustrated below). Copper electrodes are used as the cathode and anode.



Why does this reaction happen? When a clay pot is used as a “dividing” wall, separating two different compartments, the clay pot acts as a salt bridge allowing water and certain ions to migrate through it. In this reaction, dissolved magnesium ions migrate from the anode compartment to the cathode compartment forming magnesium hydroxide. The sulfate ions dissolved in the anode liquid react with the copper producing copper sulfate. During the electrolysis process, the copper anode will slowly corrode.

Hazards: Carryout this process in a ventilated area as hydrogen gas is formed, and can accumulate in enclosed areas forming an explosive mixture.

Procedure: Dissolve 250 grams of Epsom into 400 milliliters of tap water, and then pour this solution into the anode compartment. Then dissolve 50 grams of Epsom salt into 1500 milliliters of tap water, and then pour this solution into the cathode compartment. Thereafter, insert a copper pipe into the anode compartment, and a copper pipe into the cathode compartment and then attach the power clamps. Thereafter, electrolyze the apparatus at 12 volt 6 amp for about 10 hours, or until the bulk of the copper pipe anode has dissolved. After which, remove the power source, and then filter the anode liquid to remove impurities. Finally, place the filtered anode liquid into a crystallizing dish and allow it to evaporate. The result will be bluish crystals of cupric sulfate pentahydrate.



Setup for the preparation of copper-II-sulfate pentahydrate.

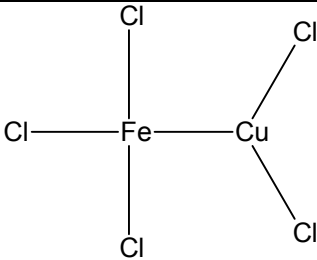
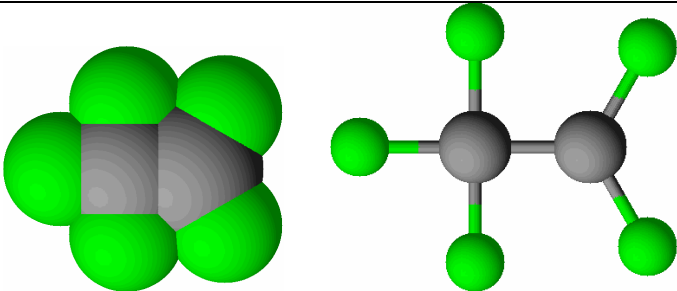
Final note: Now that you’re familiar with electrolysis, it’s time for you to go out on your own, and experiment with different anodes and cathodes of a wide variety of metals, graphite, and even special conductive polymers or ceramics and different chemicals (electrolytes). Many different electrolytes can be used such as bromides, iodides, fluorides, nitrates of various metals, permanganates of various metals, alkali hydroxides, chromates, dichromates, a variety of chlorides including chlorides of nickel, zinc, copper, iron, chromium, aluminum, calcium, and magnesium, water soluble sulfides, sulfates of a variety of metals, and water soluble organic compounds containing one or more metals such as acetates. Also, you can carryout special

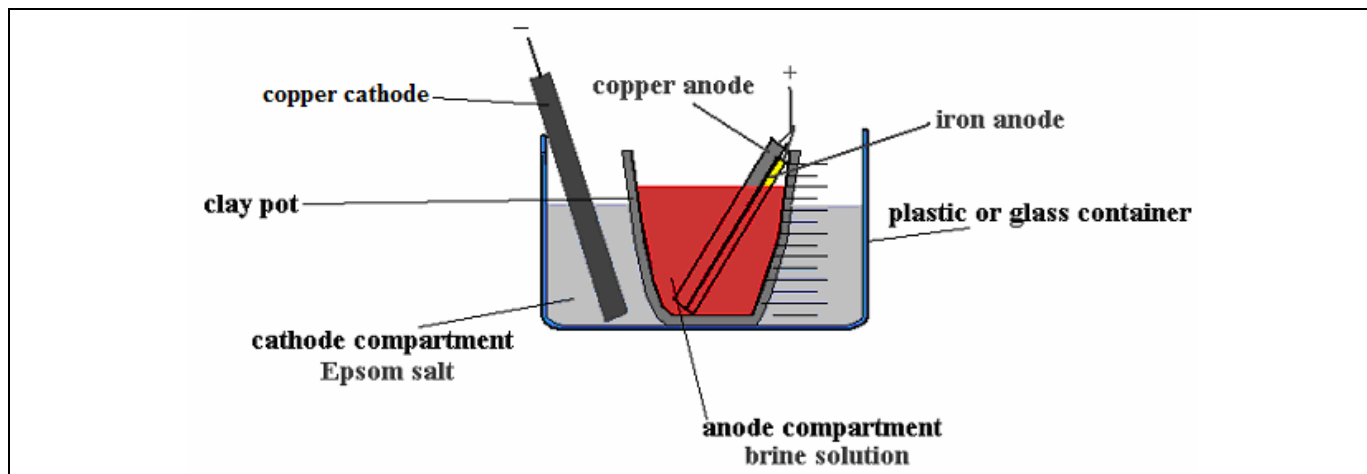
reactions using water-soluble organic compounds such as alcohols as seen in the preparation of isopropyl hypochlorite. In either case, whether using open cells or divided cells, the combinations and possibilities are virtually endless, and electrochemical reactions can produce some fascinating results and products so use your imagination and see what you can come up with. Note: in open cells, the reactions prefer reduction, and in divided cells reactions favor oxidation. Now get out there and explore the fascinating world of electrochemical chemistry!

Chapter 11: Experimental Electrochemical processes, Electro chemical methods in general chemistry Utilizing “divided Cells”

Now is the time to use the knowledge you have acquired so far and branch out into experimenting with new general techniques using divided cells. The following experiments and setups are a tad different than what you have seen in earlier chapters, but this chapter is dedicated to familiarizing yourself with how experimental divided cell electrochemical reactions are used and how the data is written down as the procedure progresses. It should be known that the following procedures are merely experimental and may not actually produce the corresponding product(s) as listed. Use this chapter to better help you understand how to carryout experimental general electrochemical processes, which can be quite exciting and maybe perhaps form new and un-known products. As in the previous chapter, we will be using a standard diaphragm salt bridge cell utilizing a clay pot. It should be noted that this chapter is designed to give you a general idea of experimentation in electrochemical chemistry utilizing divided cells, and it should be noted that many of the procedures in this chapter are basic and simply exist to give you an understanding of how experimental electrochemical reactions are conducted. Experimenting with electrochemical reactions using a divided cell is fascinating and the setups, number of electrodes, and the number of electrolytes are virtually endless, and after reading this section, feel free to experiment using your own ideas utilizing various metals for anodes and cathodes, and using many different types of electrolytes. The possibilities in these outcomes can vary greatly producing an unknown toll of products and reactions.

Procedure 70: Experimental Procedure 01: The possible formation of Cupric ferric chloride


<p style="text-align: center;">CuFeCl₅</p>

<p> Molecular Formula = Cl₅CuFe Formula Weight = 296.6581944 Composition = Cl(59.75%) Cu(21.42%) Fe(18.83%) Molar Refractivity = Not available Molar Volume = Not available Parachor = Not available Index of Refraction = Not available Surface Tension = Not available Density = Not available Dielectric Constant = Not available Polarizability = Not available Monoisotopic Mass = 293.711001 Da Nominal Mass = 294 Da Average Mass = 296.6582 Da </p>
<p>Properties: Forms brilliant blackish crystals with metallic sheen. On long exposure, the solution slowly evaporates forming cupric chloride and ferric chloride.</p>
<p>Preparation: Dissolve 50 grams of pickling salt into 400 milliliters of tap water, then pour into the anode compartment. Then dissolve 105 grams of Epsom salt into 700 milliliters of tap water, and then pour into the cathode compartment. Thereafter, electrolyze the apparatus at 12 volt 6 amp for about 10 hours. After 10 hours, remove the power source, and then filter the anode liquid to remove impurities. Finally, use a food dehydrator to remove the water from the anode liquid, by placing the filtered anode liquid into a crystallizing dish, then placing this dish into an appropriate sized food dehydrator machine until dry solid remains.</p>
<p>Set-up:</p>

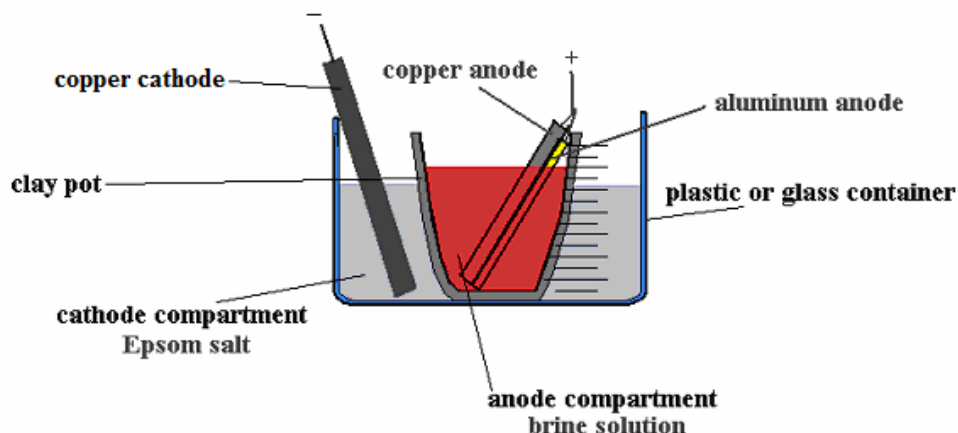


Procedure 71: Experimental Procedure 02: The possible formation of Cupric Aluminum chloride

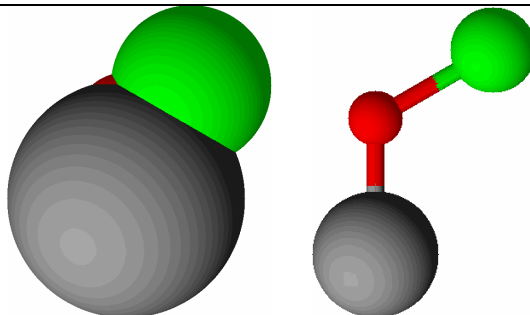
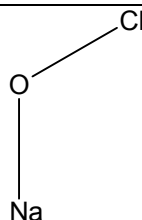
<p style="text-align: center;">AlCuCl_5</p>
<p> Molecular Formula = AlCl_5Cu Formula Weight = 267.7941844 Composition = Al(10.08%) Cl(66.19%) Cu(23.73%) Molar Refractivity = Not available Molar Volume = Not available Parachor = Not available Index of Refraction = Not available Surface Tension = Not available Density = Not available Dielectric Constant = Not available Polarizability = Not available Monoisotopic Mass = 264.757048 Da Nominal Mass = 265 Da Average Mass = 267.7942 Da </p>
<p>Properties: Forms yellowish rhombic crystals, which slowly decompose forming cupric chloride and aluminum chloride hexahydrate.</p>
<p>Preparation: Dissolve 50 grams of pickling salt into 400 milliliters of tap water, and then pour into the anode compartment. Then dissolve 105 grams of Epsom salt into 700 milliliters of tap water, and then pour into the cathode compartment. Thereafter, electrolyze the apparatus at 12 volt 6 amp for about 10 hours. After 10 hours, remove the power source, and then filter the anode liquid to remove impurities (mostly red cuprous oxide). Finally, use a food dehydrator to</p>

remove the water from the anode liquid, by placing the filtered anode liquid into a crystallizing dish, then placing this dish into an appropriate sized food dehydrator machine until dry solid remains.

Set-up:



Procedure 72: Experimental Procedure 03: The possible formation of sodium hypochlorite



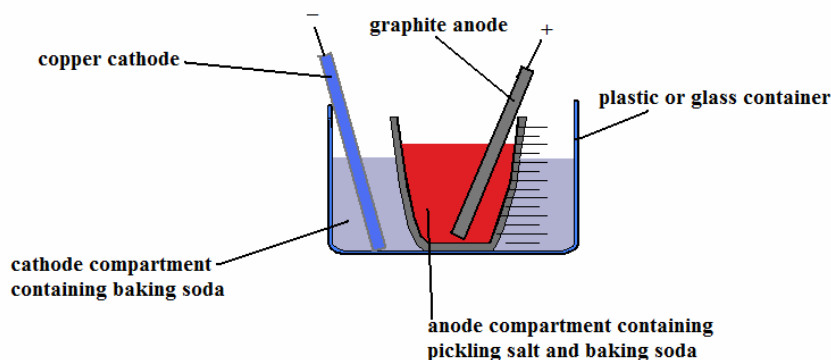
Molecular Formula = ClNaO
 Formula Weight = 74.442169
 Composition = Cl(47.62%) Na(30.88%) O(21.49%)
 Molar Refractivity = Not available
 Molar Volume = Not available
 Parachor = Not available
 Index of Refraction = Not available
 Surface Tension = Not available
 Density = Not available
 Dielectric Constant = Not available
 Polarizability = Not available
 Monoisotopic Mass = 73.953537 Da
 Nominal Mass = 74 Da
 Average Mass = 74.4422 Da

Properties: Yellowish liquid, with mild chlorine-like odor. Strong oxidizer.

Preparation: Dissolve 95 grams of pickling salt into 600 milliliters of tap water, and after the salt dissolves, add in 100 grams of baking soda, and then stir the entire mixture for about 30 minutes. Thereafter, pour the entire solution/mixture into the anode compartment. Then partially dissolve 197 grams of baking soda into 800 milliliters of tap water, and then pour into

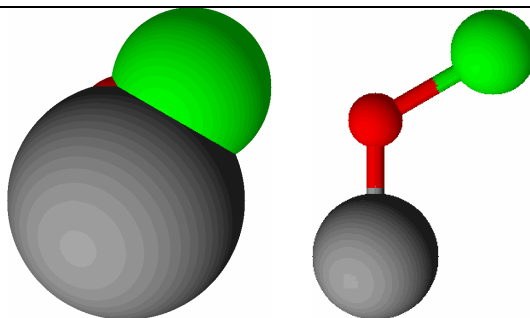
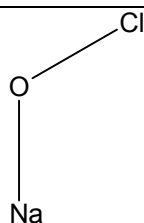
the cathode compartment. Thereafter, electrolyze the apparatus at 12 volt 6 amp for about 12 hours. After 12 hours, remove the power source, and then filter the anode liquid to remove impurities (mostly insoluble carbonates). Finally, use a food dehydrator to remove the water from the anode liquid until about only 200 milliliters of solution remains. Note: Use a solid clay pot for containing the anode liquid.

Setup:



Conclusion: Bleach formation minimal or non-existent; using a platinum coated anode may help to increase hypochlorite formation. After boiling to concentrate solution, less salt was obtained than first used; concentrated anode liquid had no reaction with Epsom salt, indicating no presence of carbonate or hydroxide ions.

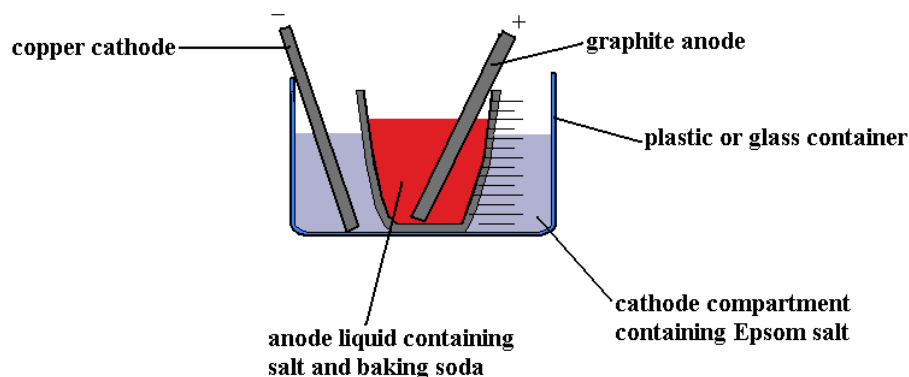
Procedure 73: Experimental Procedure 04: The possible formation of Sodium hypochlorite



Properties: Yellowish liquid, with mild chlorine-like odor. Strong oxidizer.

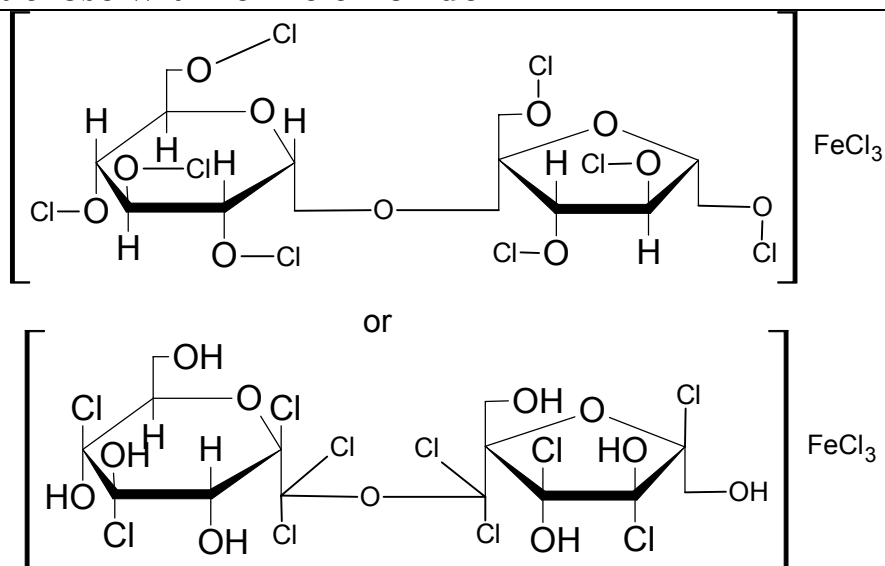
Preparation: Dissolve 95 grams of pickling salt into 600 milliliters of tap water, and after the salt dissolves, add in 100 grams of baking soda, and then stir the entire mixture for about 30 minutes. Thereafter, pour the entire solution/mixture into the anode compartment. Then dissolve 198 grams of Epsom salt into 800 milliliters of tap water, and then pour into the cathode compartment. Thereafter, electrolyze the apparatus at 12 volt 6 amp for about 12 hours. After 12 hours, remove the power source, and then filter the anode liquid to remove impurities (mostly insoluble carbonates). Finally, use a food dehydrator to remove the water from the anode liquid until about only 200 milliliters of solution remains. Note: Use a solid clay pot for containing the anode liquid.

Setup:



Conclusion: After 12 hours, the anode liquid was filtered and had no yellowish color to it. Anode liquid had no reaction with Epsom salt solution indicating no presence of carbonate or hydroxide ions in the anode liquid. Most likely, the sodium ions of the bicarbonate in the anode liquid migrate to the cathode compartment bonding with sulfate to form sodium sulfate, and precipitating magnesium hydroxide; the sodium chloride in the anode liquid remains un-disturbed until all the bicarbonate has been neutralized. Magnesium hydroxide formed in the cathode compartment, as expected. The filtered anode liquid has a very, very, slight pinkish tint to it?? Anode liquid had no reaction with potassium bromide or iodide.

Procedure 74: Experimental Procedure 05: The possible formation of chlorinated sucrose with ferric chloride

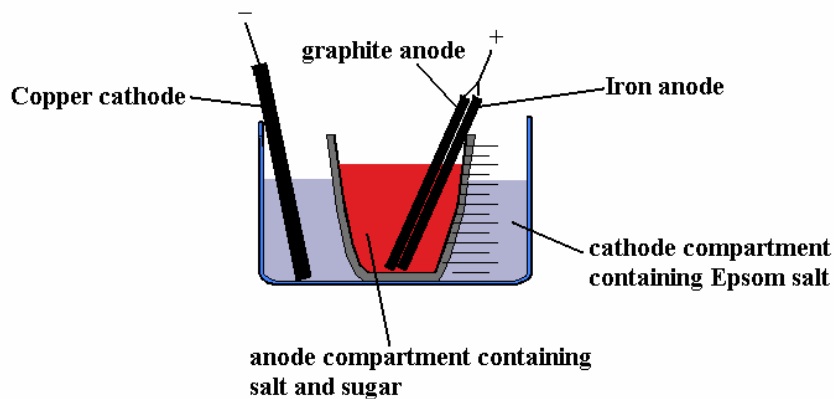


N/A

Properties: Golden brownish-black crystals.

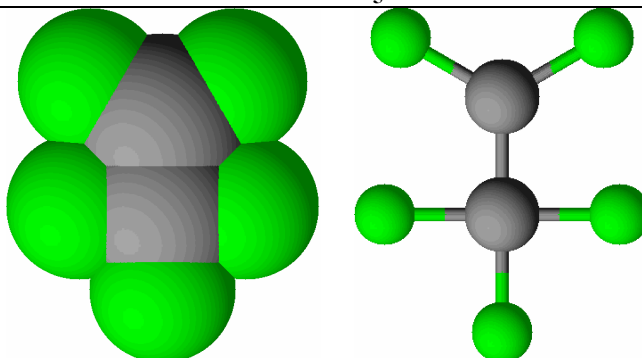
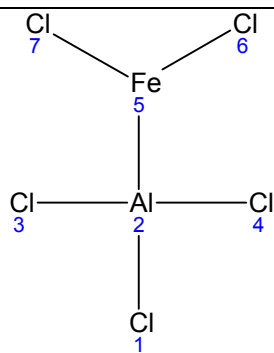
Preparation: Dissolve 100 grams of sucrose into 400 milliliters of tap water, and after the sugar has dissolved, add and dissolve 120 grams of sodium chloride. Thereafter, pour this solution into the anode compartment. Thereafter, dissolve 251 grams of Epsom salt into 700 milliliters of water, and then pour this solution into the cathode compartment. Then electrolyze the apparatus at 12 volt 6 amp for 12 hours or more, using a iron/graphite anode, and copper cathode.

Setup:



Conclusion: Note: I attempted to electrolyze sucrose using a salt/sucrose solution in the anode, and Epsom salt in the cathode, but had to stop abruptly due to chlorine evolution. Thereafter, I switched to an iron/graphite electrode about 15 minutes later, and proceeded from there. At this point, there was a mild chlorine odor at startup, but little or no chlorine could be detected after about 15 minutes. Conductivity is normal, and hydrogen gas evolution at cathode is average. After about 2 hours, a significant precipitate of magnesium hydroxide has formed in cathode compartment. At three hours, the conductivity is good, and the heat is increasing. At 6 hours, the anode liquid has become dark in appearance and has a ferric chloride-like odor; formation of ferric chloride likely; reaction with sucrose un-known. At around 8 hours, the temperature of cell decreased somewhat. More white precipitate in cathode compartment. Stopped cell at close to 12 hours; *the iron electrodes where completely dissolved*. Reaction of the chlorine and ferric chloride with the sucrose is doubtful. After filtration of the anode liquid, a brilliant yellowish-brown solution was obtained. Reaction with sucrose doubtful. Will attempt to extract the mixture with 91% isopropyl alcohol. Extraction with alcohol successful, two layers separated, with the alcohol occupying the upper layer. This clearly indicates that reaction of the sucrose with the ferric chloride and chlorine did not occur.

Procedure 75: Experimental Procedure 06: The possible formation of Aluminum ferrous chloride



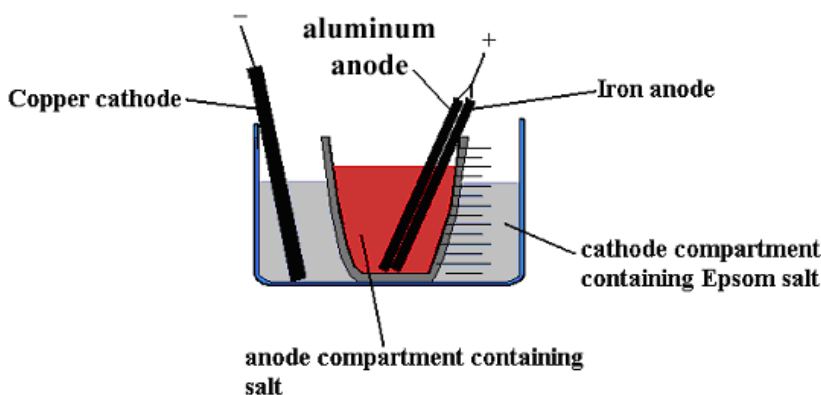
Molecular Formula = AlCl_4Fe
 Formula Weight = 224.6390872
 Composition = Al(12.01%) Cl(63.13%) Fe(24.86%)

Molar Refractivity = Not available
 Molar Volume = Not available
 Parachor = Not available
 Index of Refraction = Not available
 Surface Tension = Not available
 Density = Not available
 Dielectric Constant = Not available
 Polarizability = Not available
 Monoisotopic Mass = 222.792439 Da
 Nominal Mass = 223 Da
 Average Mass = 224.6391 Da

Properties: White to colorless crystals with a slight greenish-yellow tint. **Unknown!**

Preparation: Dissolve 110 grams of pickling salt into 400 milliliters of tap water. Thereafter, pour this solution into the anode compartment. Thereafter, dissolve 200 grams of Epsom salt into 700 milliliters of water, and then pour this solution into the cathode compartment. Then electrolyze the apparatus at 12 volt 6 amp for 12 hours or more, using a iron/aluminum anode, and copper cathode for about 12 hours.

Setup:



Conclusion: Conductivity at exact startup was average. Within 10 minutes, pieces of the aluminum foil that were used as the anode were peeling off. Had to replace aluminum anode with additional aluminum as the aluminum portion of the anode was completely dissolved around the 9th hour. The anode liquid is a grayish color with pieces of aluminum floating around. Conductivity is good, with heat. A significant amount of magnesium hydroxide has formed in the cathode compartment as expected. A little blackish precipitate has formed around the rim of the clay pot on the cathode side, and a small lump of a blackish material has formed on top of the anode liquid, but white precipitate in cathode compartment remains the same; no discolored precipitate in cathode compartment as of yet. Stopped cell at just over 11 hours. The clay pot had some blackish precipitate around the outer portion, but the cathode liquid was mostly white precipitate. Note: The clay pot may be becoming distorted and may need to be replaced? The anode liquid was a grayish liquid. Some notable corrosion of the iron anode was noted. The aluminum portion of the anode was completely dissolved for a second time. After several filtrations, a very very slight greenish-yellow solution was obtained. A sample of this solution reacted with hydrogen peroxide forming a brownish black liquid, indicating the presence of iron in the anode liquid. After this sample was allowed to stand for a little bit, a brownish precipitate settled out.

A sample of the anode liquid was shaken with 91% isopropyl alcohol in a test tube, and the resulting mixture allowed to stand; at which time, a white precipitate slowly settles out. Also, after the white precipitate settles out, a two-phase mixture results with an upper layer and a bottom layer; the white precipitate settles to the bottom of the lower layer. The upper layer is the alcohol layer. Note: This white precipitate is not crystalline, and does not appear to be sodium chloride; cannot be sodium aluminate or ferrous aluminate, which are soluble in water, but insoluble in alcohol. Formation of aluminum oxychloride possible.

After awhile, the upper alcohol layer in the test tube has a very, very slight greenish yellow tint with a cloudy layer at the up most top portion of the alcohol layer. Note: After several hours, this upper layer turned yellow. A sample of this upper alcohol layer turned a golden yellow when a little hydrogen peroxide was added. **NOTE: when 100 milliliters of alcohol was added to the bulk of the anode liquid, no precipitation or separation of layers occurred; added some more alcohol with no change; however, after about an hour, the mixture "slowly" became cloudy. Added more alcohol to bring the total volume to 600 milliliters, and stirred the entire solution for about 30 minutes. After allowing the bulk of the mixture to stand, a clear upper portion formed, and a white precipitate formed in the lower portion. After awhile, the upper clear portion turned a lemon yellow, while the white precipitate remained on the lower portion.**

A small portion of the lower aqueous phase also reacted with hydrogen peroxide forming a brownish yellow solution. Upon

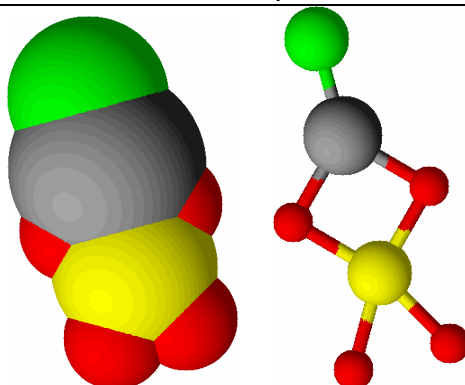
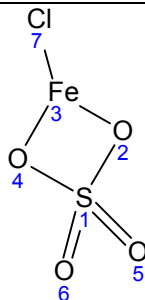
prolonged standing, a brownish precipitate forms, and slowly settles out.

1. Anhydrous ferrous chloride is a white solid. Ferrous chloride dihydrate is a white solid.

2. Formation of ferrous chloride dihydrate most likely, with formation of aluminum oxy chloride; as aluminum chloride is soluble in both water and alcohol.

3. A mixture of anhydrous ferrous chloride, ferrous chloride dihydrate, aluminum chloride, aluminum oxychloride, and sodium aluminate probably formed. However: 1. Both the upper and lower phases of the alcohol mixture in the test tube reacted with hydrogen peroxide, but only the lower aqueous phase produced a brownish precipitate that slowly settled out on prolonged standing. 2. When the alcohol was added to the anode liquid in a test tube to begin with, a white fluffy precipitate slowly separates out; aluminum oxychloride is a glassy solid, not a fluffy white precipitate. Ferrous chloride in either sense is soluble in both water and alcohol.

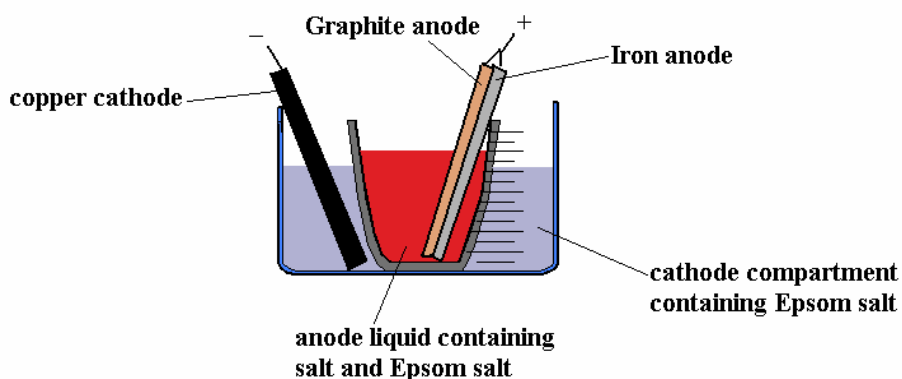
Procedure 76: Experimental Procedure 07: The possible formation of Ferric chlorosulfate



Properties: N/A

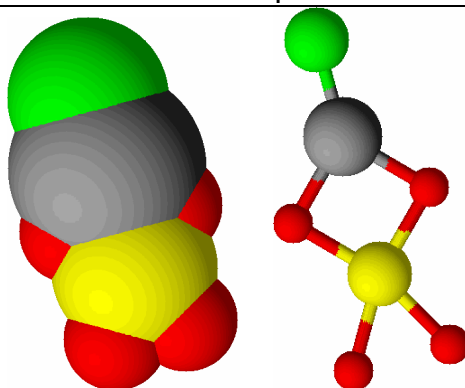
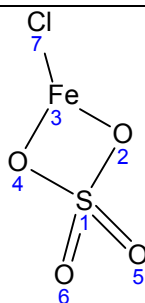
Preparation: Dissolve 50 grams of pickling salt, and 210 grams of Epsom salt into 600 milliliters of tap water. Thereafter, pour this solution into the anode compartment. Thereafter, dissolve 105 grams of Epsom salt into 700 milliliters of water, and then pour this solution into the cathode compartment. Then electrolyze the apparatus at 12 volt 6 amp for 12 hours or more, using a iron/graphite anode, and copper cathode for about 8 to 10 hours.

Setup:



Conclusion: Conductivity at startup is average; no gas evolution from graphite portion of anode. Around 1st hour, magnesium hydroxide forming in cathode compartment as expected. After several hours, a greenish black condensate on the cathode side has formed around the upper portion of the clay pot, but the white precipitate in the cathode compartment remains unchanged. A little, light brownish material appears to be floating on the surface of the anode liquid; but the majority of the anode liquid remains clear; reaction with the iron anodes is evident, but nothing appears to be emanating from the graphite anode. Stopped cell after about 12 hours. A small amount of a light bluish-gray precipitate formed in cathode compartment. The iron anodes had less corrosion then expected. After filtration of the anode liquid, a light greenish yellow solution was obtained. The cathode liquid was discarded due to iron impurities. A sample of the filtered anode liquid in a test tube reacted with hydrogen peroxide to form a brownish black liquid with a reddish tint; after awhile, a brownish precipitate eventually separates out, but does not float to the bottom, but remains suspended in the middle of the test tube. The reaction of the anode liquid with hydrogen peroxide clearly indicates no ferric ions present in anode liquid. Formation of ferrous sulfate and chloride likely, even though graphite rod was used; perhaps the graphite rod was not connected properly to the electrical clamp. The clay pot maybe coming corrupt and may need to be replaced. Added a sample of the filtered anode liquid to 91% isopropyl alcohol, shook very well for a few minutes, and then allowed to stand. On standing, a two-layer phase occurred, with the alcohol layer being the upper layer and the bottom layer the aqueous phase. *The upper alcohol layer was a little cloudy white, and the lower aqueous layer was a light yellowish color. After an hour or so, the upper layer became a light yellowish color; however, after shaking the test tube for a few moments and then allowing the test tube to stand, a two-phase mixture formed with a "colorless" upper alcohol layer, and a cloudy light yellowish lower aqueous phase; after awhile, the upper alcohol layer become a slightly cloudy yellowish color, with the lower layer being more yellowish in color.* 1. Ferrous chloride is soluble in alcohol, but ferrous sulfate is not. 2. Ferric ion should have been formed due to using a graphite anode, but it appears no ferric ion formed? *Putting the Epsom salt in the anode compartment seems to have a remarked affect on the overall reaction.*

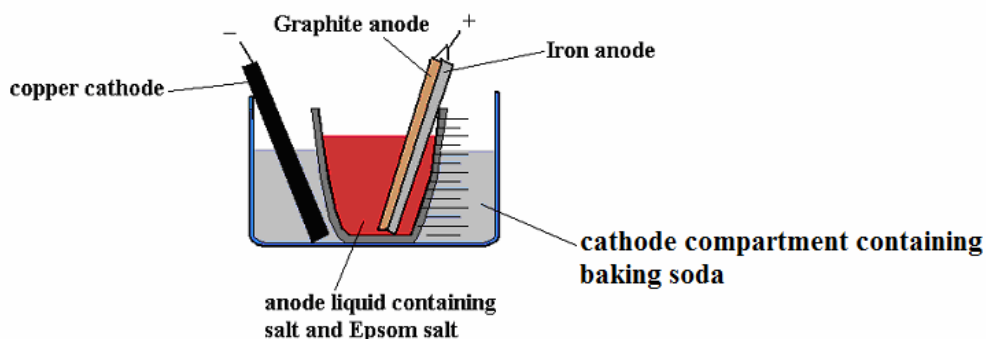
Procedure 77: Experimental Procedure 08: The possible formation of Ferric chlorosulfate



Properties: N/A

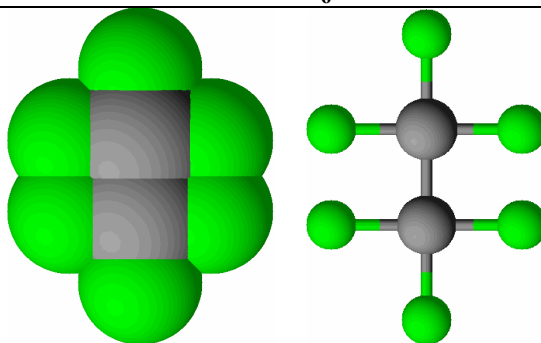
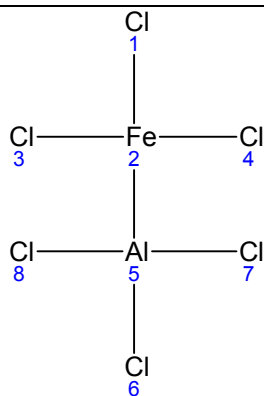
Preparation: Dissolve 44 grams of pickling salt, and 200 grams of Epsom salt into 600 milliliters of tap water. Thereafter, pour this solution into the anode compartment. Thereafter, dissolve 156 grams of Baking soda into 800 milliliters of water, and then pour this solution into the cathode compartment. Then electrolyze the apparatus at 12 volt 6 amp for 12 hours or more, using a iron/graphite anode, and copper cathode for about 8 to 10 hours.

Setup:



Conclusion: Conductivity at startup is average; no gas evolution from graphite portion of anode. No gas or refuse is emanating from the graphite and iron anodes, as in the previous process. Around the 2-hour mark, conductivity has decreased. *After 5 hours, no precipitate in cathode liquid.* Around the 6 hour mark, a little white precipitate has formed on the copper cathode, and some was floating on the surface of the cathode liquid. Stopped cell at 10 hour mark. Very little white precipitate formed in cathode compartment. *Putting the Epsom salt in the anode compartment seems to have a remarked affect on the overall reaction.*

Procedure 78: Experimental Procedure 9: The possible formation of ferric aluminum chloride



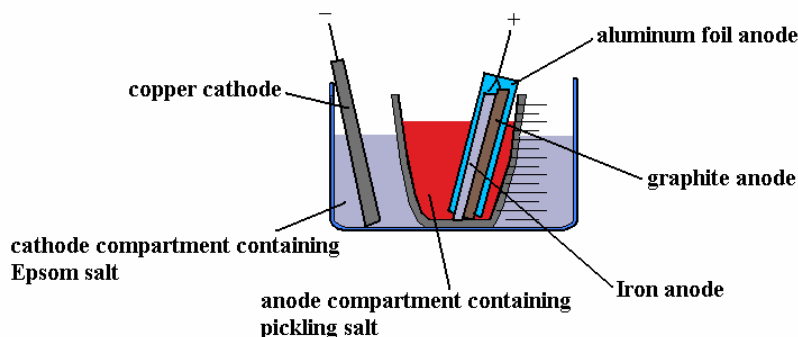
Molecular Formula = AlCl_6Fe
 Formula Weight = 295.5461844
 Composition = Al(9.13%) Cl(71.97%) Fe(18.90%)
 Molar Refractivity = Not available
 Molar Volume = Not available
 Parachor = Not available
 Index of Refraction = Not available
 Surface Tension = Not available
 Density = Not available
 Dielectric Constant = Not available
 Polarizability = Not available

Monoisotopic Mass = 292.731242 Da
Nominal Mass = 293 Da
Average Mass = 295.5462 Da

Properties: unknown

Preparation: Dissolve 110 grams of pickling salt into 400 milliliters of tap water. Thereafter, pour this solution into the anode compartment. Thereafter, dissolve 200 grams of Epsom salt into 700 milliliters of water, and then pour this solution into the cathode compartment. Then electrolyze the apparatus at 12 volt 6 amp for 12 hours or more, using a iron/aluminum/Graphite anode, and copper cathode for about 12 hours.

Setup:



Conclusion: Conductivity at exact startup is slow. As expected refuse emanating from aluminum foil anode, but no refuse from graphite. Around 3-hour mark, no white precipitate in cathode liquid. Corrosion of aluminum foil is evident. Conductivity is less then expected—clay pot might need to be replaced. Finally, at the 4-hour mark, a white precipitate is forming in cathode compartment, as expected. The anode liquid is grayish color as expected. Around 5th hour, a small amount of white gelatinous precipitate is floating on the surface of the anode liquid. Note: Made sure that the positive clamp was touching the aluminum and graphite anodes. Around 6th hour, conductivity is good, with heat. Around the 6th hour, there was some “snapping” sounds being made by the cell?? At the 8-hour mark, conductivity has increased along with the heat. The aluminum portion of the anode is giving off allot of refuse. Less white precipitate in cathode liquid then expected. Stopped cell just under 9 hours; the aluminum portion of the anode was completely dissolved.....appears to be a slight corrosion of the iron anode, but much less then expected? **Added more aluminum and proceeded with electrolysis, also added a little more tap water to anode liquid to raise its volume; conductivity very good with heat.** Finally stopped cell after a total of 11 ½ hours. Much less white precipitate formed in cathode liquid then expected. The anode liquid corroded the aluminum portion of the anode heavily, but little corrosion seems to of happened to the iron portion of the anode; reaction of the anode liquid with the graphite and iron portions of the anode seems low, and appears to be over shadowed by the aluminum portion. The unfiltered anode liquid had a slight HCl-like odor. After filtration, the anode liquid was colorless.

Preparation and Conclusion 2: 1. Pour the filtered cathode liquid back into the cathode compartment. 2. Pour the filtered anode liquid into the anode compartment. Note: the clay pot was scrubbed and cleaned prior to use. 3. Setup the anode as same as used in the first procedure, and use the same copper cathode for the cathode. 4. Electrolysize the cell at 12 volt 6 amp in the usual manner. Note: For making aluminum chloride from 110 grams of sodium chloride, approximately 16.9 grams of aluminum is needed.

Conductivity at startup is slow, but as expected. Refuse slowly emanating from graphite anode as expected. Almost immediately, within the first hour, a white scaly precipitate has formed at the bottom of the copper cathode. Conductivity has increased. Around 3 hour mark, significant precipitate in cathode compartment around the copper cathode. Note: Aluminum hydroxide may be forming in the cathode compartment. Note: The clay pot may be corrupted. Stopped cell around the 7th hour mark; the aluminum portion of the anode was completely corroded. This brings the total electrolysis time to 18+ hours. The anode liquid has a slight HCl-odor, and its volume has decreased significantly. **Significant precipitate in cathode compartment; most likely aluminum hydroxide.** Reaction of the anode liquid with the iron anodes is unlikely. **Multiple filtrations of the anode liquid, to remove carbon, resulted in a colorless liquid. A small sample was mixed with 91% isopropyl alcohol, and no phase separation occurred! However, after prolonged standing, a two-phase mixture results with a large upper layer, and a small lower layer. After a several days, a white gelatinous precipitate settled out on the bottom of the lower layer, and was difficult to remove, being mostly insoluble in water.**

Added 100 milliliters of 91% isopropyl alcohol to the bulk of the anode liquid and shaken vigorously for several moments to thoroughly mix the ingredients. At first, no phase separation occurred.

Note: Aluminum chloride and its hydrates are soluble in both alcohol and water; aluminate is insoluble in alcohol but soluble in water. **Result: Formation of aluminum chloride probable, but something else formed.**

Procedure 79: Experimental Procedure 10: The formation of ferric chloride (To test the integrity of the clay pot, and its durability after so many procedures)

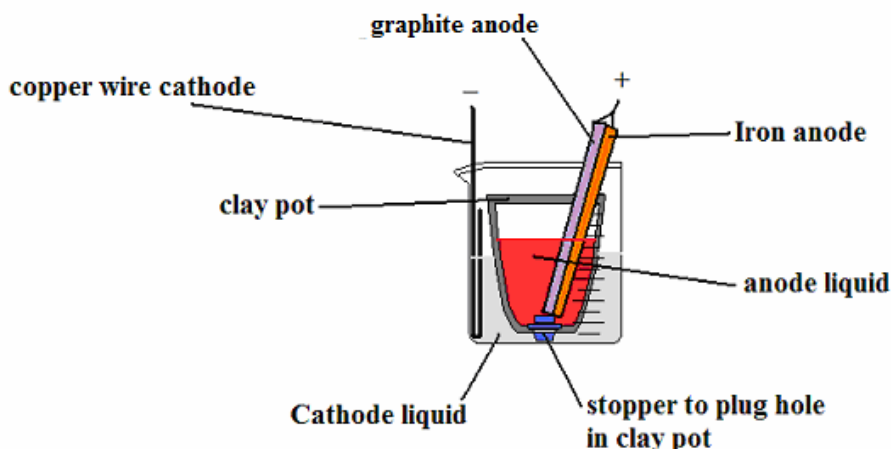
Preparation: Dissolve 110 grams of pickling salt into 400 milliliters of tap water. Thereafter, pour this solution into the anode compartment. Thereafter, dissolve 200 grams of Epsom salt into 700 milliliters of water, and then pour this solution into the cathode compartment. Then electrolyze the apparatus at 12 volt 6 amp for 12 hours or more, using a iron/Graphite anode, and copper cathode for about 12 hours.

Conclusions: Conductivity at startup was normal. Some chlorine gas was given off for a short period of time. Had to reduce power due to conductivity and heat; I used 12 volt, 2 amp rate. White precipitate forming in cathode compartment. The anode compartment has refuse floating on the surface; no chlorine odor. There appears to be a problem with the graphite anodes conducting electricity.....seemed to fix the problem around the 6th hour. I am doubtful that ferric chloride is forming! At the 6 hour mark, the white precipitate in the cathode compartment is moderate. Heavy refuse floating on the surface of the anode liquid. Stopped cell after about 10+ hours. There was a significant amount of a blackish-grayish-brown material floating on the surface of the anode liquid. White precipitate in cathode compartment as expected. A portion of one of the iron anodes was heavily corroded.....The efficiency of the clay pot seems to have decreased, but its integrity still remains useful; although should discard and use new clay port for further procedures. **Heavy blackish grayish brown solids settled to the bottom of the clay pot in the anode liquid; allot!** Looks like clay pot has reached the end of its road!!! Filtered white precipitate from the cathode liquid as usual. After several filtrations to remove brownish impurities, a light brownish liquid is obtained, leaving behind a brownish solid material on the filter paper. **This filtered anode liquid reacts with hydrogen peroxide forming a dark brownish-black liquid, with generation of heat and gas.** This clearly indicates the presence of ferrous ion, **even though a graphite anode was used along with a iron anode; conductivity of the graphite anode is questionable.** The anode liquid is probably a mixture of ferric and ferrous chlorides, with ferric oxide.

Procedure 80: Experimental Procedure 11: The formation of ferric chloride (To test the integrity of the “new” clay pot

Preparation: Dissolve 110 grams of pickling salt into 600 milliliters of tap water. Thereafter, pour this solution into the anode compartment. A brand new clay pot; used a large 1-liter capacity clay pot. Thereafter, dissolve 200 grams of Epsom salt into 1000 milliliters of water, and then pour this solution into the cathode compartment. Then electrolyze the apparatus at 12 volt 6 amp for 12 hours or more, using a iron/Graphite anode, and copper cathode for about 12 hours. Note: Use only one iron anode and one graphite anode. Use an 18 gauge copper wire for the cathode.

Set-up:



Conclusions: Conductivity at startup normal. A little chlorine odor emitting from graphite anode as expected. Note: Used a large clay pot, larger then normal. Within the first hour, a white precipitate has formed in cathode compartment. Stopped cell after about 10 hours. There was a significant white precipitate that formed in cathode compartment as expected. The Iron anode was completely dissolved. It appears the cell worked good; no leakage or seeping of the anode liquid with the cathode liquid. A little very light blue precipitate formed in cathode liquid above the white precipitate. After several filtrations, a light greenish solution is obtained; **FERROUS CHLORIDE tetrahydrate!** Note: **I made sure the positive electrical clamp was touching the graphite and iron anodes at the same time.....**Note: 1, Ferrous hydroxide is white, Ferric hydroxide is a reddish-brown, 3. Ferrous chloride is white, ferrous chloride dihydrate is white, ferrous chloride tetrahydrate is greenish; Ferric chloride is a dark blackish color with metallic luster, and ferric chloride hexahydrate is a brownish, to brownish black.

Looking back on procedure 5, perhaps if sucrose were present the ferric chloride would have formed.

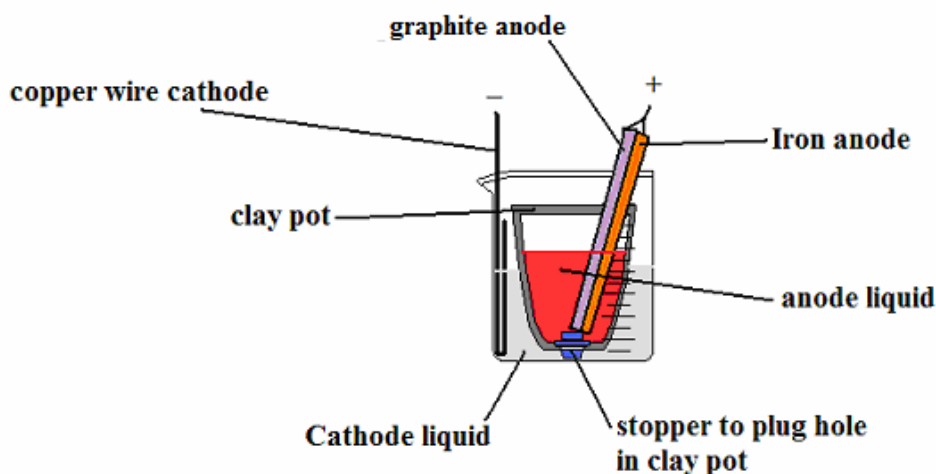
Note: I'm getting the feeling that even the composition of the cathode influences the reaction in the anode compartment. Using copper for the cathode and using a brand new clay pot forms ferrous chloride tetrahydrate.

Note: I accidentally spilled some of the anode liquid, and the spilled liquid turned brown and stained the surroundings; readily oxidized. The greenish anode liquid upon standing does not oxidize, and its color remains the same with no precipitate; unlike ferrous chloride tetrahydrate that is made when dissolving iron into dilute hydrochloric acid, which tends to oxidize and slowly deposit iron oxides!!

Procedure 81: Experimental Procedure 12: The formation of ferric chloride (to see if sucrose plays a role in the formation of ferric chloride; see procedure 5)

Preparation: Dissolve 100 grams of sucrose into 700 milliliters of tap water, and after the sucrose dissolves, add in and dissolve 110 grams of pickling salt. Thereafter, pour this solution into the anode compartment. Used the clay pot from procedure 11; large 1-liter capacity clay pot. Thereafter, dissolve 200 grams of Epsom salt into 1100 milliliters of water, and then pour this solution into the cathode compartment. Then electrolyze the apparatus at 12 volt 6 amp for 12 hours or more, using a iron/Graphite anode, and copper cathode for about 12 hours. Note: Use only one iron anode and one graphite anode. Use an 18 gauge copper wire for the cathode.

Set-up:



Conclusions: Conductivity at startup was normal. I made sure the positive clamp was touching the graphite anode, but shortly after startup, no refuse emanating from the graphite anode; the graphite anode should be conducting as the positive clamp is clearly touching it! **A greenish color is appearing in the cathode liquid almost immediately after startup. There is a dark green color in the cathode liquid that has completely discolored the entire cathode liquid. The anode liquid remains clear at this point in time.** Around the 30-minute mark, what appears to be a white precipitate has formed around the copper cathode. Note: The dark green color of the cathode liquid may just be remnants of ferrous ion trapped in the matrix of the clay pot wall? At the 30-minute mark, the anode liquid remains clear. The precipitate in the cathode liquid is probably magnesium hydroxide, but is tainted with a greenish color. Around the 3-hour mark, the dark green color of the cathode liquid has faded, and seems to slowly settle to the bottom. A white precipitate is definitely forming around the copper cathode. Around 5th hour, a definite white precipitate has formed in cathode liquid. A dark greenish material occupies the bottom of the cathode compartment. Anode liquid remains clear, but slightly tinted; ferric chloride formation un-known. **Around the 10th hour, checked anodes, and the iron anode was much less corroded then expected!!** **Note: The positioning of the anodes seems to have an affect; when the iron anode is placed in front of the graphite anode facing the copper cathode, (with the graphite anode in the rear), refuse seems to emanate from the anodes; even with the positive clamp clearly touching the graphite anode.** At the 10-hour mark, rotated the anodes so that the iron anode is in front of the graphite anode facing the direction of the copper cathode; the positive clamp is clearly touching the graphite anode. At the 10 hour mark, even with much less iron anode corrosion then expected, there is allot of white precipitate in cathode compartment, with a dark green layer occupying the bottom of the bulk of the precipitate in the cathode compartment. Note: no chlorine odor emanating from the graphite anode as would be expected at this point in time. **Within ten minutes after rotating the anodes after the 10th hour mark, the anode liquid is becoming darker!! This indicates the formation of ferric chloride.....**

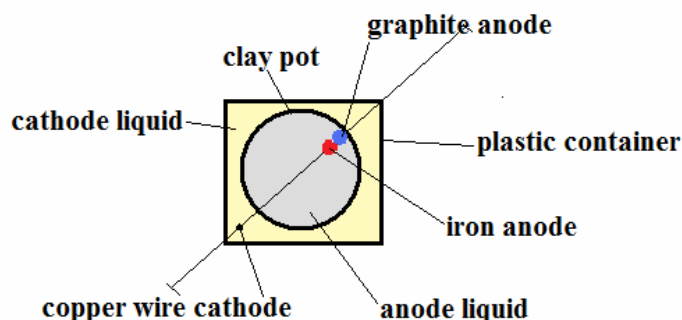


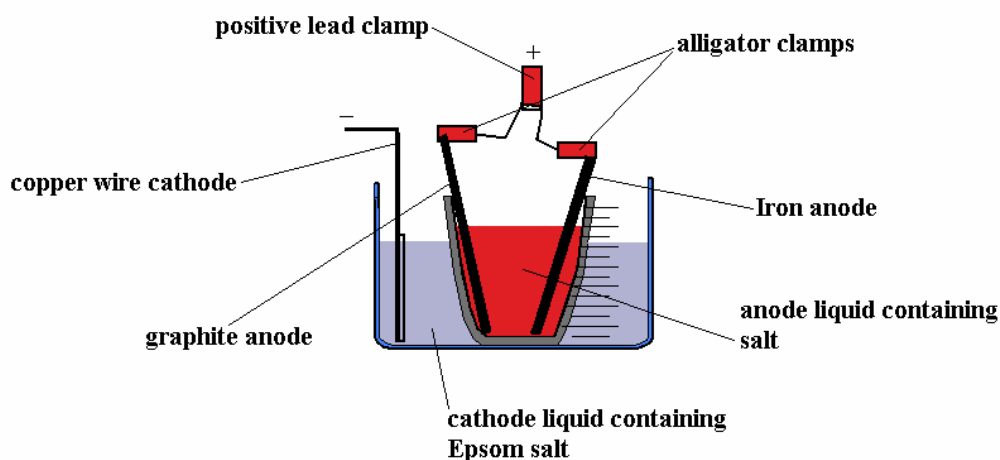
Figure. Positioning of the electrodes after the 10th hour mark

Stopped cell at around 12-hour mark. After filtration, the anode liquid was a light yellowish brown, indicating formation of “some” ferric chloride, but I am weary if all the anode liquid is ferric chloride. **Result: Probably a mixture of ferric and ferrous chlorides. Am not sure if electrode placement at the 10th hour mark had any influence on formation of the ferric chloride, or whether the sucrose played a role in the formation of ferric chloride.** A sample was mixed with 91% isopropyl alcohol and no phase separation occurred; probably because the anode liquid is too dilute for the alcohol to separate out.

Procedure 82: Experimental Procedure 13: The formation of ferric chloride minus sucrose (to determine if two different clamps attached to the graphite and iron anodes will have any affect on formation of ferric chloride)

Preparation: Dissolve 110 grams of pickling salt into 350 milliliters of water. Thereafter, pour this solution into the anode compartment. Used a brand new clay pot of about 350-milliliter capacity. Thereafter, dissolve 200 grams of Epsom salt into 700 milliliters of water, and then pour this solution into the cathode compartment. Then electrolyze the apparatus at 12 volt 6 amp for 12 hours or more, using a Graphite and iron anodes using two separate clamps for the anodes. **Note: Use only one iron anode and one graphite anode. Note: Make sure the graphite and iron anodes are perfectly lined up and not crooked.** Use an 18 gauge copper wire for the cathode.

Set-up:



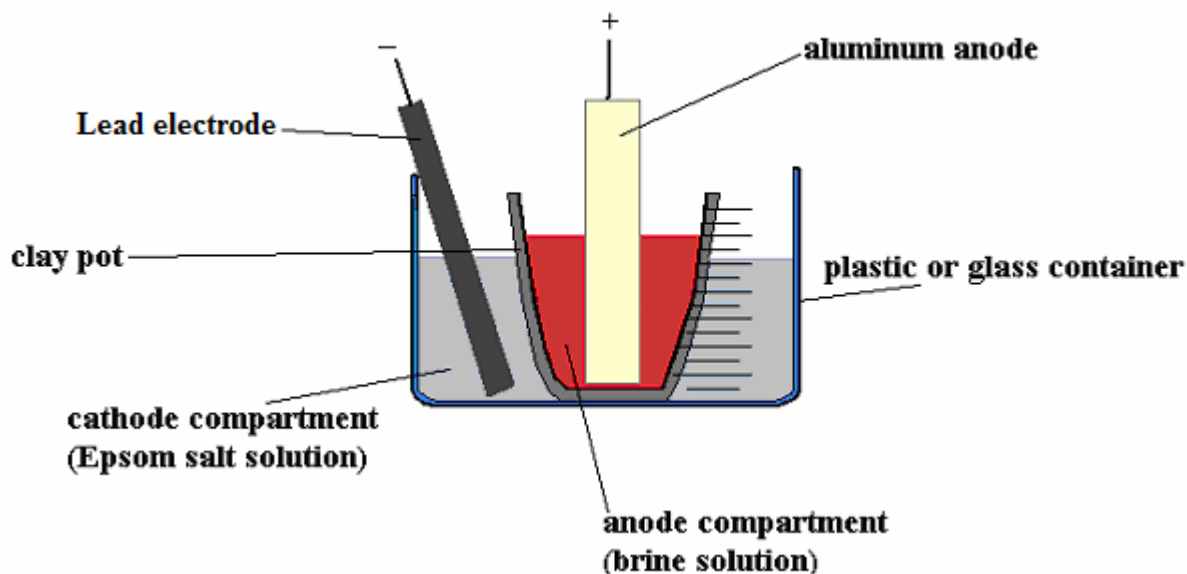
Conclusions: Conductivity at startup is average. No refuse emanating from graphite anode. At 1-hour mark conductivity has increased, so I decreased the voltage and amperage as expected. For a test, I temporarily removed the alligator clamp from the iron anode, and the graphite conducted with refuse emanating. When I re-attached the alligator clamp to the iron anode, there was conductivity to the cell, but no refuse emanated from the graphite anode..... As expected, white precipitate in the cathode compartment. Around 4th hour, a few white scales have deposited on the surface of the anode liquid. At 5th hour mark, a “skin” has appeared on top of the anode liquid, and the anode liquid looks dark. White precipitate in cathode liquid is as expected. There may be some “splashing” of the cathode liquid into the anode liquid? Stopped cell after 9 hours; the iron anode was completely corroded. The anode liquid is blackish in color and indicates formation of ferric chloride! **Note: When I took away the iron anode briefly in the beginning, may have had some affect**

on the overall outcome by acting as a promoter by the presence of a little ferric chloride; this means start cell and let it run for 15 minutes; then pull the iron anode and run the cell with just a single graphite anode for about 2 to 3 minutes, then re-connect the iron anode and proceed with the electrolysis. Around the 9th hour, a little greenish-bluish precipitate has formed into the cathode compartment. The outer wall of the clay pot has a blackish layer covering it. After filtration of the anode liquid a dark yellowish brown liquid is obtained. **Formation of ferric chloride successful!** Note: There was some crystalline material at the bottom of the clay pot. *Note: A diluted small sample of filtered anode liquid reacts vigorously with hydrogen peroxide forming a dark brownish liquid, with vigorous evolution of gas. On standing, the dark color decreases and a lightly brownish gelatinous precipitate forms.* I am not quit certain what reaction takes place, other then the decomposition of the hydrogen peroxide. Ferrous ion may exist in the anode liquid, but the anode liquids color clearly indicates the presence of ferric ion. The anode liquid is probably mostly ferric chloride with some ferrous chloride. On prolonged standing of the anode liquid, a white flocculent material separates out.

Procedure 83: Experimental Procedure 14: The formation of an un-known aluminum-containing compound, possibly a hydrated aluminum oxychloride

Preparation: Dissolve 50 grams of pickling salt into 300 milliliters of tap water, and then pour into the anode compartment. Then dissolve 105 grams of Epsom salt into 700 milliliters of tap water, and then pour into the cathode compartment. Thereafter, electrolyze the apparatus at 12 volt 6 amp for about 6 hours. After 6 hours, remove the power source, and then filter the anode liquid several times to remove impurities. Finally, use a food dehydrator to remove the water from the anode liquid, by placing the filtered anode liquid into a crystallizing dish, then placing this dish into an appropriate sized food dehydrator machine until dry solid remains. **Note: the clay pot used in this procedure was used in a previous run many months earlier.**

Set-up:



Note: Aluminum foil is used as the anode

Conclusions: Start time 6:10 PM. Conductivity at start up, normal, used cold water for both anode and cathode liquids. Corrosion of aluminum anode evident. Around 8 PM severe corrosion of the anode is evident. Heat is being generated in the cell. At 8:50 PM the cell has increased in heat. Unknown if white precipitate is forming in cathode compartment due to dirt and grim on the outer container. Significant corrosion of the aluminum anode is evident. Stopped cell at 9:50 PM to replace the corroded aluminum anode. Restarted a minute later with a fresh aluminum anode. Conductivity is good, with heat. At 11:40 PM, the cathode liquid has same consistency as the anode liquid.....the clay pot may be damaged or compromised. Stopped cell at 12:10 AM.....the cathode liquid has a grayish-black precipitate in it! Possible formation of magnesium hydroxide possible but un-known. Anode liquid is of the usual color prior to filtering, with little pieces of aluminum foil floating around.....After filtration, a colorless solution is obtained with a strange peculiar odor (kind of like aluminum and HCl). **Note: Perhaps the clay pot used, was not damaged or compromised.** Added 100 ml of 91% isopropyl alcohol at 3:30AM to the filtered anode liquid.....no separation of solids at first!

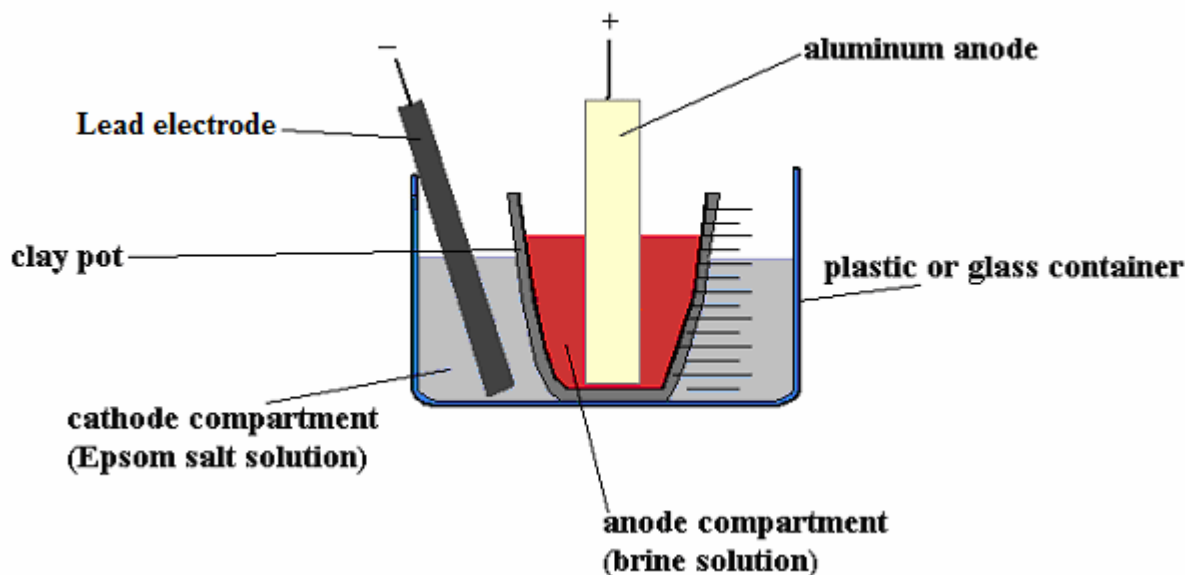
After a few hours, a grayish-black precipitate settles out of the cathode liquid (could be ferrous salts from the previous use of the clay pot?

After filtration of the cathode liquid (the pre-filtered cathode liquid has an odor similar to the anode liquid!!!), a dark grayish-black precipitate is collected, and the filtered cathode liquid is colorless. After standing for a prolonged period of time, a two-layer phase of the anode liquid took place with the alcohol forming the upper layer. A white precipitate separated out of the lower aqueous phase. Note: aluminum chloride is soluble in water and alcohol.

Procedure 84: Experimental Procedure 15: The formation of an un-known aluminum-containing compound, possibly a hydrated aluminum oxychloride using a brand new clay pot

Preparation: Dissolve 50 grams of pickling salt into 300 milliliters of tap water, and then pour into the anode compartment. Then dissolve 105 grams of Epsom salt into 700 milliliters of tap water, and then pour into the cathode compartment. Thereafter, electrolyze the apparatus at 12 volt 6 amp for about 6 to 10 hours. Note: some clay pots have holes in the bottom of them, and hence, must be plugged up airtight in order for the cell to work properly.

Set-up:



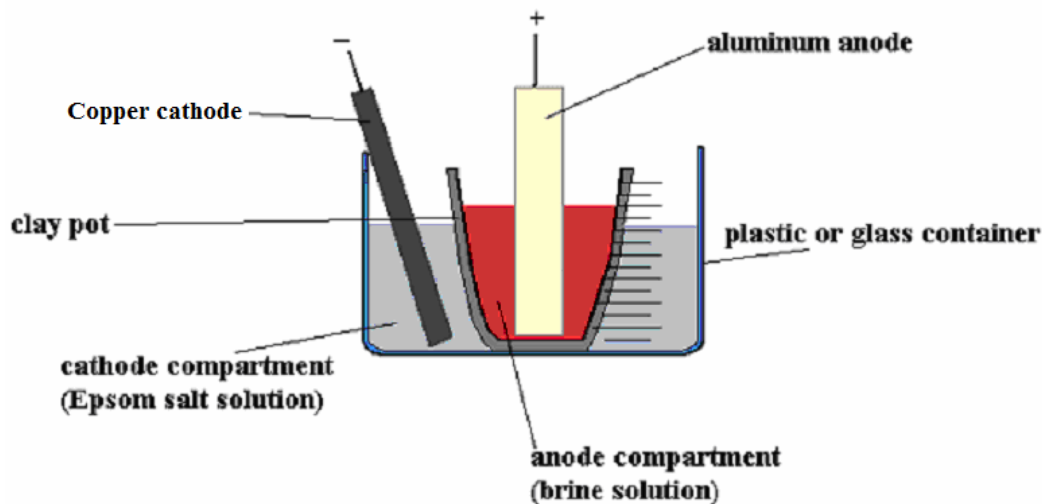
Note: Aluminum foil is used as the anode

Conclusions: Startup time, 6:35 PM, at startup, conductivity was good. Nothing out of the ordinary. About 10 minutes into the procedure, the aluminum foil anode, has begun to tear apart. At 6:47 PM, no evidence of precipitates forming in cathode compartment. There appears to be a small amount of gas emanating from the aluminum anode. At 8:24 PM, conductivity is very good, with heat increasing. No visible white precipitate forming in cathode liquid, but a grayish black solid is forming; could be magnesium hydroxide contaminated with lead? It should be noted that the lead cathode has been touching the side of the clay pot during the whole process! Stopped at 9:05 PM for just a minute to add more aluminum to the anode, then restarted, with conductivity very good. The grayish black precipitate forming in the cathode compartment appears to be magnesium hydroxide contaminated with something! It is clear that the anode compartment is sealed properly, as the water level in the anode liquid is just above that of the cathode compartment indicating that the clay pot is sealed watertight. **This procedure should be re-done using a copper cathode instead of a lead cathode.** At 11:15 PM the cathode liquid is dark in color, and has the same appearance has the anode liquid! However, the water level of the anode liquid is higher then the cathode liquid, indicating a watertight seal of the clay pot. It is obvious that a precipitate has formed in the cathode compartment, but is most likely magnesium hydroxide, contaminated with impurities, which makes it dark. Conductivity is good. At 12:15 AM Had to stop cell to replace aluminum anode for a minute. Again, at start up, conductivity has decreased a little. Cathode liquid is dark just like the anode liquid. Ended cell at 1:20 AM, cathode liquid was dark black; Filtering cathode liquid.....tones of black precipitate collecting in filter of cathode liquid. After filtration, the cathode liquid had a heavy precipitate of black unknown substance. After first filtration of anode liquid, had milky light precipitate to it, After several minutes, the cathode liquid was jet black. Anode liquid was cloudy white. BUST! The Lead cathode may have reacted with the cathode liquid? This does not explain the jet-black precipitate that was formed. Completely unusual what happened in the cell! A brand new clay pot, utilizing a lead cathode produced a jet black precipitate, which was probably

Procedure 85: Experimental Procedure 16: The formation of an un-known aluminum-containing compound, possibly a hydrated aluminum oxychloride using a brand new clay pot utilizing a copper cathode to see if there is any difference in precipitate color of cathode

Preparation: Dissolve 50 grams of pickling salt into 300 milliliters of tap water, and then pour into the anode compartment. Then dissolve 105 grams of Epsom salt into 700 milliliters of tap water, and then pour into the cathode compartment. Thereafter, electrolyze the apparatus at 12 volt 6 amp for about 6 to 10 hours. Note: some clay pots have holes in the bottom of them, and hence, must be plugged up airtight in order for the cell to work properly.

Set-up:



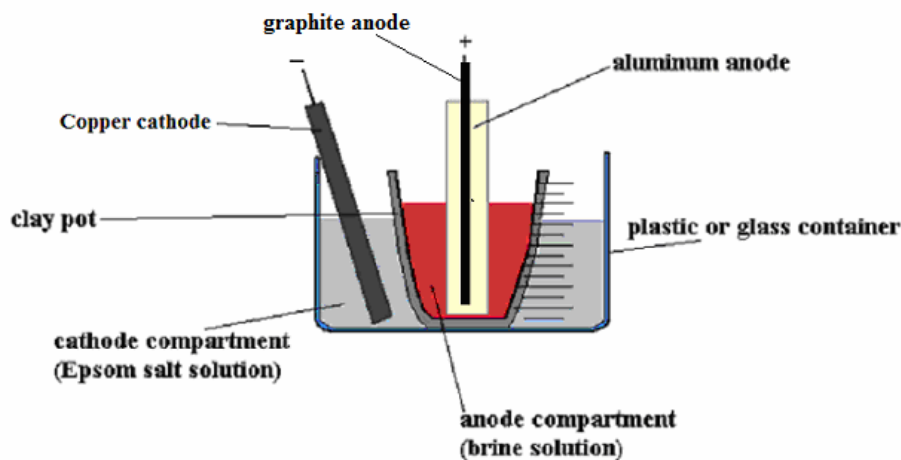
Note: Aluminum foil is used as the anode

Conclusions: Startup time, 7:05 PM. Conductivity at start up was normal. As normal, bubbles of gas emanating from the aluminum anode. At 7:47 PM, a white precipitate is clearly visible in the cathode compartment. Around 8:12 PM conductivity is normal, but less than that of the previous procedure using a lead cathode. The reaction seems to be going as planned. Significant white precipitate forming in cathode compartment. At 8:48 PM conductivity has increased. Added water to the anode liquid to increase its volume. Reaction is going smooth as expected. White precipitate in cathode compartment increasing, and the anode liquid is a grayish cloudy color due to carbon from the aluminum. Note: I have no idea what using a copper cathode over a lead cathode (as in the previous procedure) has to do with the current outcome; as with the lead cathode a dark heavy precipitate was formed, but using a copper cathode is producing a smooth clean white precipitate. Around 11:22 PM, significant white precipitate in cathode compartment. Severe corrosion to aluminum anode, will have to replace with more aluminum shortly. At 11:40 PM turned off power to add more aluminum to the anode. At 11:45 PM conductivity has decreased. **Note: For some reason, using copper as a cathode seems to have a remarkable effect upon the overall cell reaction.** Around 12:53 PM, lots of white precipitate in cathode compartment. Stopped cell at 2:00 AM. After filtration, cathode liquid has much less white precipitate than once thought. The cathode liquid after filtration is a clear odorless liquid. The amount of white precipitate is less than expected. Added 200 ml to anode liquid. The anode liquid is difficult to filter, as the carbon particles are so small, they go through the filter. A small sample of anode liquid, reacted with ammonia forming a gelatinous precipitate. No reaction of anode liquid with 3% hydrogen peroxide. Perhaps I did not electrolysis the reactants long enough! Got fed up, and mixed in 150 ml of 91% isopropyl alcohol to the anode liquid and mixed the mixture. A precipitate formed immediately. Mixed 150 ml of 91% isopropyl alcohol to the filtered cathode liquid. No precipitation occurred! However, after awhile, a few crystals precipitated in the cathode liquid. After adding and mixing the 91% isopropyl alcohol to the anode liquid, the entire mixture turned a milky white, and stayed that way for a long time. Note: Aluminum chloride is soluble in alcohol! Indicates no formation of aluminum chloride! After about ten minutes, a small amount of white precipitate is forming in the cathode liquid, and the cathode liquid is slowly precipitating a triangle shaped mass starting from the base. The anode liquid after being mixed with alcohol is totally milky white through out the whole volume for a long time! After standing overnight, all white precipitate remains in the anode liquid. Note: Aluminum chloride is soluble in alcohol. After vacuum filtration, a white solid was recovered. Now, allow this compound to dry at room temperature or vacuum dry as much as possible and then react it with other compounds to see if you get any reactions. Can you figure out what this compound is?

Procedure 86: Experimental Procedure 17: The formation of an un-known aluminum-containing compound, using a copper cathode, and an aluminum/graphite anode

Preparation: Dissolve 50 grams of pickling salt into 300 milliliters of tap water, and then pour into the anode compartment. Then dissolve 105 grams of Epsom salt into 700 milliliters of tap water, and then pour into the cathode compartment. Thereafter, electrolyze the apparatus at 12 volt 6 amp for about 6 to 10 hours. Note: some clay pots have holes in the bottom of them, and hence, must be plugged up airtight in order for the cell to work properly.

Set-up:



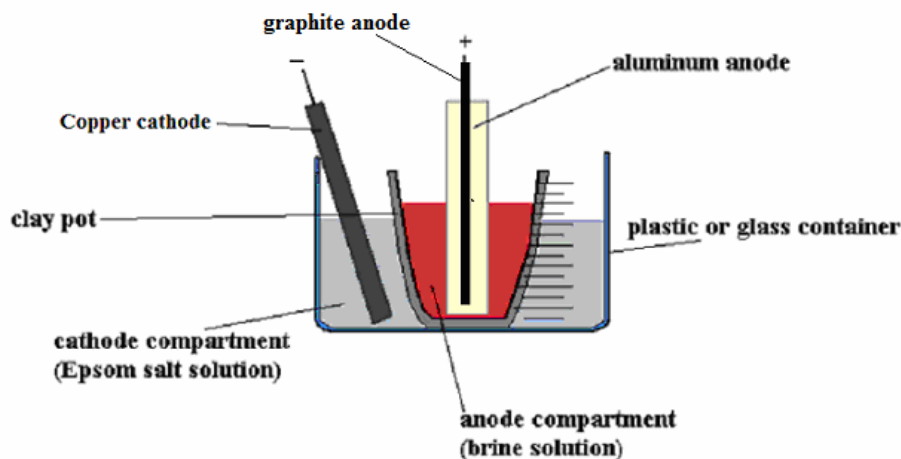
Note: Aluminum foil is used as the anode

Conclusions: Startup time, 7:47 PM, conductivity at startup rather slow. At 8:16 PM, conductivity is still rather slow, bubbles can be seen coming from the aluminum anode, but no bubbles coming off the graphite portion. At 8:49 PM conductivity has increased slightly, and there is little white flocculent mass on the surface of the anode liquid near the area of the copper cathode; note: The cell is water tight, as the water level of the anode compartment is higher than the cathode compartment indicating a water tight seal of the clay pot. Second note: I used the same clay pot as used in procedure 17, so this may be affecting the outcome, maybe! At 9:08 PM, there is little or no precipitate in the cathode liquid, but the cathode liquid is slightly cloudy when looked down upon. At 10:18 PM, the anode liquid is a grayish color, the usual corrosion of the aluminum anode, and a white precipitate is slowly collecting in the cathode compartment as expected—not sure if the graphite is having any real affect on the cell reaction. At 11:57 PM, more white precipitate in cathode compartment as expected, anode liquid is dark gray with severe corrosion to the aluminum anode, no sign the graphite anode is having any effect. Note: at 12:47 AM, I pulled out the anode until the aluminum was no longer touching the anode liquid, but the graphite was, and I got a smell of chlorine gas, I double checked this, and got another smell of chlorine gas so this tells me that the graphite is conducting in the cell!! Note: the chlorine can be smelled from a long distance. The cell conductivity is good, corrosion of the aluminum anode is less than expected at this point in time. More white precipitate in cathode compartment, with increase in heat. At 2:48 AM added more aluminum to the anode. As before, when removing the aluminum from the anode liquid and exposing only the graphite, a chlorine smell was developed. Stopped cell at 4:00 AM even after separating the cell, the anode liquid reacted with the aluminum. Removed the aluminum and graphite at 4:05 AM. After filtering the anode liquid, a clear non-odorous liquid is obtained. When this liquid is allowed to evaporate over many days, a white crystalline solid separates out, with a small amount of a yellowish liquid remaining. When a small sample of this white crystalline solid is burned using a butane lighter, it first crackles, and then evolves a pungent odor of hydrogen chloride/chlorine, with some popping of the heated material taking place.

Procedure 87: Experimental Procedure 18: The formation of an un-known aluminum-containing compound, using a copper cathode, and an aluminum/graphite anode

Preparation: Dissolve 50 grams of pickling salt into 300 milliliters of tap water, and then pour into the anode compartment. Then dissolve 105 grams of Epsom salt into 700 milliliters of tap water, and then pour into the cathode compartment. Thereafter, electrolyze the apparatus at 12 volt 6 amp for about 6 to 10 hours. Note: some clay pots have holes in the bottom of them, and hence, must be plugged up airtight in order for the cell to work properly.

Set-up:



Note: Aluminum foil is used as the anode

Conclusions: Start time: 6:15PM. Conductivity at startup is normal—bubbles of gas coming from copper cathode, bubbles forming on aluminum part of the anode. At 7:11 PM white precipitate is forming in cathode compartment as expected. Aluminum anode is corroding and small pieces of it are floating on the surface of the anode liquid. At 8:45 PM conductivity is very good. White precipitate in cathode compartment increasing as usual. Anode liquid is gray and non-clear as expected with heavy corrosion of the aluminum part of the anode. Had to stop cell at 12:27 AM to replace the aluminum anode portion, which had been completely dissolved. Note: with only the graphite inserted into the anode liquid, a small amount of chlorine gas was evolved. Stopped cell at 2:10 AM; the anode liquid is dark grayish color as expected with a slight hint of a dilute hydrogen chloride like odor. White precipitate in cathode liquid as expected—greater than previously thought after the cathode liquid is allowed to stand, a layer of white precipitate settles out. Note: The second aluminum anode portion was severely corroded. After letting the anode liquid stand for several weeks, the muddy grayish color becomes clear as the carbon impurities settle to the bottom of the beaker. Note: after allowing the anode liquid to settle, its original volume was 300 milliliters, but when time to filter the anode liquid it had a volume of 250 milliliters due to evaporation upon standing. After filtration of the anode liquid using suction filtration, a almost clear solution is obtained. After filtration, an exact volume of 91% isopropyl alcohol is added, and a two-phase mixture results, with the alcohol layer being the upper layer. Note: at first there is no separation of solids, but on prolong standing, a white precipitate slowly separates out and settles to the bottom. Now, at this point most of the alcohol would have evaporated, but some will still remain. In any sense, filter-off the insoluble precipitate using suction filtration, wash with three 150-milliliter portions of fresh 91% isopropyl alcohol, and then suction dry as best as possible. Finally, remove the washed suction dried filter cake, and place it onto a shallow plastic tray (not metal) and allow it to air dry thoroughly for at least a week. Thereafter, scrap up this dried product and then place it into a zip lock bag until use. Now it's time to perform some tests. Now is the time to use your experience you have learned by now to determine what this white precipitate may be. First, take a small sample of the product and add to 50 ml of 3% hydrogen peroxide, and note any reaction. Second, burn a small sample and smell the vapors that are evolved. Third, dissolve a small sample into 50ml of water and then add in 20ml of 10% store bought crystal clear ammonia, and note any reaction (precipitate), fourth, perform a pH test using a digital pH meter (note: conduct a pH test of the anode liquid before starting the electrolysis, and then perform a pH test after the anode liquid has been filtered) and fifth, try mixing it or mixing and heating it with any desired substance to note any potential reactions. The compounds used can be ferric chloride, sodium nitrate, potassium permanganate, sugar, sulfur, red phosphorus, potassium dichromate, dilute sulfuric acid, dilute hydrochloric acid, calcium hypochlorite, bleach, and/or sodium or potassium hydroxide. What could this substance be??? Well, it contains aluminum, we know that for sure due to the corrosion of the aluminum anode during the electrolysis. We know it contains chlorine due to chlorine evolution when the aluminum anode is removed temporarily and the graphite left in (whereby chlorine gas is evolved). Could the substance contain sodium? If it does contain sodium what explains the precipitation of magnesium hydroxide in the cathode compartment? We know the cathode liquid contains precipitated magnesium hydroxide, and sodium sulfate. Therefore, the anode product must be a hydrated aluminum oxy chloride, noting that earlier when we added the 91% isopropyl alcohol, the product slowly precipitated—aluminum chloride is soluble in alcohol!

Final wrap up

Now, at this point you should now be familiar with basic general chemistry, and it is important that you practice the techniques detailed in this book to better familiarize yourself with general chemistry. Remember and keep in mind that chemistry is a massive and never ending subject filled with trials of positives and negatives, trial by fire, and never ending chemical reactions that only happen when the appropriate reactants are carried out in the appropriate manners. Knowing these appropriate reactants and appropriate manners is the essence of chemistry knowledge, and you don't need a PhD in chemistry to master this

art of knowledge. So go on now, and practice the techniques detailed in this book, and you will find them quite exciting and fun to carryout. Many of the procedures detailed in this book use reactants (chemicals) that are readily available from store bought materials and on-line auction sites such as E-bay. In conclusion, Chemistry is the art of changing matter from one form to the next, and there are millions of ways to do so, but don't focus too much on this as truly mastering all of chemistry is virtually impossible even for a PhD in chemistry.