A NOTE ON THE PURIFICATION OF PICRIC ACID FOR CREATININE DETERMINATION.

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In a previous communication (1) the present writer described a technique for the purification of picric acid which depends upon its recrystallization from benzene. While that process yields a satisfactory product with most samples of picric acid, occasionally some may be found which show little improvement after crystallization from benzene; hence we have been lead to develop more satisfactory procedures.

Below are described two processes, either one of which has been found to be very satisfactory even with the most impure picric acids which we have ever encountered. The one of these which uses crystallization from glacial acetic acid is especially suitable for the treatment of small amounts of material, but requires that the impure picric acid be dried before the process is used. As tested by the Folin-Doisy method (2), the product obtained reads about 12.5 to 13.5 mm. The second process, crystallization as sodium picrate, is essentially a modification of the Folin-Doisy method. We use sodium carbonate in place of the hydroxide employed by Folin and Doisy and convert the sodium salt to free picric acid directly on the filter paper. The difficulty with the Folin-Doisy process probably lies in the fact that there is more or less continuous decomposition of the picric acid through action of the strong alkali. With the substitution of carbonate we have obtained a much more satisfactory final product.

Directions for the processes follow.

1. From Glacial Acetic Acid.—The technical picric acid must be dried thoroughly before being used in this procedure. Dissolve 100 gm. of dry picric acid with the aid of heat in 150 cc. of

glacial acetic acid, and continue the heating until the mixture boils.[1] Pour the hot solution upon a fluted filter contained in a dry funnel which has been previously heated, and collect the filtrate in a dry beaker. Cover the beaker with a watch-glass and allow to stand for some hours, or overnight at room temperature (not in a refrigerator). At the end of this time if picric acid has not crystallized out, stir the mixture vigorously, or better, seed with a minute crystal of pure picric acid. Crystallization will begin at once and is complete within 2 hours or less. At the end of 2 hours filter with suction on a hardened filter and wash with about 35 cc. of cold glacial acetic acid. Suck as free from acetic acid as possible and dry at about 80–90°, with occasional stirring, until there is no odor of acetic acid. It is best to conduct all of these operations in a good draft of air. The yield is about 60 gm. of pure picric acid, which should read 12.5 to 13.5 mm. by the Folin-Doisy test.

2. As Sodium Picrate.—This procedure involves handling large volumes of solution, but permits the use of moist technical picric acid and gives a good percentage recovery of pure picric acid. The product is usually slightly better (Folin-Doisy test) than the one obtained through the use of glacial acetic acid.

It is convenient in this method to use a large porcelain enamelled pail, but the enamel must be perfect at every point. Otherwise glass vessels should be used.

Place 6 liters of water in a large porcelain enamelled pail and heat to boiling. Add 250 gm. of anhydrous sodium carbonate and as soon as this has dissolved add gradually (or as fast as it dissolves) 500 gm. of the moist technical picric acid. Before all of the picric acid has dissolved the mixture should be removed from the flame and stirred for a few minutes until solution of the picric acid has been effected. Filtration is usually unnecessary. The solution may be allowed to stand for some minutes and is then decanted from some dirt which has settled to the bottom. It is then allowed to stand overnight at room temperature. In the morning the crystallized sodium picrate is filtered off on a hardened filter in a large (23 cm.) Buchner funnel, with suction. The picrate on the filter is sucked dry and then washed with 2 liters of 10 per cent sodium chloride solution, and again sucked as

[1] The mixture should be heated in an Erlenmeyer flask upon an electric plate.

dry as possible. The suction is now turned off and 500 cc. of diluted (1 part of concentrated acid plus 4 parts of water) hydrochloric acid are poured on the filter and the mixture thoroughly stirred with a porcelain spatula. This acid is then sucked into the receiving flask and the process repeated with three more portions of the hydrochloric acid (a total of 2 liters of the acid being used). After the last portion of acid is sucked through, the picric acid on the filter is washed with 2 liters of cold distilled water and sucked dry. It is then removed from the filter and dried at about 90° and powdered. This product should read about 13.5 to 14 mm. by the Folin-Doisy test.

BIBLIOGRAPHY.

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2. Folin, O., and Doisy, E. A., *J. Biol. Chem.*, **28**, 349 (1916–17).