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Communication

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A Soluble, Halogen-Free Oxalate from Methyl Salicylate for Chemiluminescence Demonstrations

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Supporting Information

ABSTRACT: A new oxalate ester made from methyl salicylate (oil of wintergreen) was found to be an excellent replacement for the typical halogenated oxalate ester (bis(2,4,6-trichlorophenyl) oxalate) used in peroxyoxalate chemiluminescence demonstrations. This new compound is easily prepared and has good solubility in organic solvents, such as ethyl acetate, which is an environmentally friendly solvent to use in the demonstration.

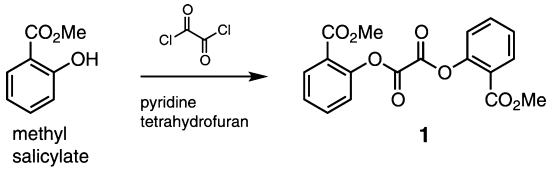
KEYWORDS: *High School/Introductory Chemistry, First-Year Undergraduate/General, Organic Chemistry, Demonstrations, Public Understanding/Outreach, Esters, Phenols, Green Chemistry*

Chemiluminescence demonstrations are a hit even among uninterested students. Most students are familiar with Cyalume glowsticks, as well as lightning bugs, which facilitate a learning connection between these types of demonstrations and the natural world. A recent chemiluminescence demonstration that appeared in this *Journal* describes the synthesis of an environmentally friendly bis(aryl) oxalate ester from the flavoring agent vanillin (3-methoxy-4-hydroxybenzaldehyde).[1] In a continuation of this theme, another "green" oxalate ester suitable for a chemiluminescence demonstration has been prepared from methyl salicylate (oil of wintergreen, methyl 2-hydroxybenzoate) (Scheme 1). This new oxalate ester (**1**) has excellent solubility in organic solvents, such as ethyl acetate. The experimental details can be found in the Supporting Information.

CHEMILUMINESCENCE PROCEDURE

A conical flask (10 mL) equipped with a stirring bar is charged with **1** (100 mg) and ethyl acetate (5 mL). All of the solids are allowed to dissolve, and then perylene (20 mg) is added (which may not completely dissolve). Next, sodium salicylate (15 mg) is added, which acts as basic catalyst, and finally one drop of H_2O_2 (30%) is added, followed by a quick shake of the flask. A brilliant blue color is generated by chemiluminescence and is best observed in a darkened room with shades drawn. Typical duration of light is about 30 min. If the flask is capped, make sure it is loosely closed to allow for a small quantity of byproduct CO_2 to escape. Other dyes can be substituted resulting in different colors of light.[2]

Scheme 1. Preparation of Bis[2-(methoxycarbonyl)phenyl] Oxalate (1) from the Natural Product Methyl Salicylate



HAZARDS

Standard protective equipment should be worn. Methyl salicylate is an eye irritant; oxalyl chloride is a lachrymator; pyridine is an irritant; tetrahydrofuran and ethyl acetate are flammable; perylene is possibly carcinogenic with chronic exposure; sodium salicylate may be caustic to mucous membranes; hydrogen peroxide is an irritant. The hazards of **1** are unknown, and therefore **1** should be handled accordingly. Details of the hazards are in the Supporting Information Part I.

ASSOCIATED CONTENT

Supporting Information

Further background discussion and description of the synthesis of bis[2-(methoxycarbonyl)phenyl] oxalate (**1**) and demonstration procedures (Part I). Additional comments on structure−activity relationships in regard to chemiluminescence of bis(aryl) oxalates, along with ^1H and ^{13}C NMR spectra of **1**

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and X-ray crystal structure plot (Part II). This material is available via the Internet at http://pubs.acs.org.

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Notes

The authors declare no competing financial interest.

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