



A Publication
of Reliable Methods
for the Preparation
of Organic Compounds

Working with Hazardous Chemicals

The procedures in *Organic Syntheses* are intended for use only by persons with proper training in experimental organic chemistry. All hazardous materials should be handled using the standard procedures for work with chemicals described in references such as "Prudent Practices in the Laboratory" (The National Academies Press, Washington, D.C., 2011; the full text can be accessed free of charge at http://www.nap.edu/catalog.php?record_id=12654). All chemical waste should be disposed of in accordance with local regulations. For general guidelines for the management of chemical waste, see Chapter 8 of Prudent Practices.

In some articles in *Organic Syntheses*, chemical-specific hazards are highlighted in red "Caution Notes" within a procedure. It is important to recognize that the absence of a caution note does not imply that no significant hazards are associated with the chemicals involved in that procedure. Prior to performing a reaction, a thorough risk assessment should be carried out that includes a review of the potential hazards associated with each chemical and experimental operation on the scale that is planned for the procedure. Guidelines for carrying out a risk assessment and for analyzing the hazards associated with chemicals can be found in Chapter 4 of Prudent Practices.

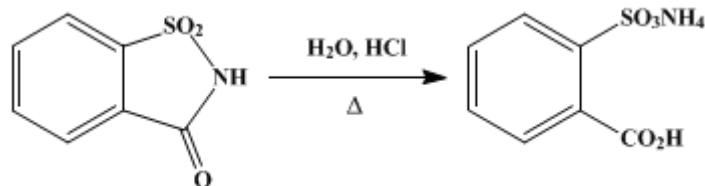
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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

Organic Syntheses, Coll. Vol. 1, p.14 (1941); Vol. 9, p.1 (1929).

ACID AMMONIUM *o*-SULFOBENZOATE

[**Benzoic acid, *o*-sulfo-, ammonium H salt**]



Submitted by H. T. Clarke and E. E. Dreger.

Checked by Henry Gilman and J. E. Kirby.

1. Procedure

In a 12-l. flask fitted with a stirrer and reflux condenser are placed 188 g. (1 mole) of *o*-sulfonylbenzimidazole ("saccharin insoluble"), 565 cc. of distilled water and 188 g. (158 cc.) of concentrated hydrochloric acid (sp. gr. 1.19). The mixture is boiled over a free flame with continual stirring (Note 1) until all the solid is in solution; this requires two and one-half to three hours. A second quantity of 188 g. of *o*-sulfonylbenzimidazole is then added, and the mixture again heated with stirring until a clear solution results, which requires one and one-half to two hours. Heating is then continued for one hour longer, whereupon the solution is poured into a crock and allowed to cool.

The crystals which separate are collected on a suction funnel, washed as free from hydrochloric acid as possible (Note 2) with ice-cold distilled water, and dried. The mother liquor and washings are concentrated on a steam bath under reduced pressure until the separation of crystals causes bumping, when the solution is again allowed to crystallize. This procedure is repeated, the final mother liquor being evaporated nearly to dryness. The main product, together with that from the mother liquors, weighs 410–427 g. (91–95 per cent of the theoretical amount) and is sufficiently pure for conversion into *o*-sulfonylbenzoic anhydride (p. 495). In order to obtain a purer product the material may be recrystallized from an equal weight of distilled water; the yield on recrystallizing is about 90 per cent.

2. Notes

1. The mixture tends to foam somewhat during the first few minutes of boiling.
2. If much hydrochloric acid is allowed to remain with the crystals, drying is extremely difficult.

3. Discussion

Acid ammonium *o*-sulfonylbenzoate can be prepared by the hydrolysis of saccharin with concentrated hydrochloric acid.¹ The procedure described is a modification of that of White and Acree.²

This preparation is referenced from:

- Org. Syn. Coll. Vol. 1, 495

References and Notes

1. Fahlberg and Barge, Ber. **22**, 755 (1889); Remsen and Linn, Am. Chem. J. **11**, 74 (1889).
2. White and Acree, J. Am. Chem. Soc. **41**, 1197 (1919).

Appendix
Chemical Abstracts Nomenclature (Collective Index Number);
(Registry Number)

ACID AMMONIUM o-SULFOBENZOATE

hydrochloric acid (7647-01-0)

saccharin (81-07-2)

o-Sulfobenzoic anhydride (81-08-3)

Benzoic acid, o-sulfo-, ammonium

o-sulfobenzoic imide

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