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](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.

The procedures described in *Organic Syntheses* are provided as published and are conducted at one's own risk. *Organic Syntheses, Inc.*, its Editors, and its Board of Directors do not warrant or guarantee the safety of individuals using these procedures and hereby disclaim any liability for any injuries or damages claimed to have resulted from or related in any way to the procedures herein.

*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.*
**p-ACETAMINOBENZENESULFINIC ACID**

*[Benzenesulfonic acid, p-acetamido-]*

Submitted by S. Smiles and C. M. Bere.

Checked by Henry Gilman and F. Schulze.

### 1. Procedure

The crude *p*-acetaminobenzenesulfonyl chloride (p. 8) obtained from 67.5 g. (0.5 mole) of acetanilide is shaken for two hours with a solution of 252 g. (1 mole) of crystallized sodium sulfite (Na₂SO₃ · 7H₂O) in 500 cc. of water. The reaction mixture is kept slightly alkaline by the addition at intervals of small portions of 50 per cent sodium hydroxide solution. The total volume of alkali used varies from 10 to 50 cc. After the alkaline mixture has been shaken for the two-hour period (Note 1) it is filtered, and the filtrate is acidified with 60 per cent sulfuric acid. If the acid is added slowly, the sulfinic acid comes down in fine crystals which, after filtering and drying, melt at 155° with decomposition (Note 2). The yield is 50–55 g. (43–47 per cent of the theoretical amount based on the acetanilide used).

The product may be purified by crystallization from 400 cc. of hot water, but this is unnecessary when the above procedure is followed carefully.

### 2. Notes

1. The solution does not clear up when reduction is complete on account of the formation of a gelatinous impurity. Two hours suffice for the completion of reduction.
2. The melting point given in the literature (180°) is incorrect, its publication being due to a typographical error. The observed melting point varies slightly with the rate of heating during the determination of the melting point.

### 3. Discussion

*p*-Acetaminobenzenesulfinic acid can be prepared by the reduction of *p*-acetaminobenzenesulfonyl chloride.¹

This preparation is referenced from:


### References and Notes


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

sulfonic acid

sulfuric acid (7664-93-9)

Acetanilide (103-84-4)

sodium sulfite (7757-83-7)

sodium hydroxide (1310-73-2)

p-Acetaminobenzenesulfonyl chloride (121-60-8)

p-Acetaminobenzenesulfonic acid,
Benzenesulfonic acid, p-acetamido- (710-24-7)