

A Publication of Reliable Methods for the Preparation of Organic Compounds

Working with Hazardous Chemicals

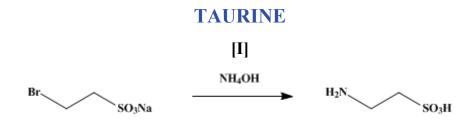
The procedures in Organic Syntheses are intended for use only by persons with proper training in experimental organic chemistry. All hazardous materials should be handled using the standard procedures for work with chemicals described in references such as "Prudent Practices in the Laboratory" (The National Academies Press, Washington, D.C., 2011; the full accessed of charge text can be free 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. 2, p.563 (1943); Vol. 18, p.77 (1938).



Submitted by C. S. Marvel and C. F. Bailey. Checked by Frank C. Whitmore and D. J. Loder.

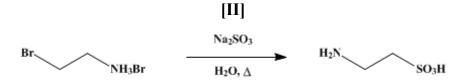
1. Procedure

A solution of 110 g. (0.52 mole) of sodium 2-bromoethanesulfonate (p. 558) in about 2 l. (28 moles) of concentrated aqueous ammonia (sp. gr. 0.9) is allowed to stand for five to seven days (Note 1) and is then evaporated to dryness. The last of the water is removed by heating on a steam bath. The residue is dissolved in the minimum quantity of hot water (about 500 cc.) and, if necessary, treated with 5 g. of Norite. The colorless solution is concentrated to 65–70 cc., and 250 cc. of 95 per cent alcohol is added. In a short time taurine mixed with some sodium bromide separates. When crystallization is complete, the crude taurine is collected on a filter and recrystallized by dissolving in 100 cc. of hot water and then adding to the solution enough 95 per cent ethyl alcohol (about 500 cc.) to give a final concentration of 80 per cent of alcohol. The taurine which separates is usually free from bromides. However, occasional runs must be recrystallized four or five times to remove all the sodium bromide. The yield of pure taurine (Note 2) is 31–36 g. (48–55 per cent of the theoretical amount).

2. Notes

1. The reaction is about 25 per cent complete in five hours, 60 per cent complete in thirty hours, and 90 per cent complete in five days, as indicated by titration of the bromide ion.

2. The purity of the taurine prepared by this method was established by analysis.



Submitted by Frank Cortese Checked by C. S. Marvel and C. L. Fleming.

1. Procedure

A solution of 615 g. (3 moles) of β -bromoethylamine hydrobromide (p. 91) and 416 g. (3.3 moles) of anhydrous u.s.p. sodium sulfite (Note 1) in 2.4 l. of water is concentrated on the steam bath to a minimum volume; thirty-six to forty-eight hours is required for this operation. After the mixture has cooled, the cold, moist cake is triturated with 1.5 l. of concentrated hydrochloric acid and collected on an asbestos mat in a Büchner funnel. The precipitate is washed ten times with 150-cc. portions of concentrated hydrochloric acid. The filtrate is mixed well, decanted from precipitated salts if necessary, and concentrated over a free flame to a volume of 600 cc.

Two and four-tenths liters of 95 per cent ethyl alcohol is added, with vigorous stirring, to the hot mixture. After fifteen minutes, the product is collected on a filter, washed with 95 per cent ethyl alcohol until it is colorless, and air-dried. The crude material is purified by dissolving it in four times its weight of hot water, adding Norite, filtering, and adding to the hot filtrate five volumes of 95 per cent ethyl alcohol.

This product is practically pure taurine; it decomposes at 305–310° (Maquenne block). The yield is 255–275 g. (68–73 per cent of the theoretical amount).

2. Notes

1. An equivalent quantity (831 g.) of the more expensive crystalline sodium sulfite may be used.

3. Discussion

Taurine is generally prepared from ox bile¹ or the large muscle of the abalone.² It has been obtained from the oxidation of cystamine³ and the decarboxylation of cysteic acid;⁴ from ethyleneimine and sulfur dioxide;⁵ from chloroethanesulfonic acid and ammonia;⁶ from 2-mercaptothiazoline by oxidation with bromine water;⁷ and from acetaldehyde by a complex set of reactions involving sulfonation, formation of the aldehyde ammonia and the imidosulfonic acid, and finally reduction.⁸ Taurine is usually synthesized either from bromoethanesulfonic acid and ammonia⁹—Procedure I, or from β-bromoethylamine hydrobromide and sodium sulfite¹⁰

This preparation is referenced from:

- Org. Syn. Coll. Vol. 2, 91
- Org. Syn. Coll. Vol. 2, 558

References and Notes

- 1. Hammarsten, Z. physiol. Chem. 32, 456 (1901); Tauber, Beitr. chem. Physiol. Path. 4, 324 (1904).
- 2. Schmidt and Watson, J. Biol. Chem. 33, 499 (1918).
- 3. Schöberl, Z. physiol. Chem. 216, 193 (1933).
- 4. Friedmann, Beitr. chem. Physiol. Path. 3, 1 (1903); White and Fishman, J. Biol. Chem. 116, 457 (1936).
- 5. Gabriel, Ber. 21, 2667 (1888).
- 6. Kolbe, Ann. 122, 42 (1862); Anschütz, ibid. 415, 97 (1918).
- 7. Gabriel, Ber. 22, 1154 (1889).
- 8. Auzies, Rev. gén. chim. 14, 278 (Chem. Zentr. 1911, II, 1433).
- 9. Marvel, Bailey, and Sparberg, J. Am. Chem. Soc. 49, 1836 (1927).
- 10. Reychler, Bull. soc. chim. Belg. 32, 247 (1923); Cortese, J. Am. Chem. Soc. 58, 191 (1936).

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

bromine water

ethyl alcohol (64-17-5)

acetaldehyde (75-07-0)

hydrochloric acid (7647-01-0)

ammonia (7664-41-7)

sodium sulfite (7757-83-7)

sulfur dioxide (7446-09-5)

sodium bromide (7647-15-6)

Norite (7782-42-5)

β-Bromoethylamine hydrobromide (2576-47-8)

Taurine (107-35-7)

ethyleneimine (9002-98-6)

Sodium 2-bromoethanesulfonate (4263-52-9)

cystamine

cysteic acid

chloroethanesulfonic acid

2-mercaptothiazoline

bromoethanesulfonic acid

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