Organic Syntheses

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.

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MUCOBROMIC ACID

[Acrylic acid, 2,3-dibromo-3-formyl-]

Submitted by G. A. Taylor[1]
Checked by B. C. McKusick, E. L. Martin, and W. R. Brasen.

1. Procedure

A mixture of 50 g. (45 ml., 0.52 mole) of freshly distilled furfural and 500 ml. of water is stirred vigorously in a 2-l. three-necked round-bottomed flask equipped with a dropping funnel and a thermometer that dips into the liquid. The flask is immersed in an ice bath, and 450 g. (144 ml., 2.81 moles) of bromine is added, while the temperature of the reaction mixture is kept below 5° (Note 1). After the addition is complete, the thermometer is replaced by a reflux condenser, and the mixture is stirred and boiled for 30 minutes. The reflux condenser is replaced by a still head and condenser, and excess bromine is removed by distilling the liquid until the distillate is almost colorless (Note 2).

The reaction mixture is evaporated to dryness under reduced pressure at a water pump on a steam bath, using a trap cooled in ice and salt to condense the hydrobromic acid (Note 3). The solid residue is cooled in an ice bath and triturated with 30–50 ml. of ice water. A few grams of sodium bisulfite, dissolved in water, is added to discharge a slight yellow discoloration. The cold mixture is filtered with suction to separate crude mucobromic acid, which is washed with two small portions of ice water. The crude mucobromic acid weighs 125–132 g. (93–99%). It is dissolved in about 110 ml. of boiling water, 2–5 g. of decolorizing carbon is added, the hot mixture is stirred for 10 minutes and filtered, and the filtrate is cooled to 0–5°. Colorless crystals of mucobromic acid separate from the filtrate; weight 100–112 g. (75–83%); m.p. 124–125°.

2. Notes

1. If the temperature is allowed to rise much above 10°, the yield is considerably reduced. Without cooling, the mixture becomes quite hot, the yield is decreased by half, and tarry material is formed.
2. Mucobromic acid can be obtained in about 63% yield (85 g.) by adding 5 g. of decolorizing carbon at this point, stirring the mixture at the boil for 10 minutes, filtering it hot, and cooling the filtrate to 0°. The crude mucobromic acid that crystallizes, weight about 105 g., is recrystallized from 120 ml. of water.
3. It is important to get rid of all the hydrobromic acid, for its presence increases the amount of mucobromic acid lost in the trituration step.

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3. Discussion

This preparation is adapted from that described by Simonis.[2] It closely follows the practical details given in *Organic Syntheses*[3] for the preparation of mucobromic acid from the relatively expensive furoic acid.

This preparation is referenced from:

- Org. Syn. Coll. Vol. 4, 844

References and Notes

1. Department of Biochemistry and Dyson Perrins Laboratory, Oxford University, Oxford, England.
2. Simonis, *Ber.*, **32**, 2085 (1899).
3. *Org. Syntheses Coll. Vol.* **3**, 621 (1955).

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

HYDROBROMIC ACID (10035-10-6)

bromine (7726-95-6)

sodium bisulfite (7631-90-5)

carbon (7782-42-5)

furoic acid (88-14-2)

Furfural (98-01-1)

Mucobromic acid,
Acrylic acid, 2,3-dibromo-3-formyl- (488-11-9)