



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.

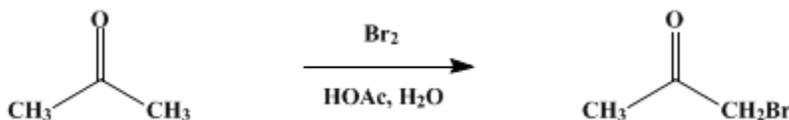
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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

Organic Syntheses, Coll. Vol. 2, p.88 (1943); Vol. 10, p.12 (1930).

BROMOACETONE

[2-Propanone, 1-bromo-]



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Checked by Frank C. Whitmore and J. Pauline Hollingshead.

1. Procedure

A 5-l., three-necked, round-bottomed flask is provided with an efficient mechanical stirrer, a 48-cm. Allihn reflux condenser, a thermometer, and a 500-cc. separatory funnel, the stem of which reaches nearly to the bottom of the flask (Note 1).

Through the separatory funnel are introduced 1.6 l. of water, 500 cc. of c. p. acetone, and 372 cc. of glacial acetic acid. The stirrer is started and the temperature of the water bath is raised to 70–80°, so that the mixture in the flask is at about 65° (Note 2). Then 354 cc. (7.3 moles) of bromine is carefully added through the separatory funnel. The addition, which requires one to two hours, is so regulated as to prevent the accumulation of unreacted bromine (Note 3). As a rule the solution is decolorized in about twenty minutes after the bromine has been added. When the solution is decolorized, it is diluted with 800 cc. of cold water, cooled to 10°, made neutral to Congo red with about 1 kg. of solid anhydrous sodium carbonate, and the oil which separates is collected in a separatory funnel and dried with 80 g. of anhydrous calcium chloride. After drying, the oil is fractionated and the fraction boiling at 38–48°/13 mm. is collected. The yield is 470–480 g. (50–51 per cent of the theoretical amount). It may be used without further purification for the preparation of acetol (p. 5); but, if a purer product is desired, the above product is refractionated and the fraction boiling at 40–42°/13 mm. is collected. The yield is 400–410 g. (43–44 per cent of the theoretical amount).

The higher-boiling fraction contains a mixture of isomeric dibromoacetones.

2. Notes

1. The apparatus should be set up with the flask in a large container (such as a 14-qt. galvanized pail) to be used as a water bath, and under a well-ventilated hood, as both the bromine and bromoacetone are powerful irritants to the skin and mucous membranes.
2. It is necessary to warm the reaction mixture to this temperature to ensure a smooth reaction
3. It is not advisable to have too great an excess of bromine present at any time, as it sometimes reacts suddenly with great violence.

3. Discussion

Bromoacetone has been prepared by the electrolysis of a mixture of acetone and hydrobromic acid,¹ and by more orthodox methods of bromination: the addition of bromine to acetone dissolved in ten times its weight of water;² the addition of bromine to acetone in which marble is suspended;³ the addition of bromine to acetone, water, and concentrated hydrochloric acid;⁴ and the introduction of bromine by means of a current of air into cold acetone.⁵

A procedure similar to the one described above, except that the reaction mixture is illuminated with a powerful light, has been published.⁶

This preparation is referenced from:

References and Notes

1. Richard, Compt. rend. **133**, 879 (1901).
 2. Sokolowsky, Ber. **9**, 1687 (1876).
 3. Scholl and Matthaiopoulos, *ibid.* **29**, 1555 (1896).
 4. Hughes, Watson, and Yates, J. Chem. Soc. **1931**, 3322.
 5. Emmerling and Wagner, Ann. **204**, 29 (1880).
 6. Gohr and Thiekötter, Biochem. Z. **305**, 374 (1940).
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Appendix Chemical Abstracts Nomenclature (Collective Index Number); (Registry Number)

Congo red

dibromoacetones

calcium chloride (10043-52-4)

hydrochloric acid (7647-01-0)

acetic acid,
Acetol (64-19-7)

HYDROBROMIC ACID (10035-10-6)

sodium carbonate (497-19-8)

bromine (7726-95-6)

acetone (67-64-1)

Bromoacetone,
2-Propanone, 1-bromo- (598-31-2)