



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.

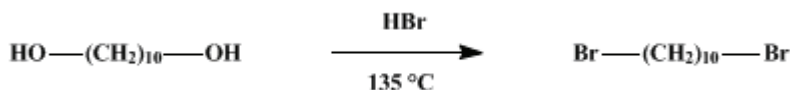
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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

Organic Syntheses, Coll. Vol. 3, p.227 (1955); Vol. 20, p.24 (1940).

DECAMETHYLENE BROMIDE

[Decane, 1,10-dibromo-]



Submitted by W. L. McEwen

Checked by Reynold C. Fuson and E. A. Cleveland.

1. Procedure

A 2-l. three-necked flask, supported in an oil bath, is fitted with a mechanical stirrer and an inlet tube which reaches almost to the bottom of the flask. In it is placed 696 g. (4 moles) of [decamethylene glycol](#), and, after the oil bath is heated to 95–100°, a rapid stream of dry [hydrogen bromide](#) is introduced, with stirring. When the mixture becomes saturated with the gas, as shown by vigorous fuming at the open neck of the flask ([Note 1](#)), the temperature of the oil bath is raised to 135° and a slow current of [hydrogen bromide](#) is passed in for 6 hours ([Note 2](#)).

After cooling, the crude product is transferred to a separatory funnel and the lower aqueous layer is drawn off and discarded. The upper layer is washed once with an equal volume of warm water, and then with successive portions of 10% [sodium carbonate](#) solution until all acid has been removed. It is then washed once with warm water, which is separated as completely as possible ([Note 3](#)). The product thus washed is distilled from a Claisen flask under reduced pressure. The first few drops of distillate containing some water are discarded: the main fraction distils at 139–142°/2 mm. The yield is 1080 g. (90%) ([Note 4](#)).

2. Notes

1. In larger runs it is advantageous to stop the stirrer and gas stream at this point and allow the lower aqueous layer to separate, after which it is siphoned out.
 2. The total quantity of [bromine](#) used is about 950 g.
 3. If difficulty is encountered with separation of layers in washing, the substance may be dissolved in an equal volume of [ether](#).
 4. This yield was almost invariably obtained by the submitter, who used quantities of glycol varying from 150 g. to 2 kg.
- By the same procedure the following dibromides have been prepared.

	% Yield	Boiling Point
Trimethylene bromide	75	165–167°
Hexamethylene bromide	90	108–112°/8 mm.
Nonamethylene bromide	93	128–130°/2 mm.

3. Discussion

[Decamethylene bromide](#) was first prepared by heating the glycol in a sealed tube with fuming [hydrobromic acid](#).¹ Later it was prepared in the manner here described,^{2,3} and by treating the glycol with [hydrobromic acid](#) in the presence of [sulfuric acid](#).⁴

This preparation is referenced from:

- [Org. Syn. Coll. Vol. 2, 598](#)

References and Notes

1. Franke and Hankam, *Monatsh.*, **31**, 181 (1910).
 2. Chuit, *Helv. Chim. Acta*, **9**, 264 (1926).
 3. Carothers, Hill, Kirby, and Jacobson, *J. Am. Chem. Soc.*, **52**, 5279 (1930).
 4. Price, Guthrie, Herbrandson, and Peel, *J. Org. Chem.*, **11**, 281 (1946).
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Appendix Chemical Abstracts Nomenclature (Collective Index Number); (Registry Number)

sulfuric acid (7664-93-9)

ether (60-29-7)

HYDROBROMIC ACID,
hydrogen bromide (10035-10-6)

sodium carbonate (497-19-8)

bromine (7726-95-6)

Trimethylene bromide (109-64-8)

Decamethylene glycol (112-47-0)

Decamethylene bromide,
Decane, 1,10-dibromo- (4101-68-2)

Hexamethylene bromide (629-03-8)

Nonamethylene bromide (4549-33-1)