



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.

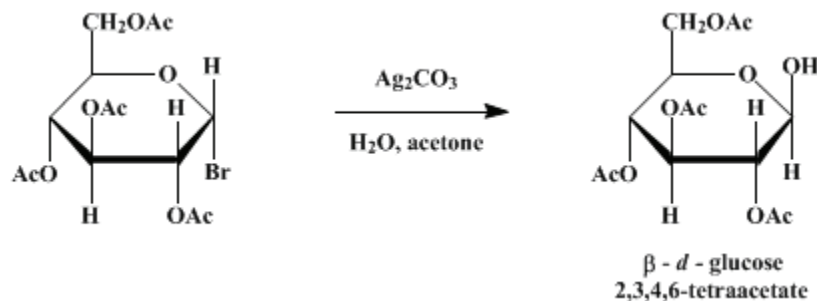
The procedures described in *Organic Syntheses* are provided as published and are conducted at one's own risk. *Organic Syntheses, Inc.*, its Editors, and its Board of Directors do not warrant or guarantee the safety of individuals using these procedures and hereby disclaim any liability for any injuries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

Organic Syntheses, Coll. Vol. 3, p.434 (1955); Vol. 25, p.53 (1945).

β -*d*-GLUCOSE-2,3,4,6-TETRAACETATE

[*D*-Glucose, β -2,3,4,6-tetraacetyl-]



Submitted by Chester M. McCloskey and George H. Coleman.
Checked by C. S. Hamilton, Robert Angier, and Ivan Baumgart.

1. Procedure

A solution of 82.2 g. (0.2 mole) of [acetobromoglucose](#) (p. 11) ([Note 1](#)) in 125 ml. of dry [acetone](#) ([Note 2](#)) in a 250-ml. flask is cooled to 0° in an ice bath. To the cold solution is added 2.3 ml. of water and then 46.5 g. (0.17 mole) of [silver carbonate](#) ([Note 3](#)) in small portions in the course of 15 minutes. The mixture is shaken well during the addition and for 30 minutes longer ([Note 4](#)). The mixture is then warmed to 50–60° and filtered. The mass of silver salts is washed with 65 ml. of dry [acetone](#) ([Note 5](#)), removed from the funnel, warmed in a flask with 65 ml. more of [acetone](#), filtered, and washed again on the funnel.

The combined filtrates are concentrated under reduced pressure in a 500-ml. filter flask ([Note 6](#)) until most of the solution is filled with crystals. The mixture is warmed to dissolve the crystals, the solution is poured into a 600-ml. beaker, and an equal volume of absolute [ether](#) and a similar volume of [ligroin](#) are added. The resulting solution is cooled in a freezing mixture with gentle stirring. The crystals of the tetraacetate form quickly and after about 10 minutes are filtered and air-dried. The crystals so obtained melt at 132–134° ([Note 7](#)). If a purer product is required, the product is dissolved in [acetone](#), and [ether](#) and [ligroin](#) are added to the solution in the manner described. The yield of once-crystallized product melting at 132–134° is 52–56 g. (75–80%).

2. Notes

1. The [acetobromoglucose](#) that was used had a melting point of 87–88°.
2. The [acetone](#) was dried over [calcium chloride](#).
3. The [silver carbonate](#) should be freshly prepared and finely ground. [Silver carbonate](#) can be prepared by the addition of a solution of [sodium carbonate](#) (53 g. in 600 ml. of water) to one of [silver nitrate](#) (172 g. in 2 l. of water). This is a very slight excess of the [silver nitrate](#). The [sodium carbonate](#) solution is added slowly (10 minutes), and the reaction mixture is stirred vigorously with a mechanical stirrer. The [silver carbonate](#) is filtered, washed with a little [acetone](#) to facilitate drying, and then air-dried. All operations are carried out in dim light.
4. At the end of this time, evolution of [carbon dioxide](#) should no longer be appreciable. The time required for the reaction depends largely on the agitation of the [silver carbonate](#). In large runs mechanical stirring is required.
5. Anhydrous chemicals are used throughout as the presence of any appreciable amount of water interferes with the crystallization of the tetraacetate.
6. The solution is not heated during the concentration, and thus the temperature is maintained below that of the room by the evaporation of the solvent. If a capillary is used it should be equipped with a drying tube.
7. β -*d*-Glucose-2,3,4,6-tetraacetate ([2,3,4,6-tetraacetyl- \$\beta\$ -*d*-glucose](#)) decomposes slightly on prolonged

standing and after 1–2 months has a melting range of 5–8 degrees. Such material can be purified by recrystallization.

3. Discussion

β -*d*-Glucose-2,3,4,6-tetraacetate has been prepared usually by the hydrolysis of acetobromoglucose.¹

References and Notes

1. Fischer and Delbruck, *Ber.*, **42**, 2776 (1909); Georg, *Helv. Chim. Acta*, **15**, 924 (1932); Hendricks, Wulf, and Liddel, *J. Am. Chem. Soc.*, **58**, 1998 (1936); McCloskey, Pyle, and Coleman, *J. Am. Chem. Soc.*, **66**, 349 (1944).

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

ligroin

calcium chloride (10043-52-4)

ether (60-29-7)

silver nitrate (7761-88-8)

sodium carbonate (497-19-8)

carbon dioxide (124-38-9)

acetone (67-64-1)

d-Glucose (492-62-6)

Acetobromoglucose (572-09-8)

β -*D*-Glucose-2,3,4,6-tetraacetate

silver carbonate (534-16-7)

2,3,4,6-tetraacetyl- β -*d*-glucose