



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.

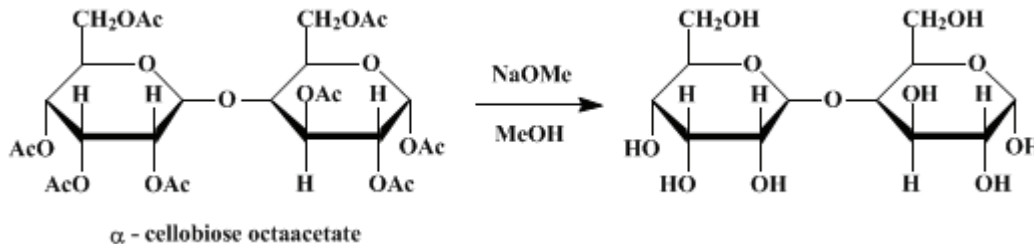
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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

Organic Syntheses, Coll. Vol. 2, p.122 (1943); Vol. 17, p.34 (1937).

CELLOBIOSE



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1. Procedure

In a 500-cc. three-necked flask, provided with a mercury-sealed stirrer and a calcium chloride tube, 68 g. (0.1 mole) of α -cellobiose octaacetate, m.p. 220–222° (p. 124), is suspended in 300 cc. of absolute methyl alcohol. A solution, prepared by dissolving 0.25 g. (0.01 gram atom) of sodium in 50 cc. of methyl alcohol, is added, and the mixture is stirred vigorously for one hour at room temperature (Note 1). The mixture becomes thin as the hydrolysis proceeds and the solvent acquires a slight color. After the time specified the crystalline solid is collected by suction filtration, washed with four 25-cc. portions of methyl alcohol, and dried at 40°. The weight of the nearly colorless crude cellobiose corresponds closely to the theoretical amount (34 g.). For purification it is dissolved in 125 cc. of hot water containing a few drops of glacial acetic acid, and the solution is clarified with 1–2 g. of Norite and filtered by suction. The colorless filtrate is concentrated under reduced pressure to a small volume, continuing until a large portion of the cellobiose has crystallized, and the crystalline magma is washed into an Erlenmeyer flask with 100 cc. of methyl alcohol. The mixture is stirred well and allowed to stand for several hours for completion of the crystallization, and the sugar is collected on a Büchner funnel, washed with 25 cc. of methyl alcohol, and dried at 40°. The yield of pure cellobiose, $[\alpha]_D^{20} + 34.8^\circ$ (in 6 per cent aqueous solution), is 31 g. (91 per cent of the theoretical amount). On concentrating the mother liquor to a small volume and adding alcohol as before, 1 g. of equally pure product is obtained, making the total yield 94 per cent of the theoretical amount (Note 2).

2. Notes

1. The reaction may be carried out equally satisfactorily by shaking the mixture mechanically in a stoppered bottle.
2. Using an earlier procedure,¹ in which a solution of the octaacetate in chloroform is treated with sodium methoxide solution and then with water, the yields of pure cellobiose amounted to 67–79 per cent of the theoretical quantity.

3. Discussion

Cellobiose was prepared first by Skraup and König² by the saponification of the octaacetate with alcoholic potassium hydroxide, and the method was improved by Pringsheim and Merkatz.³ Aqueous barium hydroxide also has been employed⁴ for the purpose, and methyl alcoholic ammonia has been used extensively for the hydrolysis of carbohydrate acetates. The method of catalytic hydrolysis with a small quantity of sodium methoxide was introduced by Zemplén,¹ who considered the action to be due to the addition of the reagent to the ester-carbonyl groups of the sugar acetate and the decomposition of the addition compound by reaction with alcohol.⁵ The present procedure, reported by Zemplén, Gerecs, and Hadácsy,⁶ is a considerable improvement over the original method¹ (see (Note 2)).

This preparation is referenced from:

References and Notes

1. Zemplén, Ber. **59**, 1254 (1926).
 2. Skraup and König, *ibid.* **34**, 1115 (1901).
 3. Pringsheim and Merkatz, Z. physiol. Chem. **105**, 173 (1919).
 4. Abderhalden and Zemplén, *ibid.* **72**, 58 (1911).
 5. Zemplén and Kunz, Ber. **56**, 1705 (1923).
 6. Zemplén, Gerecs, and Hadácsy, *ibid.* **69**, 1827 (1936).
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Appendix Chemical Abstracts Nomenclature (Collective Index Number); (Registry Number)

acetic acid (64-19-7)

methyl alcohol (67-56-1)

chloroform (67-66-3)

sodium methoxide (124-41-4)

Norite (7782-42-5)

potassium hydroxide (1310-58-3)

sodium (13966-32-0)

barium hydroxide (17194-00-2)

CELLOBIOSE (528-50-7)

α -Cellobiose octaacetate (5346-90-7)