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Note

2-Amino-2-deoxy-D-[1-^{14}C]glucitol hydrochloride

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2-Amino-2-deoxy-D-glucitol hydrochloride has been prepared from 2-amino-2-deoxy-D-glucose hydrochloride by high-pressure hydrogenation over Raney nickel catalyst[1–3], but the reaction is slow and incomplete. Borohydrides[4] are used for the reduction of reducing saccharides, with shorter reaction-times and better yields; for example, sodium borohydride[5,6] effected the reduction of 2-acetamido-2-deoxy-D-galactose, and the product was subsequently purified by use of Biodemin-rolite resin.

As a part of studies on the biosynthesis of the mitomycin antibiotics, labeled 2-amino-2-deoxy-D-glucitol hydrochloride was needed for feeding experiments. The present work reports the synthesis of 2-amino-2-deoxy-D-[1-^{14}C]glucitol hydrochloride (**2**) from 2-amino-2-deoxy-D-[1-^{14}C]glucose hydrochloride (**1**) in almost quantitative yield (95%), and purification of the product by chromatography[7–10] on Sephadex G-10.

EXPERIMENTAL

General. — Melting points are uncorrected. Evaporations were performed under diminished pressure below 50°. Thin-layer chromatography (t.l.c.) was conducted on silica gel (Kiesel gel G, Merck) with 5:5:1:3 pyridine–ethyl acetate–water–acetic acid as the solvent, and ninhydrin as the spray reagent. Liquid scintillation counting was performed with a Beckman LS-250 liquid scintillation spectrometer, and scanning of t.l.c. plates with a Packard Model 7201 radiochromatogram scanner.

Preparation of 2-amino-2-deoxy-D-[1-^{14}C]glucitol hydrochloride (**2**). — A solution of sodium borohydride (100 mg) in water (4 ml) was slowly added, with occasional shaking, to a solution of 2-amino-2-deoxy-D-glucose hydrochloride (Sigma Chemical Co.; 200 mg) in water (4 ml) containing 2-amino-2-deoxy-D-

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[1-^{14}C]glucose hydrochloride (**1**) (from New England Nuclear Co.; 100 μCi), at 0°. At the end of the addition, the mixture was kept for 30 min at 0° and then for 2 h at room temperature. The excess of borohydride was decomposed by addition of 4M hydrochloric acid, the solution was evaporated to dryness, and the boric acid removed as its methyl ester by repeated addition and evaporation of methyl alcohol. The residue was de-ionized by chromatography on a column (3 × 104 cm) of Sephadex G-10 at room temperature, with 1mM acetic acid as the eluant. Fractions (3 ml) were collected, and analyzed for sodium ions with uranyl zinc acetate, and for **2** by liquid scintillation counting and by t.l.c. Compound **2** was eluted first from the column (R_F: **2**, 0.18; **1**, 0.27). The later fractions contained traces of **2** from which sodium chloride was completely separated by rechromatography. Fractions of **2** were combined, and evaporated to a syrup which crystallized after addition of ethanol. The suspension was kept overnight in a refrigerator, and filtered, and the crystals were washed with acetone and dried; yield 191 mg, m.p. 160–161°; lit.[3] m.p. of unlabeled 2-amino-2-deoxy-D-glucitol hydrochloride, 159–160°. Liquid scintillation counting showed a specific activity of 0.11 mCi/mmole, and t.l.c. scanning revealed only one peak, having the R_F value of **2**.

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