

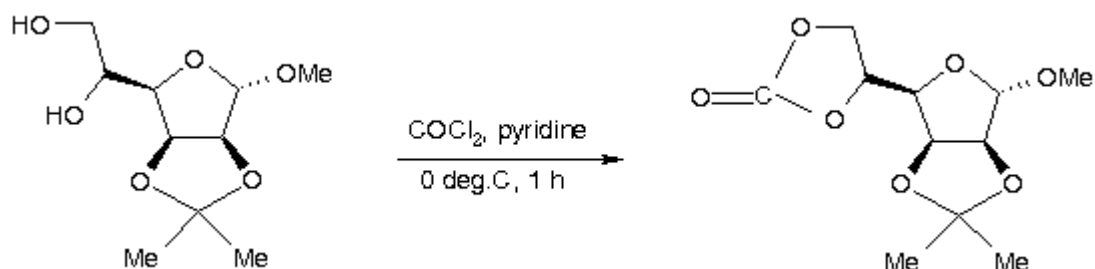
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Methyl 2,3-*O*-Isopropylidene- α -D-mannofuranoside 5,6-Carbonate

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Sugar carbonates can be prepared by the condensation of the *cis*-glycol system of the carbohydrate with phosgene or with a chloroformic ester [1,2].

A stream of phosgene was introduced into an ice-cooled, vigorously stirred solution of methyl 2,3-*O*-isopropylidene- α -D-mannofuranoside (2.34 g, 10 mmol) in dry pyridine (10 ml). After an hour, the reaction mixture was poured onto crushed ice (100 ml) and the product was extracted with chloroform (3 x 50 ml). The organic layer was washed successively with a cold saturated solution of sodium bicarbonate, cold water and dried over sodium sulfate. The solvent was co-evaporated with toluene under diminished pressure to give the crude product. Decolourizing of its ethereal solution with charcoal and recrystallization from ethyl acetate-hexane afforded the title compound (1.77 g, 68%) as white crystals.

M.p. 130-131 °C.

$[\alpha]_D^{+97}$ ($c = 10 \text{ mg cm}^{-3}$, methanol).

TLC (EtOAc/Hexane 3:2, silica gel) R_f 0.54.

$^1\text{H-NMR}$ (CDCl_3): 5.00 (m, $J=8.7$ and 7.0 and 3.3 Hz, 1H, H-5); 4.98 (s, 1H, H-1); 4.77 (dd, $J=5.9$ and 3.8 Hz, 1H, H-3); 4.64 (dd, $J=8.7$ and 7.0 Hz, 1H, H-6); 4.59 (d, $J=5.9$ Hz, 1H, H-2); 4.48 (t, $J=8.7$ Hz, 1H, H-6'); 4.33 (dd, $J=3.8$ and 3.3 Hz, 1H, H-4); 3.35 (s, 3H, OMe); 1.44 and 1.28 (2s, 2 x 3H, CMe₂).

$^{13}\text{C-NMR}$ (CDCl_3): 154.5 (C=O), 112.8 (CMe₂), 107.2 (C-1), 84.6 (C-2), 79.0 (C-3), 78.2 (C-4), 74.0 (C-5), 65.7 (C-6), 54.9 (OMe), 25.3 and 23.8 (CMe₂).

EI-MS (70 eV): 245 ($\text{M}^+ \text{CH}_3$, 100%), 185 (45), 173 (17), 141 (18), 113 (36), 98 (61), 85 (60), 60 (55), 59 (65), 57 (50), 43 (100).

Anal. calc. for C₁₁H₁₆O₇ (260.24): C 50.77, H 6.20; found: C 50.69, H 6.27.

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References and Notes

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2. Hirst, E. L.; Percival, E. *Methods Carbohydr. Chem.* **1963**, *2*, 349-353.

Sample availability: Available from the authors.

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