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Short Note

Methyl 6-*O*-tosyl-alpha-D-galactopyranoside

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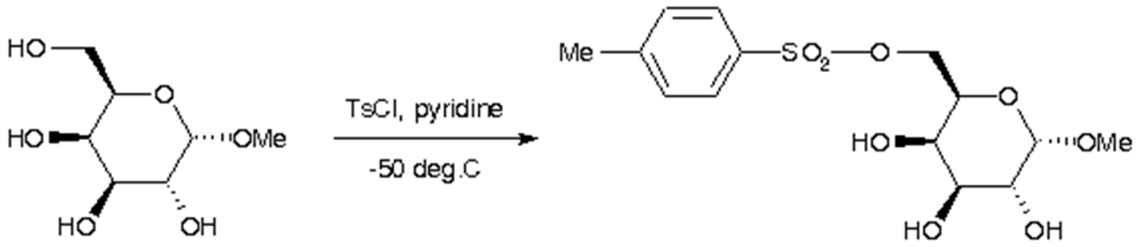

Although the title compound has already been prepared in four steps [1] or three steps [2] from D-galactose, this selective tosylation consists of only a two step synthesis avoiding the preparation of several intermediates and affording much higher overall yields. Moreover, NMR spectral data of the title compound have not yet been published.

To a well stirred solution of methyl alpha-D-galactopyranoside (9.71 g, 50 mmol) [3] in dry pyridine (40 ml) 4-toluenesulfonyl chloride (10.49 g, 55 mmol) was added in small portions at -50 deg.C. The reaction mixture was then kept in a freezer overnight and after reaching laboratory temperature it was poured into ice-water (250 ml). Filtration and recrystallization from ethanol gave the title compound (18.65 g, 90%) as white crystals.

M.p. 170-171 deg.C.

$[\alpha]_D$ +105 deg. (c = 10 mg.cm^{-3}, pyridine).

TLC (Chloroform/MeOH 4:1, silica gel): R_f 0.65.

^{1}H-NMR (DMSO-d_6): 7.78-7.46 (m, 4H, aromatics); 4.49 (d, J=3.2 Hz, 1H, H-1); 4.12 (dd, J=10.4 and 3.5 Hz, 1H, H-6); 4.03 (dd, J=10.4 and 8.2 Hz, 1H, H-6'); 3.73 (dd, J=8.2 and 3.5 Hz, 1H, H-5); 3.63 (d, J=2.8 Hz, 1H, H-4); 3.51 (dd, J=10.1 and 3.2 Hz, 1H, H-2); 3.45 (dd, J=10.1 and 2.8, 1H, H-3); 3.18 (s, 3H, OCH_3), 2.41 (s, 3H, CH_3).

^{13}C-NMR (DMSO-d_6): 145.2 (C-4'), 132.4 (C-1'), 130.3 (C-3' and C-5'), 127.7 (C-2' and C-6'), 100.0 (C-1), 71.0 (C-6), 68.9 (C-4), 68.8 (C-5), 68.3 (C-3), 67.9 (C-2), 54.6 (OCH_3), 21.2 (CH_3).

Anal. calc. for $C_{14}H_{20}O_8S$ (348.37): C C 48.27, H 5.79, S 9.20; found: C 48.39, H 5.85, S 9.01.

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References and Notes

1. Ohle, H.; Thiel, H. *Ber.* **1933**, *66B*, 525-532.
2. Bell, D. J.; Williamson, S. *J. Chem. Soc.* **1938**, 1196-1200.
3. It was dried at 90 deg.C *in vacuo* for 12 h before using.

Sample availability: Available from the authors and MDPI, MDPI Reg. No.13740.