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1,2:5,6-Di-*O*-isopropylidene-3-*O*-methylsulfonyl-α-D-glucofuranose

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.004 Å
R factor = 0.039
wR factor = 0.090
Data-to-parameter ratio = 12.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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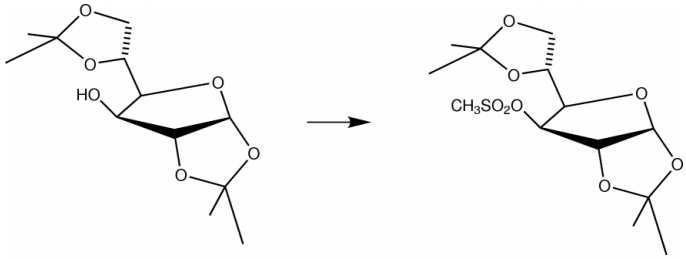
The absolute chemical configuration of the title compound, $C_{13}H_{22}O_8S$, was determined unambiguously by X-ray diffraction for the first time.

Comment

The analytical and physical data of the title compound, (II), are in accordance with those described by Gracza & Szolscanyi (2000). The absolute chemical configuration is in accordance with the NMR data (chemical shifts in the ^{1}H and ^{13}C NMR spectra as well as coupling constants in the ^{1}H NMR spectrum).



(I) (II)

Experimental

The title compound, (II), was prepared by the reaction of 1,2:5,6-di-*O*-isopropylidene-α-D-glucofuranose (I) (Hardegger *et al.*, 1957; Recondo & Rinderknecht, 1960) with methanesulfonyl chloride and pyridine at room temperature. ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.248 (*s*, 3H, $-CH_3$), 1.253 (s, 3H, $-CH_3$), 1.36 (*s*, 3H, $-CH_3$), 1.44 (*s*, 3H, $-CH_3$), 3.03 (*s*, 3H, $-SO_2CH_3$), 3.9 and 4.15 (*dd*, *J* = 4.2, 9.1 Hz, 2H, *ABX* system), 4.10 (*m*, 2H), 4.73 (*d*, *J* = 3.8 Hz, 1H), 4.91 (*d*, *J* = 2.6 Hz, 1H), 5.88 (*d*, *J* = 3.4 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 112.7, 109.6, 105.2, 83.7, 82.7, 79.8, 72.1, 67.6, 38.0, 26.9, 26.6, 26.2, 25.2.

Crystal data

$C_{13}H_{22}O_8S$
M_r = 338.37
Orthorhombic, $P2_12_12_1$
a = 8.866 (3) Å
b = 9.2962 (16) Å
c = 19.361 (3) Å
V = 1595.8 (7) Å^3
Z = 4
D_x = 1.408 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 5000 reflections
θ = 2.5–25.0°
μ = 0.24 mm^{-1}
T = 150 (2) K
Block, white
0.48 × 0.40 × 0.20 mm

Data collection

Stoe IPDS diffractometer
φ scans
6444 measured reflections
2686 independent reflections
2236 reflections with $I > 2\sigma(I)$
R_{int} = 0.086
θ_{max} = 25.3°
h = −10 → 10
k = −11 → 8
l = −19 → 23

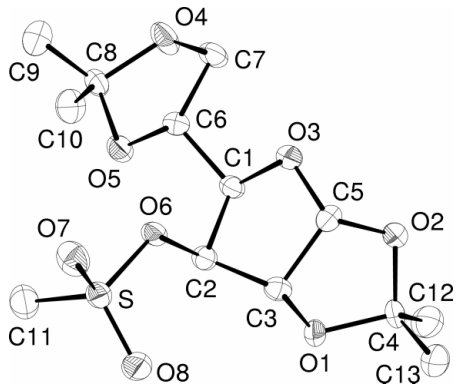


Figure 1
Molecular structure of (II), showing 60% probability displacement ellipsoids. H atoms have been omitted for clarity.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.090$
$S = 0.93$
2686 reflections
209 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0459P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.005$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.43$ e Å^{-3}
Extinction correction: *SHELXL*
Extinction coefficient: 0.010 (2)
Absolute structure: Flack (1983)
Flack parameter = −0.03 (10)

H atoms were constrained as riding atoms, with C—H = 1.00, 0.99 and 0.98 Å in the methine, methylene and methyl groups, respectively, and $U_{iso}(H) = 1.2U_{eq}$(parent atom).

Data collection: *IPDS* (Stoe & Cie, 1997); cell refinement: *IPDS*; data reduction: *IPDS*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *SHELXL*97.

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