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1,3,4,6-Tetra-*O*-acetyl-2-(trifluoromethylsulfonyl)-β-D-mannopyranose

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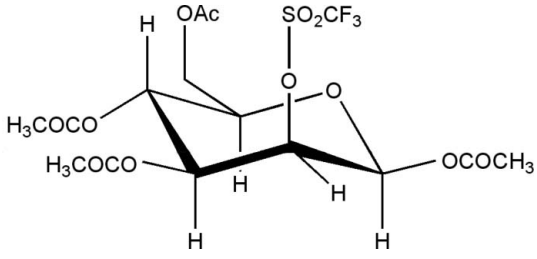
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.007 Å; disorder in main residue; R factor = 0.041; wR factor = 0.102; data-to-parameter ratio = 6.7.

Two independent molecules comprise the asymmetric unit of the title compound, $C_{15}H_{19}F_3O_{12}S$, which was synthesized by reacting 1,3,4,6-tetra-*O*-acetyl-β-D-mannopyranose with trifluoromethanesulfonic anhydride. In one molecule three F atoms and one O of the $-SO_2CF_3$ side chains are disordered equally over two positions; in the other molecule the three F atoms are equally disordered over two positions.

Related literature

For background literature, see: Fowler & Wolf (1986, 1997). For the synthesis, see Pavliak & Kováč (1991).



Experimental

Crystal data

$C_{15}H_{19}F_3O_{12}S$
M_r = 480.36
Triclinic, $P\overline{1}$
a = 7.885 (2) Å
b = 8.542 (2) Å
c = 15.629 (4) Å
α = 99.103 (4)°
β = 90.958 (4)°
γ = 90.271 (4)°
V = 1039.2 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 293 (2) K
0.26 × 0.24 × 0.20 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.940, T_{max} = 0.953
5327 measured reflections
4322 independent reflections
3520 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.102
S = 1.05
4322 reflections
647 parameters
197 restraints
H-atom paramteres constrained
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}
Absolute structure: Flack (1983), 678 Friedel Pairs
Flack parameter: 0.02 (10)

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2374).

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supplementary materials

Acta Cryst. (2007). E**63**, o2833 [**doi:10.1107/S1600536807019770**]

1,3,4,6-Tetra-*O*-acetyl-2-(trifluoromethylsulfonyl)-β-D-mannopyranose

H.-Y. Zhu and S.-D. Jiang

Comment

1,3,4,6-Tetra-*O*-acetyl-2-trifluoromethansulfonyl-beta-*D*-mannopyranose (I) (Fowler *et al.*, 1986) is used for the synthesis of 2-deoxy-2-[^{18}F]fluoro-D-glucose (Fowler *et al.*, 1997) which is a widely used radiopharmaceutical for tumor treatment using positron emission tomography (PET). We have prepared (I) from 1,3,4,6-tetra-*O*-acetyl-beta-*D*-mannopyranose and in our recently developped process to synthesize 2-deoxy-2-[^{18}F]fluoro-D-glucose using (I) as a staring material, the crystal structure of (I) has been determined. A view of the molecular structure of (I) is shown in Fig. 1. The trifluoromethansulfonyl side chains are disordered in both molecules. With no H bond interactions, the crystal structure must be stablized by van der Waals forces. The absolute configuration of the stereogenic centres are as follows: C1 S, C2 S, C3 S, C4 *R*, C5 *R*, C16 S, C17 S, C18 S, C19 *R*, C20 *R*.

Experimental

The title compound was synthesized according to the procedure of Pavliak *et al.* (1991). Crystals of (I) suitable for X-Ray analysis were grown by slow evaporation of a mixture of ethyl acetate (5 ml) and petroleum ether (10 ml) at room temperature over a period of 5 days.

Refinement

The H atoms were positioned geometrically (C—H = 0.96–0.98 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

Figures

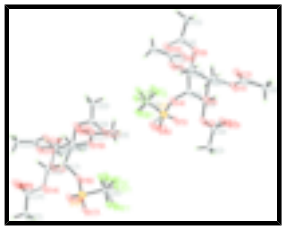


Fig. 1. The molecular structure of (I), shown with 30% probability displacement ellipsoids (arbitrary spheres for H atoms).

1,3,4,6-Tetra-*O*-acetyl-2-(trifluoromethylsulfonyl)-β-*D*-mannopyranose

Crystal data

$C_{15}H_{19}F_3O_{12}S$	$Z = 2$
$M_r = 480.36$	$F_{000} = 496$

Triclinic, *P*1	D_x = 1.535 Mg m^{-3}
Hall symbol: P 1	Melting point = 394–395 K
a = 7.885 (2) Å	Mo *K*α radiation λ = 0.71073 Å
b = 8.542 (2) Å	Cell parameters from 2166 reflections
c = 15.629 (4) Å	θ = 2.6–22.8º
α = 99.103 (4)º	μ = 0.24 mm^{-1}
β = 90.958 (4)º	*T* = 293 (2) K
γ = 90.271 (4)º	Block, colourless
V = 1039.2 (5) Å^3	0.26 × 0.24 × 0.20 mm

Data collection

Bruker SMART CCD diffractometer	4322 independent reflections
Radiation source: fine-focus sealed tube	3520 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.022
T = 293(2) K	θ_{max} = 25.0º
ω scans	θ_{min} = 1.3º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	*h* = −9→6
T_{min} = 0.940, T_{max} = 0.953	*k* = −10→9
5327 measured reflections	*l* = −17→18

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.041	$w = 1/[\sigma^2(F_o^2) + (0.0498P)^2 + 0.1781P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.102	$(\Delta/\sigma)_{max}$ = 0.003
S = 1.05	$\Delta\rho_{max}$ = 0.22 e Å^{-3}
4322 reflections	$\Delta\rho_{min}$ = −0.27 e Å^{-3}
647 parameters	Extinction correction: none
197 restraints	Absolute structure: Flack (1983), 678 Friedel Pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.02 (10)
Secondary atom site location: difference Fourier map	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
S1	−0.02275 (18)	0.39160 (14)	0.59984 (9)	0.0604 (4)	
S2	0.30286 (17)	1.08151 (14)	0.27349 (9)	0.0575 (4)	
F1	−0.2372 (11)	0.1807 (9)	0.5496 (6)	0.070 (3)	0.50
F2	−0.0571 (11)	0.1715 (9)	0.4551 (5)	0.085 (3)	0.50
F3	−0.2327 (16)	0.3640 (14)	0.4687 (8)	0.098 (5)	0.50
F1'	−0.2084 (14)	0.1471 (9)	0.5269 (7)	0.095 (4)	0.50
F2'	−0.0168 (11)	0.2427 (12)	0.4501 (5)	0.097 (3)	0.50
F3'	−0.2485 (14)	0.3694 (12)	0.4820 (8)	0.079 (4)	0.50
F4	0.4851 (15)	0.9080 (12)	0.3465 (8)	0.087 (4)	0.50
F5	0.2613 (13)	0.9564 (11)	0.4163 (7)	0.079 (3)	0.50
F6	0.4972 (12)	1.1337 (10)	0.4036 (6)	0.088 (3)	0.50
F4'	0.4747 (16)	0.8990 (12)	0.3704 (8)	0.077 (3)	0.50
F5'	0.2438 (13)	1.0102 (12)	0.4231 (7)	0.079 (3)	0.50
F6'	0.4299 (13)	1.1494 (11)	0.4317 (6)	0.087 (3)	0.50
O1	0.3293 (4)	0.2628 (3)	0.77341 (18)	0.0414 (7)	
O2	0.1262 (4)	0.4414 (3)	0.7840 (2)	0.0471 (8)	
O3	0.0199 (15)	0.4611 (11)	0.9144 (5)	0.070 (3)	0.50
O3'	−0.0585 (14)	0.4445 (12)	0.8892 (8)	0.084 (3)	0.50
O4	0.1010 (4)	0.2671 (3)	0.62718 (19)	0.0450 (8)	
O5	−0.1479 (6)	0.4347 (5)	0.6611 (3)	0.0928 (15)	
O6	0.0638 (16)	0.5204 (12)	0.5858 (7)	0.072 (3)	0.50
O6'	0.0854 (18)	0.4832 (14)	0.5478 (8)	0.084 (4)	0.50
O7	0.0675 (4)	−0.0513 (3)	0.59600 (18)	0.0435 (7)	
O8	−0.1597 (5)	−0.1509 (5)	0.6510 (3)	0.0739 (11)	
O9	0.3581 (4)	−0.1525 (3)	0.68326 (19)	0.0485 (8)	
O10	0.4592 (6)	−0.1711 (5)	0.5502 (2)	0.0789 (13)	
O11	0.6069 (4)	0.1596 (4)	0.8673 (2)	0.0589 (9)	
O12	0.8653 (5)	0.0630 (4)	0.8535 (2)	0.0682 (10)	
O13	−0.0337 (4)	0.8573 (3)	0.09217 (18)	0.0433 (7)	
O14	0.1755 (4)	1.0269 (3)	0.0859 (2)	0.0490 (8)	
O15	0.3477 (16)	0.9738 (15)	−0.0237 (9)	0.085 (4)	0.40
O15'	0.2645 (13)	0.9757 (9)	−0.0476 (4)	0.077 (3)	0.60
O16	0.1826 (4)	0.9410 (3)	0.24162 (19)	0.0437 (7)	
O17	0.2047 (6)	1.2164 (4)	0.2950 (3)	0.0832 (13)	
O18	0.4455 (5)	1.0799 (5)	0.2206 (3)	0.0852 (13)	
O19	0.2162 (4)	0.6368 (3)	0.26959 (18)	0.0445 (8)	
O20	0.4479 (6)	0.5157 (5)	0.2153 (3)	0.0816 (12)	
O21	−0.0754 (4)	0.4915 (3)	0.18026 (19)	0.0470 (8)	
O22	−0.1672 (6)	0.5425 (5)	0.3149 (2)	0.0806 (12)	
O23	−0.3150 (4)	0.7048 (4)	−0.0025 (2)	0.0603 (9)	

O24	−0.5749 (5)	0.6116 (5)	0.0092 (3)	0.0723 (11)
C1	0.1529 (6)	0.2768 (5)	0.7796 (3)	0.0400 (11)
H1	0.1131	0.2403	0.8322	0.048*
C2	0.0628 (5)	0.1897 (4)	0.7007 (3)	0.0360 (10)
H2	−0.0598	0.1909	0.7100	0.043*
C3	0.1234 (6)	0.0205 (5)	0.6802 (3)	0.0368 (10)
H3	0.0768	−0.0399	0.7228	0.044*
C4	0.3146 (6)	0.0103 (5)	0.6835 (3)	0.0388 (10)
H4	0.3645	0.0503	0.6341	0.047*
C5	0.3806 (6)	0.1027 (5)	0.7684 (3)	0.0402 (10)
H5	0.3319	0.0581	0.8166	0.048*
C6	0.5699 (6)	0.1050 (6)	0.7775 (3)	0.0504 (12)
H6A	0.6152	−0.0004	0.7600	0.061*
H6B	0.6192	0.1760	0.7419	0.061*
C7	0.7565 (6)	0.1263 (5)	0.8976 (3)	0.0462 (11)
C8	0.7708 (7)	0.1779 (8)	0.9924 (3)	0.0778 (18)
H8A	0.8816	0.1528	1.0125	0.117*
H8B	0.6864	0.1242	1.0208	0.117*
H8C	0.7537	0.2903	1.0054	0.117*
C9	0.0366 (8)	0.5174 (6)	0.8494 (3)	0.0575 (14)
C10	0.0188 (7)	0.6868 (5)	0.8422 (4)	0.0613 (14)
H10A	−0.0649	0.7337	0.8821	0.092*
H10B	−0.0160	0.6968	0.7842	0.092*
H10C	0.1257	0.7400	0.8557	0.092*
C11	−0.1335 (7)	0.2770 (6)	0.5116 (3)	0.0723 (17)
C12	−0.0774 (7)	−0.1369 (5)	0.5893 (3)	0.0465 (12)
C13	−0.1140 (7)	−0.2048 (6)	0.4981 (3)	0.0614 (14)
H13A	−0.2319	−0.2336	0.4913	0.092*
H13B	−0.0459	−0.2972	0.4820	0.092*
H13C	−0.0882	−0.1277	0.4616	0.092*
C14	0.4319 (6)	−0.2312 (6)	0.6123 (3)	0.0503 (13)
C15	0.4722 (8)	−0.3947 (6)	0.6236 (4)	0.0735 (17)
H15A	0.4723	−0.4614	0.5681	0.110*
H15B	0.3884	−0.4327	0.6594	0.110*
H15C	0.5820	−0.3968	0.6508	0.110*
C16	0.1418 (6)	0.8664 (5)	0.0881 (3)	0.0414 (11)
H16	0.1825	0.8001	0.0356	0.050*
C17	0.2274 (6)	0.8213 (5)	0.1680 (3)	0.0396 (10)
H17	0.3507	0.8182	0.1611	0.048*
C18	0.1624 (6)	0.6630 (5)	0.1861 (3)	0.0395 (11)
H18	0.2080	0.5789	0.1428	0.047*
C19	−0.0287 (6)	0.6536 (5)	0.1815 (3)	0.0381 (10)
H19	−0.0787	0.7218	0.2309	0.046*
C20	−0.0891 (6)	0.6993 (5)	0.0971 (3)	0.0412 (10)
H20	−0.0416	0.6261	0.0489	0.049*
C21	−0.2793 (6)	0.6997 (6)	0.0875 (3)	0.0502 (12)
H21A	−0.3278	0.6048	0.1043	0.060*
H21B	−0.3263	0.7916	0.1236	0.060*
C22	−0.4661 (6)	0.6519 (6)	−0.0341 (3)	0.0515 (12)

C23	−0.4770 (8)	0.6516 (8)	−0.1288 (4)	0.0798 (18)
H23A	−0.3842	0.5922	−0.1564	0.120*
H23B	−0.4715	0.7586	−0.1403	0.120*
H23C	−0.5823	0.6036	−0.1512	0.120*
C24	0.2547 (7)	1.0678 (6)	0.0179 (3)	0.0561 (13)
C25	0.2774 (8)	1.2410 (5)	0.0276 (4)	0.0614 (14)
H25A	0.3250	1.2682	−0.0243	0.092*
H25B	0.3526	1.2760	0.0759	0.092*
H25C	0.1695	1.2916	0.0375	0.092*
C26	0.3761 (7)	1.0261 (6)	0.3745 (4)	0.0744 (18)
C27	0.3637 (7)	0.5579 (5)	0.2761 (3)	0.0469 (12)
C28	0.4025 (8)	0.5389 (6)	0.3675 (3)	0.0627 (15)
H28A	0.4562	0.4385	0.3685	0.094*
H28B	0.2992	0.5431	0.3994	0.094*
H28C	0.4773	0.6228	0.3935	0.094*
C29	−0.1476 (6)	0.4521 (6)	0.2528 (3)	0.0532 (13)
C30	−0.1937 (8)	0.2835 (6)	0.2386 (4)	0.0713 (16)
H30A	−0.1988	0.2465	0.2934	0.107*
H30B	−0.1100	0.2244	0.2034	0.107*
H30C	−0.3024	0.2692	0.2098	0.107*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0637 (9)	0.0489 (7)	0.0696 (9)	0.0102 (6)	−0.0230 (7)	0.0150 (6)
S2	0.0620 (9)	0.0460 (7)	0.0648 (8)	−0.0196 (6)	−0.0161 (7)	0.0117 (5)
F1	0.068 (5)	0.051 (4)	0.087 (5)	−0.004 (4)	−0.007 (4)	0.000 (4)
F2	0.096 (6)	0.082 (5)	0.074 (5)	0.006 (4)	−0.012 (4)	0.001 (4)
F3	0.098 (8)	0.115 (8)	0.086 (7)	0.024 (6)	−0.034 (6)	0.031 (5)
F1'	0.113 (7)	0.054 (5)	0.111 (7)	0.025 (4)	−0.049 (5)	−0.009 (4)
F2'	0.105 (6)	0.121 (7)	0.058 (4)	0.036 (5)	0.001 (4)	−0.009 (5)
F3'	0.084 (7)	0.090 (6)	0.065 (5)	0.021 (5)	−0.038 (5)	0.017 (4)
F4	0.087 (6)	0.082 (5)	0.101 (7)	−0.005 (4)	−0.033 (5)	0.047 (4)
F5	0.096 (6)	0.078 (6)	0.065 (5)	0.011 (5)	−0.013 (4)	0.014 (4)
F6	0.094 (6)	0.081 (5)	0.084 (6)	−0.023 (4)	−0.034 (5)	0.000 (4)
F4'	0.083 (6)	0.085 (6)	0.067 (5)	0.008 (5)	−0.010 (4)	0.028 (4)
F5'	0.098 (6)	0.082 (6)	0.060 (5)	0.024 (5)	0.005 (4)	0.020 (4)
F6'	0.098 (6)	0.084 (5)	0.069 (5)	−0.006 (5)	−0.027 (5)	−0.015 (4)
O1	0.0347 (18)	0.0427 (16)	0.0462 (17)	0.0050 (14)	−0.0028 (14)	0.0052 (13)
O2	0.056 (2)	0.0376 (16)	0.0471 (19)	0.0096 (15)	0.0068 (16)	0.0026 (13)
O3	0.104 (7)	0.063 (5)	0.042 (4)	0.019 (5)	0.013 (4)	0.006 (4)
O3'	0.088 (7)	0.063 (5)	0.102 (7)	0.008 (5)	0.039 (6)	0.010 (5)
O4	0.0440 (19)	0.0519 (16)	0.0411 (17)	0.0108 (15)	−0.0024 (14)	0.0139 (14)
O5	0.084 (3)	0.111 (3)	0.074 (3)	0.065 (3)	−0.019 (2)	−0.014 (2)
O6	0.090 (6)	0.048 (5)	0.084 (6)	0.001 (4)	−0.008 (5)	0.026 (4)
O6'	0.091 (7)	0.079 (6)	0.091 (7)	−0.004 (5)	−0.017 (6)	0.048 (5)
O7	0.0473 (19)	0.0488 (16)	0.0327 (15)	−0.0019 (15)	−0.0050 (14)	0.0020 (13)
O8	0.063 (3)	0.097 (3)	0.059 (2)	−0.026 (2)	0.001 (2)	0.005 (2)

O9	0.058 (2)	0.0460 (16)	0.0414 (17)	0.0168 (15)	0.0015 (15)	0.0066 (14)
O10	0.097 (3)	0.092 (3)	0.046 (2)	0.040 (2)	0.009 (2)	0.002 (2)
O11	0.0363 (19)	0.090 (2)	0.0448 (19)	0.0184 (18)	−0.0064 (15)	−0.0054 (16)
O12	0.044 (2)	0.092 (3)	0.066 (2)	0.014 (2)	−0.0028 (18)	0.006 (2)
O13	0.0420 (19)	0.0479 (17)	0.0423 (16)	−0.0030 (14)	−0.0003 (14)	0.0146 (13)
O14	0.064 (2)	0.0408 (16)	0.0437 (18)	−0.0073 (15)	0.0129 (16)	0.0115 (13)
O15	0.082 (7)	0.080 (6)	0.094 (8)	−0.001 (6)	0.037 (6)	0.013 (6)
O15'	0.118 (6)	0.070 (4)	0.042 (4)	−0.033 (4)	0.016 (4)	0.007 (3)
O16	0.0446 (19)	0.0471 (16)	0.0384 (16)	−0.0102 (14)	0.0014 (14)	0.0035 (13)
O17	0.098 (3)	0.043 (2)	0.105 (3)	−0.003 (2)	−0.035 (3)	0.0041 (19)
O18	0.065 (3)	0.091 (3)	0.103 (3)	−0.042 (2)	0.003 (2)	0.026 (2)
O19	0.047 (2)	0.0536 (17)	0.0333 (16)	−0.0029 (16)	−0.0033 (14)	0.0082 (13)
O20	0.074 (3)	0.113 (3)	0.062 (2)	0.038 (3)	0.002 (2)	0.025 (2)
O21	0.052 (2)	0.0489 (18)	0.0405 (17)	−0.0148 (15)	−0.0002 (15)	0.0096 (13)
O22	0.104 (3)	0.094 (3)	0.044 (2)	−0.032 (2)	0.015 (2)	0.009 (2)
O23	0.041 (2)	0.098 (3)	0.0454 (18)	−0.0156 (19)	−0.0113 (16)	0.0230 (17)
O24	0.045 (2)	0.098 (3)	0.076 (3)	−0.014 (2)	0.002 (2)	0.024 (2)
C1	0.045 (3)	0.041 (2)	0.034 (2)	0.009 (2)	0.003 (2)	0.0040 (18)
C2	0.033 (2)	0.040 (2)	0.035 (2)	0.0086 (19)	0.0017 (19)	0.0068 (18)
C3	0.038 (3)	0.041 (2)	0.030 (2)	0.001 (2)	−0.0017 (18)	0.0031 (18)
C4	0.046 (3)	0.041 (2)	0.031 (2)	0.011 (2)	0.0026 (19)	0.0074 (18)
C5	0.035 (2)	0.049 (2)	0.037 (2)	0.004 (2)	0.0025 (19)	0.0068 (19)
C6	0.041 (3)	0.069 (3)	0.039 (3)	0.014 (2)	0.002 (2)	0.003 (2)
C7	0.034 (3)	0.054 (3)	0.051 (3)	0.001 (2)	−0.001 (2)	0.010 (2)
C8	0.052 (4)	0.123 (5)	0.056 (3)	0.007 (4)	−0.012 (3)	0.008 (3)
C9	0.071 (4)	0.046 (3)	0.056 (3)	0.011 (3)	0.016 (3)	0.007 (2)
C10	0.062 (4)	0.046 (3)	0.071 (3)	0.010 (2)	−0.004 (3)	−0.003 (2)
C11	0.076 (4)	0.077 (4)	0.065 (4)	0.029 (4)	−0.016 (3)	0.014 (3)
C12	0.056 (3)	0.033 (2)	0.050 (3)	0.004 (2)	−0.008 (3)	0.005 (2)
C13	0.070 (4)	0.056 (3)	0.054 (3)	−0.002 (3)	−0.022 (3)	−0.003 (2)
C14	0.046 (3)	0.057 (3)	0.044 (3)	0.018 (2)	−0.012 (2)	−0.006 (2)
C15	0.072 (4)	0.065 (3)	0.077 (4)	0.024 (3)	−0.014 (3)	−0.010 (3)
C16	0.041 (3)	0.040 (2)	0.045 (3)	−0.006 (2)	0.005 (2)	0.0086 (19)
C17	0.032 (2)	0.047 (2)	0.040 (2)	−0.009 (2)	0.0052 (19)	0.0048 (19)
C18	0.047 (3)	0.041 (2)	0.030 (2)	−0.002 (2)	0.0011 (19)	0.0062 (18)
C19	0.040 (3)	0.043 (2)	0.031 (2)	−0.006 (2)	0.0013 (19)	0.0060 (18)
C20	0.036 (3)	0.050 (2)	0.037 (2)	−0.004 (2)	0.0010 (19)	0.0081 (19)
C21	0.041 (3)	0.071 (3)	0.040 (3)	−0.014 (2)	0.000 (2)	0.014 (2)
C22	0.039 (3)	0.065 (3)	0.050 (3)	0.003 (3)	−0.006 (2)	0.006 (2)
C23	0.050 (4)	0.129 (5)	0.059 (4)	0.002 (4)	−0.011 (3)	0.009 (3)
C24	0.062 (4)	0.058 (3)	0.051 (3)	−0.004 (3)	0.012 (3)	0.016 (3)
C25	0.066 (4)	0.051 (3)	0.073 (4)	−0.009 (3)	0.006 (3)	0.026 (2)
C26	0.083 (5)	0.058 (3)	0.078 (4)	−0.011 (4)	−0.036 (4)	−0.001 (3)
C27	0.055 (3)	0.042 (2)	0.046 (3)	0.003 (2)	−0.005 (2)	0.012 (2)
C28	0.083 (4)	0.057 (3)	0.049 (3)	−0.004 (3)	−0.017 (3)	0.013 (2)
C29	0.050 (3)	0.072 (3)	0.039 (3)	−0.027 (3)	−0.012 (2)	0.018 (2)
C30	0.069 (4)	0.076 (4)	0.075 (4)	−0.027 (3)	−0.011 (3)	0.031 (3)

Geometric parameters (Å, °)

S1—O6	1.342 (11)	C2—C3	1.512 (5)
S1—O5	1.396 (5)	C2—H2	0.9800
S1—O6'	1.489 (13)	C3—C4	1.511 (6)
S1—O4	1.551 (3)	C3—H3	0.9800
S1—C11	1.774 (6)	C4—C5	1.516 (6)
S2—O17	1.391 (4)	C4—H4	0.9800
S2—O18	1.405 (4)	C5—C6	1.497 (6)
S2—O16	1.540 (3)	C5—H5	0.9800
S2—C26	1.805 (6)	C6—H6A	0.9700
F1—C11	1.365 (8)	C6—H6B	0.9700
F2—C11	1.313 (7)	C7—C8	1.479 (7)
F3—C11	1.326 (8)	C8—H8A	0.9600
F1'—C11	1.312 (8)	C8—H8B	0.9600
F2'—C11	1.341 (8)	C8—H8C	0.9600
F3'—C11	1.329 (8)	C9—C10	1.476 (7)
F4—C26	1.353 (9)	C10—H10A	0.9600
F5—C26	1.318 (9)	C10—H10B	0.9600
F6—C26	1.344 (8)	C10—H10C	0.9600
F4'—C26	1.332 (9)	C12—C13	1.475 (7)
F5'—C26	1.320 (9)	C13—H13A	0.9600
F6'—C26	1.331 (8)	C13—H13B	0.9600
O1—C1	1.400 (5)	C13—H13C	0.9600
O1—C5	1.418 (5)	C14—C15	1.471 (7)
O2—C9	1.335 (6)	C15—H15A	0.9600
O2—C1	1.413 (5)	C15—H15B	0.9600
O3—C9	1.201 (8)	C15—H15C	0.9600
O3'—C9	1.213 (8)	C16—C17	1.513 (6)
O4—C2	1.449 (5)	C16—H16	0.9800
O7—C12	1.347 (6)	C17—C18	1.513 (6)
O7—C3	1.423 (5)	C17—H17	0.9800
O8—C12	1.192 (6)	C18—C19	1.509 (6)
O9—C14	1.346 (6)	C18—H18	0.9800
O9—C4	1.433 (5)	C19—C20	1.504 (6)
O10—C14	1.189 (6)	C19—H19	0.9800
O11—C7	1.313 (6)	C20—C21	1.505 (6)
O11—C6	1.432 (5)	C20—H20	0.9800
O12—C7	1.187 (6)	C21—H21A	0.9700
O13—C16	1.388 (5)	C21—H21B	0.9700
O13—C20	1.431 (5)	C22—C23	1.480 (7)
O14—C24	1.334 (6)	C23—H23A	0.9599
O14—C16	1.401 (5)	C23—H23B	0.9600
O15—C24	1.208 (9)	C23—H23C	0.9600
O15'—C24	1.192 (7)	C24—C25	1.473 (7)
O16—C17	1.463 (5)	C25—H25A	0.9600
O19—C27	1.358 (6)	C25—H25B	0.9600
O19—C18	1.417 (5)	C25—H25C	0.9600

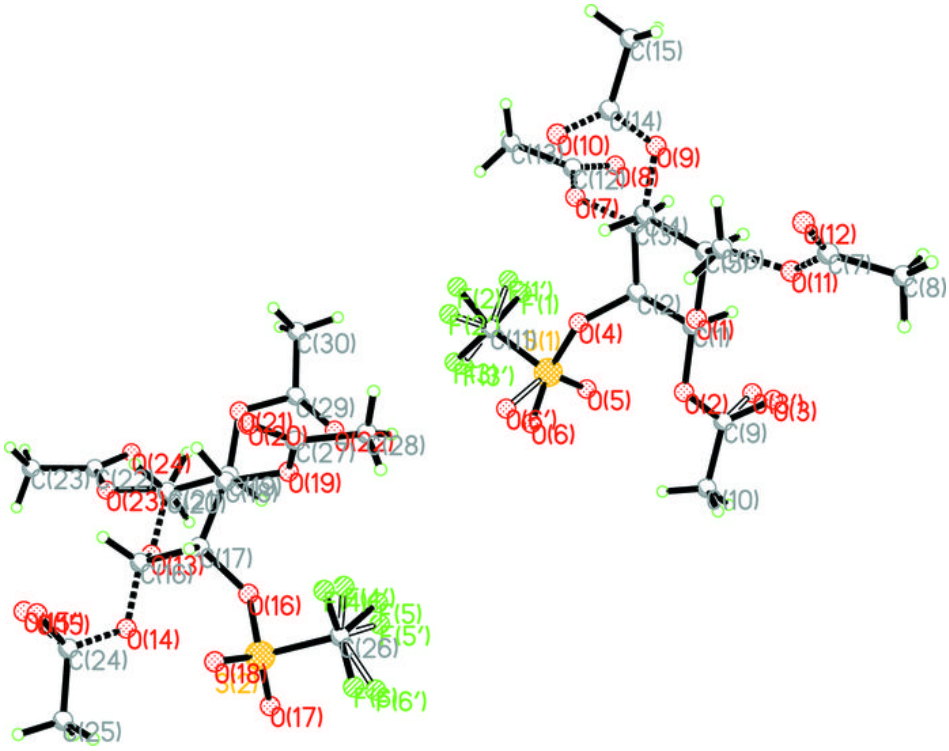
O20—C27	1.179 (6)	C27—C28	1.489 (7)
O21—C29	1.364 (6)	C28—H28A	0.9600
O21—C19	1.428 (5)	C28—H28B	0.9600
O22—C29	1.154 (6)	C28—H28C	0.9600
O23—C22	1.332 (6)	C29—C30	1.465 (7)
O23—C21	1.437 (5)	C30—H30A	0.9600
O24—C22	1.185 (6)	C30—H30B	0.9600
C1—C2	1.501 (6)	C30—H30C	0.9600
C1—H1	0.9800		
O6—S1—O5	110.1 (5)	C12—C13—H13B	109.5
O6—S1—O6'	26.0 (6)	H13A—C13—H13B	109.5
O5—S1—O6'	133.4 (5)	C12—C13—H13C	109.5
O6—S1—O4	110.0 (5)	H13A—C13—H13C	109.5
O5—S1—O4	112.4 (2)	H13B—C13—H13C	109.5
O6'—S1—O4	103.3 (5)	O10—C14—O9	122.4 (4)
O6—S1—C11	118.7 (5)	O10—C14—C15	126.6 (5)
O5—S1—C11	104.1 (3)	O9—C14—C15	111.0 (5)
O6'—S1—C11	97.2 (5)	C14—C15—H15A	109.5
O4—S1—C11	101.25 (19)	C14—C15—H15B	109.5
O17—S2—O18	122.0 (3)	H15A—C15—H15B	109.5
O17—S2—O16	108.0 (2)	C14—C15—H15C	109.5
O18—S2—O16	111.5 (2)	H15A—C15—H15C	109.5
O17—S2—C26	106.5 (3)	H15B—C15—H15C	109.5
O18—S2—C26	106.6 (3)	O13—C16—O14	104.4 (3)
O16—S2—C26	99.8 (2)	O13—C16—C17	111.8 (4)
C1—O1—C5	111.4 (3)	O14—C16—C17	108.5 (3)
C9—O2—C1	118.7 (4)	O13—C16—H16	110.7
C2—O4—S1	120.0 (3)	O14—C16—H16	110.7
C12—O7—C3	117.9 (4)	C17—C16—H16	110.7
C14—O9—C4	118.0 (4)	O16—C17—C16	107.5 (3)
C7—O11—C6	117.6 (4)	O16—C17—C18	107.4 (3)
C16—O13—C20	111.3 (3)	C16—C17—C18	111.1 (3)
C24—O14—C16	119.3 (4)	O16—C17—H17	110.3
C17—O16—S2	120.6 (3)	C16—C17—H17	110.3
C27—O19—C18	117.8 (3)	C18—C17—H17	110.3
C29—O21—C19	117.6 (4)	O19—C18—C19	108.2 (3)
C22—O23—C21	117.5 (4)	O19—C18—C17	110.6 (3)
O1—C1—O2	103.3 (3)	C19—C18—C17	111.8 (4)
O1—C1—C2	111.8 (3)	O19—C18—H18	108.7
O2—C1—C2	109.2 (3)	C19—C18—H18	108.7
O1—C1—H1	110.7	C17—C18—H18	108.7
O2—C1—H1	110.7	O21—C19—C20	107.3 (3)
C2—C1—H1	110.7	O21—C19—C18	107.3 (3)
O4—C2—C1	108.8 (3)	C20—C19—C18	108.9 (4)
O4—C2—C3	107.5 (3)	O21—C19—H19	111.1
C1—C2—C3	111.0 (3)	C20—C19—H19	111.1
O4—C2—H2	109.8	C18—C19—H19	111.1
C1—C2—H2	109.8	O13—C20—C19	109.5 (3)
C3—C2—H2	109.8	O13—C20—C21	106.1 (4)

O7—C3—C4	107.7 (3)	C19—C20—C21	113.2 (4)
O7—C3—C2	111.0 (3)	O13—C20—H20	109.3
C4—C3—C2	112.0 (3)	C19—C20—H20	109.3
O7—C3—H3	108.7	C21—C20—H20	109.3
C4—C3—H3	108.7	O23—C21—C20	106.1 (4)
C2—C3—H3	108.7	O23—C21—H21A	110.5
O9—C4—C3	107.7 (3)	C20—C21—H21A	110.5
O9—C4—C5	107.1 (3)	O23—C21—H21B	110.5
C3—C4—C5	109.3 (3)	C20—C21—H21B	110.5
O9—C4—H4	110.9	H21A—C21—H21B	108.7
C3—C4—H4	110.9	O24—C22—O23	123.3 (5)
C5—C4—H4	110.9	O24—C22—C23	125.9 (5)
O1—C5—C6	106.6 (4)	O23—C22—C23	110.8 (5)
O1—C5—C4	108.5 (3)	C22—C23—H23A	109.5
C6—C5—C4	113.9 (4)	C22—C23—H23B	109.5
O1—C5—H5	109.2	H23A—C23—H23B	109.5
C6—C5—H5	109.2	C22—C23—H23C	109.5
C4—C5—H5	109.2	H23A—C23—H23C	109.5
O11—C6—C5	106.0 (4)	H23B—C23—H23C	109.5
O11—C6—H6A	110.5	O15'—C24—O15	36.5 (7)
C5—C6—H6A	110.5	O15'—C24—O14	120.2 (6)
O11—C6—H6B	110.5	O15—C24—O14	119.3 (8)
C5—C6—H6B	110.5	O15'—C24—C25	126.5 (6)
H6A—C6—H6B	108.7	O15—C24—C25	123.7 (8)
O12—C7—O11	123.5 (5)	O14—C24—C25	111.0 (4)
O12—C7—C8	125.1 (5)	C24—C25—H25A	109.5
O11—C7—C8	111.4 (4)	C24—C25—H25B	109.5
C7—C8—H8A	109.5	H25A—C25—H25B	109.5
C7—C8—H8B	109.5	C24—C25—H25C	109.5
H8A—C8—H8B	109.5	H25A—C25—H25C	109.5
C7—C8—H8C	109.5	H25B—C25—H25C	109.5
H8A—C8—H8C	109.5	F5—C26—F5'	20.8 (7)
H8B—C8—H8C	109.5	F5—C26—F6'	104.4 (8)
O3—C9—O3'	35.1 (7)	F5'—C26—F6'	89.9 (8)
O3—C9—O2	120.2 (6)	F5—C26—F4'	90.2 (8)
O3'—C9—O2	120.4 (6)	F5'—C26—F4'	109.9 (9)
O3—C9—C10	125.5 (6)	F6'—C26—F4'	113.9 (8)
O3'—C9—C10	124.4 (7)	F5—C26—F6	131.0 (8)
O2—C9—C10	111.1 (4)	F5'—C26—F6	119.6 (8)
C9—C10—H10A	109.5	F6'—C26—F6	30.1 (6)
C9—C10—H10B	109.5	F4'—C26—F6	96.4 (8)
H10A—C10—H10B	109.5	F5—C26—F4	103.6 (8)
C9—C10—H10C	109.5	F5'—C26—F4	124.2 (9)
H10A—C10—H10C	109.5	F6'—C26—F4	119.5 (8)
H10B—C10—H10C	109.5	F4'—C26—F4	17.2 (9)
F1'—C11—F2	80.0 (7)	F6—C26—F4	95.3 (8)
F1'—C11—F3	112.8 (10)	F5—C26—S2	115.0 (6)
F2—C11—F3	108.5 (8)	F5'—C26—S2	108.9 (6)
F1'—C11—F3'	108.9 (8)	F6'—C26—S2	113.0 (6)

F2—C11—F3'	118.0 (9)	F4'—C26—S2	117.5 (6)
F3—C11—F3'	10.4 (11)	F6—C26—S2	104.5 (6)
F1'—C11—F2'	110.5 (7)	F4—C26—S2	101.5 (6)
F2—C11—F2'	30.6 (5)	O20—C27—O19	121.8 (4)
F3—C11—F2'	97.0 (8)	O20—C27—C28	126.7 (5)
F3'—C11—F2'	107.4 (8)	O19—C27—C28	111.5 (5)
F1'—C11—F1	20.5 (6)	C27—C28—H28A	109.5
F2—C11—F1	100.5 (6)	C27—C28—H28B	109.5
F3—C11—F1	107.0 (8)	H28A—C28—H28B	109.5
F3'—C11—F1	100.0 (8)	C27—C28—H28C	109.5
F2'—C11—F1	131.0 (7)	H28A—C28—H28C	109.5
F1'—C11—S1	116.8 (6)	H28B—C28—H28C	109.5
F2—C11—S1	122.2 (6)	O22—C29—O21	123.0 (5)
F3—C11—S1	112.7 (7)	O22—C29—C30	126.9 (5)
F3'—C11—S1	108.0 (6)	O21—C29—C30	110.1 (5)
F2'—C11—S1	104.7 (6)	C29—C30—H30A	109.5
F1—C11—S1	104.2 (5)	C29—C30—H30B	109.5
O8—C12—O7	122.1 (4)	H30A—C30—H30B	109.5
O8—C12—C13	127.4 (5)	C29—C30—H30C	109.5
O7—C12—C13	110.5 (5)	H30A—C30—H30C	109.5
C12—C13—H13A	109.5	H30B—C30—H30C	109.5
O6—S1—O4—C2	133.4 (5)	O6'—S1—C11—F1	178.3 (7)
O5—S1—O4—C2	10.3 (4)	O4—S1—C11—F1	73.2 (5)
O6'—S1—O4—C2	159.6 (5)	C3—O7—C12—O8	1.5 (6)
C11—S1—O4—C2	−100.2 (3)	C3—O7—C12—C13	−178.9 (4)
O17—S2—O16—C17	140.1 (3)	C4—O9—C14—O10	−1.2 (7)
O18—S2—O16—C17	3.4 (4)	C4—O9—C14—C15	178.5 (4)
C26—S2—O16—C17	−108.9 (3)	C20—O13—C16—O14	−178.5 (3)
C5—O1—C1—O2	179.9 (3)	C20—O13—C16—C17	−61.5 (4)
C5—O1—C1—C2	−62.7 (4)	C24—O14—C16—O13	−119.7 (4)
C9—O2—C1—O1	−126.7 (4)	C24—O14—C16—C17	121.0 (4)
C9—O2—C1—C2	114.2 (5)	S2—O16—C17—C16	−96.3 (4)
S1—O4—C2—C1	−95.6 (4)	S2—O16—C17—C18	144.1 (3)
S1—O4—C2—C3	144.1 (3)	O13—C16—C17—O16	−65.5 (4)
O1—C1—C2—O4	−66.7 (4)	O14—C16—C17—O16	49.1 (4)
O2—C1—C2—O4	47.1 (4)	O13—C16—C17—C18	51.7 (5)
O1—C1—C2—C3	51.5 (5)	O14—C16—C17—C18	166.3 (3)
O2—C1—C2—C3	165.3 (3)	C27—O19—C18—C19	145.2 (4)
C12—O7—C3—C4	142.6 (4)	C27—O19—C18—C17	−92.0 (4)
C12—O7—C3—C2	−94.5 (4)	O16—C17—C18—O19	−50.8 (5)
O4—C2—C3—O7	−48.1 (4)	C16—C17—C18—O19	−168.1 (4)
C1—C2—C3—O7	−167.1 (4)	O16—C17—C18—C19	69.8 (4)
O4—C2—C3—C4	72.4 (4)	C16—C17—C18—C19	−47.4 (5)
C1—C2—C3—C4	−46.6 (5)	C29—O21—C19—C20	−134.5 (4)
C14—O9—C4—C3	110.9 (4)	C29—O21—C19—C18	108.6 (4)
C14—O9—C4—C5	−131.6 (4)	O19—C18—C19—O21	−70.5 (4)
O7—C3—C4—O9	−70.7 (4)	C17—C18—C19—O21	167.5 (3)
C2—C3—C4—O9	166.9 (3)	O19—C18—C19—C20	173.7 (3)
O7—C3—C4—C5	173.3 (3)	C17—C18—C19—C20	51.6 (4)

C2—C3—C4—C5	50.9 (5)	C16—O13—C20—C19	65.9 (4)
C1—O1—C5—C6	−170.4 (3)	C16—O13—C20—C21	−171.6 (4)
C1—O1—C5—C4	66.5 (4)	O21—C19—C20—O13	−175.5 (3)
O9—C4—C5—O1	−176.0 (3)	C18—C19—C20—O13	−59.7 (4)
C3—C4—C5—O1	−59.6 (4)	O21—C19—C20—C21	66.3 (5)
O9—C4—C5—C6	65.4 (5)	C18—C19—C20—C21	−177.8 (4)
C3—C4—C5—C6	−178.2 (4)	C22—O23—C21—C20	157.2 (4)
C7—O11—C6—C5	159.1 (4)	O13—C20—C21—O23	74.3 (4)
O1—C5—C6—O11	74.9 (4)	C19—C20—C21—O23	−165.6 (4)
C4—C5—C6—O11	−165.5 (4)	C21—O23—C22—O24	5.2 (7)
C6—O11—C7—O12	5.2 (7)	C21—O23—C22—C23	−174.8 (5)
C6—O11—C7—C8	−175.1 (4)	C16—O14—C24—O15'	15.9 (9)
C1—O2—C9—O3	20.4 (10)	C16—O14—C24—O15	−26.4 (11)
C1—O2—C9—O3'	−20.5 (10)	C16—O14—C24—C25	179.7 (4)
C1—O2—C9—C10	−178.7 (4)	O17—S2—C26—F5	76.1 (6)
O6—S1—C11—F1'	176.2 (8)	O18—S2—C26—F5	−152.2 (6)
O5—S1—C11—F1'	−61.0 (7)	O16—S2—C26—F5	−36.1 (6)
O6'—S1—C11—F1'	160.8 (8)	O17—S2—C26—F5'	54.7 (6)
O4—S1—C11—F1'	55.7 (7)	O18—S2—C26—F5'	−173.7 (6)
O6—S1—C11—F2	81.2 (8)	O16—S2—C26—F5'	−57.6 (6)
O5—S1—C11—F2	−156.0 (6)	O17—S2—C26—F6'	−43.6 (7)
O6'—S1—C11—F2	65.9 (8)	O18—S2—C26—F6'	88.0 (7)
O4—S1—C11—F2	−39.2 (6)	O16—S2—C26—F6'	−155.9 (6)
O6—S1—C11—F3	−50.8 (10)	O17—S2—C26—F4'	−179.6 (8)
O5—S1—C11—F3	72.0 (8)	O18—S2—C26—F4'	−47.9 (8)
O6'—S1—C11—F3	−66.1 (9)	O16—S2—C26—F4'	68.2 (8)
O4—S1—C11—F3	−171.2 (7)	O17—S2—C26—F6	−74.2 (6)
O6—S1—C11—F3'	−60.7 (9)	O18—S2—C26—F6	57.5 (6)
O5—S1—C11—F3'	62.1 (7)	O16—S2—C26—F6	173.6 (5)
O6'—S1—C11—F3'	−76.1 (8)	O17—S2—C26—F4	−172.9 (6)
O4—S1—C11—F3'	178.8 (7)	O18—S2—C26—F4	−41.2 (7)
O6—S1—C11—F2'	53.6 (8)	O16—S2—C26—F4	74.9 (7)
O5—S1—C11—F2'	176.4 (5)	C18—O19—C27—O20	2.0 (6)
O6'—S1—C11—F2'	38.2 (7)	C18—O19—C27—C28	−179.7 (4)
O4—S1—C11—F2'	−66.9 (5)	C19—O21—C29—O22	−2.4 (7)
O6—S1—C11—F1	−166.4 (7)	C19—O21—C29—C30	177.5 (4)
O5—S1—C11—F1	−43.6 (5)		

Fig. 1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