Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3,4-*O*-Carbonyl-1,2:5,6-di-*O*-isopropylidene-D-mannitol

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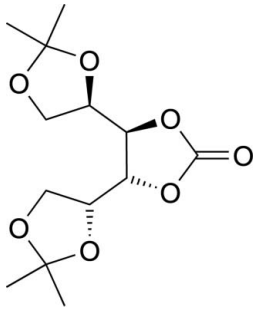
Received 23 July 2007; accepted 21 August 2007

Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.005 Å; R factor = 0.045; wR factor = 0.114; data-to-parameter ratio = 10.0.

The title compound, $C_{13}H_{20}O_7$, the carbonate of a partially protected sugar alcohol, was obtained accidentally in the attempted preparation of an orthocarbonate thereof. The five-membered 1,3-dioxolane rings adopt twist and envelope conformations.

Related literature

For related literature, see: Hough *et al.* (1962); Baker & Sachdev (1963); Mues & Buysch (1990). For analysis tools, see: Cremer & Pople (1975).



Experimental

Crystal data

$C_{13}H_{20}O_7$
M_r = 288.29
Orthorhombic, $P2_12_12_1$
a = 6.0863 (3) Å
b = 11.6958 (4) Å
c = 19.6816 (8) Å
V = 1401.02 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 200 (2) K
0.16 × 0.04 × 0.04 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Absorption correction: none
3191 measured reflections
1858 independent reflections
1153 reflections with $I > 2\sigma(I)$
R_{int} = 0.059

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.114
S = 1.00
1858 reflections
186 parameters
Only H-atom displacement parameters refined
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

Data collection: *COLLECT* (Nonius, 2004); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97* (Sheldrick, 1997) and *PLATON* (Spek, 2003).

The authors thank Phillipp Lorenz and Dr Peter Mayer for professional support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2451).

References

Baker, B. R. & Sachdev, H. S. (1963). *J. Org. Chem.* **28**, 2135–2139.
Burnett, M. N. & Johnson, C. K. (1996). *ORTEPIII*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Hough, L., Priddle, J. E. & Theobald, R. S. (1962). *J. Chem. Soc.* pp. 1934–1938.
Mues, P. & Buysch, H.-J. (1990). *Synthesis*, pp. 249–252.
Nonius (2004). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

  doi:10.1107/S1600536807041244

supplementary materials

Acta Cryst. (2007). E**63**, o3890 [**doi:10.1107/S1600536807041244**]

3,4-*O*-Carbonyl-1,2:5,6-di-*O*-isopropylidene-D-mannitol

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Comment

The title compound, $C_{13}H_{20}O_7$, the carbonate of a partially protected sugar alcohol, 1,2:5,6-di-*O*-isopropylidene-*D*-mannitol, was obtained accidentally on the attempted preparation of an orthocarbonate thereof.

The molecular structure is shown in Fig. 1. The 5-membered 1,3-dioxolane ring O1–C1–C2–O2–C12 adopts a twist conformation on O1—C1 (Q_2 = 0.324 (3) Å, φ_2 = 16.7 (6)°), whereas O5–C5–C6–O6–C22 (Q_2 = 0.302 (3) Å, φ_2 = 319.3 (6)°) shows an envelope conformation on C22. The dioxolane ring O3–C3–C4–O4–C10, which contains the carbonate group, is twisted on C3—C4 (Q_2 = 0.165 (3) Å, φ_2 = 57.8 (11)°). Ring puckering parameters (Cremer & Pople, 1975) were calculated with *PLATON* (Spek, 2003).

The molecular packing is shown in Fig. 2.

Experimental

The title compound was obtained accidentally as the sole product on the attempted preparation of an orthocarbonate by the reaction of dichlorodiphenoxymethane, $(PhO)_2CCl_2$, with 1,2:5,6-di-*O*-isopropylidene-*D*-mannitol in analogy to a literature procedure (Mues & Buysch, 1990). The crude product was recrystallized from boiling ethyl acetate.

Refinement

All H atoms were located in a difference map and refined as riding on their parent atoms. One common isotropic displacement parameter for all H atoms was refined to U_{iso}(H) = 0.059 (2) Å^2.

Due to the absence of significant anomalous scattering the absolute structure factor (Flack, 1983), which is 2.9 with an estimated standard deviation of 1.8 for the unmerged data set, is meaningless. Thus, Friedel opposites (1329 pairs) have been merged. The absolute structure has been assigned to match the known sterochemistry of the starting material 1,2:5,6-di-*O*-isopropylidene-D-mannitol.

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.

Figures

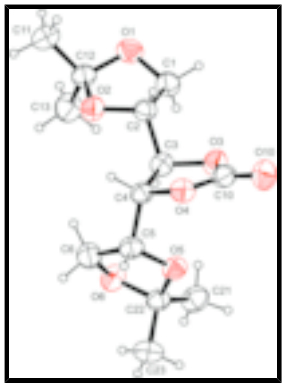

Fig. 1. The molecular structure of (I), with atom labels and anisotropic displacement ellipsoids (drawn at 50% probability level) for non-H atoms.

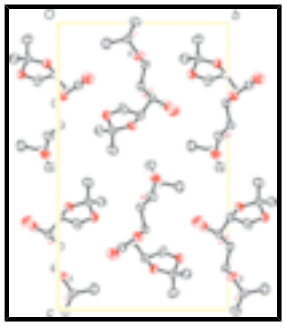

Fig. 2. The packing of (I), viewed along [100]. H atoms omitted for clarity.

3,4-*O*-Carbonyl-1,2:5,6-di-*O*-isopropylidene-D-mannitol

Crystal data

$C_{13}H_{20}O_7$	F_{000} = 616
M_r = 288.29	D_x = 1.367 (1) Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 13166 reflections
a = 6.0863 (3) Å	θ = 3.1–27.5°
b = 11.6958 (4) Å	μ = 0.11 mm^{-1}
c = 19.6816 (8) Å	T = 200 (2) K
V = 1401.02 (10) Å^3	Needle, colourless
Z = 4	0.16 × 0.04 × 0.04 mm

Data collection

Nonius KappaCCD area-detector diffractometer	1858 independent reflections
Radiation source: rotating anode	1153 reflections with $I > 2\sigma(I)$
Monochromator: MONTEL, graded multilayered X-ray optics	R_{int} = 0.059
Detector resolution: 9 pixels mm^{-1}	θ_{max} = 27.5°
T = 200(2) K	θ_{min} = 3.5°

φ and ω scans	$h = -7 \rightarrow 7$
Absorption correction: none	$k = -15 \rightarrow 15$
3191 measured reflections	$l = -25 \rightarrow 25$

Refinement

Refinement on F^2	Only H-atom displacement parameters refined
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0615P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.045$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.114$	$\Delta\rho_{max} = 0.16$ e Å^{-3}
$S = 1.00$	$\Delta\rho_{min} = -0.26$ e Å^{-3}
1858 reflections	Extinction correction: none
186 parameters	Absolute structure:
Primary atom site location: structure-invariant direct methods	Flack parameter:
Secondary atom site location: difference Fourier map	Rogers parameter:
Hydrogen 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 $F^2 > 2\sigma(F^2)$ 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}
O1	0.3167 (4)	0.4262 (2)	0.04204 (11)	0.0471 (6)
O2	0.0435 (4)	0.4772 (2)	0.11497 (10)	0.0442 (6)
O3	0.4588 (4)	0.52808 (19)	0.24498 (11)	0.0447 (6)
O4	0.2177 (4)	0.64536 (16)	0.29075 (10)	0.0392 (6)
O5	0.1688 (4)	0.43473 (18)	0.35795 (10)	0.0410 (6)
O6	−0.0359 (4)	0.28634 (17)	0.32210 (11)	0.0440 (6)
O10	0.5768 (5)	0.6762 (2)	0.30616 (13)	0.0634 (8)
C1	0.4190 (6)	0.4462 (3)	0.10539 (15)	0.0433 (8)
H11	0.5558	0.4911	0.0998	0.059 (2)*
H12	0.4537	0.3735	0.1288	0.059 (2)*
C2	0.2473 (6)	0.5135 (2)	0.14394 (14)	0.0361 (8)
H2	0.2686	0.5972	0.1360	0.059 (2)*
C3	0.2486 (5)	0.4882 (2)	0.22009 (15)	0.0370 (8)
H3	0.2329	0.4042	0.2282	0.059 (2)*

C4	0.0853 (5)	0.5539 (3)	0.26282 (14)	0.0342 (7)
H4	−0.0350	0.5856	0.2338	0.059 (2)*
C5	−0.0096 (6)	0.4859 (2)	0.32170 (15)	0.0389 (8)
H5	−0.0949	0.5374	0.3525	0.059 (2)*
C6	−0.1529 (6)	0.3852 (3)	0.29908 (17)	0.0461 (9)
H61	−0.3003	0.3893	0.3202	0.059 (2)*
H62	−0.1696	0.3842	0.2491	0.059 (2)*
C10	0.4302 (6)	0.6218 (3)	0.28301 (16)	0.0420 (8)
C11	−0.0367 (7)	0.4552 (3)	−0.00416 (16)	0.0527 (10)
H111	0.0049	0.4136	−0.0454	0.059 (2)*
H112	−0.0033	0.5366	−0.0099	0.059 (2)*
H113	−0.1944	0.4456	0.0041	0.059 (2)*
C12	0.0902 (6)	0.4089 (3)	0.05523 (15)	0.0400 (8)
C13	0.0364 (7)	0.2857 (3)	0.0715 (2)	0.0542 (10)
H131	0.1349	0.2582	0.1073	0.059 (2)*
H132	0.0554	0.2389	0.0307	0.059 (2)*
H133	−0.1162	0.2803	0.0871	0.059 (2)*
C21	0.3026 (7)	0.2459 (3)	0.37563 (17)	0.0499 (9)
H211	0.4105	0.2747	0.4084	0.059 (2)*
H212	0.3679	0.2451	0.3301	0.059 (2)*
H213	0.2590	0.1681	0.3883	0.059 (2)*
C22	0.1052 (6)	0.3218 (3)	0.37579 (15)	0.0370 (8)
C23	−0.0157 (7)	0.3221 (3)	0.44255 (17)	0.0547 (10)
H231	0.0792	0.3541	0.4780	0.059 (2)*
H232	−0.0564	0.2437	0.4547	0.059 (2)*
H233	−0.1488	0.3688	0.4384	0.059 (2)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0490 (16)	0.0524 (14)	0.0398 (12)	0.0003 (13)	0.0080 (12)	−0.0053 (11)
O2	0.0397 (14)	0.0545 (14)	0.0383 (12)	0.0032 (12)	0.0004 (11)	−0.0122 (11)
O3	0.0407 (15)	0.0514 (14)	0.0421 (12)	0.0062 (12)	−0.0042 (11)	−0.0051 (11)
O4	0.0464 (15)	0.0292 (11)	0.0420 (12)	−0.0005 (10)	0.0015 (11)	−0.0011 (10)
O5	0.0485 (15)	0.0343 (10)	0.0403 (11)	−0.0035 (11)	−0.0062 (12)	0.0049 (9)
O6	0.0522 (16)	0.0317 (11)	0.0482 (13)	−0.0018 (11)	−0.0116 (12)	0.0008 (10)
O10	0.0551 (18)	0.0756 (17)	0.0595 (16)	−0.0169 (17)	−0.0102 (15)	−0.0074 (14)
C1	0.041 (2)	0.0480 (19)	0.0414 (17)	−0.0007 (18)	0.0032 (17)	−0.0082 (16)
C2	0.040 (2)	0.0349 (15)	0.0335 (15)	−0.0019 (16)	0.0010 (15)	−0.0020 (13)
C3	0.042 (2)	0.0303 (14)	0.0391 (16)	0.0002 (15)	−0.0035 (15)	−0.0001 (13)
C4	0.0383 (19)	0.0305 (15)	0.0337 (15)	0.0013 (15)	−0.0015 (15)	0.0024 (13)
C5	0.045 (2)	0.0350 (16)	0.0369 (16)	0.0025 (16)	0.0006 (15)	0.0012 (14)
C6	0.045 (2)	0.0381 (16)	0.056 (2)	−0.0002 (16)	−0.0038 (18)	0.0109 (16)
C10	0.046 (2)	0.0435 (18)	0.0367 (17)	−0.0026 (19)	−0.0040 (18)	0.0036 (16)
C11	0.062 (3)	0.057 (2)	0.0394 (17)	0.002 (2)	−0.0045 (19)	0.0043 (16)
C12	0.043 (2)	0.0441 (19)	0.0327 (16)	0.0011 (17)	0.0027 (16)	−0.0075 (13)
C13	0.060 (3)	0.0416 (18)	0.061 (2)	−0.0052 (19)	−0.008 (2)	−0.0011 (17)
C21	0.055 (2)	0.0486 (18)	0.0464 (18)	0.0096 (19)	−0.0025 (19)	0.0013 (17)

C22	0.045 (2)	0.0330 (15)	0.0333 (16)	−0.0022 (16)	−0.0008 (15)	0.0006 (13)
C23	0.064 (3)	0.057 (2)	0.043 (2)	−0.002 (2)	0.0130 (19)	0.0072 (16)

Geometric parameters (Å, °)

O1—C1	1.413 (4)	C4—H4	1.0000
O1—C12	1.417 (4)	C5—C6	1.531 (4)
O2—C2	1.429 (4)	C5—H5	1.0000
O2—C12	1.449 (4)	C6—H61	0.9900
O3—C10	1.339 (4)	C6—H62	0.9900
O3—C3	1.447 (4)	C11—C12	1.502 (5)
O4—C10	1.331 (4)	C11—H111	0.9800
O4—C4	1.448 (4)	C11—H112	0.9800
O5—C22	1.420 (4)	C11—H113	0.9800
O5—C5	1.430 (4)	C12—C13	1.512 (5)
O6—C22	1.424 (4)	C13—H131	0.9800
O6—C6	1.431 (4)	C13—H132	0.9800
O10—C10	1.187 (4)	C13—H133	0.9800
C1—C2	1.512 (4)	C21—C22	1.494 (5)
C1—H11	0.9900	C21—H211	0.9800
C1—H12	0.9900	C21—H212	0.9800
C2—C3	1.528 (4)	C21—H213	0.9800
C2—H2	1.0000	C22—C23	1.506 (5)
C3—C4	1.512 (4)	C23—H231	0.9800
C3—H3	1.0000	C23—H232	0.9800
C4—C5	1.520 (4)	C23—H233	0.9800
C1—O1—C12	106.9 (2)	H61—C6—H62	108.9
C2—O2—C12	108.5 (2)	O10—C10—O4	125.2 (3)
C10—O3—C3	109.8 (3)	O10—C10—O3	123.8 (4)
C10—O4—C4	110.2 (2)	O4—C10—O3	111.1 (3)
C22—O5—C5	107.8 (2)	C12—C11—H111	109.5
C22—O6—C6	107.4 (2)	C12—C11—H112	109.5
O1—C1—C2	103.0 (3)	H111—C11—H112	109.5
O1—C1—H11	111.2	C12—C11—H113	109.5
C2—C1—H11	111.2	H111—C11—H113	109.5
O1—C1—H12	111.2	H112—C11—H113	109.5
C2—C1—H12	111.2	O1—C12—O2	105.1 (3)
H11—C1—H12	109.1	O1—C12—C11	107.8 (3)
O2—C2—C1	104.2 (2)	O2—C12—C11	109.4 (3)
O2—C2—C3	109.8 (2)	O1—C12—C13	112.7 (3)
C1—C2—C3	112.8 (3)	O2—C12—C13	108.1 (3)
O2—C2—H2	110.0	C11—C12—C13	113.4 (3)
C1—C2—H2	110.0	C12—C13—H131	109.5
C3—C2—H2	110.0	C12—C13—H132	109.5
O3—C3—C4	103.2 (2)	H131—C13—H132	109.5
O3—C3—C2	105.9 (2)	C12—C13—H133	109.5
C4—C3—C2	116.3 (3)	H131—C13—H133	109.5
O3—C3—H3	110.3	H132—C13—H133	109.5
C4—C3—H3	110.3	C22—C21—H211	109.5

C2—C3—H3	110.3	C22—C21—H212	109.5
O4—C4—C3	102.7 (3)	H211—C21—H212	109.5
O4—C4—C5	108.0 (2)	C22—C21—H213	109.5
C3—C4—C5	114.1 (3)	H211—C21—H213	109.5
O4—C4—H4	110.6	H212—C21—H213	109.5
C3—C4—H4	110.6	O5—C22—O6	104.6 (2)
C5—C4—H4	110.6	O5—C22—C21	109.5 (3)
O5—C5—C4	108.1 (3)	O6—C22—C21	108.1 (2)
O5—C5—C6	104.8 (2)	O5—C22—C23	110.3 (2)
C4—C5—C6	113.4 (3)	O6—C22—C23	110.7 (3)
O5—C5—H5	110.1	C21—C22—C23	113.4 (3)
C4—C5—H5	110.1	C22—C23—H231	109.5
C6—C5—H5	110.1	C22—C23—H232	109.5
O6—C6—C5	104.2 (2)	H231—C23—H232	109.5
O6—C6—H61	110.9	C22—C23—H233	109.5
C5—C6—H61	110.9	H231—C23—H233	109.5
O6—C6—H62	110.9	H232—C23—H233	109.5
C5—C6—H62	110.9		
C12—O1—C1—C2	−35.9 (3)	O4—C4—C5—C6	−179.6 (2)
C12—O2—C2—C1	−9.5 (3)	C3—C4—C5—C6	−66.0 (4)
C12—O2—C2—C3	−130.6 (2)	C22—O6—C6—C5	22.0 (3)
O1—C1—C2—O2	27.4 (3)	O5—C5—C6—O6	−2.6 (3)
O1—C1—C2—C3	146.5 (3)	C4—C5—C6—O6	115.1 (3)
C10—O3—C3—C4	−13.6 (3)	C4—O4—C10—O10	−173.1 (3)
C10—O3—C3—C2	109.1 (3)	C4—O4—C10—O3	7.3 (3)
O2—C2—C3—O3	−179.7 (2)	C3—O3—C10—O10	−175.1 (3)
C1—C2—C3—O3	64.6 (3)	C3—O3—C10—O4	4.6 (3)
O2—C2—C3—C4	−65.6 (3)	C1—O1—C12—O2	30.4 (3)
C1—C2—C3—C4	178.6 (3)	C1—O1—C12—C11	147.0 (3)
C10—O4—C4—C3	−15.2 (3)	C1—O1—C12—C13	−87.0 (3)
C10—O4—C4—C5	105.7 (3)	C2—O2—C12—O1	−11.9 (3)
O3—C3—C4—O4	16.7 (3)	C2—O2—C12—C11	−127.5 (3)
C2—C3—C4—O4	−98.9 (3)	C2—O2—C12—C13	108.6 (3)
O3—C3—C4—C5	−99.9 (3)	C5—O5—C22—O6	31.8 (3)
C2—C3—C4—C5	144.5 (3)	C5—O5—C22—C21	147.4 (3)
C22—O5—C5—C4	−139.1 (2)	C5—O5—C22—C23	−87.3 (3)
C22—O5—C5—C6	−17.8 (3)	C6—O6—C22—O5	−33.6 (3)
O4—C4—C5—O5	−63.8 (3)	C6—O6—C22—C21	−150.1 (3)
C3—C4—C5—O5	49.7 (3)	C6—O6—C22—C23	85.2 (3)

Fig. 1

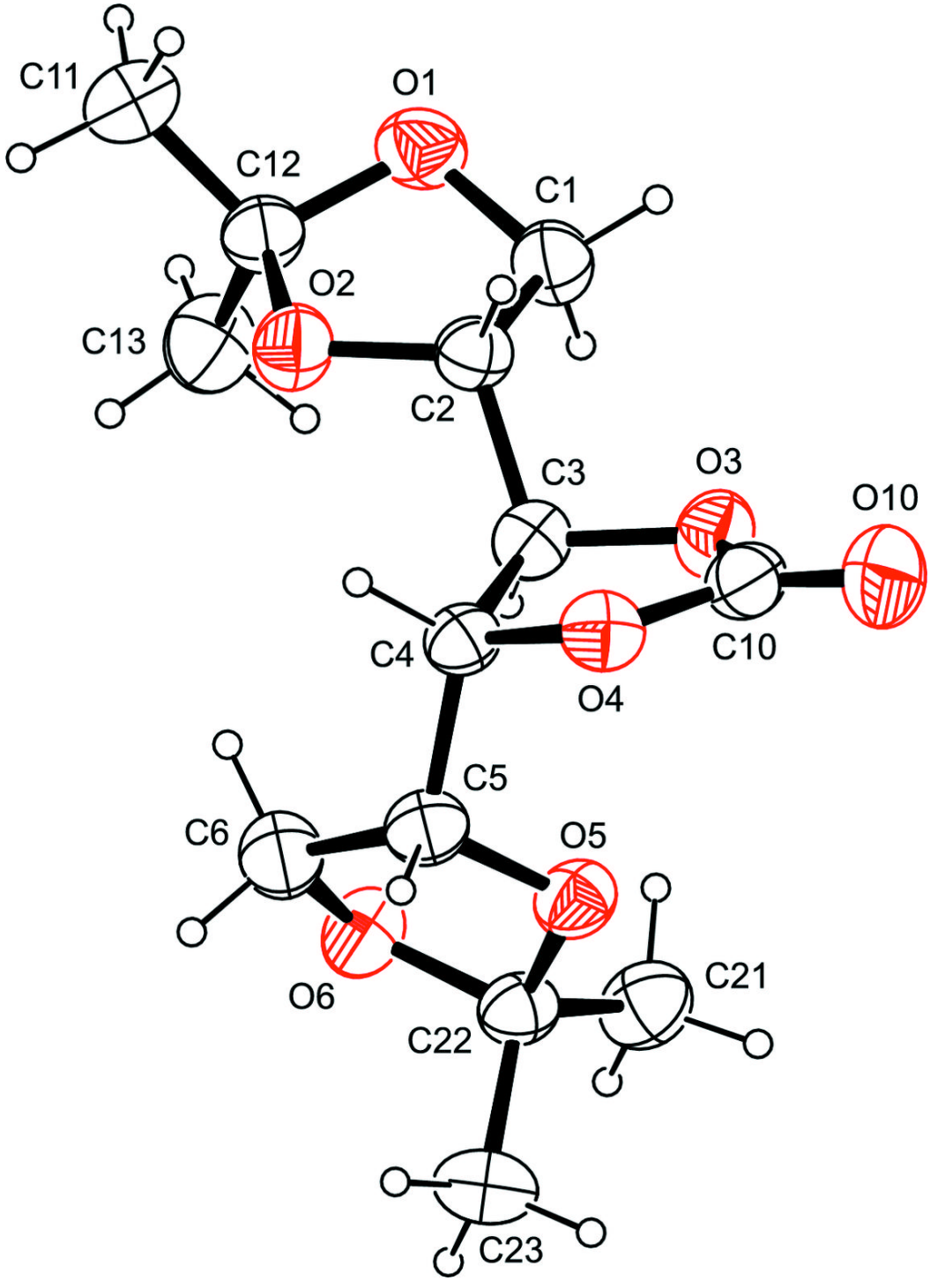

Fig. 2