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An orthorhombic polymorph of 6-deoxy-6-iodo-1,2:3,4-di-O-isopropylidene- α -D-galactopyranoside

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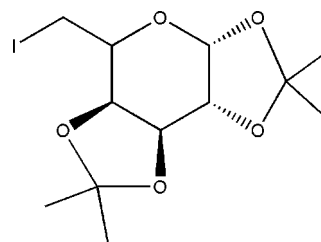
Received 20 July 2009; accepted 22 July 2009

Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.035; wR factor = 0.096; data-to-parameter ratio = 45.0.

The title compound, $\text{C}_{12}\text{H}_{19}\text{IO}_5$, is the orthorhombic polymorph of a previously reported monoclinic form [Krajewski *et al.* (1987). *Bull. Pol. Acad. Sci. Chem.* **35**, 91–102]. The dihedral angles between the six-membered ring and the two five-membered rings are 67.66 (14) and 71.79 (13)°, whereas the dihedral angle between the five-membered rings is 74.41 (12)°, indicating that all three rings are twisted from each other. The six-membered ring has a twist-boat conformation while both of the five-membered rings have envelope conformations. The crystal structure is stabilized by a network of $\text{C}-\text{H}\cdots\text{O}$ contacts linking the molecules into a two-dimensional array parallel to the ab plane.

Related literature

For the monoclinic polymorph of the title compound, see: Krajewski *et al.* (1987). For the synthesis and biological evaluation of 6-substituted purines, see: Gambogi Braga *et al.* (2007). For halogenation reagent systems, see: Clason *et al.* (1988). For the synthesis of perosamine derivatives, see: Stevens *et al.* (1970). For the synthesis of labilose, see: Westwood *et al.* (1967). For ring conformations and ring puckering analysis, see: Boeyens (1978); Cremer & Pople (1975). For the stability of the temperature controller used in the data collection, see: Cosier & Glazer (1986).



Experimental

Crystal data

$\text{C}_{12}\text{H}_{19}\text{IO}_5$	$V = 1440.13$ (4) Å ³
$M_r = 370.17$	$Z = 4$
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation
$a = 7.3595$ (1) Å	$\mu = 2.23$ mm ⁻¹
$b = 11.5145$ (2) Å	$T = 100$ K
$c = 16.9945$ (2) Å	$0.17 \times 0.11 \times 0.11$ mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	27359 measured reflections
Absorption correction: multi-scan (SADABS; Bruker, 2005)	7509 independent reflections
$T_{\min} = 0.703$, $T_{\max} = 0.785$	6211 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.046$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.035$	$\Delta\rho_{\text{max}} = 0.87$ e Å ⁻³
$wR(F^2) = 0.096$	$\Delta\rho_{\text{min}} = -1.24$ e Å ⁻³
$S = 1.07$	Absolute structure: Flack (1983),
7509 reflections	3286 Friedel pairs
167 parameters	Flack parameter: -0.020 (19)
H-atom parameters constrained	

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C}8-\text{H}8\text{B}\cdots\text{O}2^{\text{i}}$	0.97	2.42	3.377 (3)	169
$\text{C}12-\text{H}12\text{C}\cdots\text{O}2^{\text{ii}}$	0.96	2.60	3.477 (4)	152

Symmetry codes: (i) $-x + 1, y - \frac{1}{2}, -z + \frac{3}{2}$; (ii) $-x, y - \frac{1}{2}, -z + \frac{3}{2}$.

Data collection: APEX2 (Bruker, 2005); cell refinement: SAINT (Bruker, 2005); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL and PLATON (Spek, 2009).

HKF and WCL thank Universiti Sains Malaysia (USM) for a Research University Golden Goose Grant (No. 1001/PFIZIK/811012). WCL thanks USM for a student assistantship. AMI is grateful to the Director, NITK, Surathkal, India, for providing research facilities

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2509).

* Thomson Reuters Researcher ID: A-3561-2009.

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supplementary materials

Acta Cryst. (2009). E65, o1999-o2000 [doi:10.1107/S1600536809029031]

An orthorhombic polymorph of 6-deoxy-6-iodo-1,2:3,4-di-*O*-isopropylidene- α -D-galactopyranoside

H.-K. Fun, W.-C. Liew, S. Rai, P. Shetty and A. M. Isloor

Comment

Libilomycin, an antibiotic which inhibits the growth of Gram-positive bacteria (Westwood *et al.*, 1967) and which is effective against certain tumor cells, is produced by the microorganism, streptomyces albosporeas (Stevens *et al.*, 1970), and contains 6-iodo,6-deoxy-1,2,3,4-di-*O*-isopropylidene- α -D-galactopyranoside as a part of its structure (Classon *et al.*, 1988). These intermediates are used for the synthesis of 6-substituted purines which show high activity against *Leishmania amazonensis* (Gambogi Braga *et al.*, 2007). These results prompted us to synthesize the title compound, (I).

Compound (I), Fig. 1, crystallized in the orthorhombic space group $P2_12_12_1$ but has been reported previously (Krajewski *et al.*, 1987) in the monoclinic space group $P2_1$, with $a = 11.157(2)$ Å, $b = 20.047(4)$ Å, $c = 14.188(2)$ Å and $\beta = 107.67(1)^\circ$. The dihedral angles between the six-membered ring systems, ring *A* (C1/C3/C4/C6/C7/O5), and the five-membered ring systems [rings *B* (C1—C3/O1—O2) and *C* (C4—C6/O3—O4)] are $67.66(14)^\circ$ and $71.79(13)^\circ$, respectively. Moreover, the dihedral angle between rings *B* and *C* is $74.41(12)^\circ$ indicating that all the three rings are twisted from each other. Ring *A* adopts the twist-boat conformation (Boeyens, 1978; Cremer & Pople, 1975) with puckering amplitude $Q = 0.629(2)$ Å, $\varphi = 75.3(3)^\circ$ and $\theta = 325.6(2)^\circ$. On the other hand, each of rings *B* and *C* adopt an envelope conformation with flap atoms O2 and C5, respectively, but having different puckering parameters. For ring *B*, the puckering amplitude $Q = 0.285(2)$ Å and $\varphi = 294.9(5)^\circ$ whereas for ring *C*, the puckering amplitude $Q = 0.323(3)$ Å and $\varphi = 150.8(4)^\circ$.

The crystal packing (Fig. 2 & Fig. 3) is consolidated by C8—H8B \cdots O2 and C12—H12C \cdots O2 contacts (Table 1) that link the molecules into a 2-D array parallel to the *ab* plane.

Experimental

Triphenylphosphine (0.53 g, 1.9 mmol) and imidazole (0.4 g, 5.7 mmol) was added to the mixture of 1,2,3,4-di-*O*-isopropylidene- α -D-galactopyranoside (0.5 g, 1.9 mmol) in toluene: acetonitrile (2: 1, 10 ml). The mixture was heated to 70 °C. Iodine (0.6 g, 3.8 mmol) was then added portion-wise for a period of 30 min and mixture was further stirred for 2 hours. The completion of the reaction was confirmed by TLC (30% EtOAc/hexane, R_f - 0.6). The brown reaction mixture was concentrated under vacuum and the residue was purified by column chromatography using 25% ethylacetate in petroleum ether to get desired compound as white crystals. (Yield 600 mg, 83%, *m.p.* 334–336 K).

Refinement

C-bound H atoms were positioned geometrically [C—H = 0.96–0.98 Å] and refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ and $1.5U_{\text{eq}}(\text{methyl-C})$. A rotating group model was used for the methyl groups.

The maximum and minimum residual electron density peaks of 0.87 and $-1.24 \text{ e}\text{\AA}^{-3}$, respectively, were located 0.85 Å and 0.52 Å from the H8A and I1 atoms, respectively.

Figures

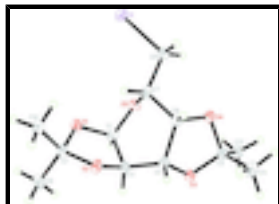


Fig. 1. The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom numbering scheme.

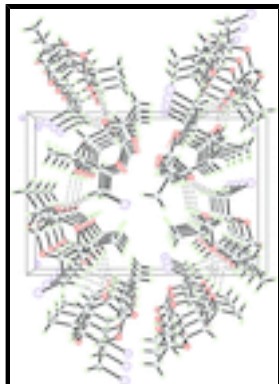


Fig. 2. Crystal packing viewed along the *a* axis. The C-H...O contacts are shown as dashed lines.

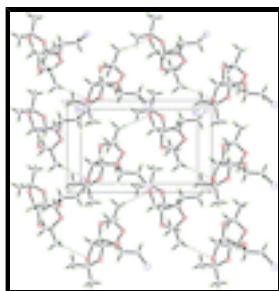


Fig. 3. Crystal packing viewed along the *c* axis. The C-H...O contacts are shown as dashed lines.

6-deoxy-6-iodo-1,2:3,4-di-*O*-isopropylidene- α -D-galactopyranoside

Crystal data

$C_{12}H_{19}IO_5$

$M_r = 370.17$

Orthorhombic, $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 7.3595$ (1) Å

$b = 11.5145$ (2) Å

$c = 16.9945$ (2) Å

$V = 1440.13$ (4) Å³

$Z = 4$

$F_{000} = 736$

$D_x = 1.707$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 6517 reflections

$\theta = 3.0$ – 30.1°

$\mu = 2.23$ mm⁻¹

$T = 100$ K

Block, colourless

$0.17 \times 0.11 \times 0.11$ mm

Data collection

Bruker SMART APEXII CCD area-detector
diffractometer

7509 independent reflections

Radiation source: fine-focus sealed tube	6211 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.046$
$T = 100$ K	$\theta_{\text{max}} = 37.5^\circ$
φ and ω scans	$\theta_{\text{min}} = 2.1^\circ$
Absorption correction: multi-scan (<i>SADABS</i> ; Bruker, 2005)	$h = -12 \rightarrow 12$
$T_{\text{min}} = 0.703$, $T_{\text{max}} = 0.785$	$k = -19 \rightarrow 17$
27359 measured reflections	$l = -28 \rightarrow 29$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.035$	$w = 1/[\sigma^2(F_o^2) + (0.0418P)^2 + 0.1348P]$
$wR(F^2) = 0.096$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.07$	$(\Delta/\sigma)_{\text{max}} = 0.003$
7509 reflections	$\Delta\rho_{\text{max}} = 0.87 \text{ e } \text{\AA}^{-3}$
167 parameters	$\Delta\rho_{\text{min}} = -1.24 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none
Secondary atom site location: difference Fourier map	Absolute structure: Flack (1983), 3286 Friedel pairs
	Flack parameter: -0.020 (19)

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cyrosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
II	0.48767 (2)	-0.062287 (16)	0.585388 (10)	0.02322 (5)
O1	0.3941 (3)	0.30650 (19)	0.54871 (11)	0.0202 (4)
O2	0.4126 (2)	0.41053 (17)	0.66190 (11)	0.0174 (4)
O3	-0.0095 (3)	0.31453 (16)	0.74636 (10)	0.0169 (3)
O4	0.0869 (3)	0.13209 (18)	0.77122 (12)	0.0181 (4)
O5	0.2158 (2)	0.16776 (16)	0.61095 (11)	0.0162 (3)
C1	0.2269 (3)	0.2856 (2)	0.58845 (16)	0.0166 (4)

supplementary materials

H1A	0.1256	0.3041	0.5532	0.020*
C2	0.4873 (3)	0.4031 (2)	0.58404 (14)	0.0167 (4)
C3	0.2281 (3)	0.3722 (2)	0.65788 (15)	0.0166 (4)
H3A	0.1476	0.4379	0.6466	0.020*
C4	0.1835 (3)	0.3209 (3)	0.73797 (15)	0.0167 (4)
H4A	0.2335	0.3710	0.7793	0.020*
C5	-0.0464 (3)	0.2160 (2)	0.79401 (15)	0.0165 (4)
C6	0.2485 (3)	0.1941 (2)	0.75132 (13)	0.0152 (5)
H6A	0.3359	0.1907	0.7948	0.018*
C7	0.3309 (3)	0.1412 (2)	0.67660 (15)	0.0165 (5)
H7A	0.4522	0.1737	0.6677	0.020*
C8	0.3433 (4)	0.0106 (3)	0.68407 (16)	0.0194 (5)
H8A	0.2220	-0.0221	0.6864	0.023*
H8B	0.4059	-0.0092	0.7325	0.023*
C9	0.4481 (4)	0.5139 (3)	0.53734 (17)	0.0235 (6)
H9A	0.3193	0.5267	0.5353	0.035*
H9B	0.4945	0.5057	0.4848	0.035*
H9C	0.5058	0.5787	0.5626	0.035*
C10	0.6874 (4)	0.3785 (3)	0.59115 (18)	0.0220 (5)
H10A	0.7049	0.3032	0.6142	0.033*
H10B	0.7430	0.4364	0.6240	0.033*
H10C	0.7421	0.3804	0.5399	0.033*
C11	-0.0254 (4)	0.2444 (3)	0.88146 (15)	0.0241 (5)
H11A	0.0946	0.2739	0.8910	0.036*
H11B	-0.0440	0.1753	0.9120	0.036*
H11C	-0.1136	0.3018	0.8963	0.036*
C12	-0.2321 (4)	0.1723 (3)	0.77260 (18)	0.0205 (5)
H12A	-0.2307	0.1435	0.7196	0.031*
H12B	-0.3183	0.2346	0.7766	0.031*
H12C	-0.2662	0.1109	0.8078	0.031*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
II	0.02406 (8)	0.02602 (9)	0.01959 (7)	0.00713 (7)	0.00446 (7)	0.00100 (6)
O1	0.0212 (9)	0.0223 (10)	0.0170 (8)	-0.0053 (8)	0.0033 (7)	-0.0029 (8)
O2	0.0136 (7)	0.0220 (10)	0.0167 (8)	-0.0033 (6)	0.0016 (6)	-0.0022 (7)
O3	0.0124 (7)	0.0161 (7)	0.0222 (7)	-0.0003 (6)	0.0014 (6)	0.0023 (6)
O4	0.0133 (8)	0.0188 (9)	0.0223 (9)	0.0002 (6)	0.0036 (7)	0.0023 (7)
O5	0.0169 (8)	0.0149 (9)	0.0166 (7)	-0.0011 (6)	-0.0038 (6)	0.0004 (7)
C1	0.0177 (9)	0.0178 (12)	0.0143 (9)	-0.0010 (8)	-0.0028 (9)	-0.0001 (9)
C2	0.0173 (9)	0.0193 (10)	0.0134 (8)	-0.0026 (8)	0.0025 (11)	0.0023 (8)
C3	0.0145 (10)	0.0180 (12)	0.0172 (10)	-0.0018 (8)	0.0006 (8)	-0.0008 (9)
C4	0.0127 (9)	0.0207 (13)	0.0167 (10)	-0.0020 (8)	0.0012 (8)	-0.0009 (9)
C5	0.0150 (10)	0.0179 (12)	0.0166 (10)	0.0003 (8)	0.0014 (8)	0.0017 (8)
C6	0.0125 (9)	0.0197 (13)	0.0135 (10)	-0.0002 (8)	-0.0011 (7)	0.0007 (9)
C7	0.0127 (10)	0.0218 (13)	0.0149 (10)	-0.0004 (8)	-0.0003 (8)	0.0005 (9)
C8	0.0186 (11)	0.0227 (14)	0.0170 (11)	0.0044 (9)	0.0011 (9)	0.0041 (10)

C9	0.0251 (13)	0.0236 (14)	0.0218 (12)	-0.0012 (10)	0.0004 (10)	0.0054 (11)
C10	0.0175 (10)	0.0292 (15)	0.0193 (11)	0.0012 (9)	0.0031 (10)	0.0007 (11)
C11	0.0227 (13)	0.0313 (15)	0.0182 (10)	0.0003 (11)	0.0021 (10)	-0.0010 (10)
C12	0.0170 (11)	0.0200 (14)	0.0245 (13)	-0.0021 (9)	0.0014 (9)	0.0036 (11)

Geometric parameters (Å, °)

I1—C8	2.155 (3)	C5—C11	1.530 (4)
O1—C1	1.424 (3)	C6—C7	1.533 (4)
O1—C2	1.438 (3)	C6—H6A	0.9800
O2—C3	1.430 (3)	C7—C8	1.512 (4)
O2—C2	1.435 (3)	C7—H7A	0.9800
O3—C5	1.420 (3)	C8—H8A	0.9700
O3—C4	1.429 (3)	C8—H8B	0.9700
O4—C6	1.428 (3)	C9—H9A	0.9600
O4—C5	1.431 (3)	C9—H9B	0.9600
O5—C1	1.412 (3)	C9—H9C	0.9600
O5—C7	1.434 (3)	C10—H10A	0.9600
C1—C3	1.545 (4)	C10—H10B	0.9600
C1—H1A	0.9800	C10—H10C	0.9600
C2—C10	1.505 (4)	C11—H11A	0.9600
C2—C9	1.530 (4)	C11—H11B	0.9600
C3—C4	1.519 (4)	C11—H11C	0.9600
C3—H3A	0.9800	C12—H12A	0.9600
C4—C6	1.554 (4)	C12—H12B	0.9600
C4—H4A	0.9800	C12—H12C	0.9600
C5—C12	1.501 (4)		
C1—O1—C2	110.17 (19)	C7—C6—H6A	110.4
C3—O2—C2	107.52 (19)	C4—C6—H6A	110.4
C5—O3—C4	106.8 (2)	O5—C7—C8	108.2 (2)
C6—O4—C5	107.3 (2)	O5—C7—C6	109.1 (2)
C1—O5—C7	112.42 (19)	C8—C7—C6	110.4 (2)
O5—C1—O1	109.9 (2)	O5—C7—H7A	109.7
O5—C1—C3	114.4 (2)	C8—C7—H7A	109.7
O1—C1—C3	104.4 (2)	C6—C7—H7A	109.7
O5—C1—H1A	109.3	C7—C8—H1	110.61 (18)
O1—C1—H1A	109.3	C7—C8—H8A	109.5
C3—C1—H1A	109.3	I1—C8—H8A	109.5
O2—C2—O1	104.41 (18)	C7—C8—H8B	109.5
O2—C2—C10	108.2 (2)	I1—C8—H8B	109.5
O1—C2—C10	110.8 (2)	H8A—C8—H8B	108.1
O2—C2—C9	110.8 (2)	C2—C9—H9A	109.5
O1—C2—C9	109.8 (2)	C2—C9—H9B	109.5
C10—C2—C9	112.5 (2)	H9A—C9—H9B	109.5
O2—C3—C4	106.4 (2)	C2—C9—H9C	109.5
O2—C3—C1	104.0 (2)	H9A—C9—H9C	109.5
C4—C3—C1	115.6 (2)	H9B—C9—H9C	109.5
O2—C3—H3A	110.2	C2—C10—H10A	109.5
C4—C3—H3A	110.2	C2—C10—H10B	109.5

supplementary materials

C1—C3—H3A	110.2	H10A—C10—H10B	109.5
O3—C4—C3	108.9 (2)	C2—C10—H10C	109.5
O3—C4—C6	104.1 (2)	H10A—C10—H10C	109.5
C3—C4—C6	115.4 (2)	H10B—C10—H10C	109.5
O3—C4—H4A	109.4	C5—C11—H11A	109.5
C3—C4—H4A	109.4	C5—C11—H11B	109.5
C6—C4—H4A	109.4	H11A—C11—H11B	109.5
O3—C5—O4	104.70 (19)	C5—C11—H11C	109.5
O3—C5—C12	107.7 (2)	H11A—C11—H11C	109.5
O4—C5—C12	109.4 (2)	H11B—C11—H11C	109.5
O3—C5—C11	111.4 (2)	C5—C12—H12A	109.5
O4—C5—C11	109.8 (2)	C5—C12—H12B	109.5
C12—C5—C11	113.5 (2)	H12A—C12—H12B	109.5
O4—C6—C7	109.1 (2)	C5—C12—H12C	109.5
O4—C6—C4	104.4 (2)	H12A—C12—H12C	109.5
C7—C6—C4	112.0 (2)	H12B—C12—H12C	109.5
O4—C6—H6A	110.4		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C8—H8B \cdots O2 ⁱ	0.97	2.42	3.377 (3)	169
C12—H12C \cdots O2 ⁱⁱ	0.96	2.60	3.477 (4)	152

Symmetry codes: (i) $-x+1, y-1/2, -z+3/2$; (ii) $-x, y-1/2, -z+3/2$.

Fig. 1

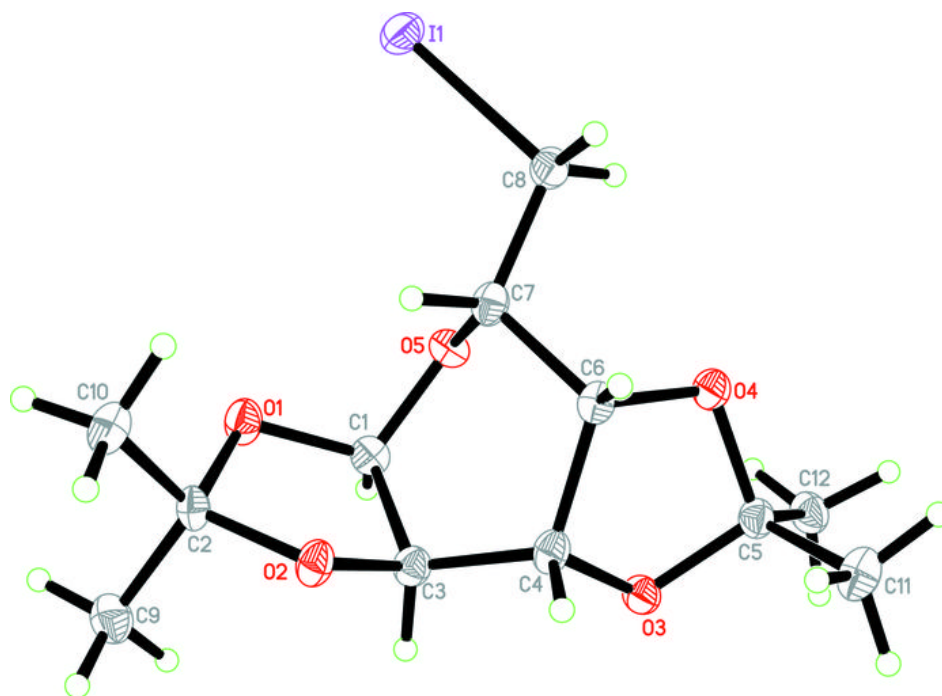


Fig. 2

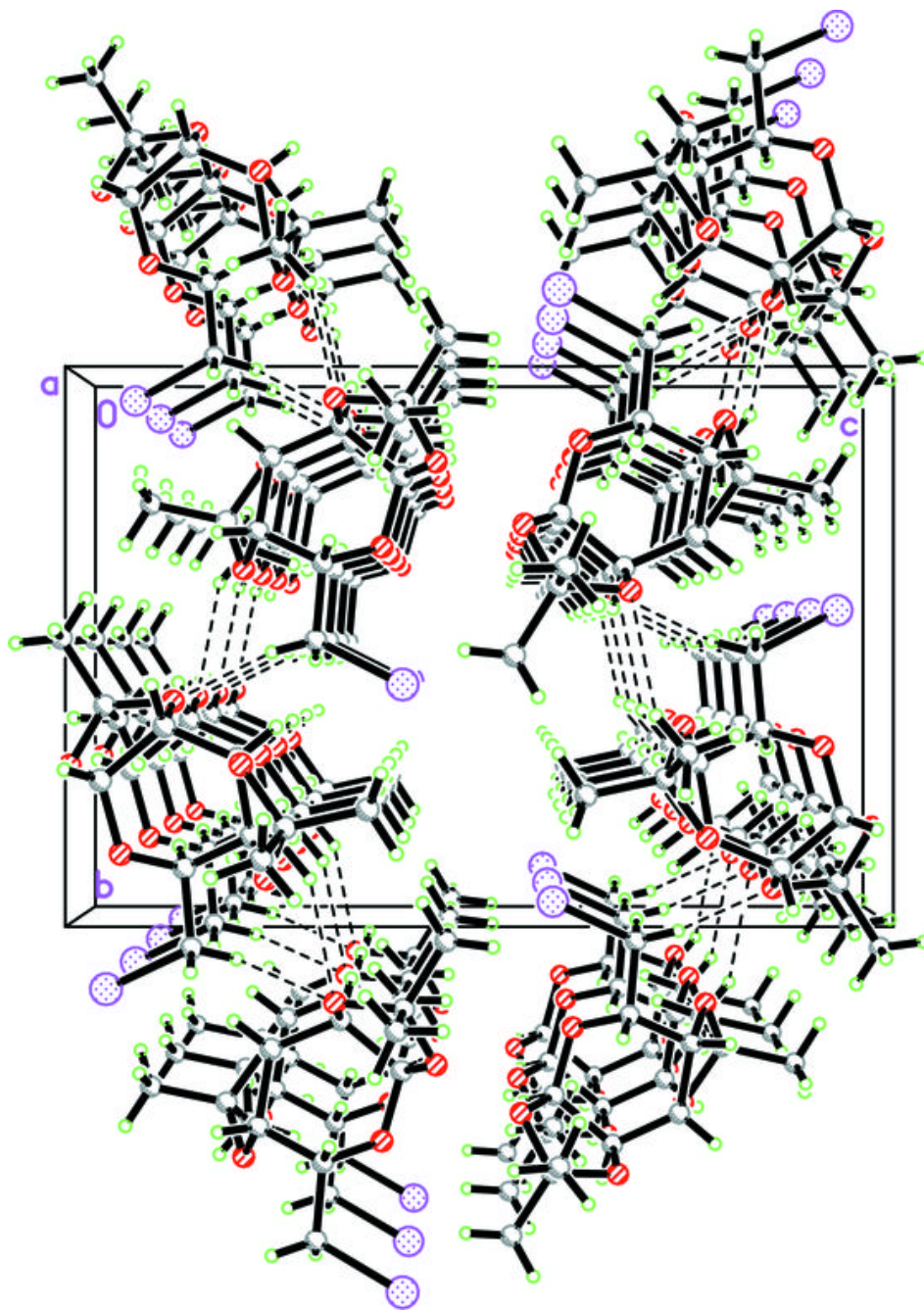


Fig. 3

