Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

7,11,15,28-Tetramethyl-1,21,23,25-tetrakis(2-phenylethyl)resorcin[4]arene ethyl acetate clathrate

Michael G. Mc Kay, Holger B. Friedrich and Glenn E. M. Maguire*

School of Chemistry, University of KwaZulu-Natal, Durban 4041, South Africa
Correspondence e-mail: maguireg@ukzn.ac.za

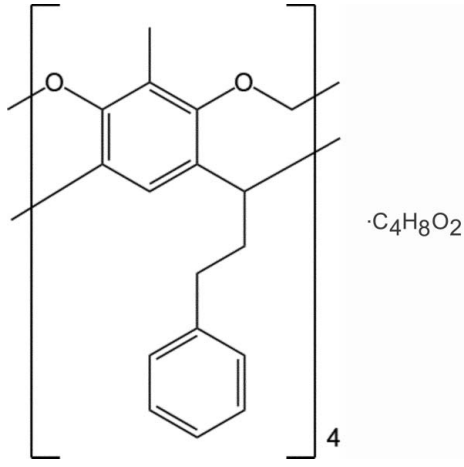
Received 18 October 2007; accepted 8 November 2007

Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.004 Å; disorder in main residue; R factor = 0.063; wR factor = 0.209; data-to-parameter ratio = 13.1.

The title compound, $C_{68}H_{64}O_8{\cdot}C_4H_8O_2$, is a new resorcin[4]-arene cavitand synthetic precursor, obtained by alkylation of a previously reported resorcin[4]arene. The additional alkyl bridges significantly rigidify the structure and enforce a 'bowl' shape on the molecular cavity. In the crystal structure, the molecule lies on a crystallographic mirror plane, and a single ethyl acetate molecule (also lying on the mirror plane) is present within the compound cavity, illustrating the host capabilities of the molecule.

Related literature

For related literature, see: Cram *et al.* (1988); Eisler *et al.* (2002); Friedrich *et al.* (2007); Piepers & Kellog (1978); Roman *et al.* (1999); Sebo *et al.* (2000).



Experimental

Crystal data

$C_{68}H_{64}O_8{\cdot}C_4H_8O_2$
M_r = 1097.30
Orthorhombic, *Pnma*
a = 24.3288 (4) Å
b = 20.6279 (4) Å
c = 11.7828 (2) Å
V = 5913.22 (18) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 173 (2) K
0.39 × 0.27 × 0.19 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: none
49448 measured reflections
5981 independent reflections
3934 reflections with $I > 2\sigma(I)$
R_{int} = 0.062

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.209
S = 1.04
5981 reflections
455 parameters
131 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.55 e Å^{-3}

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT-NT* (Bruker, 2005); data reduction: *SAINT-NT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXTL* (Bruker, 1997).

The financial support of the DST–NRF Centre of Excellence in Catalysis, c*change, is duly acknowledged. Our thanks go to Dr Manuel Fernandes at the University of the Witwatersrand for performing the data acquisition and structure solution.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BI2251).

References

Bruker (1997). *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2005). *APEX2* (Version 2.0-1) and *SAINT-NT* (Version 6.0). Bruker AXS Inc., Madison, Wisconsin, USA.
Cram, D. J., Karbach, S., Kim, H.-E., Knobler, C. B., Maverick, E. F., Ericson, J. L. & Helgeson, R. C. (1988). *J. Am. Chem. Soc.* **110**, 2229–2237.
Eisler, D., Hong, W., Jennings, M. C. & Pudephatt, R. J. (2002). *Organometallics*, **21**, 3955–3960.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Friedrich, H. B., Howie, R. A., Maguire, G. E. M. & Mc Kay, M. G. (2007). *Acta Cryst.* E**63**, o4346.
Piepers, O. & Kellog, R. M. (1978). *J. Chem. Soc. Chem. Commun.* pp. 383–384.
Roman, E., Peinador, C., Mendoza, S. & Kaifer, A. E. (1999). *J. Org. Chem.* **64**, 2577–2578.
Sebo, L., Diederich, F. & Gramlich, V. (2000). *Helv. Chim. Acta*, **83**, 93–96.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

  doi:10.1107/S160053680705711X

supporting information

Acta Cryst. (2008). E**64**, o98 [https://doi.org/10.1107/S160053680705711X]

7,11,15,28-Tetramethyl-1,21,23,25-tetrakis(2-phenylethyl)resorcin[4]arene ethyl acetate clathrate

Michael G. Mc Kay, Holger B. Friedrich and Glenn E. M. Maguire

S1. Comment

The title compound is obtained by alkylation of the hydroxyl groups of a resorcin[4]arene octol material whose structure has been previously reported (Friedrich *et al.*, 2007). The alkylating agent, CH_2BrCl, results in the formation of four OCH_2O ether bridges between the four aromatic units of the resorcin[4]arene starting material (Fig. 1). The bridges rigidify the molecular structure, fixing the compound in a "bowl" shape (Fig. 2) with an enforced cavity that can accommodate guest molecules.

The 2-phenylethyl "feet" of the compound are orientated such that the aromatic rings are arranged in an edge-to-face manner. Such C—H···π interactions have also been observed in the resorcin[4]arene octol precursor (Friedrich *et al.*, 2007). Interestingly, only two of the feet orientate in such a manner (Fig. 3). The presence of the ethyl acetate molecule within the molecular cavity forces the remaining two aromatic units apart, disrupting complete C—H···π interaction comparable to that as seen for the precursor. Similar disruption has also been reported previously for related structures.

S2. Experimental

The synthesis of the resorcin[4]arene octol precursor can proceed *via* two different synthetic approaches. The older protocol as set out by Cram *et al.* (1988) involves heating a solution of the resorcin[4]arene starting material and the CH_2BrCl in the presence of K_2CO_3 at atmospheric pressure. Reaction proceeds over a number of days, often accompanied by addition of further equivalents of the alkylating reagent. However, since CH_2BrCl is volatile and boils at 341 K, the reaction temperature is limited to a range between 333 and 343 K. This results in yields of 40–60%. More recently, Roman *et al.* (1999) reported a procedure which made use of a sealed tube as a reaction vessel, heated to 361 K. The reagents are heated in the presence of Cs_2CO_3, which is used instead of K_2CO_3 due to the templating ability of the caesium cation, which aids in the formation of macrocyclic assemblies such as cavitands (Piepers & Kellog, 1978). Under these conditions, yields are in excess of 80%; indeed, the use of the protocol of Kaifer *et al.* in the synthesis of the resorcin[4]arene octol precursor gave a yield of 92%.

To prepare the title compound (Scheme 2): dry resorcin[4]arene octol (1.00 g, 1.66 mmol) and Cs_2CO_3 (3.00 g, 9.21 mmol) were added with stirring to dry DMSO (10 ml) in a pressure tube (ACE pressure tube, Aldrich). To the resulting pink solution, CH_2BrCl (3.00 ml, 46.0 mmol) was added followed by further DMSO (10 ml). The tube was sealed and heated at 360 K for 16 h. After cooling to room temperature, the tube contents were poured into 2% HCl (200 ml) and the voluminous solid formed was filtered and washed with water. The cream coloured solid was chromatographed on silica gel using a mobile phase of 70:30 hexane-ethyl acetate (R_f = 0.59). The fractions collected were concentrated on a rotary evaporator to yield a cream coloured solid. The solid was stirred in methanol overnight, and filtered to yield the product as a white powder (0.97 g, 92%, m.p. 418–420 K). Crystals suitable for X-ray diffraction analysis were grown by slow liquid diffusion of methanol into a solution of the title compound in 1:1 ethyl acetate:hexane.

S3. Refinement

H atoms were visible in difference Fourier maps but were positioned geometrically and allowed to ride on their respective parent atoms, with C—H bond lengths of 1.00 (CH), 0.99 (CH_2), or 0.98 (CH_3) Å. They were then refined with a riding model with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(CH_3). One of the phenylethyl 'feet′ was found to be disordered and was refined over two positions using isotropic displacement parameters and with SADI, SIMU, DELU, *DFIX* and SADI restraints applied. The refined site occupancy factors for the two positions are 0.461 (5) and 0.539 (5), respectively.

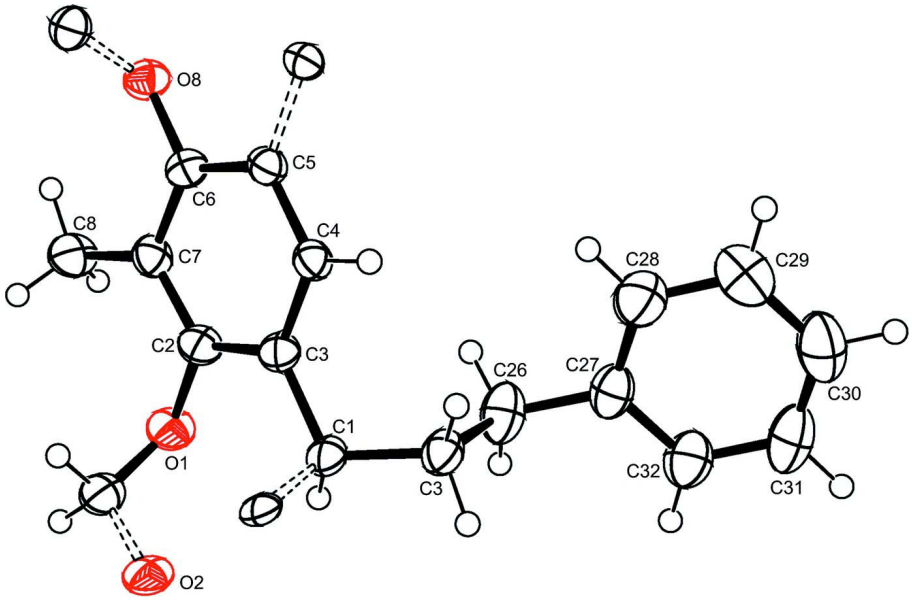


Figure 1

A view of one quarter of the cyclic tetramer. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as spheres of arbitrary radii. Dashed bonds indicate links to the neighbouring units.

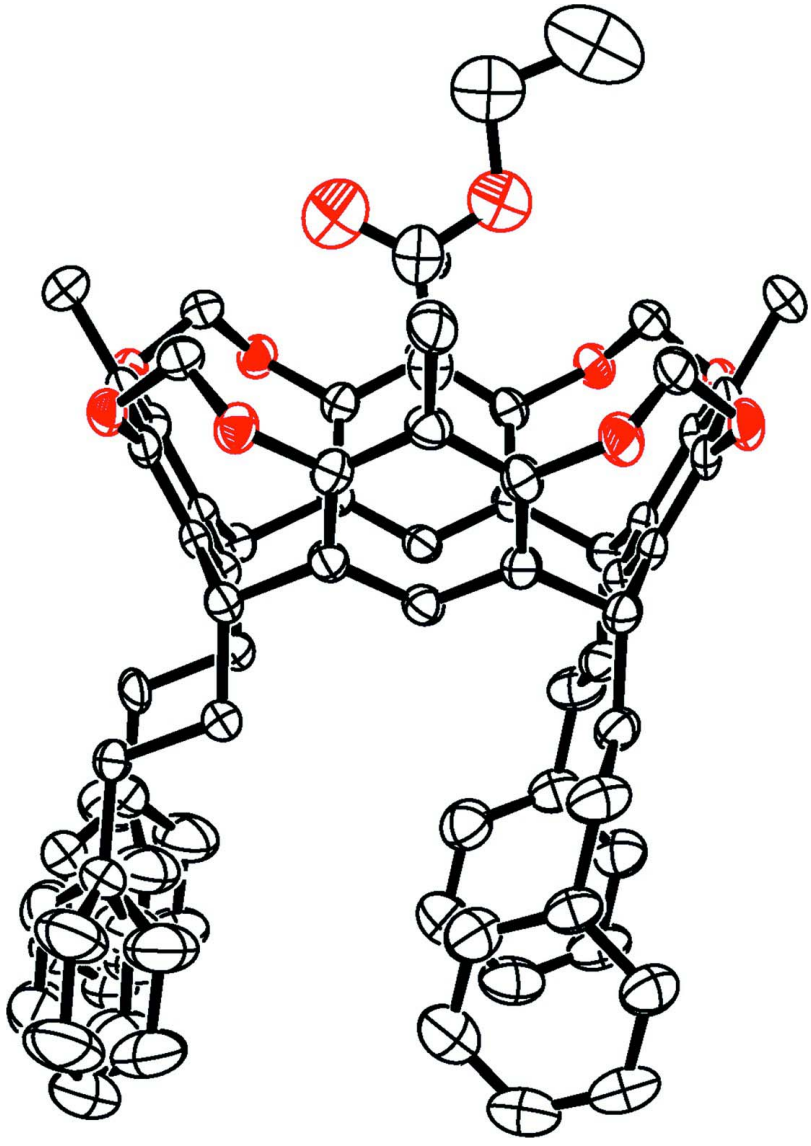


Figure 2

The molecular structure with displacement ellipsoids drawn at the 50% probability level. H atoms are omitted. The "bowl" shape of the molecule is evident, with the ethyl acetate molecule present as a guest in the molecular cavity.

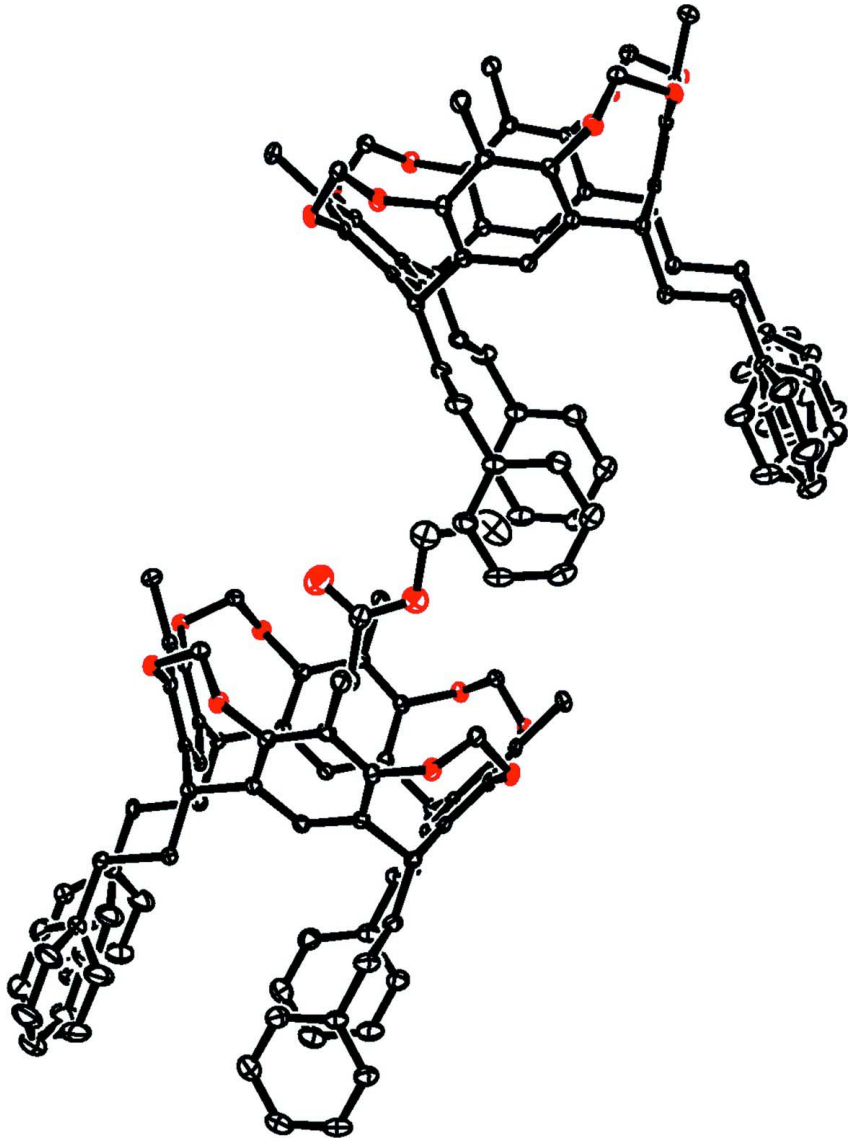


Figure 3

The relative orientation of the feet in neighbouring resorcin[4]arene units. Displacement ellipsoids are drawn at the 10% probability level. The inclusion of the ethyl acetate solvent molecule between aromatic groups is shown. H atoms are omitted.

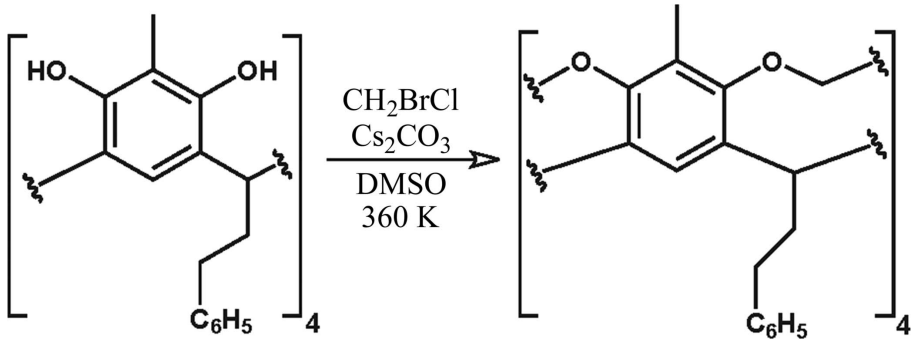


Figure 4

The formation of the title compound.

7,11,15,28-Tetramethyl-1,21,23,25-tetrakis(2-phenylethyl)-2,20:3,19- dimetheno-1H,21*H*,23H,25*H*-bis[1,3]dioxocino[5,4 - i:5′,4′-i′]benzo[1,2 - d:5,4 - d′] bis[1,3]benzodioxocin stereoisomer

Crystal data

$C_{68}H_{64}O_8 \cdot C_4H_8O_2$
$M_r = 1097.30$
Orthorhombic, *Pnma*
Hall symbol: -P 2ac 2n
$a = 24.3288$ (4) Å
$b = 20.6279$ (4) Å
$c = 11.7828$ (2) Å
$V = 5913.22$ (18) Å^3
$Z = 4$
$F(000) = 2336$
$D_x = 1.233$ Mg m^{-3}
$D_m = 1.233$ Mg m^{-3}
D_m measured by ?
Melting point: 419 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 8201 reflections
$\theta = 2.2$–$27.7°$
$\mu = 0.08$ mm^{-1}
$T = 173$ K
Block, colourless
0.39 × 0.27 × 0.19 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
49448 measured reflections
5981 independent reflections
3934 reflections with $I > 2\sigma(I)$
$R_{int} = 0.062$
$\theta_{max} = 26.0°$, $\theta_{min} = 1.7°$
$h = -29 \rightarrow 30$
$k = -25 \rightarrow 25$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.063$
$wR(F^2) = 0.209$
$S = 1.04$
5981 reflections
455 parameters
131 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.132P)^2 + 1.0228P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 1.16$ e Å^{-3}
$\Delta\rho_{min} = -0.55$ e Å^{-3}

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.21767 (10)	0.05927 (12)	−0.0930 (2)	0.0315 (6)	
C2	0.20814 (10)	0.07645 (12)	0.0196 (2)	0.0303 (5)	
C3	0.16055 (10)	0.10805 (11)	0.05431 (19)	0.0281 (5)	
C4	0.12187 (10)	0.12419 (11)	−0.02821 (19)	0.0282 (5)	
H4	0.0899	0.1475	−0.0064	0.034*	
C5	0.12871 (10)	0.10715 (12)	−0.1421 (2)	0.0296 (5)	
C6	0.17667 (10)	0.07484 (12)	−0.1710 (2)	0.0325 (6)	
C7	0.26893 (11)	0.02447 (15)	−0.1284 (2)	0.0429 (7)	
H7A	0.2680	0.0168	−0.2104	0.064*	0.50
H7B	0.2712	−0.0171	−0.0885	0.064*	0.50
H7C	0.3011	0.0510	−0.1094	0.064*	0.50
H7D	0.2922	0.0170	−0.0617	0.064*	0.50
H7E	0.2890	0.0509	−0.1837	0.064*	0.50
H7F	0.2591	−0.0172	−0.1628	0.064*	0.50
C8	0.20493 (11)	0.09993 (15)	−0.3597 (2)	0.0408 (7)	
H8A	0.2284	0.0775	−0.4161	0.049*	
H8B	0.2283	0.1311	−0.3179	0.049*	
C9	0.08732 (10)	0.12684 (12)	−0.2322 (2)	0.0317 (6)	
H9	0.0892	0.0936	−0.2938	0.038*	
C10	0.16631 (14)	0.2500	−0.4127 (3)	0.0384 (9)	
C11	0.14567 (10)	0.19219 (14)	−0.36868 (19)	0.0341 (6)	
C12	0.10640 (9)	0.19097 (13)	−0.28304 (19)	0.0312 (6)	
C13	0.08815 (13)	0.2500	−0.2412 (3)	0.0286 (7)	
H13	0.0620	0.2500	−0.1812	0.034*	
C14	0.20920 (17)	0.2500	−0.5051 (3)	0.0487 (11)	
H14A	0.2431	0.2699	−0.4765	0.073*	0.50
H14B	0.1957	0.2748	−0.5703	0.073*	0.50
H14C	0.2168	0.2053	−0.5285	0.073*	0.50
C15	0.26710 (13)	0.2500	0.2326 (3)	0.0304 (8)	
C16	0.23792 (9)	0.19246 (12)	0.22008 (18)	0.0279 (5)	
C17	0.18192 (9)	0.19104 (11)	0.19712 (18)	0.0260 (5)	
C18	0.15503 (13)	0.2500	0.1853 (3)	0.0266 (7)	
H18	0.1168	0.2500	0.1684	0.032*	
C19	0.32741 (15)	0.2500	0.2608 (3)	0.0411 (9)	
H19A	0.3402	0.2052	0.2698	0.062*	0.50

H19B	0.3334	0.2738	0.3318	0.062*	0.50
H19C	0.3479	0.2710	0.1994	0.062*	0.50
C20	0.28457 (10)	0.10094 (13)	0.1404 (2)	0.0343 (6)	
H20A	0.2923	0.1328	0.0797	0.041*	
H20B	0.3194	0.0783	0.1584	0.041*	
C21	0.15301 (9)	0.12615 (12)	0.17830 (19)	0.0278 (5)	
H21	0.1734	0.0931	0.2239	0.033*	
C22	0.02798 (10)	0.12884 (12)	−0.1903 (2)	0.0352 (6)	
H22A	0.0254	0.1599	−0.1264	0.042*	
H22B	0.0043	0.1452	−0.2523	0.042*	
C23	0.00637 (11)	0.06397 (14)	−0.1517 (3)	0.0493 (8)	
H23A	0.0018	0.0354	−0.2185	0.059*	
H23B	0.0336	0.0436	−0.1005	0.059*	
C24	−0.04747 (11)	0.06929 (13)	−0.0909 (3)	0.0411 (7)	
C25	−0.04889 (13)	0.08805 (16)	0.0217 (3)	0.0550 (8)	
H25	−0.0153	0.0962	0.0603	0.066*	
C26	−0.09799 (15)	0.09521 (18)	0.0792 (3)	0.0638 (9)	
H26	−0.0979	0.1084	0.1565	0.077*	
C27	−0.14716 (14)	0.08316 (18)	0.0247 (3)	0.0644 (9)	
H27	−0.1811	0.0880	0.0638	0.077*	
C28	−0.14632 (13)	0.06401 (17)	−0.0874 (4)	0.0638 (10)	
H28	−0.1799	0.0552	−0.1256	0.077*	
C29	−0.09712 (12)	0.05752 (14)	−0.1446 (3)	0.0496 (7)	
H29	−0.0973	0.0448	−0.2221	0.060*	
C30	0.09333 (10)	0.12443 (13)	0.21862 (19)	0.0315 (6)	
H30A	0.0728	0.1603	0.1824	0.038*	
H30B	0.0763	0.0831	0.1942	0.038*	
C31	0.0885 (2)	0.1305 (6)	0.3468 (3)	0.046 (3)	0.461 (5)
H31A	0.1150	0.1004	0.3826	0.055*	0.461 (5)
H31B	0.0989	0.1751	0.3692	0.055*	0.461 (5)
C32	0.0316 (5)	0.1161 (4)	0.393 (2)	0.048 (2)	0.461 (5)
C33	−0.0079 (3)	0.1621 (4)	0.3928 (8)	0.077 (2)	0.461 (5)
H33	0.0007	0.2041	0.3648	0.092*	0.461 (5)
C34	−0.0606 (3)	0.1497 (5)	0.4325 (10)	0.093 (3)	0.461 (5)
H34	−0.0881	0.1823	0.4277	0.112*	0.461 (5)
C35	−0.0726 (4)	0.0909 (6)	0.4783 (12)	0.093 (3)	0.461 (5)
H35	−0.1069	0.0837	0.5144	0.112*	0.461 (5)
C36	−0.0355 (3)	0.0434 (4)	0.4718 (9)	0.096 (3)	0.461 (5)
H36	−0.0452	0.0009	0.4959	0.115*	0.461 (5)
C37	0.0170 (3)	0.0552 (4)	0.4304 (8)	0.077 (2)	0.461 (5)
H37	0.0431	0.0210	0.4280	0.092*	0.461 (5)
C31A	0.0893 (2)	0.1308 (5)	0.3470 (3)	0.049 (3)	0.539 (5)
H31C	0.1101	0.0949	0.3823	0.058*	0.539 (5)
H31D	0.1071	0.1719	0.3701	0.058*	0.539 (5)
C32A	0.0310 (4)	0.1300 (4)	0.3930 (18)	0.049 (2)	0.539 (5)
C33A	0.0156 (2)	0.1708 (4)	0.4765 (5)	0.0705 (18)	0.539 (5)
H33A	0.0416	0.2008	0.5057	0.085*	0.539 (5)
C34A	−0.0371 (3)	0.1703 (4)	0.5210 (6)	0.088 (2)	0.539 (5)

H34A	−0.0471	0.2007	0.5781	0.105*	0.539 (5)
C35A	−0.0746 (3)	0.1265 (6)	0.4831 (9)	0.081 (2)	0.539 (5)
H35A	−0.1099	0.1236	0.5172	0.097*	0.539 (5)
C36A	−0.0609 (2)	0.0877 (5)	0.3972 (7)	0.088 (2)	0.539 (5)
H36A	−0.0874	0.0582	0.3680	0.105*	0.539 (5)
C37A	−0.0088 (2)	0.0896 (4)	0.3497 (6)	0.076 (2)	0.539 (5)
H37A	−0.0004	0.0626	0.2866	0.091*	0.539 (5)
C38	0.2894 (2)	0.2500	−0.1267 (4)	0.0743 (16)	
C39	0.22971 (16)	0.2500	−0.1041 (3)	0.0427 (9)	
H39A	0.2233	0.2500	−0.0220	0.064*	
H39B	0.2131	0.2112	−0.1378	0.064*	
C40	0.3641 (3)	0.2500	−0.2541 (6)	0.159 (5)	
H40	0.3816	0.2111	−0.2208	0.190*	
C41	0.3703 (4)	0.2500	−0.3809 (6)	0.131 (3)	
H41A	0.4096	0.2500	−0.3991	0.197*	
H41B	0.3531	0.2112	−0.4132	0.197*	
O1	0.18418 (7)	0.05391 (9)	−0.28283 (14)	0.0395 (5)	
O2	0.16347 (7)	0.13445 (10)	−0.41755 (14)	0.0413 (5)	
O3	0.26603 (7)	0.13439 (8)	0.23871 (14)	0.0326 (4)	
O4	0.24595 (7)	0.05531 (8)	0.10003 (14)	0.0337 (4)	
O5	0.30466 (17)	0.2500	−0.2313 (3)	0.1016 (15)	
O6	0.3238 (2)	0.2500	−0.0520 (5)	0.223 (5)	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0301 (13)	0.0327 (13)	0.0318 (13)	0.0041 (10)	0.0041 (10)	−0.0017 (10)
C2	0.0305 (12)	0.0308 (12)	0.0296 (12)	0.0014 (10)	−0.0035 (10)	0.0013 (10)
C3	0.0303 (12)	0.0295 (12)	0.0245 (12)	−0.0034 (10)	0.0016 (10)	−0.0013 (10)
C4	0.0278 (12)	0.0301 (12)	0.0267 (12)	−0.0002 (10)	0.0031 (10)	−0.0021 (10)
C5	0.0297 (13)	0.0323 (13)	0.0269 (12)	−0.0018 (10)	−0.0012 (10)	−0.0050 (10)
C6	0.0368 (14)	0.0347 (13)	0.0260 (12)	0.0013 (11)	0.0017 (10)	−0.0081 (10)
C7	0.0418 (15)	0.0517 (17)	0.0351 (14)	0.0139 (13)	0.0023 (12)	−0.0069 (13)
C8	0.0334 (14)	0.0619 (19)	0.0272 (13)	0.0097 (12)	0.0024 (11)	−0.0095 (13)
C9	0.0267 (12)	0.0423 (14)	0.0260 (12)	−0.0013 (10)	−0.0026 (10)	−0.0098 (10)
C10	0.0302 (19)	0.071 (3)	0.0140 (15)	0.000	−0.0018 (14)	0.000
C11	0.0295 (13)	0.0571 (17)	0.0155 (11)	0.0039 (11)	−0.0042 (9)	−0.0055 (11)
C12	0.0247 (12)	0.0508 (16)	0.0180 (11)	0.0003 (10)	−0.0069 (9)	−0.0043 (10)
C13	0.0234 (16)	0.043 (2)	0.0195 (15)	0.000	−0.0005 (13)	0.000
C14	0.043 (2)	0.079 (3)	0.0242 (18)	0.000	0.0088 (17)	0.000
C15	0.0264 (17)	0.046 (2)	0.0186 (16)	0.000	0.0009 (13)	0.000
C16	0.0303 (12)	0.0368 (13)	0.0165 (11)	0.0041 (10)	−0.0006 (9)	0.0011 (9)
C17	0.0282 (12)	0.0357 (13)	0.0141 (10)	−0.0017 (10)	0.0019 (9)	0.0006 (9)
C18	0.0245 (16)	0.0385 (19)	0.0167 (15)	0.000	0.0016 (12)	0.000
C19	0.0290 (19)	0.053 (2)	0.041 (2)	0.000	−0.0064 (16)	0.000
C20	0.0271 (12)	0.0414 (15)	0.0346 (13)	0.0056 (11)	−0.0004 (10)	0.0007 (11)
C21	0.0288 (12)	0.0323 (13)	0.0223 (11)	0.0006 (10)	0.0015 (9)	0.0031 (10)
C22	0.0292 (13)	0.0400 (14)	0.0363 (14)	−0.0018 (11)	−0.0026 (11)	−0.0073 (11)

C23	0.0362 (15)	0.0434 (17)	0.068 (2)	−0.0046 (12)	0.0020 (14)	−0.0102 (15)
C24	0.0340 (14)	0.0331 (14)	0.0563 (18)	−0.0047 (11)	−0.0008 (13)	0.0004 (13)
C25	0.0474 (18)	0.060 (2)	0.058 (2)	−0.0049 (15)	−0.0041 (15)	0.0011 (16)
C26	0.067 (2)	0.069 (2)	0.056 (2)	−0.0032 (18)	0.0135 (18)	0.0028 (17)
C27	0.0484 (19)	0.064 (2)	0.081 (3)	−0.0070 (16)	0.0186 (18)	0.0097 (19)
C28	0.0338 (16)	0.068 (2)	0.090 (3)	−0.0104 (15)	−0.0009 (17)	0.000 (2)
C29	0.0420 (16)	0.0476 (17)	0.0592 (19)	−0.0079 (13)	−0.0026 (14)	−0.0054 (14)
C30	0.0292 (13)	0.0404 (14)	0.0249 (12)	−0.0038 (11)	0.0008 (10)	0.0008 (10)
C31	0.033 (4)	0.085 (8)	0.020 (7)	−0.019 (4)	−0.001 (4)	−0.015 (6)
C32	0.034 (3)	0.081 (4)	0.031 (4)	−0.010 (3)	0.005 (3)	0.003 (5)
C33	0.056 (4)	0.097 (4)	0.079 (5)	0.010 (3)	0.024 (4)	0.021 (4)
C34	0.048 (4)	0.133 (6)	0.098 (6)	0.019 (4)	0.020 (4)	0.027 (5)
C35	0.053 (4)	0.148 (6)	0.079 (6)	−0.011 (4)	0.019 (4)	0.029 (6)
C36	0.067 (4)	0.117 (5)	0.102 (6)	−0.023 (3)	0.023 (4)	0.044 (5)
C37	0.060 (4)	0.091 (4)	0.079 (5)	−0.007 (3)	0.022 (4)	0.028 (4)
C31A	0.038 (4)	0.075 (7)	0.033 (7)	0.000 (4)	0.008 (4)	0.010 (5)
C32A	0.033 (3)	0.082 (4)	0.030 (4)	−0.002 (3)	0.003 (3)	0.006 (4)
C33A	0.041 (3)	0.129 (5)	0.042 (3)	0.010 (3)	−0.001 (2)	−0.019 (3)
C34A	0.048 (3)	0.163 (6)	0.053 (4)	0.017 (3)	0.008 (3)	−0.021 (4)
C35A	0.036 (3)	0.150 (6)	0.058 (4)	0.010 (3)	0.011 (3)	0.013 (4)
C36A	0.038 (3)	0.130 (6)	0.095 (5)	−0.014 (3)	0.010 (3)	−0.008 (4)
C37A	0.048 (3)	0.097 (5)	0.083 (4)	−0.017 (3)	0.019 (3)	−0.018 (3)
C38	0.059 (3)	0.119 (5)	0.046 (3)	0.000	−0.006 (2)	0.000
C39	0.047 (2)	0.046 (2)	0.035 (2)	0.000	−0.0041 (17)	0.000
C40	0.076 (5)	0.308 (15)	0.092 (6)	0.000	0.006 (4)	0.000
C41	0.134 (7)	0.119 (7)	0.140 (8)	0.000	0.055 (6)	0.000
O1	0.0422 (11)	0.0478 (11)	0.0286 (9)	0.0067 (8)	0.0018 (8)	−0.0136 (8)
O2	0.0390 (10)	0.0638 (13)	0.0210 (8)	0.0101 (9)	−0.0026 (7)	−0.0125 (8)
O3	0.0328 (9)	0.0400 (10)	0.0249 (8)	0.0070 (7)	−0.0022 (7)	0.0038 (7)
O4	0.0351 (9)	0.0342 (9)	0.0317 (9)	0.0057 (7)	−0.0045 (7)	0.0020 (7)
O5	0.065 (2)	0.173 (5)	0.067 (3)	0.000	0.004 (2)	0.000
O6	0.074 (3)	0.515 (16)	0.081 (4)	0.000	−0.010 (3)	0.000

Geometric parameters (Å, º)

C1—C2	1.392 (3)	C23—C24	1.497 (4)
C1—C6	1.394 (3)	C23—H23A	0.990
C1—C7	1.498 (3)	C23—H23B	0.990
C2—C3	1.390 (3)	C24—C25	1.382 (4)
C2—O4	1.391 (3)	C24—C29	1.385 (4)
C3—C4	1.394 (3)	C25—C26	1.382 (4)
C3—C21	1.519 (3)	C25—H25	0.950
C4—C5	1.397 (3)	C26—C27	1.380 (5)
C4—H4	0.950	C26—H26	0.950
C5—C6	1.387 (3)	C27—C28	1.379 (5)
C5—C9	1.519 (3)	C27—H27	0.950
C6—O1	1.398 (3)	C28—C29	1.380 (4)
C7—H7A	0.980	C28—H28	0.950

C7—H7B	0.980	C29—H29	0.950
C7—H7C	0.980	C30—C31	1.521 (4)
C7—H7D	0.980	C30—C31A	1.522 (4)
C7—H7E	0.980	C30—H30A	0.990
C7—H7F	0.980	C30—H30B	0.990
C8—O1	1.406 (3)	C31—C32	1.517 (5)
C8—O2	1.410 (3)	C31—H31A	0.990
C8—H8A	0.990	C31—H31B	0.990
C8—H8B	0.990	C32—C33	1.349 (13)
C9—C12	1.525 (4)	C32—C37	1.378 (13)
C9—C22	1.526 (3)	C33—C34	1.388 (7)
C9—H9	1.000	C33—H33	0.950
C10—C11	1.394 (3)	C34—C35	1.360 (12)
C10—C11^{i}	1.394 (3)	C34—H34	0.950
C10—C14	1.508 (5)	C35—C36	1.334 (12)
C11—C12	1.390 (3)	C35—H35	0.950
C11—O2	1.392 (3)	C36—C37	1.389 (7)
C12—C13	1.387 (3)	C36—H36	0.950
C13—C12^{i}	1.387 (3)	C37—H37	0.950
C13—H13	0.950	C31A—C32A	1.517 (5)
C14—H14A	0.980	C31A—H31C	0.990
C14—H14B	0.980	C31A—H31D	0.990
C14—H14C	0.980	C32A—C33A	1.350 (13)
C15—C16	1.391 (3)	C32A—C37A	1.375 (13)
C15—C16^{i}	1.391 (3)	C33A—C34A	1.384 (7)
C15—C19	1.505 (5)	C33A—H33A	0.950
C16—C17	1.389 (3)	C34A—C35A	1.360 (12)
C16—O3	1.397 (3)	C34A—H34A	0.950
C17—C18	1.388 (3)	C35A—C36A	1.332 (12)
C17—C21	1.528 (3)	C35A—H35A	0.950
C18—C17^{i}	1.388 (3)	C36A—C37A	1.388 (7)
C18—H18	0.950	C36A—H36A	0.950
C19—H19A	0.980	C37A—H37A	0.950
C19—H19B	0.980	C38—O6	1.213 (7)
C19—H19C	0.980	C38—O5	1.286 (6)
C20—O4	1.412 (3)	C38—C39	1.477 (6)
C20—O3	1.422 (3)	C39—H39A	0.980
C20—H20A	0.990	C39—H39B	0.980
C20—H20B	0.990	C40—O5	1.470 (8)
C21—C30	1.528 (3)	C40—C41	1.503 (5)
C21—H21	1.000	C40—H40	0.990
C22—C23	1.508 (4)	C41—H41A	0.980
C22—H22A	0.990	C41—H41B	0.980
C22—H22B	0.990		

C2—C1—C6	116.8 (2)	C23—C22—H22B	108.8
C2—C1—C7	121.7 (2)	C9—C22—H22B	108.8
C6—C1—C7	121.5 (2)	H22A—C22—H22B	107.7

C3—C2—O4	119.8 (2)	C24—C23—C22	112.6 (2)
C3—C2—C1	122.6 (2)	C24—C23—H23A	109.1
O4—C2—C1	117.3 (2)	C22—C23—H23A	109.1
C2—C3—C4	118.0 (2)	C24—C23—H23B	109.1
C2—C3—C21	119.9 (2)	C22—C23—H23B	109.1
C4—C3—C21	122.1 (2)	H23A—C23—H23B	107.8
C3—C4—C5	122.0 (2)	C25—C24—C29	117.8 (3)
C3—C4—H4	119.0	C25—C24—C23	120.1 (3)
C5—C4—H4	119.0	C29—C24—C23	122.1 (3)
C6—C5—C4	117.2 (2)	C26—C25—C24	121.5 (3)
C6—C5—C9	121.0 (2)	C26—C25—H25	119.2
C4—C5—C9	121.7 (2)	C24—C25—H25	119.2
C5—C6—C1	123.4 (2)	C27—C26—C25	120.1 (3)
C5—C6—O1	119.4 (2)	C27—C26—H26	119.9
C1—C6—O1	117.2 (2)	C25—C26—H26	119.9
C1—C7—H7A	109.5	C28—C27—C26	119.0 (3)
C1—C7—H7B	109.5	C28—C27—H27	120.5
H7A—C7—H7B	109.5	C26—C27—H27	120.5
C1—C7—H7C	109.5	C27—C28—C29	120.6 (3)
H7A—C7—H7C	109.5	C27—C28—H28	119.7
H7B—C7—H7C	109.5	C29—C28—H28	119.7
C1—C7—H7D	109.5	C28—C29—C24	121.1 (3)
H7A—C7—H7D	141.1	C28—C29—H29	119.5
H7B—C7—H7D	56.3	C24—C29—H29	119.5
H7C—C7—H7D	56.3	C31—C30—C21	112.4 (3)
C1—C7—H7E	109.5	C31A—C30—C21	111.6 (3)
H7A—C7—H7E	56.3	C31—C30—H30A	109.1
H7B—C7—H7E	141.1	C31A—C30—H30A	109.3
H7C—C7—H7E	56.3	C21—C30—H30A	109.1
H7D—C7—H7E	109.5	C31—C30—H30B	109.1
C1—C7—H7F	109.5	C31A—C30—H30B	109.7
H7A—C7—H7F	56.3	C21—C30—H30B	109.1
H7B—C7—H7F	56.3	H30A—C30—H30B	107.9
H7C—C7—H7F	141.1	C32—C31—C30	114.2 (10)
H7D—C7—H7F	109.5	C32—C31—H31A	108.7
H7E—C7—H7F	109.5	C30—C31—H31A	108.7
O1—C8—O2	113.3 (2)	C32—C31—H31B	108.7
O1—C8—H8A	108.9	C30—C31—H31B	108.7
O2—C8—H8A	108.9	H31A—C31—H31B	107.6
O1—C8—H8B	108.9	C33—C32—C37	117.3 (5)
O2—C8—H8B	108.9	C33—C32—C31	120.8 (10)
H8A—C8—H8B	107.7	C37—C32—C31	121.8 (9)
C5—C9—C12	107.77 (19)	C32—C33—C34	121.8 (7)
C5—C9—C22	114.1 (2)	C32—C33—H33	119.1
C12—C9—C22	113.1 (2)	C34—C33—H33	119.1
C5—C9—H9	107.2	C35—C34—C33	119.7 (7)
C12—C9—H9	107.2	C35—C34—H34	120.2
C22—C9—H9	107.2	C33—C34—H34	120.2

C11—C10—C11^{i}	117.6 (3)	C36—C35—C34	119.2 (6)
C11—C10—C14	121.18 (15)	C36—C35—H35	120.4
C11^{i}—C10—C14	121.18 (15)	C34—C35—H35	120.4
C12—C11—O2	119.9 (2)	C35—C36—C37	120.8 (7)
C12—C11—C10	122.2 (2)	C35—C36—H36	119.6
O2—C11—C10	117.8 (2)	C37—C36—H36	119.6
C13—C12—C11	117.6 (2)	C32—C37—C36	120.6 (7)
C13—C12—C9	121.6 (2)	C32—C37—H37	119.7
C11—C12—C9	120.7 (2)	C36—C37—H37	119.7
C12^{i}—C13—C12	122.8 (3)	C32A—C31A—C30	114.5 (9)
C12^{i}—C13—H13	118.6	C32A—C31A—H31C	108.6
C12—C13—H13	118.6	C30—C31A—H31C	108.6
C10—C14—H14A	109.5	C32A—C31A—H31D	108.6
C10—C14—H14B	109.5	C30—C31A—H31D	108.6
H14A—C14—H14B	109.5	H31C—C31A—H31D	107.6
C10—C14—H14C	109.5	C33A—C32A—C37A	116.9 (5)
H14A—C14—H14C	109.5	C33A—C32A—C31A	120.9 (9)
H14B—C14—H14C	109.5	C37A—C32A—C31A	122.2 (9)
C16—C15—C16^{i}	117.2 (3)	C32A—C33A—C34A	122.0 (7)
C16—C15—C19	121.41 (15)	C32A—C33A—H33A	119.0
C16^{i}—C15—C19	121.41 (15)	C34A—C33A—H33A	119.0
C17—C16—C15	122.6 (2)	C35A—C34A—C33A	120.1 (7)
C17—C16—O3	119.5 (2)	C35A—C34A—H34A	119.9
C15—C16—O3	117.7 (2)	C33A—C34A—H34A	119.9
C18—C17—C16	117.6 (2)	C36A—C35A—C34A	118.8 (5)
C18—C17—C21	122.4 (2)	C36A—C35A—H35A	120.6
C16—C17—C21	119.9 (2)	C34A—C35A—H35A	120.6
C17^{i}—C18—C17	122.4 (3)	C35A—C36A—C37A	121.2 (7)
C17^{i}—C18—H18	118.8	C35A—C36A—H36A	119.4
C17—C18—H18	118.8	C37A—C36A—H36A	119.4
C15—C19—H19A	109.5	C32A—C37A—C36A	120.7 (7)
C15—C19—H19B	109.5	C32A—C37A—H37A	119.6
H19A—C19—H19B	109.5	C36A—C37A—H37A	119.6
C15—C19—H19C	109.5	O6—C38—O5	119.8 (5)
H19A—C19—H19C	109.5	O6—C38—C39	123.1 (5)
H19B—C19—H19C	109.5	O5—C38—C39	117.1 (4)
O4—C20—O3	112.75 (19)	C38—C39—H39A	109.5
O4—C20—H20A	109.0	C38—C39—H39B	109.4
O3—C20—H20A	109.0	H39A—C39—H39B	109.5
O4—C20—H20B	109.0	O5—C40—C41	106.3 (6)
O3—C20—H20B	109.0	O5—C40—H40	110.6
H20A—C20—H20B	107.8	C41—C40—H40	110.5
C3—C21—C30	114.09 (19)	C40—C41—H41A	108.4
C3—C21—C17	107.41 (18)	C40—C41—H41B	110.0
C30—C21—C17	114.4 (2)	H41A—C41—H41B	109.5
C3—C21—H21	106.8	C6—O1—C8	116.5 (2)
C30—C21—H21	106.8	C11—O2—C8	117.04 (19)
C17—C21—H21	106.8	C16—O3—C20	116.33 (17)

C23—C22—C9	113.8 (2)	C2—O4—C20	117.43 (18)
C23—C22—H22A	108.8	C38—O5—C40	117.2 (5)
C9—C22—H22A	108.8		
C6—C1—C2—C3	−0.5 (4)	C5—C9—C22—C23	−62.8 (3)
C7—C1—C2—C3	−179.2 (2)	C12—C9—C22—C23	173.6 (2)
C6—C1—C2—O4	173.5 (2)	C9—C22—C23—C24	169.1 (2)
C7—C1—C2—O4	−5.2 (4)	C22—C23—C24—C25	−79.7 (4)
O4—C2—C3—C4	−175.4 (2)	C22—C23—C24—C29	98.6 (3)
C1—C2—C3—C4	−1.6 (4)	C29—C24—C25—C26	−0.3 (5)
O4—C2—C3—C21	6.9 (3)	C23—C24—C25—C26	178.1 (3)
C1—C2—C3—C21	−179.3 (2)	C24—C25—C26—C27	0.4 (5)
C2—C3—C4—C5	2.7 (3)	C25—C26—C27—C28	0.0 (5)
C21—C3—C4—C5	−179.6 (2)	C26—C27—C28—C29	−0.6 (5)
C3—C4—C5—C6	−1.8 (4)	C27—C28—C29—C24	0.7 (5)
C3—C4—C5—C9	−177.9 (2)	C25—C24—C29—C28	−0.3 (4)
C4—C5—C6—C1	−0.4 (4)	C23—C24—C29—C28	−178.7 (3)
C9—C5—C6—C1	175.8 (2)	C3—C21—C30—C31	169.4 (5)
C4—C5—C6—O1	176.8 (2)	C17—C21—C30—C31	−66.4 (6)
C9—C5—C6—O1	−7.0 (4)	C3—C21—C30—C31A	169.6 (5)
C2—C1—C6—C5	1.5 (4)	C17—C21—C30—C31A	−66.1 (5)
C7—C1—C6—C5	−179.8 (2)	C31A—C30—C31—C32	176 (100)
C2—C1—C6—O1	−175.8 (2)	C21—C30—C31—C32	−168.8 (7)
C7—C1—C6—O1	2.9 (4)	C30—C31—C32—C33	−82.8 (17)
C6—C5—C9—C12	−83.5 (3)	C30—C31—C32—C37	93 (2)
C4—C5—C9—C12	92.5 (3)	C37—C32—C33—C34	2 (3)
C6—C5—C9—C22	150.1 (2)	C31—C32—C33—C34	178.7 (13)
C4—C5—C9—C22	−33.9 (3)	C32—C33—C34—C35	3 (2)
C11^{i}—C10—C11—C12	−0.8 (5)	C33—C34—C35—C36	−8 (2)
C14—C10—C11—C12	179.6 (3)	C34—C35—C36—C37	7 (2)
C11^{i}—C10—C11—O2	175.83 (17)	C33—C32—C37—C36	−3 (3)
C14—C10—C11—O2	−3.8 (4)	C31—C32—C37—C36	−179.8 (13)
O2—C11—C12—C13	−176.9 (2)	C35—C36—C37—C32	−1 (2)
C10—C11—C12—C13	−0.4 (4)	C31—C30—C31A—C32A	−15 (67)
O2—C11—C12—C9	7.2 (3)	C21—C30—C31A—C32A	179.6 (6)
C10—C11—C12—C9	−176.3 (2)	C30—C31A—C32A—C33A	−138.5 (14)
C5—C9—C12—C13	−92.7 (3)	C30—C31A—C32A—C37A	39.3 (17)
C22—C9—C12—C13	34.3 (3)	C37A—C32A—C33A—C34A	3 (2)
C5—C9—C12—C11	83.1 (3)	C31A—C32A—C33A—C34A	−179.1 (10)
C22—C9—C12—C11	−149.9 (2)	C32A—C33A—C34A—C35A	2.0 (16)
C11—C12—C13—C12^{i}	1.6 (5)	C33A—C34A—C35A—C36A	−4.9 (15)
C9—C12—C13—C12^{i}	177.53 (19)	C34A—C35A—C36A—C37A	2.7 (17)
C16^{i}—C15—C16—C17	−0.6 (4)	C33A—C32A—C37A—C36A	−5 (2)
C19—C15—C16—C17	178.2 (3)	C31A—C32A—C37A—C36A	176.9 (11)
C16^{i}—C15—C16—O3	−176.47 (16)	C35A—C36A—C37A—C32A	2.5 (17)
C19—C15—C16—O3	2.2 (4)	C5—C6—O1—C8	84.0 (3)
C15—C16—C17—C18	0.7 (4)	C1—C6—O1—C8	−98.7 (3)
O3—C16—C17—C18	176.6 (2)	O2—C8—O1—C6	−92.1 (3)

C15—C16—C17—C21	176.8 (2)	C12—C11—O2—C8	−82.9 (3)
O3—C16—C17—C21	−7.3 (3)	C10—C11—O2—C8	100.4 (3)
C16—C17—C18—C17^{i}	−0.9 (4)	O1—C8—O2—C11	91.4 (3)
C21—C17—C18—C17^{i}	−176.90 (17)	C17—C16—O3—C20	84.4 (3)
C2—C3—C21—C30	−147.6 (2)	C15—C16—O3—C20	−99.6 (3)
C4—C3—C21—C30	34.7 (3)	O4—C20—O3—C16	−91.5 (2)
C2—C3—C21—C17	84.5 (3)	C3—C2—O4—C20	−83.6 (3)
C4—C3—C21—C17	−93.1 (3)	C1—C2—O4—C20	102.3 (3)
C18—C17—C21—C3	91.1 (3)	O3—C20—O4—C2	91.1 (2)
C16—C17—C21—C3	−84.8 (2)	O6—C38—O5—C40	0.0
C18—C17—C21—C30	−36.6 (3)	C39—C38—O5—C40	180.0
C16—C17—C21—C30	147.5 (2)	C41—C40—O5—C38	180.0

Symmetry code: (i) x, $-y+1/2$, z.