Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

5,11,17,23-Tetramethyl-2,8,14,20-tetrakis(2-phenylethyl)-4,6,10,12,16,-18,22,24-octahydroxycalix[4]arene methanol pentasolvate 0.10-hydrate

Holger B. Friedrich,[a] R. Alan Howie,[b] Glenn E. M. Maguire[a]* and Michael G. Mc Kay[a]

[a]School of Chemistry, University of KwaZulu-Natal, Durban 4000, South Africa, and [b]Department of Chemistry, University of Aberdeen, Aberdeen AB24 3UE, Scotland
Correspondence e-mail: maguireg@ukzn.ac.za

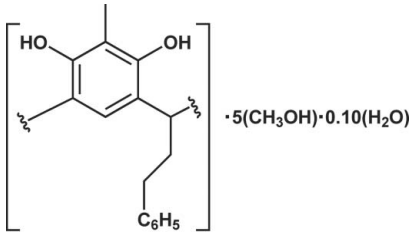
Received 28 September 2007; accepted 12 October 2007

Key indicators: single-crystal X-ray study; T = 105 K; mean σ(C–C) = 0.003 Å; disorder in solvent or counterion; R factor = 0.058; wR factor = 0.175; data-to-parameter ratio = 18.6.

The title compound [systematic name: 5,11,17,23-tetramethyl-2,8,14,16-tetrakis(2-phenylethyl)pentacyclo[19.3.1.1^{3,7}.1^{9,13}.1^{15,19}]octacosa-1(25),3,5,7(28),9,11,13 (27),15,17,19(26),21,23-dodecaene-4,6,10,12,16,18,22,24-octol methanol pentasolvate 0.10-hydrate], $C_{64}H_{64}O_8\cdot5CH_4O\cdot0.103H_2O$, was synthesized as a new synthetic intermediate for resorcin[4]arene cavitand formation. The structure displays extensive O—H···O intra- and intermolecular hydrogen bonding.

Related literature

For related literature, see: Cram *et al.* (1988); Tunstad *et al.* (1989).



Experimental

Crystal data

$C_{64}H_{64}O_8\cdot5CH_4O\cdot0.103H_2O$
M_r = 1123.22
Monoclinic, $P2_1/n$
a = 12.3109 (3) Å
b = 30.6151 (7) Å
c = 16.5024 (3) Å
β = 92.8385 (19)°
V = 6212.1 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 105 (2) K
0.65 × 0.50 × 0.50 mm

Data collection

Oxford Xcalibur-2 area-detector diffractometer
Absorption correction: multi-scan (*ABSPACK* in *CrysAlis RED*; Oxford Diffraction, 2006)
T_{min} = 0.785, T_{max} = 0.960
74273 measured reflections
14218 independent reflections
10324 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.175
S = 1.09
14218 reflections
765 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.67 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O8	0.84	1.87	2.709 (2)	178
O2—H2···O6^{i}	0.84	1.85	2.6442 (19)	157
O3—H3···O2	0.84	1.85	2.687 (2)	171
O4—H4···O5	0.84	1.98	2.805 (2)	169
O5—H5···O10	0.84	1.82	2.643 (2)	165
O6—H6···O7	0.84	1.86	2.7001 (19)	177
O7—H7···O9^{ii}	0.84	1.86	2.639 (2)	154
O8—H8···O12	0.84	1.90	2.631 (2)	145
O10—H10···O13^{ii}	0.84	1.92	2.754 (3)	173
O11—H11···O1	0.84	1.93	2.716 (2)	156
O12—H12···O13	0.84	1.91	2.748 (3)	174
O13—H13···O11	0.84	1.84	2.671 (3)	172

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; 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: *SHELXL97* and *PLATON* (Spek, 2003).

The authors acknowledge the DST-NRF Centre of Excellence in Catalysis, c*change, for financial support and Professor Orde Munro at the University of KwaZulu-Natal (Pietermaritzburg) for data acquisition.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2307).

References

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.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Oxford Diffraction (2006). *CrysAlis CCD* and *CrysAlis RED*. Version 1.171.29.9. Oxford Diffraction Ltd, Abingdon, Oxfordshire, England.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Tunstad, L. M., Tucker, J. A., Dalcanale, E., Weiser, J., Bryant, J. A., Sherman, J. C., Helgeson, R. C., Knobler, C. B. & Cram, D. J. (1989). *J. Org. Chem.* **54**, 1305–1312.

  doi:10.1107/S160053680705012X

supplementary materials

Acta Cryst. (2007). E**63**, o4346 [**doi:10.1107/S160053680705012X**]

5,11,17,23-Tetramethyl-2,8,14,20-tetrakis(2-phenylethyl)-4,6,10,12,16,18,22,24-octahydroxycalix[4]arene methanol pentasolvate 0.10-hydrate

H. B. Friedrich, R. A. Howie, G. E. M. Maguire and M. G. Mc Kay

Comment

In the title compound, (I), the [4]arene moiety, (I A), is a cyclic tetramer. The labelling scheme for one of the monomers, shown in Fig.1, extends over the whole molecule such that for monomer n the numbers p and q associated with particular C and O atoms, respectively, become p' = p + 16 *x* (n − 1) and q' = q + 2 *x* (n − 1). The asymmetric unit of (I) is shown in Fig. 2. The bond lengths and bond angles all fall within the normal ranges and are not discussed further. In molecule (I A) the 2-phenylethyl 'feet' all lie to one side of the macrocyclic ring surrounding the site of the partially occupied water molecule, O1W, with their phenyl groups in a cyclic edge-to-face arrangement. All but one of the OH groups participate in O—H···O hydrogen-bond formation (Table 1) and all but three of those listed occur within the asymmetric unit (Fig.2). The acceptor for methanolic O9 is the benzene ring, with centroid *Cg*, defined by C17—C22. The parameters for this O—H..π contact include the H..*Cg* distance and O—H..*Cg* angle of 2.48 Å and 173°, respectively. The O2—H2.·O6^{i} (symmetry code as in Table 1) hydrogen-bond provides direct connection between molecules (1 A) in the propagation of chains in the direction of the cell edge a. The hydrogen-bonds O7—H7.·O9^{ii} and O10—H10.·O13^{ii} link chains, related to one another by crystallographic centres of symmetry, in pairs as shown in Fig. 3.

The partial occupancy of the water molecule associated with (I) is not exclusive to this structure, and has been noted before in related structures. For related literature, see Tunstad *et al.* (1989) and Cram *et al.* (1988).

Experimental

2-Methylresorcinol (12.41 g, 0.10 mol) was added to a stirred mixture of water (100 ml), ethanol (200 ml) and 32% aqueous HCl (50 ml). The reaction vessel and its contents were cooled to a temperature in the range of 273–278 K, by immersion in an ice-salt bath before 3-phenylpropionaldehyde (13.42 g, 0.10 mol) was added slowly over 30 minutes. Once addition was complete, the solution was allowed to slowly attain room temperature, after which time it was refluxed at 353 K for 48 h. The solution was cooled to room temperature, and water added in order to precipitate out the crude product. The brown material that separated was filtered from the solution, washed with cold 1:1 e thanol:water until the washings were light yellow and then redissolved in warm methanol and recrystallized overnight. The resulting peach-coloured material was then filtered from the methanol solution and thereafter stirred in hexane to remove residual aldehyde. The solid was filtered from hexane and dried, to yield the title compound as a peach-coloured microcrystalline solid. (13.93 g, 58%). mp 555–559 K. ^{1}H NMR [d_6-DMSO, 300 MHz]: d= 1.97 (s, 12 H, Ar CH_3), 2.49–2.50 (m, 28 H, CH_2CH$_2$Ar, and DMSO as solvent signal), 4.26–4.30 (t, *J* = 7.0 Hz, 4 H, C*H*CH$_2$), 7.13–7.29 (m, 20 H, C$_6H_5$), 7.39 (s, 4 H, Ar *H*), 8.73 (s, 8 H, O*H*). ^{13}C NMR [$CDCl_3$, 75 MHz]: d = 149.34, 142.40, 128.70, 128.38, 125.81, 125.00, 111.88, 40.533, 40.250, 39.134, 38.859, 10.264. Anal Calcd for $C_{64}H_{64}O_8$ (961.195): C 79.97, H 6.71. Found: C 80.42, H 6.79.

A solution of the compound in hot methanol, subsequently cooled in a refrigerator at 277 K, provided crystals suitable for X-ray crystallography.

Refinement

In the final stages of refinement H atoms were placed in calculated positions with C—H distances of 0.95, 0.98, 0.99 and 1.00 Å for H bonded to aryl, methyl, methylene and tertiary C atoms, respectively, and at O—H distances of 0.84 Å. They were than refined with a riding model with U_{iso}(H) = 1.5U_{eq}(X) for X = O or methyl C and U_{iso}(H) = 1.2U_{eq}(C) otherwise. The torsion angles of the methyl and hydroxyl groups were also refined. The atom O1W was introduced into the structural model to account for an electron density feature of approximately 1 e/Å^3. Its coordinates and sof were refined with its U_{iso} fixed, somewhat arbitrarily, at 0.05. This feature is interpreted as the O atom of a water molecule but its low occupancy factor [0.103 (5)] has prevented discovery of the associated H atoms which are, therefore, absent from the structural model. Rather large and significantly anisotropic displacement parameters for atoms C60—C62 are indicative of libration in this part of the molecule. The largest residual electron density peak of 0.67 e/Å^3 is 0.94 Å from C11.

Figures

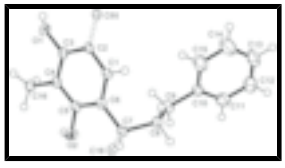


Fig. 1. A view of one component of the cyclic tetramer. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as spheres of arbitrary radii. Dashed bonds indicate links to the neighbouring monomer units. This is the key to the labelling of the entire molecule - see text.

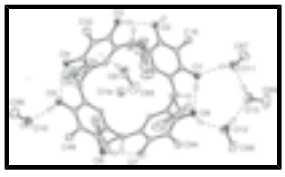


Fig. 2. The asymmetric unit of (1). Displacement ellipsoids are drawn at the 50% probability level and H atoms, where shown, are drawn as spheres of arbitrary radii. Dashed lines represent O—H···O and, for O9, O—H···π hydrogen bonds. Selected atoms are labelled.

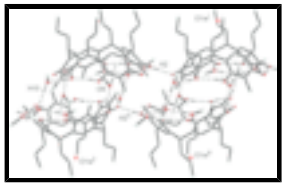


Fig. 3. Hydrogen bonding in the double chain in (1). Displacement ellipsoids are drawn at the 10% probability level and H atoms, where shown, are drawn as spheres of arbitrary radii. Dashed lines represent O—H···O and, for O9, O—H···π hydrogen bonds. Selected atoms are labelled. The drawing has been reduced in size and complexity by representing the phenyl groups of the 2-phenylethyl substituents by only those C atoms forming C—C bonds. [Symmetry codes: (i) x + 1, y, z; (ii) −x + 1, −y, −z + 1; (iii) −x + 2, −y, −z + 1.]

5,11,17,23-tetramethyl-2,8,14,16-tetrakis(2-phenylethyl)pentacyclo [19.3.1.1^{3,7}.1^{9,13}.1^{15,19}]octacosa-1(25),3,5,7(28),9,11,13 (27),15,17,19 (26),21,23- dodecaene-4,6,10,12,16,18,22,24-octol

Crystal data

$C_{64}H_{64}O_8$·5CH_4O·0.103H_2O	F_{000} = 2412
M_r = 1123.22	D_x = 1.201 Mg m^{-3}
Monoclinic, $P2_1/n$	Melting point: 555-559 K
Hall symbol: -P 2yn	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 12.3109 (3) Å	Cell parameters from 39787 reflections
b = 30.6151 (7) Å	θ = 3.7–34.1°
c = 16.5024 (3) Å	μ = 0.08 mm^{-1}
β = 92.8385 (19)°	T = 105 (2) K

V = 6212.1 (2) Å^3	Block, light orange
Z = 4	0.65 × 0.50 × 0.50 mm

Data collection

Oxford Xcalibur-2 area-detector diffractometer	14218 independent reflections
Radiation source: Enhance (Mo) X-ray Source	10324 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.028
Detector resolution: 0 pixels mm^{-1}	θ_{max} = 27.5º
T = 105(2) K	θ_{min} = 3.7º
φ and ω scans	h = −15→15
Absorption correction: multi-scan [empirical (using intensity measurements) absorption correction using spherical harmonics, implemented in the SCALE3 ABSPACK scaling algorithm of CrysAlis RED (Oxford Diffraction, 2006)]	k = −39→39
T_{min} = 0.785, T_{max} = 0.960	l = −21→21
74273 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.058	H-atom parameters constrained
$wR(F^2)$ = 0.175	$w = 1/[\sigma^2(F_o^2) + (0.0811P)^2 + 4.858P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.09	$(\Delta/\sigma)_{max}$ = 0.001
14218 reflections	$\Delta\rho_{max}$ = 0.67 e Å^{-3}
765 parameters	$\Delta\rho_{min}$ = −0.39 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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.

O—H..pi geometry (extract from *PLATON* output listing)

O—H H.·*Cg* H-Perp Gamma O—H.·*Cg* O.·*Cg* O—H,Pi

O9—H9.·*Cg* 0.84 2.48 2.409 13.89 173 3.3182 (18) 80

Cg is the centroid of the ring defined by C17—C22

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 $F^2 > 2\text{sigma}(F^2)$ 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)
O1	0.85729 (11)	0.09144 (5)	0.32812 (8)	0.0246 (3)	
H1	0.7916	0.0919	0.3116	0.037*	
O2	1.03772 (12)	0.08075 (5)	0.59094 (9)	0.0300 (3)	
H2	1.0931	0.0746	0.5655	0.045*	
O3	0.96781 (12)	0.05706 (5)	0.73621 (9)	0.0286 (3)	
H3	0.9920	0.0671	0.6933	0.043*	
O4	0.63258 (12)	0.03653 (5)	0.85613 (8)	0.0252 (3)	
H4	0.5664	0.0424	0.8474	0.038*	
O5	0.40942 (12)	0.04559 (5)	0.81674 (9)	0.0273 (3)	
H5	0.3490	0.0392	0.8351	0.041*	
O6	0.23362 (10)	0.05510 (4)	0.55231 (8)	0.0210 (3)	
H6	0.2554	0.0596	0.5056	0.032*	
O7	0.30719 (11)	0.06709 (4)	0.40248 (8)	0.0227 (3)	
H7	0.3129	0.0413	0.3856	0.034*	
O8	0.64617 (12)	0.09095 (5)	0.27331 (8)	0.0260 (3)	
H8	0.6123	0.0842	0.2296	0.039*	
O9	0.70597 (16)	0.01875 (5)	0.60865 (10)	0.0411 (4)	
H9	0.7256	0.0358	0.6465	0.062*	
O10	0.23037 (16)	0.03459 (6)	0.89641 (11)	0.0454 (4)	
H10	0.2039	0.0095	0.9020	0.068*	
O11	0.98560 (14)	0.07699 (6)	0.20163 (12)	0.0434 (4)	
H11	0.9627	0.0846	0.2466	0.065*	
O12	0.62774 (17)	0.06765 (7)	0.11976 (11)	0.0535 (5)	
H12	0.6909	0.0610	0.1071	0.080*	
O13	0.84004 (16)	0.05017 (6)	0.08692 (11)	0.0468 (5)	
H13	0.8876	0.0562	0.1235	0.070*	
O1W	0.6027 (15)	0.2344 (6)	0.6306 (12)	0.050*	0.103 (5)
C1	0.80760 (15)	0.14614 (6)	0.51960 (11)	0.0188 (4)	
H1A	0.7578	0.1662	0.5413	0.023*	
C2	0.79593 (15)	0.13561 (6)	0.43786 (11)	0.0184 (4)	
C3	0.86596 (15)	0.10398 (6)	0.40833 (11)	0.0187 (4)	
C4	0.94785 (15)	0.08440 (6)	0.45757 (12)	0.0197 (4)	
C5	0.95919 (15)	0.09790 (6)	0.53830 (12)	0.0208 (4)	
C6	0.88912 (15)	0.12854 (6)	0.57123 (11)	0.0198 (4)	
C7	0.90150 (15)	0.14173 (6)	0.66068 (11)	0.0200 (4)	
H7A	0.9787	0.1361	0.6793	0.024*	
C8	0.87922 (16)	0.19076 (6)	0.67358 (12)	0.0224 (4)	
H8A	0.8028	0.1970	0.6557	0.027*	
H8B	0.8875	0.1972	0.7323	0.027*	
C9	0.95389 (17)	0.22143 (7)	0.62824 (13)	0.0266 (4)	

H9A	1.0263	0.2222	0.6572	0.032*
H9B	0.9635	0.2097	0.5732	0.032*
C10	0.90986 (17)	0.26746 (7)	0.62107 (12)	0.0251 (4)
C11	0.9589 (2)	0.30254 (9)	0.66018 (17)	0.0498 (7)
H11A	1.0245	0.2981	0.6919	0.060*
C12	0.9150 (3)	0.34435 (9)	0.6546 (2)	0.0586 (8)
H12A	0.9489	0.3674	0.6848	0.070*
C13	0.8263 (2)	0.35265 (8)	0.60759 (17)	0.0451 (6)
H13A	0.7988	0.3816	0.6026	0.054*
C14	0.7747 (3)	0.31839 (10)	0.5659 (2)	0.0648 (9)
H14	0.7111	0.3236	0.5324	0.078*
C15	0.8169 (3)	0.27649 (9)	0.5738 (2)	0.0576 (8)
H15	0.7806	0.2532	0.5456	0.069*
C16	1.02018 (16)	0.04978 (7)	0.42312 (12)	0.0242 (4)
H16A	1.0795	0.0638	0.3955	0.036*
H16B	0.9773	0.0317	0.3843	0.036*
H16C	1.0505	0.0314	0.4672	0.036*
C17	0.72402 (15)	0.12586 (6)	0.72762 (11)	0.0197 (4)
H17	0.6972	0.1522	0.7038	0.024*
C18	0.82944 (15)	0.11320 (6)	0.71213 (11)	0.0197 (4)
C19	0.86595 (16)	0.07334 (6)	0.74615 (11)	0.0213 (4)
C20	0.79892 (16)	0.04773 (6)	0.79338 (11)	0.0227 (4)
C21	0.69420 (16)	0.06269 (6)	0.80808 (11)	0.0202 (4)
C22	0.65541 (15)	0.10231 (6)	0.77587 (11)	0.0184 (4)
C23	0.54167 (15)	0.11898 (6)	0.79234 (11)	0.0190 (4)
H23	0.5216	0.1063	0.8454	0.023*
C24	0.53656 (16)	0.16896 (6)	0.80089 (11)	0.0217 (4)
H24A	0.5586	0.1825	0.7497	0.026*
H24B	0.4604	0.1777	0.8091	0.026*
C25	0.60952 (19)	0.18671 (7)	0.87140 (13)	0.0289 (5)
H25A	0.5798	0.1776	0.9234	0.035*
H25B	0.6832	0.1740	0.8687	0.035*
C26	0.61727 (18)	0.23588 (7)	0.86898 (12)	0.0278 (4)
C27	0.54097 (19)	0.26162 (8)	0.90601 (14)	0.0337 (5)
H27	0.4835	0.2481	0.9330	0.040*
C28	0.5477 (2)	0.30689 (8)	0.90412 (15)	0.0414 (6)
H28	0.4960	0.3241	0.9307	0.050*
C29	0.6291 (2)	0.32680 (8)	0.86384 (16)	0.0455 (7)
H29	0.6333	0.3578	0.8624	0.055*
C30	0.7051 (2)	0.30190 (9)	0.82532 (17)	0.0474 (6)
H30	0.7610	0.3156	0.7969	0.057*
C31	0.6987 (2)	0.25636 (8)	0.82873 (15)	0.0394 (5)
H31	0.7512	0.2392	0.8029	0.047*
C32	0.8400 (2)	0.00431 (7)	0.82479 (14)	0.0331 (5)
H32A	0.8684	−0.0126	0.7801	0.050*
H32B	0.7802	−0.0118	0.8481	0.050*
H32C	0.8981	0.0090	0.8666	0.050*
C33	0.44583 (15)	0.12271 (6)	0.65276 (11)	0.0180 (4)
H33	0.4909	0.1470	0.6417	0.022*

C34	0.45880 (15)	0.10231 (6)	0.72794 (11)	0.0191 (4)
C35	0.39305 (15)	0.06632 (6)	0.74253 (11)	0.0206 (4)
C36	0.31748 (15)	0.05023 (6)	0.68409 (12)	0.0205 (4)
C37	0.30842 (14)	0.07197 (6)	0.60963 (11)	0.0180 (4)
C38	0.36957 (15)	0.10907 (6)	0.59312 (11)	0.0175 (4)
C39	0.34967 (14)	0.13512 (6)	0.51486 (11)	0.0166 (3)
H39	0.2751	0.1273	0.4926	0.020*
C40	0.34971 (15)	0.18472 (6)	0.53140 (12)	0.0203 (4)
H40A	0.4218	0.1933	0.5555	0.024*
H40B	0.3384	0.2004	0.4792	0.024*
C41	0.26174 (16)	0.19876 (6)	0.58852 (13)	0.0241 (4)
H41A	0.1893	0.1954	0.5605	0.029*
H41B	0.2645	0.1795	0.6368	0.029*
C42	0.27684 (16)	0.24562 (6)	0.61530 (12)	0.0224 (4)
C43	0.35539 (18)	0.25666 (7)	0.67442 (14)	0.0316 (5)
H43	0.3986	0.2343	0.6996	0.038*
C44	0.3725 (2)	0.29988 (8)	0.69781 (15)	0.0367 (5)
H44	0.4273	0.3068	0.7383	0.044*
C45	0.3100 (2)	0.33255 (7)	0.66223 (14)	0.0355 (5)
H45	0.3214	0.3621	0.6780	0.043*
C46	0.2313 (2)	0.32216 (8)	0.60406 (16)	0.0435 (6)
H46	0.1879	0.3446	0.5794	0.052*
C47	0.2143 (2)	0.27888 (7)	0.58070 (14)	0.0359 (5)
H47	0.1590	0.2721	0.5405	0.043*
C48	0.24737 (17)	0.01105 (7)	0.69962 (13)	0.0277 (4)
H48A	0.2850	−0.0080	0.7396	0.042*
H48B	0.2333	−0.0050	0.6488	0.042*
H48C	0.1782	0.0207	0.7205	0.042*
C49	0.52746 (15)	0.14412 (6)	0.44485 (11)	0.0171 (3)
H49	0.5457	0.1666	0.4828	0.021*
C50	0.42873 (15)	0.12269 (6)	0.45053 (11)	0.0175 (4)
C51	0.40527 (15)	0.08872 (6)	0.39549 (11)	0.0184 (4)
C52	0.47517 (15)	0.07751 (6)	0.33467 (11)	0.0193 (4)
C53	0.57277 (15)	0.10076 (6)	0.33119 (11)	0.0188 (4)
C54	0.60091 (15)	0.13416 (6)	0.38607 (11)	0.0174 (4)
C55	0.70990 (15)	0.15767 (6)	0.38214 (11)	0.0177 (4)
H55	0.7338	0.1543	0.3254	0.021*
C56	0.70088 (16)	0.20715 (6)	0.39854 (12)	0.0208 (4)
H56A	0.6709	0.2118	0.4524	0.025*
H56B	0.7742	0.2205	0.3992	0.025*
C57	0.62734 (17)	0.22929 (6)	0.33338 (12)	0.0252 (4)
H57A	0.6613	0.2264	0.2805	0.030*
H57B	0.5569	0.2136	0.3292	0.030*
C58	0.6057 (2)	0.27697 (7)	0.34883 (13)	0.0317 (5)
C59	0.6691 (3)	0.30944 (9)	0.31820 (16)	0.0539 (8)
H59	0.7307	0.3021	0.2885	0.065*
C60	0.6428 (4)	0.35360 (10)	0.3308 (2)	0.0811 (14)
H60	0.6861	0.3759	0.3085	0.097*
C61	0.5566 (4)	0.36466 (11)	0.3742 (2)	0.0849 (15)

H61	0.5404	0.3946	0.3831	0.102*
C62	0.4934 (3)	0.33305 (14)	0.4051 (3)	0.0876 (15)
H62	0.4325	0.3409	0.4352	0.105*
C63	0.5171 (2)	0.28891 (10)	0.3929 (2)	0.0554 (8)
H63	0.4725	0.2670	0.4149	0.066*
C64	0.44652 (17)	0.04193 (7)	0.27437 (13)	0.0276 (4)
H64A	0.4880	0.0461	0.2258	0.041*
H64B	0.3685	0.0431	0.2595	0.041*
H64C	0.4644	0.0134	0.2986	0.041*
C65	0.6480 (3)	0.04221 (9)	0.54767 (19)	0.0532 (7)
H65A	0.5834	0.0554	0.5701	0.080*
H65B	0.6257	0.0224	0.5033	0.080*
H65C	0.6945	0.0653	0.5270	0.080*
C66	0.2449 (3)	0.05482 (11)	0.97324 (17)	0.0541 (7)
H66A	0.2831	0.0826	0.9675	0.081*
H66B	0.2879	0.0357	1.0100	0.081*
H66C	0.1737	0.0602	0.9955	0.081*
C67	1.0729 (2)	0.10443 (9)	0.18161 (19)	0.0473 (6)
H67A	1.0485	0.1349	0.1805	0.071*
H67B	1.0977	0.0963	0.1281	0.071*
H67C	1.1330	0.1011	0.2224	0.071*
C68	0.5532 (3)	0.05356 (11)	0.05928 (16)	0.0518 (7)
H68A	0.5055	0.0314	0.0814	0.078*
H68B	0.5921	0.0409	0.0145	0.078*
H68C	0.5094	0.0784	0.0393	0.078*
C69	0.8670 (3)	0.07095 (13)	0.01281 (19)	0.0721 (10)
H69A	0.8737	0.1025	0.0216	0.108*
H69B	0.8095	0.0652	−0.0291	0.108*
H69C	0.9361	0.0593	−0.0048	0.108*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0263 (7)	0.0256 (7)	0.0220 (7)	−0.0026 (6)	0.0011 (5)	−0.0051 (6)
O2	0.0242 (7)	0.0421 (9)	0.0236 (7)	0.0154 (7)	0.0016 (6)	0.0012 (6)
O3	0.0277 (7)	0.0281 (8)	0.0304 (8)	0.0100 (6)	0.0045 (6)	0.0046 (6)
O4	0.0307 (7)	0.0216 (7)	0.0234 (7)	0.0007 (6)	0.0016 (6)	0.0044 (5)
O5	0.0289 (7)	0.0278 (8)	0.0253 (7)	−0.0037 (6)	0.0015 (6)	0.0084 (6)
O6	0.0196 (6)	0.0204 (7)	0.0229 (7)	−0.0027 (5)	0.0009 (5)	−0.0025 (5)
O7	0.0232 (7)	0.0178 (6)	0.0272 (7)	−0.0045 (5)	0.0026 (5)	−0.0070 (5)
O8	0.0291 (7)	0.0276 (8)	0.0216 (7)	0.0004 (6)	0.0050 (6)	−0.0064 (6)
O9	0.0655 (12)	0.0222 (8)	0.0349 (9)	−0.0091 (8)	−0.0030 (8)	−0.0058 (7)
O10	0.0569 (11)	0.0409 (10)	0.0396 (10)	−0.0131 (9)	0.0153 (8)	−0.0077 (8)
O11	0.0363 (9)	0.0382 (10)	0.0570 (12)	0.0048 (7)	0.0150 (8)	−0.0054 (8)
O12	0.0586 (12)	0.0630 (13)	0.0380 (10)	0.0010 (10)	−0.0055 (9)	−0.0196 (9)
O13	0.0572 (12)	0.0488 (11)	0.0350 (9)	−0.0158 (9)	0.0092 (8)	−0.0053 (8)
C1	0.0171 (8)	0.0158 (8)	0.0236 (9)	0.0002 (7)	0.0039 (7)	0.0000 (7)
C2	0.0184 (9)	0.0136 (8)	0.0231 (9)	−0.0022 (7)	0.0010 (7)	0.0016 (7)

C3	0.0191 (9)	0.0157 (8)	0.0217 (9)	−0.0040 (7)	0.0035 (7)	−0.0002 (7)
C4	0.0187 (9)	0.0156 (8)	0.0249 (9)	−0.0016 (7)	0.0038 (7)	0.0003 (7)
C5	0.0177 (9)	0.0209 (9)	0.0238 (9)	0.0012 (7)	0.0019 (7)	0.0030 (7)
C6	0.0195 (9)	0.0194 (9)	0.0206 (9)	−0.0015 (7)	0.0027 (7)	−0.0002 (7)
C7	0.0198 (9)	0.0202 (9)	0.0197 (9)	0.0025 (7)	−0.0009 (7)	−0.0006 (7)
C8	0.0240 (9)	0.0207 (9)	0.0225 (9)	−0.0017 (8)	0.0020 (7)	−0.0019 (7)
C9	0.0250 (10)	0.0242 (10)	0.0306 (11)	−0.0026 (8)	0.0034 (8)	0.0005 (8)
C10	0.0281 (10)	0.0223 (10)	0.0251 (10)	−0.0039 (8)	0.0020 (8)	−0.0001 (8)
C11	0.0610 (17)	0.0344 (13)	0.0509 (16)	−0.0015 (12)	−0.0270 (13)	−0.0012 (11)
C12	0.085 (2)	0.0276 (13)	0.0608 (18)	−0.0049 (14)	−0.0238 (16)	−0.0105 (12)
C13	0.0567 (16)	0.0236 (11)	0.0555 (16)	0.0038 (11)	0.0073 (13)	0.0057 (11)
C14	0.0617 (19)	0.0400 (16)	0.089 (2)	0.0092 (14)	−0.0299 (18)	0.0011 (16)
C15	0.0624 (18)	0.0298 (13)	0.077 (2)	0.0014 (12)	−0.0309 (16)	−0.0089 (13)
C16	0.0229 (9)	0.0223 (10)	0.0275 (10)	0.0034 (8)	0.0022 (8)	−0.0021 (8)
C17	0.0244 (9)	0.0170 (9)	0.0176 (8)	0.0021 (7)	−0.0012 (7)	0.0002 (7)
C18	0.0229 (9)	0.0191 (9)	0.0169 (8)	0.0012 (7)	−0.0008 (7)	−0.0020 (7)
C19	0.0238 (9)	0.0206 (9)	0.0193 (9)	0.0051 (7)	−0.0016 (7)	−0.0031 (7)
C20	0.0291 (10)	0.0186 (9)	0.0201 (9)	0.0061 (8)	−0.0018 (8)	−0.0006 (7)
C21	0.0270 (10)	0.0180 (9)	0.0155 (8)	−0.0006 (7)	−0.0007 (7)	−0.0011 (7)
C22	0.0219 (9)	0.0172 (9)	0.0159 (8)	0.0013 (7)	−0.0009 (7)	−0.0038 (7)
C23	0.0232 (9)	0.0165 (9)	0.0173 (8)	0.0015 (7)	0.0008 (7)	−0.0002 (7)
C24	0.0254 (10)	0.0196 (9)	0.0199 (9)	0.0044 (7)	−0.0015 (7)	−0.0031 (7)
C25	0.0412 (12)	0.0199 (10)	0.0247 (10)	0.0055 (9)	−0.0069 (9)	−0.0057 (8)
C26	0.0348 (11)	0.0231 (10)	0.0244 (10)	0.0067 (8)	−0.0094 (8)	−0.0075 (8)
C27	0.0365 (12)	0.0324 (12)	0.0314 (11)	0.0104 (9)	−0.0077 (9)	−0.0073 (9)
C28	0.0534 (15)	0.0318 (12)	0.0374 (13)	0.0202 (11)	−0.0143 (11)	−0.0097 (10)
C29	0.0700 (18)	0.0222 (11)	0.0417 (14)	0.0085 (11)	−0.0220 (13)	−0.0077 (10)
C30	0.0589 (17)	0.0359 (14)	0.0469 (15)	−0.0119 (12)	−0.0023 (13)	0.0005 (11)
C31	0.0466 (14)	0.0303 (12)	0.0413 (13)	0.0022 (10)	0.0031 (11)	−0.0106 (10)
C32	0.0388 (12)	0.0240 (11)	0.0367 (12)	0.0112 (9)	0.0041 (10)	0.0065 (9)
C33	0.0189 (8)	0.0150 (8)	0.0204 (9)	−0.0003 (7)	0.0027 (7)	−0.0025 (7)
C34	0.0200 (9)	0.0174 (9)	0.0200 (9)	0.0025 (7)	0.0017 (7)	−0.0025 (7)
C35	0.0223 (9)	0.0189 (9)	0.0208 (9)	0.0029 (7)	0.0029 (7)	0.0011 (7)
C36	0.0207 (9)	0.0158 (9)	0.0251 (9)	0.0003 (7)	0.0037 (7)	0.0000 (7)
C37	0.0154 (8)	0.0164 (8)	0.0222 (9)	0.0018 (7)	0.0005 (7)	−0.0044 (7)
C38	0.0193 (9)	0.0140 (8)	0.0193 (9)	0.0032 (7)	0.0027 (7)	−0.0017 (7)
C39	0.0159 (8)	0.0145 (8)	0.0191 (8)	0.0001 (6)	−0.0002 (7)	−0.0018 (7)
C40	0.0226 (9)	0.0148 (8)	0.0235 (9)	0.0008 (7)	0.0016 (7)	−0.0024 (7)
C41	0.0231 (9)	0.0178 (9)	0.0317 (10)	0.0011 (7)	0.0042 (8)	−0.0058 (8)
C42	0.0243 (9)	0.0189 (9)	0.0243 (9)	0.0027 (7)	0.0051 (8)	−0.0033 (7)
C43	0.0331 (11)	0.0252 (11)	0.0355 (12)	0.0025 (9)	−0.0068 (9)	−0.0005 (9)
C44	0.0416 (13)	0.0322 (12)	0.0356 (12)	−0.0094 (10)	−0.0062 (10)	−0.0090 (10)
C45	0.0498 (14)	0.0216 (10)	0.0355 (12)	−0.0041 (9)	0.0051 (10)	−0.0098 (9)
C46	0.0630 (17)	0.0199 (11)	0.0462 (14)	0.0093 (11)	−0.0120 (12)	−0.0019 (10)
C47	0.0475 (14)	0.0238 (11)	0.0349 (12)	0.0044 (10)	−0.0134 (10)	−0.0047 (9)
C48	0.0279 (10)	0.0213 (10)	0.0337 (11)	−0.0058 (8)	0.0006 (9)	0.0040 (8)
C49	0.0221 (9)	0.0121 (8)	0.0169 (8)	0.0012 (7)	−0.0020 (7)	−0.0015 (6)
C50	0.0214 (9)	0.0134 (8)	0.0174 (8)	0.0029 (7)	−0.0007 (7)	0.0003 (7)
C51	0.0197 (9)	0.0146 (8)	0.0206 (9)	0.0002 (7)	−0.0017 (7)	0.0007 (7)

C52	0.0231 (9)	0.0153 (8)	0.0192 (9)	−0.0001 (7)	−0.0025 (7)	−0.0021 (7)
C53	0.0218 (9)	0.0165 (8)	0.0181 (9)	0.0019 (7)	0.0009 (7)	−0.0006 (7)
C54	0.0198 (9)	0.0131 (8)	0.0190 (8)	0.0010 (7)	−0.0017 (7)	0.0011 (7)
C55	0.0193 (9)	0.0147 (8)	0.0189 (8)	−0.0002 (7)	0.0005 (7)	−0.0006 (7)
C56	0.0262 (10)	0.0139 (8)	0.0222 (9)	−0.0008 (7)	−0.0006 (7)	−0.0003 (7)
C57	0.0312 (10)	0.0185 (9)	0.0254 (10)	0.0009 (8)	−0.0043 (8)	0.0005 (8)
C58	0.0456 (13)	0.0209 (10)	0.0272 (11)	0.0064 (9)	−0.0121 (9)	0.0006 (8)
C59	0.100 (2)	0.0266 (13)	0.0350 (13)	−0.0102 (14)	0.0022 (14)	0.0023 (10)
C60	0.169 (4)	0.0267 (14)	0.0451 (17)	−0.014 (2)	−0.021 (2)	0.0089 (13)
C61	0.142 (4)	0.0341 (17)	0.073 (2)	0.037 (2)	−0.051 (3)	−0.0189 (16)
C62	0.073 (2)	0.067 (3)	0.119 (3)	0.044 (2)	−0.037 (2)	−0.052 (2)
C63	0.0439 (15)	0.0458 (16)	0.075 (2)	0.0129 (12)	−0.0107 (14)	−0.0228 (15)
C64	0.0299 (11)	0.0262 (10)	0.0271 (10)	−0.0064 (8)	0.0056 (8)	−0.0117 (8)
C65	0.072 (2)	0.0281 (13)	0.0588 (18)	0.0036 (13)	−0.0049 (15)	0.0052 (12)
C66	0.0652 (19)	0.0569 (18)	0.0409 (15)	−0.0057 (15)	0.0089 (13)	−0.0073 (13)
C67	0.0417 (14)	0.0381 (14)	0.0630 (17)	0.0005 (11)	0.0119 (13)	−0.0096 (12)
C68	0.0584 (17)	0.0664 (19)	0.0301 (13)	−0.0032 (14)	−0.0018 (12)	−0.0091 (12)
C69	0.102 (3)	0.076 (2)	0.0399 (16)	−0.026 (2)	0.0234 (17)	−0.0057 (15)

Geometric parameters (Å, °)

O1—C3	1.377 (2)	C30—C31	1.398 (4)
O1—H1	0.8400	C30—H30	0.9500
O2—C5	1.372 (2)	C31—H31	0.9500
O2—H2	0.8400	C32—H32A	0.9800
O3—C19	1.367 (2)	C32—H32B	0.9800
O3—H3	0.8400	C32—H32C	0.9800
O4—C21	1.380 (2)	C33—C38	1.390 (3)
O4—H4	0.8400	C33—C34	1.391 (3)
O5—C35	1.385 (2)	C33—H33	0.9500
O5—H5	0.8400	C34—C35	1.395 (3)
O6—C37	1.387 (2)	C35—C36	1.396 (3)
O6—H6	0.8400	C36—C37	1.397 (3)
O7—C51	1.387 (2)	C36—C48	1.507 (3)
O7—H7	0.8400	C37—C38	1.397 (3)
O8—C53	1.380 (2)	C38—C39	1.527 (2)
O8—H8	0.8400	C39—C50	1.524 (2)
O9—C65	1.402 (3)	C39—C40	1.543 (2)
O9—H9	0.8400	C39—H39	1.0000
O10—C66	1.414 (3)	C40—C41	1.532 (3)
O10—H10	0.8400	C40—H40A	0.9900
O11—C67	1.416 (3)	C40—H40B	0.9900
O11—H11	0.8400	C41—C42	1.510 (3)
O12—C68	1.391 (3)	C41—H41A	0.9900
O12—H12	0.8400	C41—H41B	0.9900
O13—C69	1.432 (4)	C42—C43	1.381 (3)
O13—H13	0.8400	C42—C47	1.383 (3)
C1—C2	1.387 (3)	C43—C44	1.391 (3)
C1—C6	1.392 (3)	C43—H43	0.9500

C1—H1A	0.9500	C44—C45	1.376 (4)
C2—C3	1.400 (3)	C44—H44	0.9500
C2—C55	1.525 (2)	C45—C46	1.367 (4)
C3—C4	1.398 (3)	C45—H45	0.9500
C4—C5	1.395 (3)	C46—C47	1.393 (3)
C4—C16	1.513 (3)	C46—H46	0.9500
C5—C6	1.402 (3)	C47—H47	0.9500
C6—C7	1.531 (3)	C48—H48A	0.9800
C7—C18	1.531 (3)	C48—H48B	0.9800
C7—C8	1.543 (3)	C48—H48C	0.9800
C7—H7A	1.0000	C49—C50	1.388 (3)
C8—C9	1.534 (3)	C49—C54	1.392 (3)
C8—H8A	0.9900	C49—H49	0.9500
C8—H8B	0.9900	C50—C51	1.402 (2)
C9—C10	1.512 (3)	C51—C52	1.397 (3)
C9—H9A	0.9900	C52—C53	1.400 (3)
C9—H9B	0.9900	C52—C64	1.506 (3)
C10—C11	1.377 (3)	C53—C54	1.398 (2)
C10—C15	1.381 (3)	C54—C55	1.527 (2)
C11—C12	1.391 (4)	C55—C56	1.544 (2)
C11—H11A	0.9500	C55—H55	1.0000
C12—C13	1.331 (4)	C56—C57	1.529 (3)
C12—H12A	0.9500	C56—H56A	0.9900
C13—C14	1.391 (4)	C56—H56B	0.9900
C13—H13A	0.9500	C57—C58	1.508 (3)
C14—C15	1.387 (4)	C57—H57A	0.9900
C14—H14	0.9500	C57—H57B	0.9900
C15—H15	0.9500	C58—C59	1.376 (4)
C16—H16A	0.9800	C58—C63	1.390 (4)
C16—H16B	0.9800	C59—C60	1.408 (5)
C16—H16C	0.9800	C59—H59	0.9500
C17—C18	1.390 (3)	C60—C61	1.352 (6)
C17—C22	1.391 (3)	C60—H60	0.9500
C17—H17	0.9500	C61—C62	1.358 (7)
C18—C19	1.408 (3)	C61—H61	0.9500
C19—C20	1.402 (3)	C62—C63	1.399 (4)
C20—C21	1.401 (3)	C62—H62	0.9500
C20—C32	1.505 (3)	C63—H63	0.9500
C21—C22	1.399 (3)	C64—H64A	0.9800
C22—C23	1.527 (3)	C64—H64B	0.9800
C23—C34	1.524 (3)	C64—H64C	0.9800
C23—C24	1.538 (3)	C65—H65A	0.9800
C23—H23	1.0000	C65—H65B	0.9800
C24—C25	1.534 (3)	C65—H65C	0.9800
C24—H24A	0.9900	C66—H66A	0.9800
C24—H24B	0.9900	C66—H66B	0.9800
C25—C26	1.509 (3)	C66—H66C	0.9800
C25—H25A	0.9900	C67—H67A	0.9800
C25—H25B	0.9900	C67—H67B	0.9800

C26—C31	1.380 (3)	C67—H67C	0.9800
C26—C27	1.390 (3)	C68—H68A	0.9800
C27—C28	1.389 (3)	C68—H68B	0.9800
C27—H27	0.9500	C68—H68C	0.9800
C28—C29	1.372 (4)	C69—H69A	0.9800
C28—H28	0.9500	C69—H69B	0.9800
C29—C30	1.386 (4)	C69—H69C	0.9800
C29—H29	0.9500		
C3—O1—H1	109.5	C35—C36—C37	117.46 (17)
C5—O2—H2	109.5	C35—C36—C48	122.06 (17)
C19—O3—H3	109.5	C37—C36—C48	120.48 (17)
C21—O4—H4	109.5	O6—C37—C36	116.36 (16)
C35—O5—H5	109.5	O6—C37—C38	121.06 (16)
C37—O6—H6	109.5	C36—C37—C38	122.58 (17)
C51—O7—H7	109.5	C33—C38—C37	117.24 (17)
C53—O8—H8	109.5	C33—C38—C39	121.07 (16)
C65—O9—H9	109.5	C37—C38—C39	121.61 (16)
C66—O10—H10	109.5	C50—C39—C38	112.18 (14)
C67—O11—H11	109.5	C50—C39—C40	111.99 (15)
C68—O12—H12	109.5	C38—C39—C40	111.45 (15)
C69—O13—H13	109.5	C50—C39—H39	106.9
C2—C1—C6	122.80 (17)	C38—C39—H39	106.9
C2—C1—H1A	118.6	C40—C39—H39	106.9
C6—C1—H1A	118.6	C41—C40—C39	113.01 (15)
C1—C2—C3	117.60 (17)	C41—C40—H40A	109.0
C1—C2—C55	121.22 (16)	C39—C40—H40A	109.0
C3—C2—C55	121.18 (16)	C41—C40—H40B	109.0
O1—C3—C4	117.27 (16)	C39—C40—H40B	109.0
O1—C3—C2	120.51 (17)	H40A—C40—H40B	107.8
C4—C3—C2	122.21 (17)	C42—C41—C40	111.48 (16)
C5—C4—C3	117.59 (17)	C42—C41—H41A	109.3
C5—C4—C16	122.23 (17)	C40—C41—H41A	109.3
C3—C4—C16	120.18 (17)	C42—C41—H41B	109.3
O2—C5—C4	121.68 (17)	C40—C41—H41B	109.3
O2—C5—C6	116.01 (17)	H41A—C41—H41B	108.0
C4—C5—C6	122.27 (17)	C43—C42—C47	117.87 (19)
C1—C6—C5	117.41 (17)	C43—C42—C41	120.73 (18)
C1—C6—C7	121.48 (16)	C47—C42—C41	121.39 (19)
C5—C6—C7	121.10 (16)	C42—C43—C44	121.3 (2)
C6—C7—C18	110.57 (15)	C42—C43—H43	119.3
C6—C7—C8	112.33 (15)	C44—C43—H43	119.3
C18—C7—C8	111.52 (15)	C45—C44—C43	119.9 (2)
C6—C7—H7A	107.4	C45—C44—H44	120.0
C18—C7—H7A	107.4	C43—C44—H44	120.0
C8—C7—H7A	107.4	C46—C45—C44	119.5 (2)
C9—C8—C7	114.41 (16)	C46—C45—H45	120.2
C9—C8—H8A	108.7	C44—C45—H45	120.2
C7—C8—H8A	108.7	C45—C46—C47	120.5 (2)
C9—C8—H8B	108.7	C45—C46—H46	119.8

C7—C8—H8B	108.7	C47—C46—H46	119.8
H8A—C8—H8B	107.6	C42—C47—C46	120.9 (2)
C10—C9—C8	112.78 (17)	C42—C47—H47	119.6
C10—C9—H9A	109.0	C46—C47—H47	119.6
C8—C9—H9A	109.0	C36—C48—H48A	109.5
C10—C9—H9B	109.0	C36—C48—H48B	109.5
C8—C9—H9B	109.0	H48A—C48—H48B	109.5
H9A—C9—H9B	107.8	C36—C48—H48C	109.5
C11—C10—C15	116.1 (2)	H48A—C48—H48C	109.5
C11—C10—C9	122.9 (2)	H48B—C48—H48C	109.5
C15—C10—C9	121.0 (2)	C50—C49—C54	122.98 (16)
C10—C11—C12	121.8 (2)	C50—C49—H49	118.5
C10—C11—H11A	119.1	C54—C49—H49	118.5
C12—C11—H11A	119.1	C49—C50—C51	117.38 (16)
C13—C12—C11	121.3 (3)	C49—C50—C39	121.37 (16)
C13—C12—H12A	119.4	C51—C50—C39	121.24 (16)
C11—C12—H12A	119.4	O7—C51—C52	120.82 (16)
C12—C13—C14	119.1 (3)	O7—C51—C50	116.92 (16)
C12—C13—H13A	120.5	C52—C51—C50	122.24 (17)
C14—C13—H13A	120.5	C51—C52—C53	117.72 (16)
C15—C14—C13	119.4 (3)	C51—C52—C64	121.34 (17)
C15—C14—H14	120.3	C53—C52—C64	120.94 (17)
C13—C14—H14	120.3	O8—C53—C54	117.25 (16)
C10—C15—C14	122.3 (3)	O8—C53—C52	120.69 (16)
C10—C15—H15	118.9	C54—C53—C52	122.06 (17)
C14—C15—H15	118.9	C49—C54—C53	117.58 (17)
C4—C16—H16A	109.5	C49—C54—C55	121.91 (16)
C4—C16—H16B	109.5	C53—C54—C55	120.49 (16)
H16A—C16—H16B	109.5	C2—C55—C54	110.54 (14)
C4—C16—H16C	109.5	C2—C55—C56	112.50 (15)
H16A—C16—H16C	109.5	C54—C55—C56	112.60 (15)
H16B—C16—H16C	109.5	C2—C55—H55	106.9
C18—C17—C22	124.03 (17)	C54—C55—H55	106.9
C18—C17—H17	118.0	C56—C55—H55	106.9
C22—C17—H17	118.0	C57—C56—C55	110.91 (15)
C17—C18—C19	116.81 (17)	C57—C56—H56A	109.5
C17—C18—C7	120.96 (16)	C55—C56—H56A	109.5
C19—C18—C7	122.23 (17)	C57—C56—H56B	109.5
O3—C19—C20	115.56 (17)	C55—C56—H56B	109.5
O3—C19—C18	123.02 (18)	H56A—C56—H56B	108.0
C20—C19—C18	121.42 (17)	C58—C57—C56	114.43 (16)
C21—C20—C19	119.04 (17)	C58—C57—H57A	108.7
C21—C20—C32	121.71 (18)	C56—C57—H57A	108.7
C19—C20—C32	119.23 (18)	C58—C57—H57B	108.7
O4—C21—C22	122.31 (17)	C56—C57—H57B	108.7
O4—C21—C20	116.51 (17)	H57A—C57—H57B	107.6
C22—C21—C20	121.17 (17)	C59—C58—C63	118.5 (2)
C17—C22—C21	117.49 (17)	C59—C58—C57	122.0 (2)
C17—C22—C23	121.12 (16)	C63—C58—C57	119.5 (2)

C21—C22—C23	121.39 (17)	C58—C59—C60	120.0 (3)
C34—C23—C22	110.60 (14)	C58—C59—H59	120.0
C34—C23—C24	111.60 (15)	C60—C59—H59	120.0
C22—C23—C24	113.00 (15)	C61—C60—C59	120.7 (4)
C34—C23—H23	107.1	C61—C60—H60	119.6
C22—C23—H23	107.1	C59—C60—H60	119.6
C24—C23—H23	107.1	C60—C61—C62	120.0 (3)
C25—C24—C23	113.39 (16)	C60—C61—H61	120.0
C25—C24—H24A	108.9	C62—C61—H61	120.0
C23—C24—H24A	108.9	C61—C62—C63	120.4 (4)
C25—C24—H24B	108.9	C61—C62—H62	119.8
C23—C24—H24B	108.9	C63—C62—H62	119.8
H24A—C24—H24B	107.7	C58—C63—C62	120.3 (4)
C26—C25—C24	111.64 (17)	C58—C63—H63	119.9
C26—C25—H25A	109.3	C62—C63—H63	119.9
C24—C25—H25A	109.3	C52—C64—H64A	109.5
C26—C25—H25B	109.3	C52—C64—H64B	109.5
C24—C25—H25B	109.3	H64A—C64—H64B	109.5
H25A—C25—H25B	108.0	C52—C64—H64C	109.5
C31—C26—C27	118.4 (2)	H64A—C64—H64C	109.5
C31—C26—C25	121.0 (2)	H64B—C64—H64C	109.5
C27—C26—C25	120.6 (2)	O9—C65—H65A	109.5
C28—C27—C26	120.9 (2)	O9—C65—H65B	109.5
C28—C27—H27	119.6	H65A—C65—H65B	109.5
C26—C27—H27	119.6	O9—C65—H65C	109.5
C29—C28—C27	120.0 (2)	H65A—C65—H65C	109.5
C29—C28—H28	120.0	H65B—C65—H65C	109.5
C27—C28—H28	120.0	O10—C66—H66A	109.5
C28—C29—C30	120.3 (2)	O10—C66—H66B	109.5
C28—C29—H29	119.9	H66A—C66—H66B	109.5
C30—C29—H29	119.9	O10—C66—H66C	109.5
C29—C30—C31	119.3 (3)	H66A—C66—H66C	109.5
C29—C30—H30	120.4	H66B—C66—H66C	109.5
C31—C30—H30	120.4	O11—C67—H67A	109.5
C26—C31—C30	121.1 (2)	O11—C67—H67B	109.5
C26—C31—H31	119.4	H67A—C67—H67B	109.5
C30—C31—H31	119.4	O11—C67—H67C	109.5
C20—C32—H32A	109.5	H67A—C67—H67C	109.5
C20—C32—H32B	109.5	H67B—C67—H67C	109.5
H32A—C32—H32B	109.5	O12—C68—H68A	109.5
C20—C32—H32C	109.5	O12—C68—H68B	109.5
H32A—C32—H32C	109.5	H68A—C68—H68B	109.5
H32B—C32—H32C	109.5	O12—C68—H68C	109.5
C38—C33—C34	122.69 (17)	H68A—C68—H68C	109.5
C38—C33—H33	118.7	H68B—C68—H68C	109.5
C34—C33—H33	118.7	O13—C69—H69A	109.5
C33—C34—C35	117.81 (17)	O13—C69—H69B	109.5
C33—C34—C23	120.98 (16)	H69A—C69—H69B	109.5
C35—C34—C23	121.20 (16)	O13—C69—H69C	109.5

O5—C35—C34	117.01 (17)	H69A—C69—H69C	109.5
O5—C35—C36	120.79 (17)	H69B—C69—H69C	109.5
C34—C35—C36	122.12 (17)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O8	0.84	1.87	2.709 (2)	178
O2—H2···O6^{i}	0.84	1.85	2.6442 (19)	157
O3—H3···O2	0.84	1.85	2.687 (2)	171
O4—H4···O5	0.84	1.98	2.805 (2)	169
O5—H5···O10	0.84	1.82	2.643 (2)	165
O6—H6···O7	0.84	1.86	2.7001 (19)	177
O7—H7···O9^{ii}	0.84	1.86	2.639 (2)	154
O8—H8···O12	0.84	1.90	2.631 (2)	145
O10—H10···O13^{ii}	0.84	1.92	2.754 (3)	173
O11—H11···O1	0.84	1.93	2.716 (2)	156
O12—H12···O13	0.84	1.91	2.748 (3)	174
O13—H13···O11	0.84	1.84	2.671 (3)	172

Symmetry codes: (i) *x*+1, *y*, *z*; (ii) −*x*+1, −*y*, −*z*+1.

Fig. 1

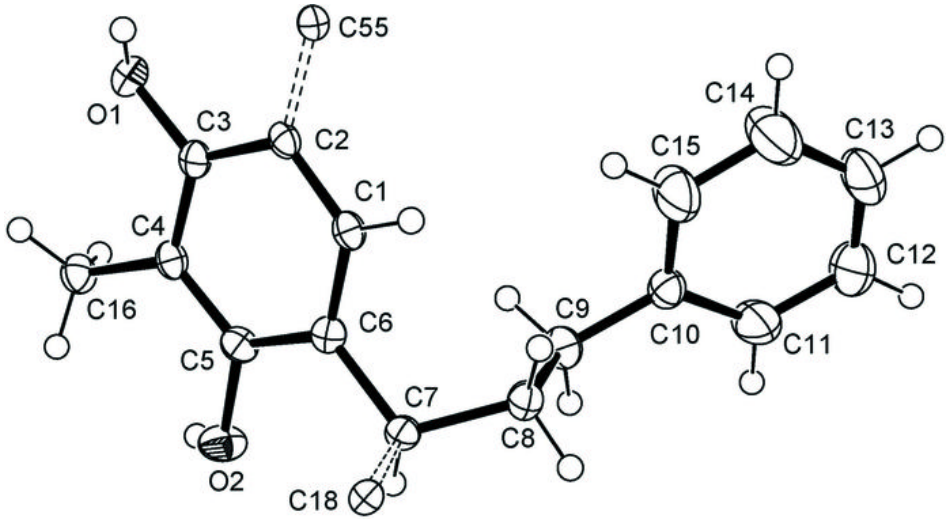


Fig. 2

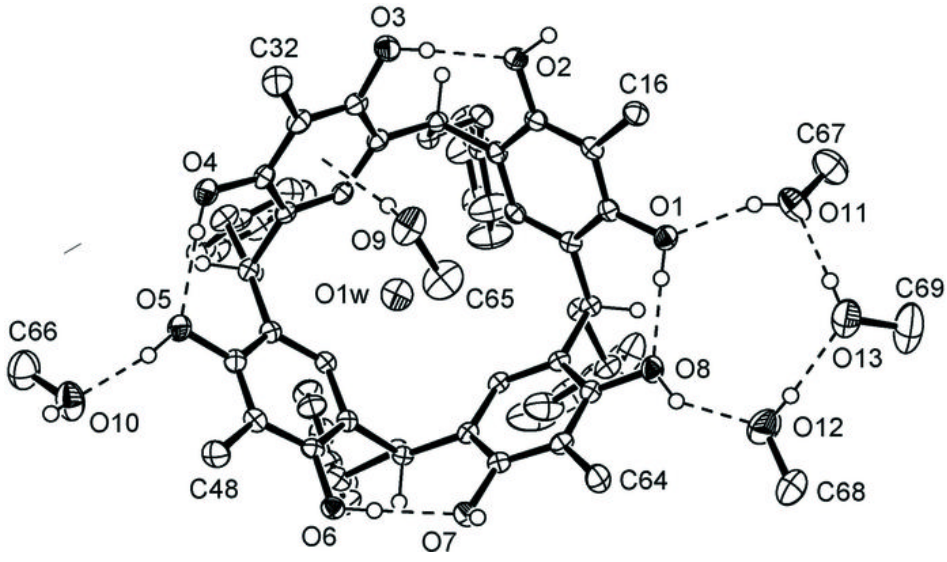

O3
C32
O2
C16
C67
O4
O1
O11
O9
O1w
C65
C69
O5
C66
O13
O8
O10
O12
C48
C64
C68
O6
O7

Fig. 3