CRYSTALLOGRAPHIC COMMUNICATIONS

ISSN 2056-9890

Received 18 November 2014
Accepted 27 November 2014

Edited by H. Ishida, Okayama University, Japan

Keywords: crystal structure; hydrogen bonds; taxane skeleton; paclitaxel

CCDC reference: 1036428
Supporting information: this article has supporting information at journals.iucr.org/e

Crystal structure of (±)-(4*RS*,5*RS*,7*SR*)-4-[(1*RS*,2*RS*,3*RS*,6*RS*)-3-benzoyloxy-2-(2-hydroxyethyl)-6-methoxymethoxy-2-methylcyclohexyl]-8,10,10-trimethyl-2-oxo-1,3-dioxaspiro[4.5]dec-8-en-7-yl benzoate benzene monosolvate

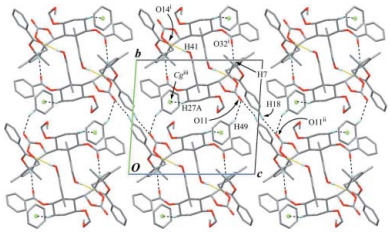
Takeshi Oishi,[a]* Yuu Yamaguchi,[b] Keisuke Fukaya,[b] Tomoya Sugai,[b] Ami Watanabe,[b] Takaaki Sato[b] and Noritaka Chida[b]

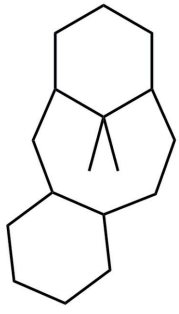
[a]School of Medicine, Keio University, Hiyoshi 4-1-1, Kohoku-ku, Yokohama 223-8521, Japan, and [b]Department of Applied Chemistry, Faculty of Science and Technology, Keio University, Hiyoshi 3-14-1, Kohoku-ku, Yokohama 223-8522, Japan. *Correspondence e-mail: oec@a6.keio.jp

In the title compound, $C_{36}H_{44}O_{10}\cdot C_6H_6$, the dioxolane ring adopts an envelope conformation with the C atom bonded to the H atom as the flap, while the cyclohexene and cyclohexane rings are in half-chair and chair conformations, respectively. In the crystal, a pair of O—H···O hydrogen bonds with an $R_2^2(26)$ graph-set motif connect the benzoate molecules into an inversion dimer. The dimers are linked by a weak C—H···O interaction into a tape structure along $[01\bar{1}]$. The benzene molecule links the tapes through C—H···O and C—H···π interactions, forming a sheet parallel to (100).

1. Chemical context

Paclitaxel is a well-known natural diterpenoid containing a taxane framework (tricyclo[9.3.1.$0^{3,8}$]pentadecane; Fig. 1), with potent antitumor activity (Wall & Wani, 1995). This unique and complicated structure has attracted significant interest, and a large number of synthetic studies have been reported. In these researches, whereas some structure data *after* cyclization into taxane or taxoid derivatives are available (§ 4), precursors *just before* cyclization are very few. The title compound has been obtained in our synthetic study of paclitaxel as a cyclization precursor to build the taxane skeleton (Fukaya *et al.*, 2014).





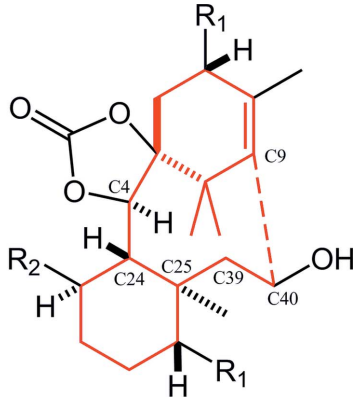
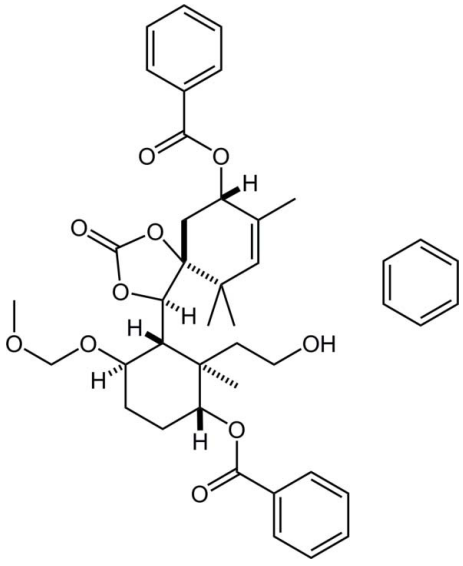


Figure 1
Left: the structure of the tricyclo[9.3.1.$0^{3,8}$]pentadecane (taxane) skeleton. Right: the title compound. Red lines indicate the taxane skeleton with the expected bond (red dashed line). R_1 = –OC(=O)Ph, R_2 = –OCH_2OCH_3.



2. Structural commentary

The molecular structure of the title compound is shown in Fig. 2. The dioxolane ring (O1/C2/O3/C4/C5) is in an envelope conformation with puckering parameters of $Q(2)$ = 0.165 (2) Å and $\varphi(2)$ = 114.5 (6)°. The flap atom C4 deviates from the mean plane of other atoms by 0.270 (3) Å. The cyclohexene ring (C5–C10), which is spiro-fused to the dioxolane ring, is in a half-chair conformation with puckering

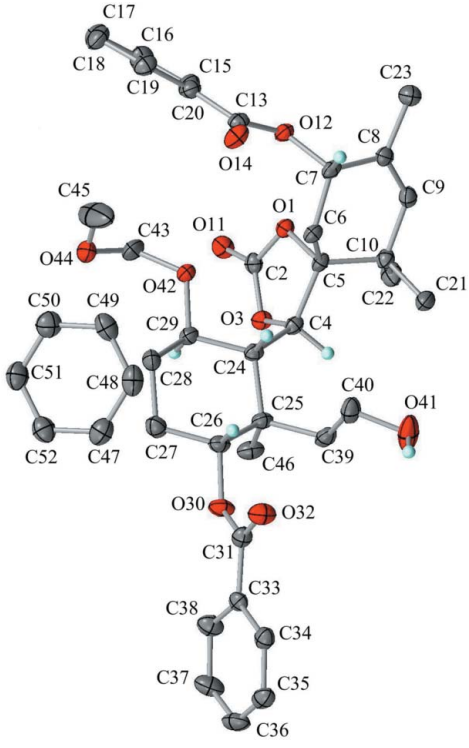


Figure 2
The asymmetric unit of the title compound with atom labelling. Displacement ellipsoids are drawn at the 50% probability level. Only H atoms connected to O and chiral C atoms are shown for clarity.

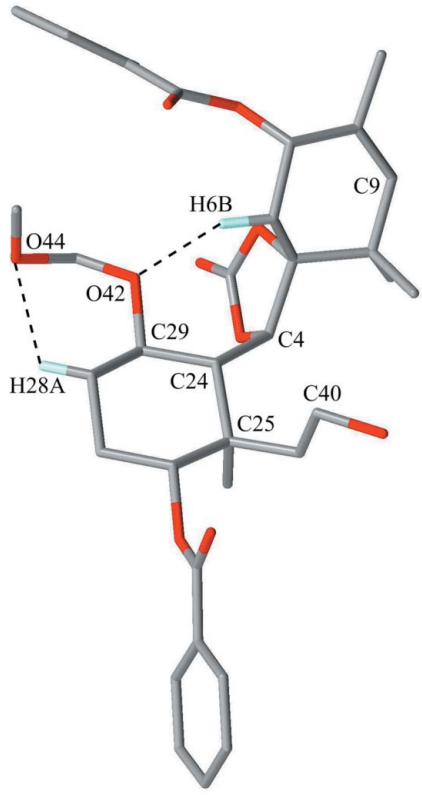


Figure 3
The molecular conformation indicating the intramolecular C—H···O interactions with dashed lines. Only H atoms involved in hydrogen bonds are shown for clarity. The benzene solvent molecule has been omitted.

parameters of Q = 0.469 (2) Å, θ = 127.5 (2)°, $\varphi(2)$ = 197.2 (3)°, $Q(2)$ = 0.372 (2) Å and $Q(3)$ = −0.285 (2) Å. Atoms C5 and C6 deviate from the mean plane of the other atoms by −0.493 (4) and 0.212 (4) Å, respectively. The cyclohexane ring (C24–C29) is in a chair conformation with puckering parameters Q = 0.587 (2) Å, θ = 4.6 (2)°, φ = 246 (3)°, Q(2) = 0.042 (2) Å and Q(3) = 0.585 (2) Å. The large substituents (C24—C4, C25—C39, C26—O30 and C29—O42) are in the equatorial positions. The methoxymethoxy group (O42/C43/O44/C45) shows a helical form with torsion angles of 76.5 (3)° for C29—O42—C43—O44 and 64.8 (3)° for O42—C43—O44—C45 held by weak intramolecular C—H···O interactions (Fig. 3, Table 1). The atom pairs which may be

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

Cg is the centroid of the C47–C52 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6*B*···O42	0.99	2.32	3.095 (2)	135
C28—H28*A*···O44	0.99	2.37	2.989 (3)	120
O41—H41···O14^{i}	0.84	2.06	2.888 (2)	170
C7—H7···O32^{i}	1.00	2.34	3.269 (2)	155
C18—H18···O11^{ii}	0.95	2.53	3.465 (2)	168
C49—H49···O11	0.95	2.46	3.300 (3)	147
C27—H27*A*···*Cg*^{iii}	0.95	2.64	3.514 (2)	147

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

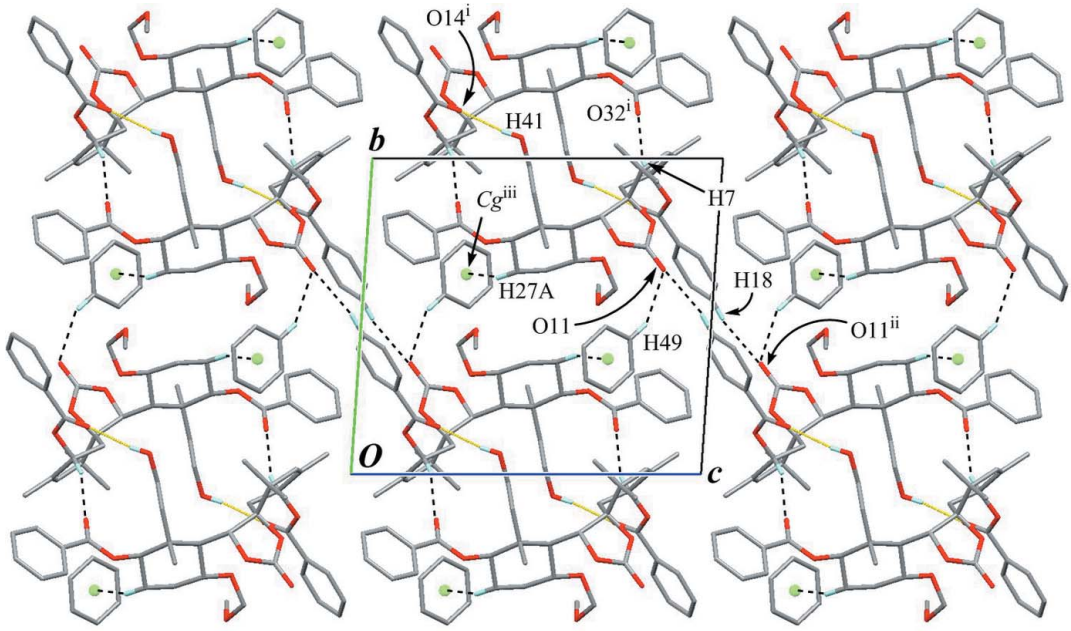


Figure 4
The crystal packing viewed along the *a* axis. Dotted yellow lines indicate the intermolecular O—H···O hydrogen bonds which form the inversion dimers. Black dashed lines indicate the intermolecular C—H···O and C—H···π interactions. *Cg* is the centroid of the benzene solvent molecule. Only H atoms involved in hydrogen bonds are shown for clarity. [Symmetry codes: (i) $-x+1, -y+2, -z+1$; (ii) $-x+1, -y+1, -z+2$; (iii) $-x+1, -y+1, -z+1$.]

connected by cyclization into a taxane framework are C9 and C40 (Figs. 1 and 3) with their distance being 5.831 (3) Å in the present conformation. They are expected to approach each other by rotation about the C4–C24, C25–C39 and C39–C40 bonds.

3. Supramolecular features

The crystal packing is stabilized by a pair of intermolecular O—H···O hydrogen bonds (O41—H41···O14^{i}; Table 1) with an $R_2^2(26)$ graph-set motif, forming an inversion dimer (Fig. 4). In the dimer, a pair of C—H···O hydrogen bonds (C7—H7···O32^{i}; Table 1) are also observed. The dimers are further linked by a weak intermolecular C—H···O hydrogen bond (C18—H18···O11^{ii}; Table 1) into a tape along [01$\bar{1}$]. The benzene molecule links adjacent tapes through C—H···O and C—H···π interactions (C49—H49···O11 and C27—H27*A*···*Cg*^{iii}; Table 1), forming a sheet parallel to (100).

4. Database survey

In the Cambridge Structural Database (CSD, Version 5.35, November 2013; Groom & Allen, 2014), four compounds possessing a core of 6,6,8-trimethyl-1,3-dioxaspiro[4.5]dec-7-ene are found (Fig. 5). These include its derivatives with 2-one (PUQLAO; Nishizawa *et al.*, 1998) and 2,2-dimethyl (NEGBOQ; Poujol *et al.*, 1997) substitutes. Another tetracyclic taxoid (ILIQUP; Ohba *et al.*, 2003) with a core of 6,6,8-trimethyl-1,3-dioxaspiro[4.5]decan-2-one, obtained in our previous study, is closely related to the title compound. Only one crystalline compound just before cyclization is found in the literature (Nicolaou *et al.*, 1995), however it is not registered in the CSD.

5. Synthesis and crystallization

The title compound was obtained in a synthetic study on paclitaxel. The cyclohexene unit (C5–C10) was provided according to the reported procedure (Nicolaou *et al.*, 1995), and coupled with the substituted cyclohexane unit (C24–C29) synthesized from 3-methylanisole (Fukaya *et al.*, 2014) by a Shapiro reaction (Nicolaou *et al.*, 1995). Further manipulation of the functional groups afforded the title compound, which was purified by silica gel column chromatography. Colorless crystals were grown from a benzene solution under a pentane-saturated atmosphere by slow evaporation at ambient temperature.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. C-bound H atoms were positioned

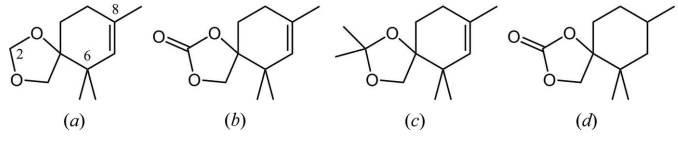


Figure 5
Core substructures for database survey; (*a*) 6,6,8-trimethyl-1,3-dioxaspiro[4.5]dec-7-ene, (*b*) its 2-one derivative, (*c*) the 2,2-dimethyl derivative and (*d*) 6,6,8-trimethyl-1,3-dioxaspiro[4.5]decan-2-one.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{36}H_{44}O_{10}\cdot C_6H_6$
M_r	714.82
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	90
a, b, c (Å)	9.6397 (6), 13.6008 (8), 15.0461 (10)
α, β, γ (°)	83.6966 (19), 77.488 (2), 77.9768 (18)
V (Å^3)	1879.2 (2)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.09
Crystal size (mm)	0.50 × 0.37 × 0.19
Data collection	
Diffractometer	Bruker D8 Venture
Absorption correction	Multi-scan (*SADABS*; Bruker, 2013)
T_{min}, T_{max}	0.96, 0.98
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	25389, 6526, 5180
R_{int}	0.043
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.042, 0.151, 0.93
No. of reflections	6526
No. of parameters	475
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.25, −0.25

Computer programs: *APEX2* and *SAINT* (Bruker, 2013), *SHELXS2013* and *SHELXL2014* (Sheldrick, 2008), *Mercury* (Macrae *et al.*, 2006), *publCIF* (Westrip, 2010) and *PLATON* (Spek, 2009).

geometrically with C—H = 0.95–1.00 Å, and constrained to ride on their parent atoms with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C). The H atom of hydroxy group (O41) was placed guided by difference maps and then treated as riding, with O—H = 0.84 Å and with U_{iso}(H) = 1.5U_{eq}(O). 13 problematic reflections were omitted from the final refinement.

Acknowledgements

We thank Professor S. Ohba (Keio University, Japan) and Dr K. Yoza (Bruker AXS Inc.) for providing valuable advice.

References

Bruker (2013). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Fukaya, K., Sugai, T., Yamaguchi, Y., Watanabe, A., Sato, T. & Chida, N. (2014). In preparation.
Groom, C. R. & Allen, F. H. (2014). *Angew. Chem. Int. Ed.* **53**, 662–671.
Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.
Nicolaou, K. C., Liu, J.-J., Yang, Z., Ueno, H., Sorensen, E. J., Claiborne, C. F., Guy, R. K., Hwang, C.-K., Nakada, M. & Nantermet, P. G. (1995). *J. Am. Chem. Soc.* **117**, 634–644.
Nishizawa, M., Imagawa, H., Hyodo, I., Takeji, M., Morikuni, E., Asoh, K. & Yamada, H. (1998). *Tetrahedron Lett.* **39**, 389–392.
Ohba, S., Chinen, A., Matsumoto, Y. & Chida, N. (2003). *Acta Cryst.* E**59**, o1476–o1477.
Poujol, H., Ahond, A., Al Mourabit, A., Chiaroni, A., Poupat, C., Riche, C. & Potier, P. (1997). *Tetrahedron*, **53**, 5169–5184.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Wall, M. E. & Wani, M. C. (1995). *ACS Symp. Ser.* **583**, 18–30.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supporting information

Acta Cryst. (2015). E**71**, 8-11 [doi:10.1107/S2056989014026048]

Crystal structure of (±)-(4*RS*,5*RS*,7*SR*)-4-[(1*RS*,2*RS*,3*RS*,6*RS*)-3-benzoyloxy-2-(2-hydroxyethyl)-6-methoxymethoxy-2-methylcyclohexyl]-8,10,10-trimethyl-2-oxo-1,3-dioxaspiro[4.5]dec-8-en-7-yl benzoate benzene monosolvate

Takeshi Oishi, Yuu Yamaguchi, Keisuke Fukaya, Tomoya Sugai, Ami Watanabe, Takaaki Sato and Noritaka Chida

Computing details

Data collection: *APEX2* (Bruker, 2013); cell refinement: *SAINT* (Bruker, 2013); data reduction: *SAINT* (Bruker, 2013); program(s) used to solve structure: *SHELXS2013* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010) and *PLATON* (Spek, 2009).

(±)-(4*RS*,5*RS*,7*SR*)-4-[(1*RS*,2*RS*,3*RS*,6*RS*)-3-Benzoyloxy-2-(2-hydroxyethyl)-6-methoxymethoxy-2-methylcyclohexyl]-8,10,10-trimethyl-2-oxo-1,3-dioxaspiro[4.5]dec-8-en-7-yl benzoate benzene monosolvate

Crystal data

$C_{36}H_{44}O_{10}\cdot C_6H_6$
M_r = 714.82
Triclinic, $P\overline{1}$
a = 9.6397 (6) Å
b = 13.6008 (8) Å
c = 15.0461 (10) Å
α = 83.6966 (19)°
β = 77.488 (2)°
γ = 77.9768 (18)°
V = 1879.2 (2) Å^3
Z = 2
$F(000)$ = 764
D_x = 1.263 Mg m^{-3}
Melting point: 459.2 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9980 reflections
θ = 2.2–25.1°
μ = 0.09 mm^{-1}
T = 90 K
Plate, colorless
0.50 × 0.37 × 0.19 mm

Data collection

Bruker D8 Venture diffractometer
Radiation source: fine-focus sealed tube
Multilayered confocal mirror monochromator
Detector resolution: 8.333 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2013)
T_{min} = 0.96, T_{max} = 0.98
25389 measured reflections
6526 independent reflections
5180 reflections with $I > 2\sigma(I)$
R_{int} = 0.043
θ_{max} = 25.0°, θ_{min} = 2.2°
h = −11→11
k = −16→16
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.151$
$S = 0.93$
6526 reflections
475 parameters
0 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
$w = 1/[\sigma^2(F_o^2) + (0.0996P)^2 + 1.0739P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.023$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}

Special details

Experimental. Recrystallization from benzene, toluene, chloroform, dichloromethane, diethyl ether, tetrahydrofuran, ethyl acetate, acetonitrile and methanol solutions under the air were failed. These solutions under hexane atmosphere also gave unsatisfactory results. Only the condition mentioned above has been quite effective to afford the single crystals suitable for X-ray analysis.; *M*.p. 458.7–459.2 K (not corrected); IR (film): 3524, 2945, 2890, 1790, 1715, 1274, 714 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): δ (p.p.m.) 8.13–8.10 (m, 2H), 8.03–7.99 (m, 2H), 7.60–7.53 (m, 2H), 7.48–7.42 (m, 4H), 5.56 (d, J = 6.0 Hz, 1H), 5.41 (t, J = 0.9 Hz, 1H), 5.09 (dd, J = 11.5, 4.6 Hz, 1H), 4.84 (s, 1H), 4.49 (d, J = 7.7 Hz, 1H), 4.13 (d, J = 7.7 Hz, 1H), 3.76–3.68 (m, 2H), 3.63 (ddd, J = 10.6, 7.2, 7.2 Hz, 1H), 3.08 (d, J = 14.9 Hz, 1H), 2.66 (s, 3H), 2.40 (dddd, J = 12.9, 4.3, 4.0, 4.0 Hz, 1H), 2.34 (d, J = 10.3 Hz, 1H), 2.29 (dd, J = 15.5, 6.3 Hz, 1H), 1.93 (dddd, J = 12.9, 4.3, 4.3, 4.0 Hz, 1H), 1.75 (d, J = 0.9 Hz, 3H), 1.73–1.60 (m, 3H), 1.59–1.45 (m, 2H), 1.22 (s, 3H), 1.20 (s, 3H), 1.11 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ (p.p.m.) 166.4 (C), 166.0 (C), 155.2 (C), 135.1 (CH), 133.4 (CH), 133.3 (CH), 130.5 (C), 130.2 (C), 130.0 (CH), 129.7 (CH), 129.0 (C), 128.7 (CH), 128.7 (CH), 97.9 (CH_2), 87.1 (C), 76.9 (CH), 76.5 (CH), 75.1 (CH), 68.7 (CH), 58.3 (CH_2), 54.7 (CH_3), 45.9 (CH), 42.0 (C), 41.2 (C), 38.1 (CH_2), 31.5 (CH_2), 30.6 (CH_2), 25.7 (CH_3), 25.1 (CH_2), 22.3 (CH_3), 20.2 (CH_3), 16.8 (CH_3); HRMS (ESI): *m/z* calcd for $C_{36}H_{44}O_{10}Na^+$ $[M+Na]^+$ 659.2832, found 659.2836.

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. Problematic 13 reflections with $|I(\text{obs})-I(\text{calc})|/\sigma W(I)$ greater than 10 (–8 –2 1, –8 –2 2, –8 –1 2, –7 –4 3, –8 –3 6, 3 11 7, 3 10 8, 0 9 9, 2 10 9, 2 8 10, 4 9 10, 2 7 11, 3 8 11) have been omitted in the final refinement.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.25606 (14)	0.79849 (9)	0.81366 (8)	0.0191 (3)
C2	0.2032 (2)	0.71963 (14)	0.79948 (13)	0.0201 (4)
O3	0.16170 (14)	0.73096 (9)	0.71879 (9)	0.0206 (3)
C4	0.2098 (2)	0.81686 (14)	0.66496 (13)	0.0185 (4)
H4	0.1247	0.8597	0.6428	0.022*
C5	0.2481 (2)	0.87501 (14)	0.73783 (12)	0.0181 (4)
C6	0.3931 (2)	0.90852 (14)	0.71464 (13)	0.0194 (4)
H6A	0.3918	0.9613	0.6638	0.023*
H6B	0.4698	0.8506	0.6934	0.023*
C7	0.4298 (2)	0.94956 (14)	0.79519 (13)	0.0204 (4)
H7	0.5042	0.9925	0.7716	0.024*

C8	0.3023 (2)	1.01014 (14)	0.85459 (13)	0.0204 (4)
C9	0.1692 (2)	1.01625 (14)	0.84154 (13)	0.0219 (4)
H9	0.094	1.0582	0.8799	0.026*
C10	0.1244 (2)	0.96338 (14)	0.77197 (13)	0.0206 (4)
O11	0.19139 (15)	0.64864 (10)	0.85222 (9)	0.0258 (3)
O12	0.48877 (14)	0.86671 (9)	0.85533 (9)	0.0206 (3)
C13	0.6265 (2)	0.82130 (14)	0.82802 (13)	0.0204 (4)
O14	0.70642 (15)	0.84907 (11)	0.76027 (10)	0.0279 (3)
C15	0.6684 (2)	0.73187 (15)	0.88941 (13)	0.0221 (4)
C16	0.8142 (2)	0.68716 (16)	0.88007 (14)	0.0264 (5)
H16	0.8853	0.7165	0.8378	0.032*
C17	0.8556 (2)	0.60012 (16)	0.93234 (16)	0.0324 (5)
H17	0.955	0.5699	0.9263	0.039*
C18	0.7518 (3)	0.55705 (16)	0.99346 (15)	0.0328 (5)
H18	0.7799	0.4965	1.0285	0.039*
C19	0.6074 (3)	0.60200 (16)	1.00363 (15)	0.0321 (5)
H19	0.5367	0.5729	1.0465	0.039*
C20	0.5651 (2)	0.68926 (15)	0.95176 (14)	0.0269 (5)
H20	0.4656	0.7198	0.9588	0.032*
C21	0.0916 (2)	1.04115 (15)	0.69334 (15)	0.0282 (5)
H21A	0.0245	1.1008	0.7182	0.042*
H21B	0.0476	1.0116	0.6524	0.042*
H21C	0.1818	1.0606	0.6593	0.042*
C22	−0.0155 (2)	0.92602 (16)	0.81716 (16)	0.0299 (5)
H22A	0.0011	0.8803	0.8705	0.045*
H22B	−0.045	0.8902	0.7734	0.045*
H22C	−0.0921	0.9837	0.8365	0.045*
C23	0.3361 (2)	1.06479 (16)	0.92679 (14)	0.0274 (5)
H23A	0.2466	1.1054	0.9587	0.041*
H23B	0.4042	1.1089	0.8981	0.041*
H23C	0.3795	1.0157	0.9705	0.041*
C24	0.3249 (2)	0.78089 (14)	0.58056 (13)	0.0191 (4)
H24	0.3926	0.8297	0.5659	0.023*
C25	0.2548 (2)	0.78407 (15)	0.49501 (13)	0.0213 (4)
C26	0.3765 (2)	0.74420 (15)	0.41478 (13)	0.0224 (4)
H26	0.4423	0.7939	0.3956	0.027*
C27	0.4645 (2)	0.64260 (15)	0.43479 (14)	0.0269 (5)
H27A	0.5411	0.623	0.3809	0.032*
H27B	0.4018	0.5915	0.4485	0.032*
C28	0.5328 (2)	0.64693 (15)	0.51624 (14)	0.0248 (4)
H28A	0.5916	0.5802	0.5296	0.03*
H28B	0.5975	0.6967	0.5017	0.03*
C29	0.4158 (2)	0.67659 (14)	0.59900 (13)	0.0208 (4)
H29	0.3513	0.6257	0.6133	0.025*
O30	0.30900 (16)	0.73471 (10)	0.33928 (9)	0.0268 (3)
C31	0.3358 (2)	0.79233 (15)	0.26237 (13)	0.0223 (4)
O32	0.40856 (17)	0.85680 (11)	0.25168 (10)	0.0316 (4)
C33	0.2631 (2)	0.76877 (15)	0.19191 (13)	0.0208 (4)

C34	0.2657 (2)	0.83032 (15)	0.11194 (13)	0.0247 (5)
H34	0.3158	0.8849	0.1024	0.03*
C35	0.1955 (2)	0.81243 (17)	0.04616 (14)	0.0286 (5)
H35	0.1968	0.855	−0.0084	0.034*
C36	0.1237 (2)	0.73291 (18)	0.05975 (14)	0.0330 (5)
H36	0.075	0.7209	0.0147	0.04*
C37	0.1222 (3)	0.67046 (18)	0.13876 (15)	0.0344 (5)
H37	0.0732	0.6153	0.1476	0.041*
C38	0.1919 (2)	0.68816 (16)	0.20503 (14)	0.0296 (5)
H38	0.1909	0.6452	0.2593	0.036*
C39	0.1840 (2)	0.89252 (15)	0.46698 (14)	0.0253 (5)
H39A	0.1477	0.8901	0.4107	0.03*
H39B	0.0986	0.9141	0.5153	0.03*
C40	0.2753 (2)	0.97374 (15)	0.44990 (15)	0.0303 (5)
H40A	0.3069	0.982	0.5066	0.036*
H40B	0.3627	0.954	0.4023	0.036*
O41	0.1917 (2)	1.06636 (12)	0.42091 (11)	0.0461 (5)
H41	0.2305	1.0848	0.3679	0.069*
O42	0.47536 (14)	0.68229 (10)	0.67742 (9)	0.0223 (3)
C43	0.5313 (3)	0.58881 (17)	0.71882 (16)	0.0364 (6)
H43A	0.5402	0.599	0.7814	0.044*
H43B	0.4618	0.5432	0.724	0.044*
O44	0.6660 (2)	0.54241 (13)	0.67142 (12)	0.0538 (5)
C45	0.7765 (3)	0.5978 (3)	0.6691 (3)	0.0826 (13)
H45A	0.7436	0.6683	0.6492	0.124*
H45B	0.8639	0.5691	0.6262	0.124*
H45C	0.7983	0.5943	0.7301	0.124*
C46	0.1359 (2)	0.72061 (16)	0.51442 (14)	0.0273 (5)
H46A	0.096	0.7225	0.4595	0.041*
H46B	0.0588	0.748	0.5644	0.041*
H46C	0.1771	0.6508	0.5318	0.041*
C47	0.1268 (2)	0.40365 (17)	0.63928 (16)	0.0345 (5)
H47	0.0799	0.4303	0.59	0.041*
C48	0.1481 (2)	0.46772 (17)	0.69805 (16)	0.0343 (5)
H48	0.1157	0.5383	0.6892	0.041*
C49	0.2161 (2)	0.42938 (16)	0.76949 (16)	0.0335 (5)
H49	0.2298	0.4736	0.8102	0.04*
C50	0.2644 (3)	0.32710 (17)	0.78217 (16)	0.0356 (5)
H50	0.3119	0.3008	0.8312	0.043*
C51	0.2433 (3)	0.26278 (17)	0.72304 (16)	0.0350 (5)
H51	0.2769	0.1923	0.7314	0.042*
C52	0.1735 (2)	0.30103 (17)	0.65192 (16)	0.0329 (5)
H52	0.1578	0.2569	0.6119	0.04*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0219 (7)	0.0173 (7)	0.0177 (7)	−0.0039 (5)	−0.0048 (5)	0.0020 (5)

C2	0.0178 (10)	0.0194 (10)	0.0209 (10)	−0.0022 (8)	−0.0001 (8)	−0.0021 (8)
O3	0.0242 (7)	0.0203 (7)	0.0186 (7)	−0.0082 (6)	−0.0052 (6)	0.0023 (5)
C4	0.0169 (10)	0.0187 (10)	0.0202 (10)	−0.0046 (7)	−0.0050 (8)	0.0029 (8)
C5	0.0185 (10)	0.0174 (9)	0.0168 (9)	−0.0019 (7)	−0.0045 (8)	0.0045 (8)
C6	0.0198 (10)	0.0191 (10)	0.0190 (10)	−0.0024 (8)	−0.0063 (8)	0.0030 (8)
C7	0.0214 (10)	0.0179 (10)	0.0223 (10)	−0.0037 (8)	−0.0089 (8)	0.0056 (8)
C8	0.0252 (11)	0.0160 (9)	0.0202 (10)	−0.0040 (8)	−0.0065 (8)	0.0021 (8)
C9	0.0240 (11)	0.0171 (10)	0.0229 (10)	−0.0012 (8)	−0.0040 (8)	−0.0005 (8)
C10	0.0165 (10)	0.0198 (10)	0.0255 (10)	−0.0021 (8)	−0.0060 (8)	−0.0011 (8)
O11	0.0350 (8)	0.0217 (7)	0.0201 (7)	−0.0099 (6)	−0.0021 (6)	0.0025 (6)
O12	0.0216 (7)	0.0199 (7)	0.0212 (7)	−0.0036 (6)	−0.0088 (6)	0.0026 (6)
C13	0.0185 (10)	0.0228 (10)	0.0232 (10)	−0.0073 (8)	−0.0083 (8)	−0.0012 (8)
O14	0.0221 (8)	0.0314 (8)	0.0292 (8)	−0.0074 (6)	−0.0062 (6)	0.0090 (6)
C15	0.0234 (11)	0.0225 (10)	0.0220 (10)	−0.0046 (8)	−0.0083 (8)	0.0000 (8)
C16	0.0227 (11)	0.0294 (11)	0.0286 (11)	−0.0074 (9)	−0.0074 (9)	0.0006 (9)
C17	0.0279 (12)	0.0309 (12)	0.0389 (13)	−0.0003 (9)	−0.0163 (10)	0.0041 (10)
C18	0.0432 (14)	0.0257 (11)	0.0294 (12)	−0.0017 (10)	−0.0157 (10)	0.0064 (9)
C19	0.0397 (13)	0.0277 (12)	0.0258 (11)	−0.0066 (10)	−0.0023 (10)	0.0034 (9)
C20	0.0264 (11)	0.0275 (11)	0.0248 (11)	−0.0033 (9)	−0.0046 (9)	0.0022 (9)
C21	0.0295 (11)	0.0220 (11)	0.0359 (12)	0.0001 (9)	−0.0170 (10)	−0.0024 (9)
C22	0.0181 (11)	0.0296 (12)	0.0413 (13)	−0.0026 (9)	−0.0032 (9)	−0.0092 (10)
C23	0.0284 (11)	0.0287 (11)	0.0265 (11)	−0.0050 (9)	−0.0080 (9)	−0.0042 (9)
C24	0.0206 (10)	0.0190 (10)	0.0188 (10)	−0.0053 (8)	−0.0060 (8)	0.0013 (8)
C25	0.0249 (11)	0.0238 (10)	0.0171 (10)	−0.0059 (8)	−0.0087 (8)	0.0022 (8)
C26	0.0293 (11)	0.0230 (10)	0.0180 (10)	−0.0079 (8)	−0.0093 (8)	0.0001 (8)
C27	0.0346 (12)	0.0238 (11)	0.0212 (10)	−0.0040 (9)	−0.0041 (9)	−0.0024 (9)
C28	0.0275 (11)	0.0201 (10)	0.0248 (11)	0.0016 (8)	−0.0056 (9)	−0.0038 (8)
C29	0.0228 (10)	0.0209 (10)	0.0195 (10)	−0.0027 (8)	−0.0083 (8)	0.0003 (8)
O30	0.0391 (9)	0.0297 (8)	0.0164 (7)	−0.0147 (7)	−0.0100 (6)	0.0027 (6)
C31	0.0229 (10)	0.0226 (10)	0.0191 (10)	−0.0027 (8)	−0.0018 (8)	0.0011 (8)
O32	0.0425 (9)	0.0319 (8)	0.0260 (8)	−0.0188 (7)	−0.0114 (7)	0.0054 (6)
C33	0.0200 (10)	0.0236 (10)	0.0165 (10)	−0.0013 (8)	−0.0003 (8)	−0.0039 (8)
C34	0.0237 (11)	0.0251 (11)	0.0227 (10)	−0.0014 (8)	−0.0022 (8)	−0.0013 (9)
C35	0.0271 (11)	0.0364 (12)	0.0180 (10)	0.0023 (9)	−0.0036 (9)	−0.0017 (9)
C36	0.0323 (12)	0.0495 (14)	0.0196 (11)	−0.0069 (10)	−0.0068 (9)	−0.0109 (10)
C37	0.0412 (13)	0.0407 (13)	0.0272 (12)	−0.0188 (11)	−0.0063 (10)	−0.0072 (10)
C38	0.0351 (12)	0.0334 (12)	0.0218 (11)	−0.0120 (10)	−0.0039 (9)	−0.0012 (9)
C39	0.0263 (11)	0.0296 (11)	0.0181 (10)	0.0001 (9)	−0.0077 (8)	0.0029 (8)
C40	0.0350 (12)	0.0222 (11)	0.0282 (11)	0.0008 (9)	−0.0038 (9)	0.0043 (9)
O41	0.0580 (11)	0.0276 (9)	0.0340 (9)	0.0095 (8)	0.0067 (8)	0.0132 (7)
O42	0.0253 (7)	0.0208 (7)	0.0203 (7)	0.0018 (6)	−0.0096 (6)	−0.0009 (6)
C43	0.0510 (15)	0.0277 (12)	0.0318 (12)	0.0042 (11)	−0.0239 (11)	0.0017 (10)
O44	0.0635 (12)	0.0453 (10)	0.0521 (11)	0.0305 (9)	−0.0381 (10)	−0.0268 (9)
C45	0.0367 (17)	0.125 (3)	0.092 (3)	0.0260 (18)	−0.0341 (17)	−0.067 (2)
C46	0.0288 (11)	0.0358 (12)	0.0211 (10)	−0.0119 (9)	−0.0099 (9)	0.0026 (9)
C47	0.0288 (12)	0.0347 (13)	0.0369 (13)	−0.0037 (10)	−0.0053 (10)	0.0033 (10)
C48	0.0286 (12)	0.0248 (11)	0.0432 (14)	−0.0022 (9)	0.0024 (10)	0.0006 (10)
C49	0.0366 (13)	0.0272 (12)	0.0351 (13)	−0.0088 (10)	0.0008 (10)	−0.0063 (10)

C50	0.0390 (13)	0.0334 (13)	0.0345 (13)	−0.0102 (10)	−0.0070 (10)	0.0028 (10)
C51	0.0368 (13)	0.0249 (12)	0.0415 (14)	−0.0055 (10)	−0.0063 (11)	0.0020 (10)
C52	0.0337 (13)	0.0287 (12)	0.0365 (13)	−0.0090 (10)	−0.0034 (10)	−0.0043 (10)

Geometric parameters (Å, º)

O1—C2	1.334 (2)	C27—C28	1.523 (3)
O1—C5	1.461 (2)	C27—H27A	0.99
C2—O11	1.189 (2)	C27—H27B	0.99
C2—O3	1.342 (2)	C28—C29	1.516 (3)
O3—C4	1.446 (2)	C28—H28A	0.99
C4—C24	1.544 (3)	C28—H28B	0.99
C4—C5	1.566 (3)	C29—O42	1.435 (2)
C4—H4	1.0	C29—H29	1.0
C5—C6	1.517 (3)	O30—C31	1.333 (2)
C5—C10	1.550 (3)	C31—O32	1.209 (2)
C6—C7	1.524 (3)	C31—C33	1.486 (3)
C6—H6A	0.99	C33—C38	1.385 (3)
C6—H6B	0.99	C33—C34	1.387 (3)
C7—O12	1.464 (2)	C34—C35	1.381 (3)
C7—C8	1.501 (3)	C34—H34	0.95
C7—H7	1.0	C35—C36	1.375 (3)
C8—C9	1.324 (3)	C35—H35	0.95
C8—C23	1.507 (3)	C36—C37	1.382 (3)
C9—C10	1.513 (3)	C36—H36	0.95
C9—H9	0.95	C37—C38	1.383 (3)
C10—C22	1.534 (3)	C37—H37	0.95
C10—C21	1.538 (3)	C38—H38	0.95
O12—C13	1.338 (2)	C39—C40	1.519 (3)
C13—O14	1.213 (2)	C39—H39A	0.99
C13—C15	1.485 (3)	C39—H39B	0.99
C15—C20	1.388 (3)	C40—O41	1.427 (3)
C15—C16	1.393 (3)	C40—H40A	0.99
C16—C17	1.383 (3)	C40—H40B	0.99
C16—H16	0.95	O41—H41	0.84
C17—C18	1.385 (3)	O42—C43	1.408 (2)
C17—H17	0.95	C43—O44	1.396 (3)
C18—C19	1.381 (3)	C43—H43A	0.99
C18—H18	0.95	C43—H43B	0.99
C19—C20	1.385 (3)	O44—C45	1.420 (4)
C19—H19	0.95	C45—H45A	0.98
C20—H20	0.95	C45—H45B	0.98
C21—H21A	0.98	C45—H45C	0.98
C21—H21B	0.98	C46—H46A	0.98
C21—H21C	0.98	C46—H46B	0.98
C22—H22A	0.98	C46—H46C	0.98
C22—H22B	0.98	C47—C52	1.380 (3)
C22—H22C	0.98	C47—C48	1.380 (3)

C23—H23A	0.98	C47—H47	0.95
C23—H23B	0.98	C48—C49	1.378 (3)
C23—H23C	0.98	C48—H48	0.95
C24—C29	1.536 (3)	C49—C50	1.378 (3)
C24—C25	1.571 (2)	C49—H49	0.95
C24—H24	1.0	C50—C51	1.386 (3)
C25—C46	1.535 (3)	C50—H50	0.95
C25—C26	1.547 (3)	C51—C52	1.383 (3)
C25—C39	1.545 (3)	C51—H51	0.95
C26—O30	1.455 (2)	C52—H52	0.95
C26—C27	1.500 (3)	C9—C40	5.831 (3)
C26—H26	1.0		

C2—O1—C5	110.64 (14)	C27—C26—C25	114.64 (16)
O11—C2—O1	124.53 (18)	O30—C26—H26	109.1
O11—C2—O3	123.74 (17)	C27—C26—H26	109.1
O1—C2—O3	111.71 (16)	C25—C26—H26	109.1
C2—O3—C4	110.32 (14)	C26—C27—C28	109.18 (16)
O3—C4—C24	109.71 (15)	C26—C27—H27A	109.8
O3—C4—C5	102.47 (14)	C28—C27—H27A	109.8
C24—C4—C5	120.64 (15)	C26—C27—H27B	109.8
O3—C4—H4	107.8	C28—C27—H27B	109.8
C24—C4—H4	107.8	H27A—C27—H27B	108.3
C5—C4—H4	107.8	C29—C28—C27	110.00 (16)
O1—C5—C6	106.35 (14)	C29—C28—H28A	109.7
O1—C5—C10	107.29 (14)	C27—C28—H28A	109.7
C6—C5—C10	110.80 (15)	C29—C28—H28B	109.7
O1—C5—C4	101.92 (14)	C27—C28—H28B	109.7
C6—C5—C4	117.25 (15)	H28A—C28—H28B	108.2
C10—C5—C4	112.21 (15)	O42—C29—C28	111.95 (15)
C5—C6—C7	112.96 (16)	O42—C29—C24	106.93 (15)
C5—C6—H6A	109.0	C28—C29—C24	110.66 (16)
C7—C6—H6A	109.0	O42—C29—H29	109.1
C5—C6—H6B	109.0	C28—C29—H29	109.1
C7—C6—H6B	109.0	C24—C29—H29	109.1
H6A—C6—H6B	107.8	C31—O30—C26	119.33 (15)
O12—C7—C8	105.62 (15)	O32—C31—O30	124.21 (18)
O12—C7—C6	110.35 (14)	O32—C31—C33	124.19 (18)
C8—C7—C6	114.15 (16)	O30—C31—C33	111.59 (16)
O12—C7—H7	108.9	C38—C33—C34	119.78 (19)
C8—C7—H7	108.9	C38—C33—C31	121.56 (18)
C6—C7—H7	108.9	C34—C33—C31	118.65 (18)
C9—C8—C7	120.99 (18)	C35—C34—C33	120.20 (19)
C9—C8—C23	122.92 (19)	C35—C34—H34	119.9
C7—C8—C23	116.07 (17)	C33—C34—H34	119.9
C8—C9—C10	126.79 (18)	C36—C35—C34	119.9 (2)
C8—C9—H9	116.6	C36—C35—H35	120.1
C10—C9—H9	116.6	C34—C35—H35	120.1

C9—C10—C22	108.36 (17)	C35—C36—C37	120.3 (2)
C9—C10—C21	108.05 (16)	C35—C36—H36	119.9
C22—C10—C21	108.13 (16)	C37—C36—H36	119.9
C9—C10—C5	109.25 (15)	C38—C37—C36	120.1 (2)
C22—C10—C5	111.47 (16)	C38—C37—H37	119.9
C21—C10—C5	111.47 (16)	C36—C37—H37	119.9
C13—O12—C7	117.11 (14)	C37—C38—C33	119.7 (2)
O14—C13—O12	123.85 (17)	C37—C38—H38	120.1
O14—C13—C15	124.22 (18)	C33—C38—H38	120.1
O12—C13—C15	111.92 (16)	C40—C39—C25	118.35 (17)
C20—C15—C16	119.80 (18)	C40—C39—H39A	107.7
C20—C15—C13	121.31 (18)	C25—C39—H39A	107.7
C16—C15—C13	118.79 (18)	C40—C39—H39B	107.7
C17—C16—C15	120.06 (19)	C25—C39—H39B	107.7
C17—C16—H16	120.0	H39A—C39—H39B	107.1
C15—C16—H16	120.0	O41—C40—C39	109.16 (18)
C18—C17—C16	119.9 (2)	O41—C40—H40A	109.8
C18—C17—H17	120.0	C39—C40—H40A	109.8
C16—C17—H17	120.0	O41—C40—H40B	109.8
C19—C18—C17	120.1 (2)	C39—C40—H40B	109.8
C19—C18—H18	120.0	H40A—C40—H40B	108.3
C17—C18—H18	120.0	C40—O41—H41	109.5
C18—C19—C20	120.4 (2)	C43—O42—C29	115.15 (15)
C18—C19—H19	119.8	O44—C43—O42	113.8 (2)
C20—C19—H19	119.8	O44—C43—H43A	108.8
C15—C20—C19	119.7 (2)	O42—C43—H43A	108.8
C15—C20—H20	120.1	O44—C43—H43B	108.8
C19—C20—H20	120.1	O42—C43—H43B	108.8
C10—C21—H21A	109.5	H43A—C43—H43B	107.7
C10—C21—H21B	109.5	C43—O44—C45	112.71 (19)
H21A—C21—H21B	109.5	O44—C45—H45A	109.5
C10—C21—H21C	109.5	O44—C45—H45B	109.5
H21A—C21—H21C	109.5	H45A—C45—H45B	109.5
H21B—C21—H21C	109.5	O44—C45—H45C	109.5
C10—C22—H22A	109.5	H45A—C45—H45C	109.5
C10—C22—H22B	109.5	H45B—C45—H45C	109.5
H22A—C22—H22B	109.5	C25—C46—H46A	109.5
C10—C22—H22C	109.5	C25—C46—H46B	109.5
H22A—C22—H22C	109.5	H46A—C46—H46B	109.5
H22B—C22—H22C	109.5	C25—C46—H46C	109.5
C8—C23—H23A	109.5	H46A—C46—H46C	109.5
C8—C23—H23B	109.5	H46B—C46—H46C	109.5
H23A—C23—H23B	109.5	C52—C47—C48	120.1 (2)
C8—C23—H23C	109.5	C52—C47—H47	119.9
H23A—C23—H23C	109.5	C48—C47—H47	119.9
H23B—C23—H23C	109.5	C49—C48—C47	120.1 (2)
C29—C24—C4	112.45 (15)	C49—C48—H48	120.0
C29—C24—C25	111.56 (15)	C47—C48—H48	120.0

C4—C24—C25	111.32 (15)	C48—C49—C50	120.2 (2)
C29—C24—H24	107.1	C48—C49—H49	119.9
C4—C24—H24	107.1	C50—C49—H49	119.9
C25—C24—H24	107.1	C49—C50—C51	119.7 (2)
C46—C25—C26	110.71 (16)	C49—C50—H50	120.2
C46—C25—C39	107.03 (16)	C51—C50—H50	120.2
C26—C25—C39	108.36 (15)	C52—C51—C50	120.2 (2)
C46—C25—C24	111.22 (15)	C52—C51—H51	119.9
C26—C25—C24	107.84 (15)	C50—C51—H51	119.9
C39—C25—C24	111.66 (15)	C47—C52—C51	119.7 (2)
O30—C26—C27	106.89 (15)	C47—C52—H52	120.2
O30—C26—C25	107.73 (15)	C51—C52—H52	120.2
C5—O1—C2—O11	−176.77 (18)	C5—C4—C24—C29	87.2 (2)
C5—O1—C2—O3	1.9 (2)	O3—C4—C24—C25	94.64 (17)
O11—C2—O3—C4	−171.43 (18)	C5—C4—C24—C25	−146.76 (16)
O1—C2—O3—C4	9.9 (2)	C29—C24—C25—C46	69.7 (2)
C2—O3—C4—C24	113.11 (16)	C4—C24—C25—C46	−56.9 (2)
C2—O3—C4—C5	−16.21 (18)	C29—C24—C25—C26	−51.9 (2)
C2—O1—C5—C6	−134.83 (16)	C4—C24—C25—C26	−178.43 (15)
C2—O1—C5—C10	106.55 (16)	C29—C24—C25—C39	−170.86 (16)
C2—O1—C5—C4	−11.50 (18)	C4—C24—C25—C39	62.6 (2)
O3—C4—C5—O1	16.00 (16)	C46—C25—C26—O30	50.78 (19)
C24—C4—C5—O1	−106.16 (17)	C39—C25—C26—O30	−66.31 (18)
O3—C4—C5—C6	131.61 (16)	C24—C25—C26—O30	172.66 (14)
C24—C4—C5—C6	9.4 (2)	C46—C25—C26—C27	−68.1 (2)
O3—C4—C5—C10	−98.47 (16)	C39—C25—C26—C27	174.86 (16)
C24—C4—C5—C10	139.36 (17)	C24—C25—C26—C27	53.8 (2)
O1—C5—C6—C7	−57.91 (19)	O30—C26—C27—C28	−177.59 (15)
C10—C5—C6—C7	58.4 (2)	C25—C26—C27—C28	−58.3 (2)
C4—C5—C6—C7	−171.06 (15)	C26—C27—C28—C29	59.7 (2)
C5—C6—C7—O12	81.26 (19)	C27—C28—C29—O42	−179.61 (15)
C5—C6—C7—C8	−37.5 (2)	C27—C28—C29—C24	−60.4 (2)
O12—C7—C8—C9	−114.50 (19)	C4—C24—C29—O42	−54.6 (2)
C6—C7—C8—C9	6.9 (3)	C25—C24—C29—O42	179.52 (14)
O12—C7—C8—C23	67.2 (2)	C4—C24—C29—C28	−176.75 (15)
C6—C7—C8—C23	−171.41 (16)	C25—C24—C29—C28	57.4 (2)
C7—C8—C9—C10	2.7 (3)	C27—C26—O30—C31	−120.95 (19)
C23—C8—C9—C10	−179.15 (18)	C25—C26—O30—C31	115.37 (18)
C8—C9—C10—C22	139.3 (2)	C26—O30—C31—O32	−3.6 (3)
C8—C9—C10—C21	−103.8 (2)	C26—O30—C31—C33	177.38 (16)
C8—C9—C10—C5	17.7 (3)	O32—C31—C33—C38	174.2 (2)
O1—C5—C10—C9	69.18 (18)	O30—C31—C33—C38	−6.9 (3)
C6—C5—C10—C9	−46.5 (2)	O32—C31—C33—C34	−6.7 (3)
C4—C5—C10—C9	−179.68 (15)	O30—C31—C33—C34	172.23 (17)
O1—C5—C10—C22	−50.6 (2)	C38—C33—C34—C35	1.1 (3)
C6—C5—C10—C22	−166.25 (16)	C31—C33—C34—C35	−178.07 (18)
C4—C5—C10—C22	60.6 (2)	C33—C34—C35—C36	−0.5 (3)

O1—C5—C10—C21	−171.49 (14)	C34—C35—C36—C37	−0.4 (3)
C6—C5—C10—C21	72.82 (19)	C35—C36—C37—C38	0.6 (3)
C4—C5—C10—C21	−60.3 (2)	C36—C37—C38—C33	0.0 (3)
C8—C7—O12—C13	−158.97 (15)	C34—C33—C38—C37	−0.8 (3)
C6—C7—O12—C13	77.20 (19)	C31—C33—C38—C37	178.25 (19)
C7—O12—C13—O14	5.0 (3)	C46—C25—C39—C40	175.91 (17)
C7—O12—C13—C15	−173.77 (15)	C26—C25—C39—C40	−64.7 (2)
O14—C13—C15—C20	−163.81 (19)	C24—C25—C39—C40	54.0 (2)
O12—C13—C15—C20	15.0 (3)	C25—C39—C40—O41	177.32 (17)
O14—C13—C15—C16	12.6 (3)	C28—C29—O42—C43	−74.0 (2)
O12—C13—C15—C16	−168.65 (17)	C24—C29—O42—C43	164.63 (17)
C20—C15—C16—C17	0.5 (3)	C29—O42—C43—O44	76.5 (2)
C13—C15—C16—C17	−175.95 (19)	O42—C43—O44—C45	64.8 (3)
C15—C16—C17—C18	0.5 (3)	C52—C47—C48—C49	0.1 (3)
C16—C17—C18—C19	−1.3 (3)	C47—C48—C49—C50	0.6 (3)
C17—C18—C19—C20	1.2 (3)	C48—C49—C50—C51	−0.4 (3)
C16—C15—C20—C19	−0.6 (3)	C49—C50—C51—C52	−0.3 (3)
C13—C15—C20—C19	175.70 (19)	C48—C47—C52—C51	−0.8 (3)
C18—C19—C20—C15	−0.2 (3)	C50—C51—C52—C47	0.9 (3)
O3—C4—C24—C29	−31.4 (2)		

Hydrogen-bond geometry (Å, º)

Cg is the centroid of the C47–C52 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6*B*···O42	0.99	2.32	3.095 (2)	135
C28—H28*A*···O44	0.99	2.37	2.989 (3)	120
O41—H41···O14^{i}	0.84	2.06	2.888 (2)	170
C7—H7···O32^{i}	1.00	2.34	3.269 (2)	155
C18—H18···O11^{ii}	0.95	2.53	3.465 (2)	168
C49—H49···O11	0.95	2.46	3.300 (3)	147
C27—H27*A*···*Cg*^{iii}	0.95	2.64	3.514 (2)	147

Symmetry codes: (i) −*x*+1, −*y*+2, −*z*+1; (ii) −*x*+1, −*y*+1, −*z*+2; (iii) −*x*+1, −*y*+1, −*z*+1.