Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

ISSN 2056-9890

Received 24 March 2015
Accepted 6 April 2015

Edited by H. Ishida, Okayama University, Japan

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

CCDC references: 1057985; 1057984; 1057983

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

Crystal structures of (±)-(1*SR*,5*SR*,6*SR*,7*SR*,10*SR*,-11*SR*,13*RS*,14*SR*)-13-hydroxy-7-methoxymethoxy-11,15,18,18-tetramethyl-3-oxo-2,4-dioxatetracyclo[12.3.1.$0^{1,5}$.$0^{6,11}$]octadec-15-en-10-yl benzoate, its 13-epimer and 13-one derivative

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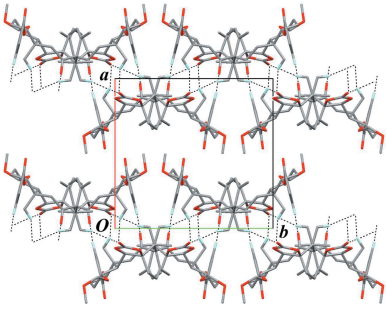
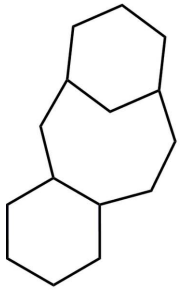
The title compounds, $C_{29}H_{38}O_8{\cdot}0.25C_5H_{12}$, (**A**), $C_{29}H_{38}O_8$, (**B**), and $C_{29}H_{36}O_8$, (**C**), are tetracyclic benzoates possessing a taxane skeleton with a fused dioxolane ring as the core structure. In the asymmetric unit of (**A**), there are two independent benzoate molecules (A and A′) and a half molecule of solvent pentane disordered about an inversion center. The molecular conformations of (**A**), (**B**) and (**C**) are similar except for the flexible methoxymethoxy group. The cyclohexane, cyclohexene and central cyclooctane rings adopt chair, half-chair and chair–chair (extended crown) forms, respectively. The dioxolane rings are essentially planar, while the dioxolane ring of A′ is slightly twisted from the mean plane. In the crystal of (**A**), intermolecular O—H···O, C—H···O and C—H···π interactions link the independent benzoates alternately, forming a chain structure. In the crystals of (**B**) and (**C**), molecules are linked through O—H···O and C—H···π interactions, and C—H···O hydrogen bonds, respectively, into similar chains. Further, weak intermolecular C—H···O interactions connect the chains into a three-dimensional network in (**A**) and a sheet in (**B**), whereas no other interactions are observed for (**C**).

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). The complicated structure and significant bioactivity have attracted chemical and medicinal interest. Recently, we





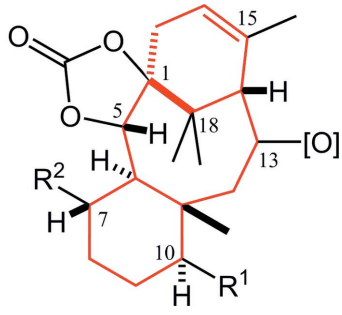
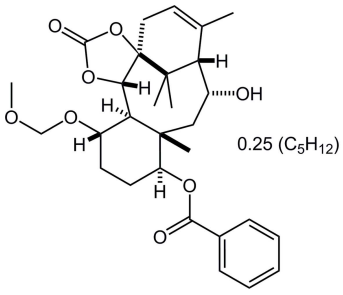
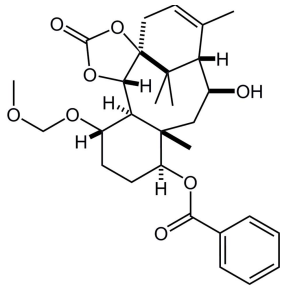


Figure 1
Left: Structure of the tricyclo[9.3.1.$0^{3,8}$]pentadecane (taxane) skeleton; Right: Core structure of the title compounds. Red lines indicate the taxane skeleton. R^1 = OC(=O)Ph, R^2 = OCH_2OCH_3.

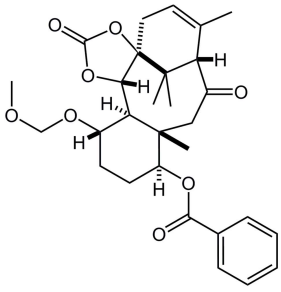
reported the crystal structure of the precursor for cyclization to build the taxane skeleton (Oishi *et al.*, 2015; §4), which was obtained in a synthetic study of paclitaxel. The cyclization reaction was accomplished (Fukaya *et al.*, 2015) to afford strained tetracyclic benzoates (**A**) and its 13-epimer (**B**), then further oxidation gave a ketone (**C**).



compound (**A**)



compound (**B**)



compound (**C**)

2. Structural commentary

The asymmetric units of the title compounds, (**A**), (**B**) and (**C**), are shown in Figs. 2, 3 and 4, respectively. Their molecular conformations are similar except for the flexible methoxymethoxy group (Fig. 5).

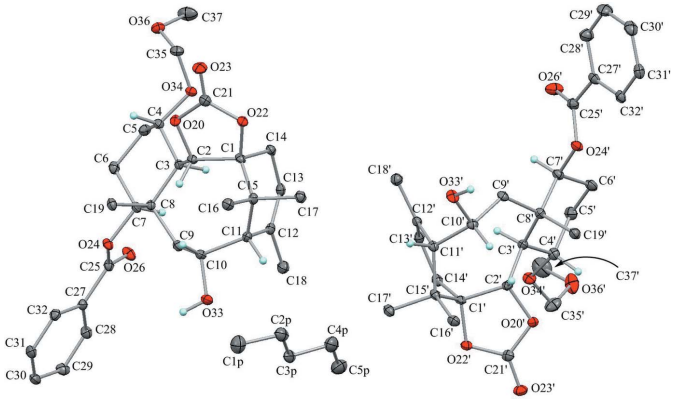


Figure 2
The asymmetric unit of compound (**A**) with the atom labelling. Displacement ellipsoids are drawn at the 30% probability level. The left benzoate molecule has been moved by a symmetry operation of $(-x+1, -y+1, -z+1)$ from its original position. The pentane solvent molecule is disordered by symmetry over two sites with occupancy 0.50. Only H atoms connected to O and chiral C atoms are shown for clarity.

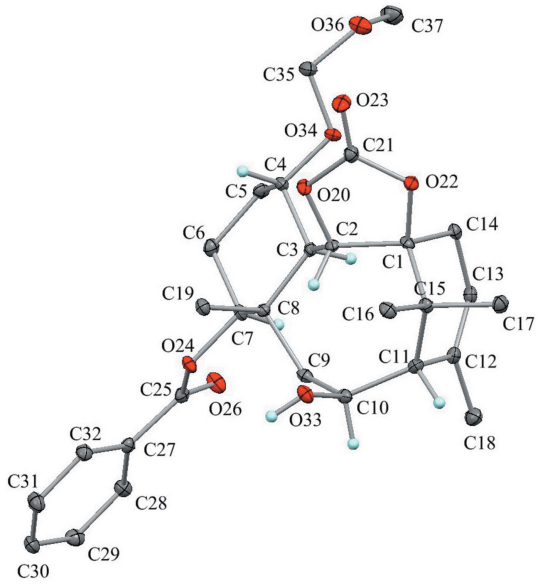


Figure 3
The asymmetric unit of compound (**B**) with the atom labelling. Displacement ellipsoids are drawn at the 30% probability level. Only H atoms connected to O and chiral C atoms are shown for clarity.

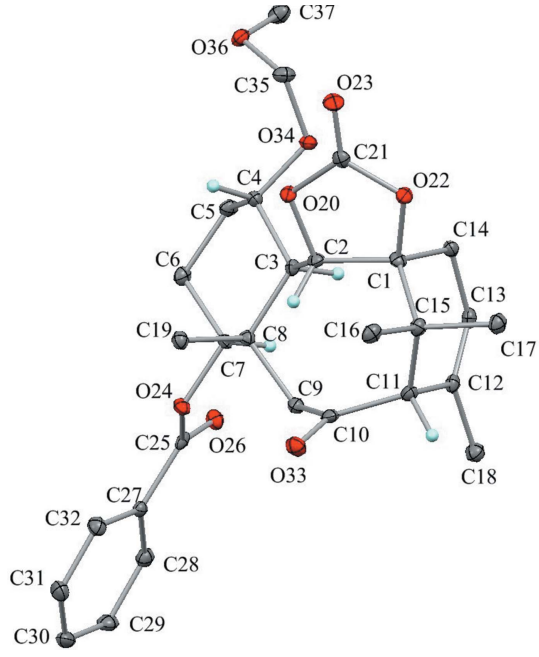


Figure 4
The asymmetric unit of compound (**C**) with the atom labelling. Displacement ellipsoids are drawn at the 30% probability level. Only H atoms connected to O and chiral C atoms are shown for clarity.

2.1. (±)-(1*SR*,5*SR*,6*SR*,7*SR*,10*SR*,11*SR*,13*RS*,14*SR*)-13-Hydroxy-7-methoxymethoxy-3-oxo-11,15,18,18-tetramethyl-2,4-dioxatetracyclo[12.3.1.$0^{1,5}$.$0^{6,11}$]octadec-15-en-10-yl benzoate, (A)

The two independent molecules, A (C1–C37) and A′ (C1′–C37′), adopt slightly different conformations. The pentane solvent molecule is disordered around the center of symmetry.

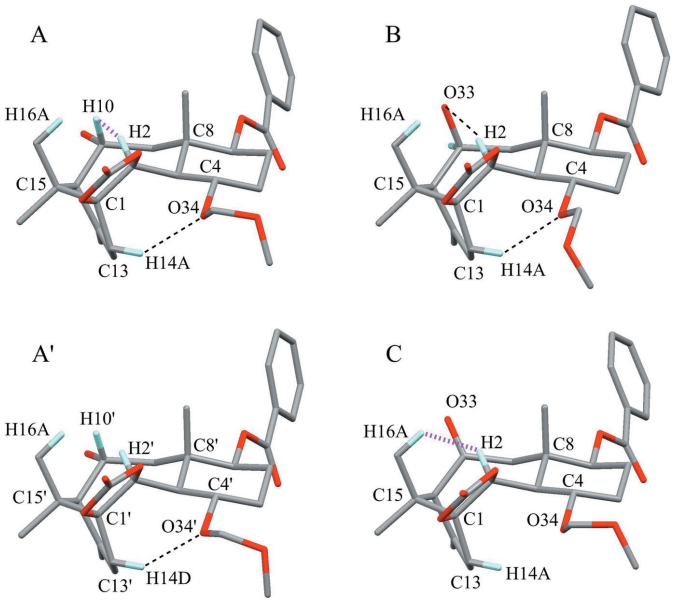


Figure 5
The molecular conformations of compounds, (**A**), (**B**) and (**C**). In (**A**), there are two independent benzoates indicated as A (C1–C37) and A′ (C1′–C37′). Black dashed lines indicate the intramolecular C—H···O interactions. Purple dotted lines indicate intramolecular H···H short contacts. For clarity, only H atoms involved in these interactions are shown.

The dioxolane ring in A (C1/C2/O20/C21/O22) is essentially planar with a maximum deviation of 0.0434 (18) Å for atom C1, while the dioxolane ring in A′ (C1′/C2′/O20′/C21′/O22′) shows a flattened twisted form with puckering parameters of $Q(2)$ = 0.0713 (17) Å and $\varphi(2)$ = 47.8 (14)°. Atoms C1′ and C2′ deviate from the mean plane of the other atoms by 0.104 (4) and −0.019 (4) Å, respectively.

The cyclohexane rings (C3–C8 in A and C3′–C8′ in A′) adopt chair forms with puckering parameters of Q = 0.5728 (19) Å, θ = 174.96 (19)°, φ = 352 (2)°, $Q(2)$ = 0.0508 (19) Å and $Q(3)$ = – 0.5705 (19) Å for the C3–C8 ring, and Q = 0.570 (2) Å, θ = 6.68 (19)°, φ = 181.2 (16)°, $Q(2)$ = 0.0691 (19) Å and $Q(3)$ = 0.5656 (19) Å for the C3′–C8′ ring. The larger substituents (C3—C2 and C3′—C2′; C4—O34 and C4′—C34′; C7—O24 and C7′—C24′) are in the equatorial positions, but substituents on quaternary carbons (C8—C9 and C8′—C9′) are slightly tilted from the equatorial positions with angles to the *Cremer & Pople plane* of 59.74 (13) and 59.59 (13)°, respectively.

The cyclohexene ring in A (C1/C14/C13/C12/C11/C15) adopts a half-chair form with puckering parameters of Q = 0.5419 (18) Å, θ = 50.2 (2)°, φ = 318.9 (3)°, $Q(2)$ = 0.4162 (19) Å and $Q(3)$ = −0.3470 (19) Å. Atoms C1 and C15 deviate from the mean plane of the other four atoms by 0.272 (3) and −0.547 (3) Å, respectively. The cyclohexene ring in A′ (C1′/C14′/C13′/C12′/C11′/C15′) also adopts a half-chair form with puckering parameters of Q = 0.5364 (19) Å, θ = 129.8 (2)°, φ = 138.8 (3)°, $Q(2)$ = 0.4124 (19) Å and $Q(3)$ = −0.3431 (19) Å. Atoms C1′ and C15′ deviate from the mean plane of other atoms by −0.268 (3) and 0.543 (3) Å, respectively.

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

*Cg*1 is the centroid of the C27–C32 benzene ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C14—H14*A*···O34	0.99	2.47	3.320 (2)	143
C14′—H14*D*···O34′	0.99	2.36	3.221 (2)	145
O33—H33···O22′^{i}	0.84	2.05	2.8563 (17)	160
O33′—H33′···O22^{ii}	0.84	2.05	2.8839 (16)	169
C7—H7···O26^{iii}	1.00	2.28	3.236 (2)	159
C4′—H4′···O36^{iv}	1.00	2.40	3.311 (2)	151
C17′—H17*F*···O33^{i}	0.98	2.48	3.431 (2)	164
C30′—H30′···O23′^{v}	0.95	2.53	3.453 (2)	163
C3*P*—H3*PB*···O33	0.99	2.49	3.369 (9)	149
C4*P*—H4*PA*···O33^{i}	0.99	2.41	3.291 (12)	148
C16′—H16*D*···*Cg*1^{i}	0.98	2.85	3.5315 (19)	127

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

The central cyclooctane ring in A (C1–C3/C8–C11/C15) adopts a chair–chair (an extended crown) form with puckering parameters of Q = 0.8995 (18) Å, $Q(2)$ = 0.3441 (18) Å, $\varphi(2)$ = 305.1 (3)°, $Q(3)$ = 0.0632 (18) Å, $\varphi(3)$ = 180.8 (16)° and $Q(4)$ = −0.8286 (18) Å. The cyclooctane ring in A′ (C1′–C3′/C8′–C11′) also adopts a similar form with puckering parameters of Q = 0.8940 (18) Å, $Q(2)$ = 0.3432 (18) Å, $\varphi(2)$ = 130.3 (3)°, $Q(3)$ = 0.0866 (19) Å, $\varphi(3)$ = 11.1 (12)° and $Q(4)$ = 0.8209 (18) Å. There is a short intramolecular contact of 1.93 Å between atoms H2 and H10 in A, while the distance between H2′ and H10′ in A′ is 2.05 Å. The methoxymethoxy groups (O34/C35/O36/C37 in A and O34′/C35′/O36′/C37′ in A′) show helical forms with weak intramolecular C—H···O interactions (Fig. 5, Table 1).

2.2. (±)-(1*SR*,5*SR*,6*SR*,7*SR*,10*SR*,11*SR*,13*SR*,14*SR*)-13-Hydroxy-7-methoxymethoxy-3-oxo-11,15,18,18-tetramethyl-2,4-dioxatetracyclo[12.3.1.0^{1,5}.0^{6,11}]octadec-15-en-10-yl benzoate, (B)

Compound (**B**) is the C10-epimer of (**A**). The dioxolane ring in (**B**) (C1/C2/O20/C21/O22) is essentially planar with a maximum deviation of 0.0124 (11) Å for atom O22. The cyclohexane ring (C3–C8) adopts a chair form with puckering parameters of Q = 0.564 (2) Å, θ = 4.1 (2)°, φ = 124 (3)°, $Q(2)$ = 0.039 (2) Å and $Q(3)$ = 0.562 (2) Å. The larger substituents (C3—C2, C4—O34 and C7—O24) are in the equatorial positions, while the substituents on quaternary carbon (C8—C9) is slightly tilted from the equatorial position with an angle to the *Cremer & Pople plane* of 57.89 (13)°.

The cyclohexene ring (C1/C14/C13/C12/C11/C15) adopts a half-chair form with puckering parameters of Q = 0.540 (2) Å, θ = 130.1 (2)°, φ = 136.3 (3)°, $Q(2)$ = 0.413 (2) Å and $Q(3)$ = −0.348 (2) Å. Atoms C1 and C15 deviate from the mean plane of the other four atoms by 0.237 (4) and −0.575 (4) Å, respectively. The central cyclooctane ring (C1–C3/C8–C11/C15) adopts a chair-chair form with puckering parameters of Q = 0.863 (2) Å, $Q(2)$ = 0.283 (2) Å, $\varphi(2)$ = 126.7 (4)°, $Q(3)$ =

Table 2
Hydrogen-bond geometry (Å, °) for **B**.

*Cg*2 is the centroid of the C27–C32 benzene ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O33	0.98	2.27	3.200 (2)	157
C14—H14*A*···O34	0.97	2.47	3.293 (2)	142
O33—H33···O23^{i}	0.82	1.96	2.7823 (19)	179
C7—H7···O26^{ii}	0.98	2.50	3.353 (2)	145
C16—H16*A*···*Cg*2^{iii}	0.98	2.93	3.594 (2)	128

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

0.113 (2) Å, $\varphi(3)$ = 23.1 (10)° and $Q(4)$ = 0.807 (2) Å. The elongated bond lengths of 1.584 (3) Å for C10—C11 and 1.571 (3) Å for C11—C15, and the extraordinary sp^3 angle of 126.80 (17)° for C8—C9—C10 suggest strain in the fused ring system. There are intramolecular C—H···O interactions (C2—H2···O33 and C14—H14*A*···O34; Table 2).

2.3. (±)-(1*SR*,5*SR*,6*SR*,7*SR*,10*SR*,11*SR*,14*SR*)-3,13-Dioxo-7-methoxymethoxy-11,15,18,18-tetramethyl-2,4-dioxatetracyclo[12.3.1.0^{1,5}.0^{6,11}]octadec-15-en-10-yl benzoate, (C)

Compound (**C**) is the C10-oxo derivative of (**A**) and (**B**). The dioxolane ring in (**C**) (C1/C2/O20/C21/O22) is essentially planar with a maximum deviation of 0.0280 (12) Å for atom O22. The cyclohexane ring (C3–C8) adopts a chair form with puckering parameters of Q = 0.563 (2) Å, θ = 5.9 (2)°, φ = 227 (2)°, $Q(2)$ = 0.056 (2) Å and $Q(3)$ = 0.560 (2) Å. The substituents including that on the quaternary carbon (C3—C2, C4—O34, C7—O24 and C8—C9) are in the equatorial positions.

The cyclohexene ring (C1/C14/C13/C12/C11/C15) adopts a half-chair form with puckering parameters of Q = 0.533 (2) Å,

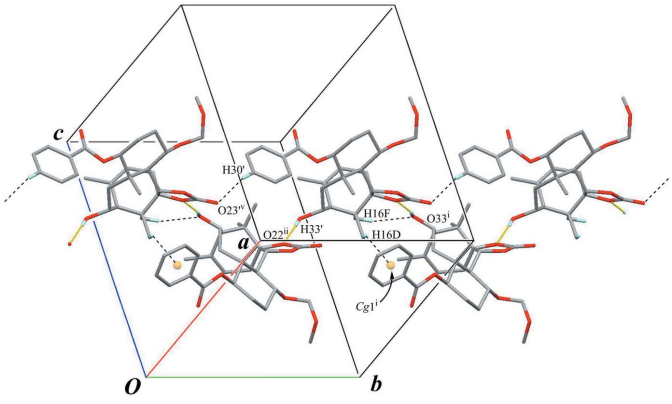


Figure 6
A partial packing view of (**A**) showing the chain structure. Yellow lines indicate the intermolecular O—H···O hydrogen bonds, generating $C(7)$ chains. Black dashed lines indicate the weak intermolecular C—H···O and C—H···π interactions. *Cg*1 is the centroid of the C27–C32 benzene ring. Only H atoms involved in hydrogen bonds are shown for clarity. The pentane solvent molecules have been omitted. [Symmetry codes: (i) $-x+1, -y+2, -z+1$; (ii) $-x+1, -y+1, -z+1$; (v) $x, y-1, z$.]

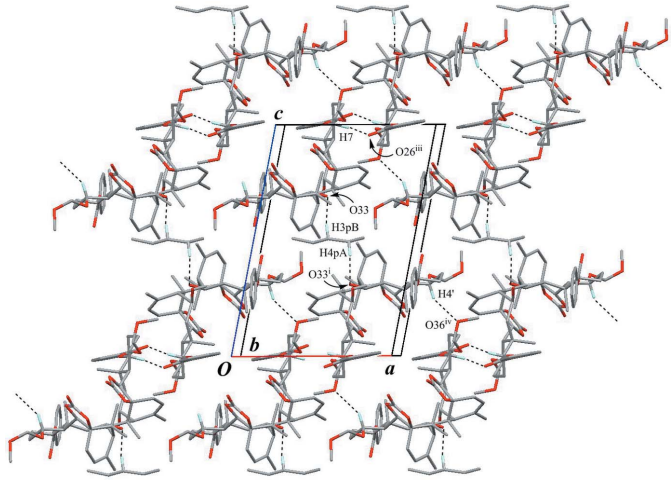


Figure 7
A packing diagram of (**A**) viewed down to *b* axis showing a three-dimensional network. The chains (projected as *butterfly*-like shapes) are connected by the intermolecular C—H···O interactions (black dashed lines). Only H atoms involved in hydrogen bonds are shown for clarity. [Symmetry codes: (iii) $-x+1, -y+2, -z+2$; (iv) $x+1, y, z-1$.]

θ = 131.7 (2)°, φ = 135.3 (3)°, $Q(2)$ = 0.398 (2) Å and $Q(3)$ = −0.354 (2) Å. Atoms C1 and C15 deviate from the mean plane of the other four atoms by 0.222 (4) and −0.577 (4) Å, respectively. The central cyclooctane ring (C1–C3/C8–C11/C15) adopts a chair-chair form with puckering parameters of Q = 0.898 (2) Å, $Q(2)$ = 0.311 (2) Å, $\varphi(2)$ = 113.2 (4)°, $Q(3)$ = 0.066 (2) Å, $\varphi(3)$ = 353 (2)° and $Q(4)$ = 0.839 (2) Å. There is a short intramolecular contact of 1.88 Å between the atoms H2 and H16*A*.

3. Supramolecular features

3.1. Compound (A)

The crystal packing is stabilized by intermolecular O—H···O hydrogen bonds (O33—H33···O22′^{i} and O33′—H33′···O22^{ii}; Table 1) connecting the A and A′ molecules alternately to form a chain with a $C(7)$ motif running along the *b* axis (Fig. 6). Further intermolecular weak C—H···O and C—H···π interactions (C17′—C17F···O33^{i}, C30′—H30′···O23′^{v} and C16′—H16*D*···*Cg*1^{i}; Table 1) support the chain structure. Interestingly, the geometric data for the corresponding interactions (C17—H17*C*···O33′^{ii}, C30—H30···O23^{vi} and C16—H16*A*···*Cg*1′) are 2.76 Å for H17*C*···O33′^{ii}, 2.80 Å for H30···O23^{vi} and 2.95 Å for H16—*Cg*1′, and 118.8° for C30—H30···O23^{vi} and 119° for C16—H16*A*···*Cg*1′, which are out of the range for proper values of a hydrogen bond [symmetry code: (vi) $x, y+1, z$; *Cg*1′ is the centroid of the C27′–C32′ benzene ring.]

The chains are interlocked by a pair of intermolecular C—H···O hydrogen bonds (C7—H7···O26^{iii}; Table 1) with an $R_2^2(10)$ graph-set motif, forming a tape parallel to ($\bar{1}$01) and along the *b* axis (Fig. 7). The adjacent tapes are connected by intermolecular C—H···O interactions (C4′—H4′···O36^{iv};

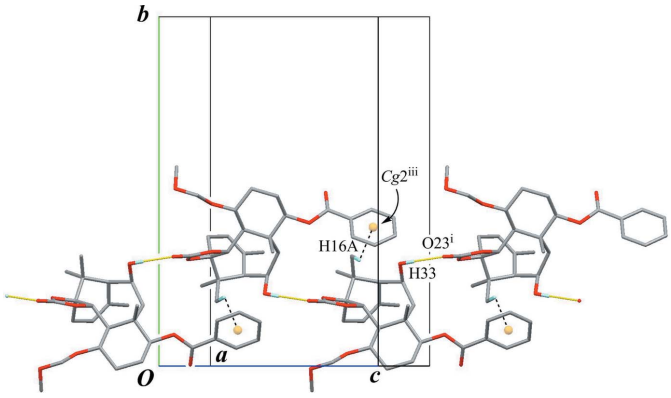


Figure 8
A partial packing view of (**B**) showing a chain structure. The intermolecular O—H···O hydrogen bonds (yellow lines) link the enantiomers alternately, generating *C*(9) chains. In the chain, further intermolecular weak C—H···π interactions (black dashed lines) are also observed. *Cg*2 is the centroid of the C27–C32 phenyl ring. Only H atoms involved in hydrogen bonds are shown for clarity. [Symmetry codes: (i) $x+\frac{1}{2}, -y+\frac{1}{2}, -z+\frac{1}{2}$; (iii) $x-\frac{1}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$.]

Table 1), forming a layer parallel to (001). Among the layers, disordered solvent pentane molecules are held by weak intermolecular C—H···O interactions (C3*P*—H3*PB*···O33 and C4*P*—H4*PA*···O33^{i}; Table 1), constructing a three-dimensional architecture.

3.2. Compound (B)

The crystal packing is stabilized by an intermolecular O—H···O hydrogen bond (O33—H33···O23^{i}; Table 2) connecting the enantiomers alternately to form a chain with a

Table 3
Hydrogen-bond geometry (Å, °) for **C**.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C31—H31···O33^{i}	0.95	2.35	3.147 (3)	141
C19—H19*C*···O23^{ii}	0.98	2.43	3.310 (3)	149
C16—H16*A*···O23^{ii}	0.98	2.56	3.491 (3)	158

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

C(9) motif along [101] (Fig. 8). Further, an intermolecular weak C—H···π interaction (C16—H16*A*···*Cg*2^{iii}; Table 2) supports the chain formation. The chains are connected by a pair of intermolecular C—H···O hydrogen bonds (C7—H7···O26^{ii}; Fig. 9, Table 2) with an $R_2^2(10)$ graph-set motif, forming a sheet parallel to (10$\bar{1}$).

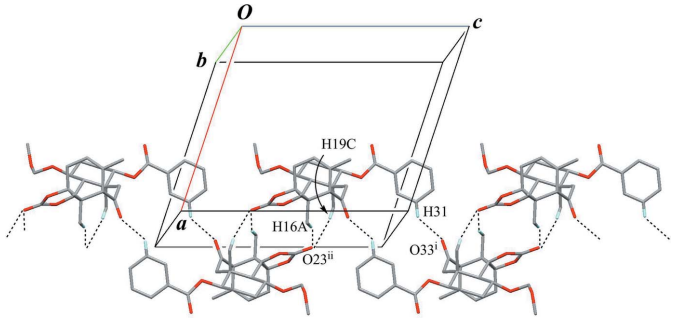


Figure 10
A partial packing view of (**C**) showing the chain structure. Intermolecular C—H···O interactions (blacked dashed lines) link the enantiomers. Only H atoms involved in hydrogen bonds are shown for clarity. [Symmetry codes: (i) $-x+2, -y+1, -z+2$; (ii) $-x+2, -y+1, -z+1$.]

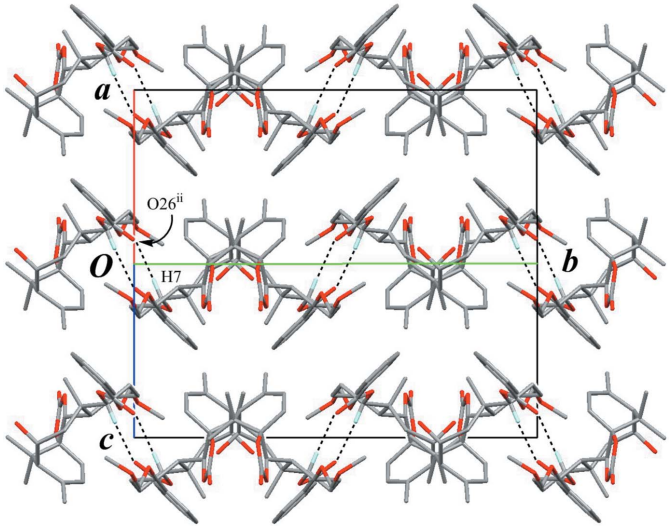


Figure 9
A packing diagram of (**B**) viewed along [$\bar{1}$0$\bar{1}$], showing parallel sheets. The chains (projected as *fly*-like shapes) are connected by pairs of intermolecular C—H···O interactions (black dashed lines), forming sheets parallel to (10$\bar{1}$). Only H atoms involved in hydrogen bonds are shown for clarity. [Symmetry codes: (ii) $-x+2, -y, -z+2$.]

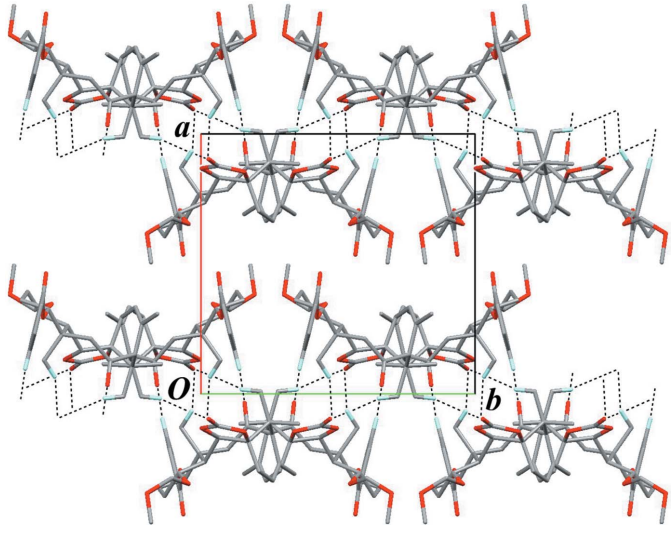


Figure 11
A packing diagram of (**C**) viewed down the *c* axis. Black dashed lines indicate the intermolecular C—H···O interactions. Overlapped molecules (projection as a *spider*-like shape) do not constitute the same chain. A half body of the *spider* is only linked to the adjacent inverted one.

Table 4
Experimental details.

	A	B	C
Crystal data			
Chemical formula	$C_{29}H_{38}O_8 \cdot 0.25C_5H_{12}$	$C_{29}H_{38}O_8$	$C_{29}H_{36}O_8$
M_r	532.64	514.59	512.59
Crystal system, space group	Triclinic, $P\overline{1}$	Monoclinic, $P2_1/n$	Monoclinic, $P2_1/c$
Temperature (K)	90	90	90
a, b, c (Å)	11.3343 (5), 15.4666 (7), 16.4870 (8)	9.3612 (6), 19.6336 (15), 14.1965 (9)	13.2416 (8), 13.1779 (8), 15.2428 (8)
α, β, γ (°)	85.1124 (14), 78.3773 (14), 78.5231 (15)	90, 101.762 (2), 90	90, 109.387 (2), 90
V (Å^3)	2771.3 (2)	2554.4 (3)	2509.0 (3)
Z	4	4	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.09	0.10	0.10
Crystal size (mm)	0.32 × 0.21 × 0.17	0.23 × 0.23 × 0.14	0.22 × 0.14 × 0.09
Data collection			
Diffractometer	Bruker D8 Venture	Bruker D8 Venture	Bruker D8 Venture
Absorption correction	Multi-scan (*SADABS*; Bruker, 2014)	Multi-scan (*SADABS*; Bruker, 2014)	Multi-scan (*SADABS*; Bruker, 2014)
T_{min}, T_{max}	0.97, 0.98	0.98, 0.99	0.92, 0.99
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	50997, 9735, 7339	23252, 4480, 3212	22512, 4395, 3050
R_{int}	0.041	0.058	0.063
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.595	0.595	0.595
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.040, 0.099, 1.02	0.043, 0.111, 1.02	0.044, 0.105, 0.96
No. of reflections	9735	4480	4395
No. of parameters	726	340	339
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.27, −0.24	0.51, −0.21	0.29, −0.25

Computer programs: *APEX2* and *SAINT* (Bruker, 2014), *SHELXS2013* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *Mercury* (Macrae *et al.*, 2006), *publCIF* (Westrip, 2010) and *PLATON* (Spek, 2009).

3.3. Compound (C)

The crystal packing is stabilized by a pair of intermolecular C—H···O interactions (C31—H31···O33^{i}; Table 3) with an $R_2^2(22)$ graph-set motif, forming an inversion dimer (Figs. 10 and 11). The dimers are further linked into a chain along the *c* axis by intermolecular C—H···O interactions (C19—H19*C*···O23^{ii} and C16—H16*A*···O23^{ii}; Table 3) with $R_2^2(16)$ and $R_2^2(14)$ graph-set motifs, respectively. There is an intermolecular O36···C25^{iii} short contact of 3.012 (3) Å involving the carbonyl group of the benzoyl moiety [symmetry code: (iii) $x, -y+\frac{3}{2}, z-\frac{1}{2}$].

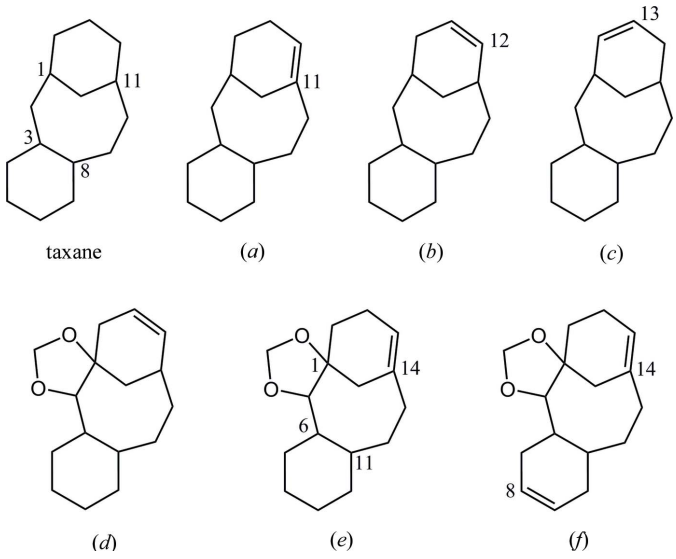


Figure 12
Core structures for database survey; tricyclo[9.3.1.0^{3,8}]pentadecane (taxane) and its (*a*) 11-ene, (*b*) 12-ene and (*c*) 13-ene derivatives, (*d*) tetracyclic core of the title compounds and (*e*) its regioisomer of olefin and (*f*) dehydro derivative of regioisomer.

4. Database survey

In the Cambridge Structural Database (CSD, Version 5.36, November 2014; Groom & Allen, 2014), 85 structures containing a tricyclo[9.3.1.0^{3,8}]pentadec-11-ene skeleton, (*a*), are found (Fig. 12). These include a large number of paclitaxels and its analogues, and one compound (NEGBOQ; Poujol *et al.*, 1997) containing a 2,4-dioxatetracyclo-[12.3.1.0^{1,5}.0^{6,11}]octadec-14-ene skeleton, (*e*), which is an olefin regioisomer for the tetracyclic core of the title compound, (*d*). On the other hand, there are two related structures (PAHTEZ; Mendoza *et al.*, 2011, and RIYTAW; Wilde *et al.*, 2014) containing a tricyclo[9.3.1.0^{3,8}]pentadec-12-ene skeleton, (*b*), and one related structure (SOJWOD; Paquette & Zhao, 1998) for a tricyclo[9.3.1.0^{3,8}]pentadec-13-ene skeleton, (*c*).

Another tetracyclic taxoid (ILIQUP; Ohba *et al.*, 2003), which was unexpectedly generated by a cyclization reaction in our previous study, is closely related to the title compound. Additionally, a precursor of cyclization obtained in our previous study is also available (NOTROF; Oishi *et al.*, 2015). Another compound, closely related to the title compounds with a 2,4-dioxatetracyclo[12.3.1.0^{1,5}.0^{6,11}]octadeca-8,14-diene skeleton, (*f*), was reported in the literature (Nicolaou *et al.*, 1995), but was not deposited in the CSD.

5. Synthesis and crystallization

The title compounds were obtained in a synthetic study on paclitaxel (Fukaya *et al.*, 2015). The cyclohexene unit (C1/C14/C13/C12/C11/C15) was synthesized according to a reported procedure (Nicolaou *et al.*, 1995), and coupled with the substituted cyclohexane unit (C3–C8) prepared from 3-methylanisole by a Shapiro reaction (Nicolaou *et al.*, 1995). Further manipulation of the functional groups and cyclization reaction afforded the tetracyclic benzoates (**A**) and its C10-epimer (**B**), which were oxidized into ketone (**C**). Each compound was purified by silica gel chromatography. Colorless crystals of (**A**) were grown from a benzene solution under a pentane-saturated atmosphere by slow evaporation at ambient temperature. Similarly, colorless crystals of (**B**) and (**C**) were obtained in the same manner.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 4. C-bound H atoms were positioned 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 was placed guided by difference maps, with O—H = 0.84 Å and with U_{iso}(H) = 1.5U_{eq}(O).

Acknowledgements

This research was partially supported by the Keio Gijuku Fukuzawa Memorial Fund for the Advancement of Education and Research. We also thank Professor S. Ohba (Keio University, Japan) for providing valuable advice.

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supporting information

Acta Cryst. (2015). E**71**, 466-472 [doi:10.1107/S2056989015006854]

Crystal structures of (±)-(1*SR*,5*SR*,6*SR*,7*SR*,10*SR*,11*SR*,13*RS*,14*SR*)-13-hydroxy-7-methoxymethoxy-11,15,18,18-tetramethyl-3-oxo-2,4-dioxatetracyclo-[12.3.1.$0^{1,5}$.$0^{6,11}$]octadec-15-en-10-yl benzoate, its 13-epimer and 13-one derivative

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

Computing details

For all compounds, data collection: *APEX2* (Bruker, 2014); cell refinement: *SAINT* (Bruker, 2014); data reduction: *SAINT* (Bruker, 2014); program(s) used to solve structure: *SHELXS2013* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010) and *PLATON* (Spek, 2009).

(A) (±)-(1*SR*,5*SR*,6*SR*,7*SR*,10*SR*,11*SR*,13*RS*,14*SR*)-13-Hydroxy-7-methoxymethoxy-11,15,18,18-tetramethyl-3-oxo-2,4-dioxatetracyclo[12.3.1.$0^{1,5}$.$0^{6,11}$]octadec-15-en-10-yl benzoate pentane 0.25-solvate

Crystal data

$C_{29}H_{38}O_8 \cdot 0.25C_5H_{12}$
M_r = 532.64
Triclinic, $P\overline{1}$
a = 11.3343 (5) Å
b = 15.4666 (7) Å
c = 16.4870 (8) Å
α = 85.1124 (14)°
β = 78.3773 (14)°
γ = 78.5231 (15)°
V = 2771.3 (2) Å^3
Z = 4
$F(000)$ = 1146
D_x = 1.277 Mg m^{-3}
Melting point: 509.2 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9428 reflections
θ = 2.4–25.0°
μ = 0.09 mm^{-1}
T = 90 K
Prism, colorless
0.32 × 0.21 × 0.17 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, 2014)
T_{min} = 0.97, T_{max} = 0.98
50997 measured reflections
9735 independent reflections
7339 reflections with $I > 2\sigma(I)$
R_{int} = 0.041
θ_{max} = 25.0°, θ_{min} = 2.2°
h = −13→13
k = −18→18
l = −19→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.099$
$S = 1.02$
9735 reflections
726 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
$w = 1/[\sigma^2(F_o^2) + (0.039P)^2 + 1.4256P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.008$
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}

Special details

Experimental. *M.p.* 507.2–509.2 K (not corrected); IR (film) 3502, 2950, 1799, 1717, 1451, 1272, 1098, 1055, 713 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ (p.p.m.) 8.03–8.00 (m, 2H), 7.60–7.55 (m, 1H), 7.48–7.43 (m, 2H), 5.50 (bs, 1H), 4.78 (d, J = 7.2 Hz, 1H), 4.71 (dd, J = 11.3, 4.3 Hz, 1H), 4.58 (d, J = 7.2 Hz, 1H), 4.50 (d, J = 3.7 Hz, 1H), 4.31 (dd, J = 9.0, 2.0 Hz, 1H), 3.56 (ddd, J = 10.7, 10.7, 5.2 Hz, 1H), 3.35 (s, 3H), 2.87 (bd, J = 18.3 Hz, 1H), 2.41–2.30 (m, 2H), 2.11 (s, 1H), 2.08 (dd, J = 10.7, 3.7 Hz, 1H), 1.89 (dddd, J = 12.7, 4.3, 4.0, 4.0 Hz, 1H), 1.77 (s, 3H), 1.72–1.50 (m, 4H), 1.29 (s, 3H), 1.25 (s, 3H), 1.17 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ (p.p.m.) 165.9 (C), 154.1 (C), 136.7 (C), 133.4 (CH), 130.3 (C), 129.7 (CH), 128.6 (CH), 120.9 (CH), 97.8 (CH_2), 87.1 (C), 80.0 (CH), 78.1 (CH), 75.0 (CH), 67.1 (CH), 60.6 (CH), 55.9 (CH_3), 47.8 (CH_2), 46.2 (CH), 42.6 (C), 40.2 (C), 31.9 (CH_2), 31.2 (CH_2), 25.9 (CH_3), 25.1 (CH_3), 24.7 (CH_2), 19.8 (CH_3), 13.6 (CH_3); LRMS (EI) *m/z* 514 (M^+, 4%), 483 (1), 469 (1), 453 (1), 409 (4), 393 (12), 348 (1), 332 (1), 121 (83), 105 (100), 77 (67); HRMS (EI) *m/z* calcd for $C_{29}H_{38}O_8^+$ $[M]^+$ 514.2567, found 514.2545.

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 three reflections with $|I(\text{obs})-I(\text{calc})|/\sigma W(I)$ greater than 10 (1 1 0, 9 9 11 and 5 3 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}	Occ. (<1)
C1	0.39192 (15)	0.71883 (11)	0.80546 (10)	0.0182 (4)	
C2	0.29323 (15)	0.76771 (11)	0.87419 (10)	0.0178 (4)	
H2	0.2366	0.8123	0.8455	0.021*	
C3	0.33237 (15)	0.81694 (11)	0.93856 (10)	0.0167 (4)	
H3	0.4227	0.8146	0.9212	0.02*	
C4	0.31165 (16)	0.77206 (11)	1.02534 (11)	0.0199 (4)	
H4	0.2234	0.767	1.0428	0.024*	
C5	0.34803 (17)	0.82311 (11)	1.08803 (11)	0.0228 (4)	
H5A	0.4373	0.8225	1.0739	0.027*	
H5B	0.3297	0.7943	1.1439	0.027*	
C6	0.27850 (17)	0.91845 (11)	1.08866 (11)	0.0231 (4)	
H6A	0.1893	0.9196	1.1049	0.028*	
H6B	0.3035	0.9513	1.1293	0.028*	
C7	0.30713 (16)	0.96120 (11)	1.00303 (10)	0.0192 (4)	
H7	0.3973	0.9604	0.9885	0.023*	

C8	0.27009 (15)	0.91625 (11)	0.93435 (10)	0.0169 (4)
C9	0.32337 (16)	0.96195 (11)	0.85154 (10)	0.0196 (4)
H9B	0.285	1.0253	0.8542	0.024*
H9A	0.4116	0.9585	0.8511	0.024*
C10	0.31409 (16)	0.93271 (11)	0.76616 (10)	0.0196 (4)
H10	0.2326	0.9153	0.7703	0.024*
C11	0.41687 (15)	0.85809 (11)	0.72360 (10)	0.0184 (4)
H11	0.4345	0.8795	0.6642	0.022*
C12	0.53785 (16)	0.85012 (11)	0.75254 (10)	0.0201 (4)
C13	0.58295 (16)	0.77927 (11)	0.79574 (11)	0.0211 (4)
H13	0.659	0.7782	0.812	0.025*
C14	0.52260 (15)	0.70092 (11)	0.82067 (11)	0.0201 (4)
H14A	0.522	0.6854	0.8802	0.024*
H14B	0.5706	0.6499	0.7888	0.024*
C15	0.38464 (15)	0.76528 (11)	0.72002 (10)	0.0191 (4)
C16	0.25946 (16)	0.77064 (12)	0.69574 (11)	0.0225 (4)
H16A	0.1941	0.7916	0.7423	0.034*
H16B	0.2515	0.712	0.6819	0.034*
H16C	0.2529	0.8118	0.6476	0.034*
C17	0.47951 (17)	0.71477 (12)	0.65165 (11)	0.0237 (4)
H17B	0.4675	0.742	0.5973	0.036*
H17C	0.4692	0.6531	0.6547	0.036*
H17A	0.5623	0.7171	0.6594	0.036*
C18	0.60684 (17)	0.92447 (12)	0.72667 (12)	0.0278 (4)
H18A	0.6397	0.9233	0.667	0.042*
H18B	0.6745	0.9177	0.7569	0.042*
H18C	0.5513	0.9809	0.7392	0.042*
C19	0.13055 (15)	0.92911 (11)	0.94717 (11)	0.0214 (4)
H19C	0.1076	0.9005	0.9034	0.032*
H19A	0.0958	0.9924	0.945	0.032*
H19B	0.0988	0.9027	1.0013	0.032*
O20	0.22400 (11)	0.70057 (8)	0.91336 (7)	0.0214 (3)
C21	0.26240 (16)	0.62580 (12)	0.87360 (11)	0.0216 (4)
O22	0.35432 (11)	0.63298 (7)	0.80929 (7)	0.0215 (3)
O23	0.22106 (12)	0.56013 (8)	0.89208 (8)	0.0298 (3)
O24	0.24272 (10)	1.05356 (7)	1.00196 (7)	0.0201 (3)
C25	0.30152 (16)	1.11360 (11)	1.02153 (10)	0.0197 (4)
O26	0.39773 (11)	1.09511 (8)	1.04546 (8)	0.0283 (3)
C27	0.23598 (16)	1.20589 (11)	1.01040 (10)	0.0194 (4)
C28	0.29728 (17)	1.27379 (12)	1.01677 (11)	0.0231 (4)
H28	0.3782	1.2602	1.0278	0.028*
C29	0.24120 (18)	1.36072 (12)	1.00719 (11)	0.0272 (4)
H29	0.2837	1.4068	1.0113	0.033*
C30	0.12343 (18)	1.38076 (12)	0.99168 (11)	0.0275 (4)
H30	0.0847	1.4407	0.9855	0.033*
C31	0.06173 (17)	1.31401 (12)	0.98513 (12)	0.0276 (4)
H31	−0.0195	1.3281	0.9748	0.033*
C32	0.11788 (16)	1.22643 (12)	0.99364 (11)	0.0226 (4)

H32	0.0758	1.1806	0.988	0.027*
O33	0.32219 (12)	1.00634 (8)	0.70805 (8)	0.0276 (3)
H33	0.2664	1.049	0.7246	0.041*
O34	0.38701 (11)	0.68492 (7)	1.01813 (7)	0.0218 (3)
C35	0.33439 (17)	0.61646 (12)	1.06299 (11)	0.0248 (4)
H35A	0.2477	0.625	1.0568	0.03*
H35B	0.3774	0.5597	1.0387	0.03*
O36	0.33867 (12)	0.61094 (8)	1.14683 (7)	0.0288 (3)
C37	0.4588 (2)	0.58010 (15)	1.16272 (15)	0.0452 (6)
H37B	0.5132	0.6193	1.1335	0.068*
H37C	0.489	0.5201	1.1432	0.068*
H37A	0.457	0.5795	1.2224	0.068*
C1′	0.80525 (16)	0.74195 (11)	0.31038 (11)	0.0190 (4)
C2′	0.90913 (15)	0.67289 (11)	0.26359 (10)	0.0185 (4)
H2′	0.8711	0.6423	0.2274	0.022*
C3′	0.98195 (15)	0.60049 (11)	0.31378 (10)	0.0182 (4)
H3′	0.9397	0.6052	0.3731	0.022*
C4′	1.11310 (16)	0.61437 (11)	0.30982 (11)	0.0225 (4)
H4′	1.1551	0.6192	0.2506	0.027*
C5′	1.18605 (17)	0.53955 (12)	0.35504 (12)	0.0274 (4)
H5′A	1.1505	0.5397	0.415	0.033*
H5′B	1.2718	0.5483	0.3478	0.033*
C6′	1.18424 (17)	0.45122 (12)	0.32163 (12)	0.0265 (4)
H6′A	1.2218	0.4501	0.262	0.032*
H6′B	1.2321	0.4028	0.3514	0.032*
C7′	1.05332 (16)	0.43877 (11)	0.33368 (11)	0.0216 (4)
H7′	1.0172	0.4405	0.3942	0.026*
C8′	0.97153 (15)	0.50882 (11)	0.28678 (10)	0.0188 (4)
C9′	0.83902 (16)	0.49154 (11)	0.31629 (11)	0.0205 (4)
H9′A	0.8245	0.4886	0.3776	0.025*
H9′B	0.84	0.4314	0.2995	0.025*
C10′	0.72291 (15)	0.55088 (11)	0.29171 (11)	0.0193 (4)
H10′	0.7401	0.5667	0.2309	0.023*
C11′	0.66133 (16)	0.63631 (11)	0.33978 (10)	0.0189 (4)
H11′	0.5715	0.6377	0.3454	0.023*
C12′	0.68111 (15)	0.62936 (11)	0.42879 (11)	0.0191 (4)
C13′	0.74589 (16)	0.68057 (11)	0.45440 (11)	0.0211 (4)
H13′	0.7548	0.6732	0.5108	0.025*
C14′	0.80599 (16)	0.74896 (12)	0.40147 (11)	0.0220 (4)
H14D	0.8917	0.7418	0.4094	0.026*
H14E	0.7625	0.8084	0.4191	0.026*
C15′	0.67950 (16)	0.72807 (11)	0.29790 (11)	0.0199 (4)
C16′	0.66754 (17)	0.73683 (12)	0.20653 (11)	0.0237 (4)
H16D	0.7362	0.6974	0.1743	0.036*
H16E	0.6686	0.798	0.1857	0.036*
H16F	0.5902	0.7207	0.2012	0.036*
C17′	0.57506 (16)	0.79806 (11)	0.34144 (12)	0.0237 (4)
H17D	0.5714	0.7911	0.4015	0.036*

H17E	0.497	0.7904	0.3288	0.036*	
H17F	0.5903	0.8572	0.3218	0.036*	
C18′	0.61846 (17)	0.56509 (12)	0.48620 (11)	0.0264 (4)	
H18D	0.5294	0.5838	0.4921	0.04*	
H18E	0.6417	0.5631	0.5406	0.04*	
H18F	0.6433	0.5063	0.4635	0.04*	
C19′	1.01302 (17)	0.49893 (12)	0.19299 (11)	0.0237 (4)	
H19D	1.0253	0.4366	0.1806	0.035*	
H19E	1.0902	0.5205	0.1742	0.035*	
H19F	0.9501	0.5335	0.1642	0.035*	
O20′	0.98878 (11)	0.72516 (7)	0.20899 (7)	0.0218 (3)	
C21′	0.93856 (16)	0.81059 (12)	0.21184 (11)	0.0219 (4)	
O22′	0.83303 (11)	0.82463 (7)	0.26720 (7)	0.0230 (3)	
O23′	0.98135 (12)	0.86793 (8)	0.17053 (8)	0.0291 (3)	
O24′	1.05092 (11)	0.35259 (7)	0.30544 (7)	0.0232 (3)	
C25′	1.05285 (16)	0.28473 (12)	0.36162 (11)	0.0230 (4)	
O26′	1.05309 (14)	0.29155 (9)	0.43359 (8)	0.0366 (4)	
C27′	1.05285 (16)	0.20029 (11)	0.32452 (11)	0.0226 (4)	
C28′	1.03407 (19)	0.12820 (13)	0.37777 (13)	0.0328 (5)	
H28′	1.0236	0.1332	0.4359	0.039*	
C29′	1.0305 (2)	0.04866 (13)	0.34638 (14)	0.0403 (5)	
H29′	1.0167	−0.0006	0.3831	0.048*	
C30′	1.04693 (18)	0.04084 (13)	0.26221 (13)	0.0343 (5)	
H30′	1.044	−0.0137	0.2409	0.041*	
C31′	1.06757 (16)	0.11189 (12)	0.20896 (12)	0.0270 (4)	
H31′	1.0804	0.1059	0.1508	0.032*	
C32′	1.06975 (15)	0.19203 (12)	0.23947 (11)	0.0231 (4)	
H32′	1.0828	0.2412	0.2024	0.028*	
O33′	0.62847 (11)	0.49828 (8)	0.30885 (8)	0.0254 (3)	
H33′	0.6435	0.4595	0.2736	0.038*	
O34′	1.09963 (11)	0.69699 (8)	0.34778 (8)	0.0255 (3)	
C35′	1.1978 (2)	0.74151 (14)	0.32338 (13)	0.0370 (5)	
H35D	1.2264	0.7375	0.2628	0.044*	
H35F	1.1685	0.8047	0.335	0.044*	
O36′	1.29654 (12)	0.70912 (10)	0.36255 (10)	0.0446 (4)	
C37′	1.2687 (2)	0.72296 (17)	0.44817 (15)	0.0529 (7)	
H37D	1.3415	0.6998	0.4725	0.079*	
H37E	1.2021	0.6923	0.4747	0.079*	
H37F	1.2432	0.7864	0.4569	0.079*	
C1P	0.2437 (5)	0.9991 (4)	0.5096 (4)	0.0466 (14)	0.5
H1PA	0.2192	1.0095	0.569	0.07*	0.5
H1PB	0.1943	0.9599	0.4945	0.07*	0.5
H1PC	0.2307	1.0555	0.4777	0.07*	0.5
C2P	0.3778 (6)	0.9566 (6)	0.4905 (6)	0.0345 (17)	0.5
H2PA	0.4002	0.942	0.4314	0.041*	0.5
H2PB	0.3906	0.9007	0.5242	0.041*	0.5
C3P	0.4624 (5)	1.0156 (5)	0.5077 (5)	0.036 (3)	0.5
H3PA	0.4536	1.0696	0.471	0.043*	0.5

H3PB	0.4357	1.0338	0.5656	0.043*	0.5
C4P	0.5962 (7)	0.9719 (8)	0.4947 (7)	0.044 (2)	0.5
H4PA	0.6228	0.9528	0.437	0.053*	0.5
H4PB	0.6055	0.9184	0.5322	0.053*	0.5
C5P	0.6788 (7)	1.0321 (6)	0.5108 (6)	0.047 (2)	0.5
H5PA	0.6741	1.0834	0.4715	0.07*	0.5
H5PB	0.7636	0.9998	0.5038	0.07*	0.5
H5PC	0.6521	1.0522	0.5675	0.07*	0.5

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0196 (9)	0.0124 (9)	0.0231 (9)	−0.0029 (7)	−0.0042 (7)	−0.0035 (7)
C2	0.0196 (9)	0.0145 (9)	0.0193 (9)	−0.0040 (7)	−0.0026 (7)	−0.0005 (7)
C3	0.0138 (9)	0.0148 (9)	0.0206 (9)	−0.0020 (7)	−0.0009 (7)	−0.0039 (7)
C4	0.0203 (10)	0.0151 (9)	0.0226 (10)	−0.0024 (7)	−0.0008 (8)	−0.0021 (7)
C5	0.0287 (11)	0.0216 (10)	0.0192 (9)	−0.0069 (8)	−0.0045 (8)	−0.0011 (8)
C6	0.0247 (10)	0.0228 (10)	0.0223 (10)	−0.0053 (8)	−0.0021 (8)	−0.0077 (8)
C7	0.0174 (9)	0.0146 (9)	0.0248 (10)	−0.0008 (7)	−0.0030 (8)	−0.0042 (7)
C8	0.0149 (9)	0.0166 (9)	0.0186 (9)	−0.0031 (7)	−0.0010 (7)	−0.0029 (7)
C9	0.0190 (9)	0.0154 (9)	0.0241 (10)	−0.0014 (7)	−0.0038 (8)	−0.0037 (7)
C10	0.0195 (9)	0.0159 (9)	0.0219 (9)	−0.0012 (7)	−0.0034 (7)	0.0019 (7)
C11	0.0189 (9)	0.0188 (9)	0.0163 (9)	−0.0027 (7)	−0.0013 (7)	−0.0010 (7)
C12	0.0188 (9)	0.0220 (10)	0.0183 (9)	−0.0031 (7)	0.0001 (7)	−0.0049 (8)
C13	0.0153 (9)	0.0255 (10)	0.0219 (9)	−0.0019 (7)	−0.0030 (7)	−0.0048 (8)
C14	0.0200 (10)	0.0172 (9)	0.0211 (9)	0.0022 (7)	−0.0035 (8)	−0.0035 (7)
C15	0.0185 (9)	0.0194 (9)	0.0188 (9)	−0.0027 (7)	−0.0025 (7)	−0.0033 (7)
C16	0.0255 (10)	0.0225 (10)	0.0209 (9)	−0.0042 (8)	−0.0070 (8)	−0.0026 (8)
C17	0.0263 (10)	0.0220 (10)	0.0220 (10)	−0.0020 (8)	−0.0038 (8)	−0.0044 (8)
C18	0.0232 (10)	0.0267 (11)	0.0327 (11)	−0.0050 (8)	−0.0034 (8)	−0.0006 (9)
C19	0.0178 (9)	0.0188 (9)	0.0269 (10)	−0.0013 (7)	−0.0025 (8)	−0.0071 (8)
O20	0.0205 (7)	0.0192 (7)	0.0249 (7)	−0.0077 (5)	0.0003 (5)	−0.0052 (5)
C21	0.0230 (10)	0.0206 (10)	0.0225 (10)	−0.0041 (8)	−0.0071 (8)	−0.0023 (8)
O22	0.0245 (7)	0.0153 (6)	0.0241 (7)	−0.0046 (5)	−0.0015 (6)	−0.0038 (5)
O23	0.0360 (8)	0.0211 (7)	0.0353 (8)	−0.0141 (6)	−0.0050 (6)	−0.0013 (6)
O24	0.0193 (6)	0.0147 (6)	0.0271 (7)	−0.0027 (5)	−0.0042 (5)	−0.0065 (5)
C25	0.0187 (10)	0.0205 (10)	0.0204 (9)	−0.0044 (8)	−0.0021 (8)	−0.0051 (7)
O26	0.0252 (8)	0.0221 (7)	0.0403 (8)	−0.0016 (6)	−0.0127 (6)	−0.0069 (6)
C27	0.0233 (10)	0.0180 (9)	0.0158 (9)	−0.0026 (7)	−0.0012 (7)	−0.0033 (7)
C28	0.0240 (10)	0.0229 (10)	0.0236 (10)	−0.0040 (8)	−0.0059 (8)	−0.0054 (8)
C29	0.0372 (12)	0.0194 (10)	0.0271 (10)	−0.0077 (8)	−0.0070 (9)	−0.0044 (8)
C30	0.0347 (12)	0.0173 (10)	0.0254 (10)	0.0029 (8)	−0.0011 (9)	−0.0009 (8)
C31	0.0184 (10)	0.0287 (11)	0.0314 (11)	0.0018 (8)	−0.0019 (8)	0.0013 (9)
C32	0.0212 (10)	0.0233 (10)	0.0233 (10)	−0.0071 (8)	−0.0011 (8)	−0.0018 (8)
O33	0.0342 (8)	0.0182 (7)	0.0259 (7)	0.0014 (6)	−0.0026 (6)	0.0019 (6)
O34	0.0255 (7)	0.0151 (6)	0.0237 (7)	−0.0036 (5)	−0.0030 (5)	0.0006 (5)
C35	0.0298 (11)	0.0187 (10)	0.0274 (10)	−0.0073 (8)	−0.0076 (8)	0.0029 (8)
O36	0.0321 (8)	0.0298 (7)	0.0229 (7)	−0.0051 (6)	−0.0048 (6)	0.0051 (6)

C37	0.0452 (14)	0.0441 (14)	0.0534 (15)	−0.0124 (11)	−0.0287 (12)	0.0146 (11)
C1′	0.0211 (10)	0.0112 (9)	0.0243 (10)	−0.0038 (7)	−0.0033 (8)	0.0000 (7)
C2′	0.0175 (9)	0.0180 (9)	0.0209 (9)	−0.0068 (7)	−0.0034 (7)	0.0008 (7)
C3′	0.0185 (9)	0.0171 (9)	0.0181 (9)	−0.0020 (7)	−0.0031 (7)	−0.0003 (7)
C4′	0.0204 (10)	0.0221 (10)	0.0261 (10)	−0.0044 (8)	−0.0056 (8)	−0.0031 (8)
C5′	0.0193 (10)	0.0299 (11)	0.0341 (11)	−0.0042 (8)	−0.0097 (8)	0.0017 (9)
C6′	0.0237 (10)	0.0219 (10)	0.0318 (11)	0.0030 (8)	−0.0085 (8)	0.0011 (8)
C7′	0.0242 (10)	0.0176 (9)	0.0224 (10)	−0.0009 (7)	−0.0048 (8)	−0.0031 (7)
C8′	0.0189 (9)	0.0175 (9)	0.0199 (9)	−0.0019 (7)	−0.0043 (7)	−0.0022 (7)
C9′	0.0240 (10)	0.0144 (9)	0.0238 (10)	−0.0033 (7)	−0.0053 (8)	−0.0034 (7)
C10′	0.0207 (10)	0.0168 (9)	0.0221 (9)	−0.0063 (7)	−0.0045 (8)	−0.0030 (7)
C11′	0.0170 (9)	0.0172 (9)	0.0231 (9)	−0.0039 (7)	−0.0032 (7)	−0.0038 (7)
C12′	0.0179 (9)	0.0163 (9)	0.0214 (9)	−0.0003 (7)	−0.0010 (7)	−0.0039 (7)
C13′	0.0213 (10)	0.0232 (10)	0.0181 (9)	−0.0021 (8)	−0.0031 (8)	−0.0043 (8)
C14′	0.0216 (10)	0.0195 (10)	0.0260 (10)	−0.0042 (7)	−0.0041 (8)	−0.0067 (8)
C15′	0.0202 (10)	0.0153 (9)	0.0245 (10)	−0.0023 (7)	−0.0047 (8)	−0.0042 (7)
C16′	0.0235 (10)	0.0203 (10)	0.0271 (10)	−0.0014 (8)	−0.0074 (8)	0.0002 (8)
C17′	0.0211 (10)	0.0181 (10)	0.0319 (11)	−0.0022 (7)	−0.0052 (8)	−0.0039 (8)
C18′	0.0265 (11)	0.0252 (10)	0.0263 (10)	−0.0050 (8)	−0.0014 (8)	−0.0033 (8)
C19′	0.0258 (10)	0.0194 (10)	0.0243 (10)	−0.0001 (8)	−0.0046 (8)	−0.0029 (8)
O20′	0.0205 (7)	0.0184 (7)	0.0248 (7)	−0.0039 (5)	−0.0021 (5)	0.0033 (5)
C21′	0.0204 (10)	0.0216 (10)	0.0260 (10)	−0.0064 (8)	−0.0079 (8)	0.0007 (8)
O22′	0.0226 (7)	0.0148 (6)	0.0303 (7)	−0.0043 (5)	−0.0021 (6)	0.0009 (5)
O23′	0.0298 (8)	0.0242 (7)	0.0345 (8)	−0.0115 (6)	−0.0059 (6)	0.0067 (6)
O24′	0.0294 (7)	0.0154 (6)	0.0235 (7)	0.0007 (5)	−0.0069 (5)	−0.0016 (5)
C25′	0.0204 (10)	0.0235 (10)	0.0255 (11)	−0.0023 (8)	−0.0077 (8)	0.0011 (8)
O26′	0.0626 (10)	0.0264 (8)	0.0268 (8)	−0.0135 (7)	−0.0178 (7)	0.0013 (6)
C27′	0.0184 (10)	0.0195 (10)	0.0301 (11)	−0.0006 (7)	−0.0075 (8)	−0.0021 (8)
C28′	0.0420 (13)	0.0270 (11)	0.0291 (11)	−0.0067 (9)	−0.0059 (9)	−0.0012 (9)
C29′	0.0523 (14)	0.0217 (11)	0.0457 (14)	−0.0112 (10)	−0.0027 (11)	0.0000 (10)
C30′	0.0337 (12)	0.0260 (11)	0.0440 (13)	−0.0073 (9)	−0.0034 (10)	−0.0121 (10)
C31′	0.0191 (10)	0.0304 (11)	0.0315 (11)	−0.0022 (8)	−0.0030 (8)	−0.0115 (9)
C32′	0.0141 (9)	0.0242 (10)	0.0305 (11)	−0.0008 (7)	−0.0048 (8)	−0.0029 (8)
O33′	0.0259 (7)	0.0194 (7)	0.0340 (8)	−0.0078 (5)	−0.0057 (6)	−0.0095 (6)
O34′	0.0230 (7)	0.0230 (7)	0.0334 (7)	−0.0082 (5)	−0.0084 (6)	−0.0017 (6)
C35′	0.0419 (13)	0.0394 (13)	0.0365 (12)	−0.0245 (10)	−0.0065 (10)	−0.0009 (10)
O36′	0.0221 (8)	0.0521 (10)	0.0644 (11)	−0.0103 (7)	−0.0081 (7)	−0.0220 (8)
C37′	0.0500 (15)	0.0592 (16)	0.0602 (17)	−0.0112 (12)	−0.0312 (13)	−0.0112 (13)
C1P	0.040 (3)	0.054 (4)	0.045 (3)	−0.006 (3)	−0.007 (3)	−0.007 (3)
C2P	0.047 (5)	0.029 (3)	0.026 (3)	−0.002 (4)	−0.006 (4)	−0.005 (2)
C3P	0.057 (9)	0.022 (5)	0.021 (3)	0.000 (6)	0.008 (6)	−0.008 (3)
C4P	0.054 (7)	0.042 (4)	0.031 (3)	−0.003 (5)	0.002 (4)	−0.004 (3)
C5P	0.047 (6)	0.051 (5)	0.040 (3)	−0.011 (5)	−0.003 (5)	0.008 (3)

Geometric parameters (Å, º)

C1—O22	1.466 (2)	C3′—C8′	1.555 (2)
C1—C14	1.519 (2)	C3′—H3′	1.0

C1—C15	1.536 (2)	C4′—O34′	1.438 (2)
C1—C2	1.551 (2)	C4′—C5′	1.516 (2)
C2—O20	1.455 (2)	C4′—H4′	1.0
C2—C3	1.538 (2)	C5′—C6′	1.521 (3)
C2—H2	1.0	C5′—H5′A	0.99
C3—C4	1.531 (2)	C5′—H5′B	0.99
C3—C8	1.560 (2)	C6′—C7′	1.506 (3)
C3—H3	1.0	C6′—H6′A	0.99
C4—O34	1.445 (2)	C6′—H6′B	0.99
C4—C5	1.517 (2)	C7′—O24′	1.456 (2)
C4—H4	1.0	C7′—C8′	1.542 (2)
C5—C6	1.527 (2)	C7′—H7′	1.0
C5—H5A	0.99	C8′—C19′	1.534 (2)
C5—H5B	0.99	C8′—C9′	1.552 (2)
C6—C7	1.509 (2)	C9′—C10′	1.552 (2)
C6—H6A	0.99	C9′—H9′A	0.99
C6—H6B	0.99	C9′—H9′B	0.99
C7—O24	1.470 (2)	C10′—O33′	1.440 (2)
C7—C8	1.544 (2)	C10′—C11′	1.565 (2)
C7—H7	1.0	C10′—H10′	1.0
C8—C19	1.527 (2)	C11′—C12′	1.520 (2)
C8—C9	1.550 (2)	C11′—C15′	1.557 (2)
C9—C10	1.544 (2)	C11′—H11′	1.0
C9—H9B	0.99	C12′—C13′	1.326 (2)
C9—H9A	0.99	C12′—C18′	1.496 (2)
C10—O33	1.429 (2)	C13′—C14′	1.495 (2)
C10—C11	1.567 (2)	C13′—H13′	0.95
C10—H10	1.0	C14′—H14D	0.99
C11—C12	1.520 (2)	C14′—H14E	0.99
C11—C15	1.559 (2)	C15′—C16′	1.532 (2)
C11—H11	1.0	C15′—C17′	1.539 (2)
C12—C13	1.327 (2)	C16′—H16D	0.98
C12—C18	1.502 (2)	C16′—H16E	0.98
C13—C14	1.496 (2)	C16′—H16F	0.98
C13—H13	0.95	C17′—H17D	0.98
C14—H14A	0.99	C17′—H17E	0.98
C14—H14B	0.99	C17′—H17F	0.98
C15—C16	1.535 (2)	C18′—H18D	0.98
C15—C17	1.538 (2)	C18′—H18E	0.98
C16—H16A	0.98	C18′—H18F	0.98
C16—H16B	0.98	C19′—H19D	0.98
C16—H16C	0.98	C19′—H19E	0.98
C17—H17B	0.98	C19′—H19F	0.98
C17—H17C	0.98	O20′—C21′	1.331 (2)
C17—H17A	0.98	C21′—O23′	1.192 (2)
C18—H18A	0.98	C21′—O22′	1.342 (2)
C18—H18B	0.98	O24′—C25′	1.339 (2)
C18—H18C	0.98	C25′—O26′	1.201 (2)

C19—H19C	0.98	C25′—C27′	1.489 (3)
C19—H19A	0.98	C27′—C28′	1.383 (3)
C19—H19B	0.98	C27′—C32′	1.390 (3)
O20—C21	1.330 (2)	C28′—C29′	1.385 (3)
C21—O23	1.193 (2)	C28′—H28′	0.95
C21—O22	1.343 (2)	C29′—C30′	1.375 (3)
O24—C25	1.340 (2)	C29′—H29′	0.95
C25—O26	1.207 (2)	C30′—C31′	1.374 (3)
C25—C27	1.487 (2)	C30′—H30′	0.95
C27—C32	1.391 (2)	C31′—C32′	1.383 (3)
C27—C28	1.391 (2)	C31′—H31′	0.95
C28—C29	1.379 (3)	C32′—H32′	0.95
C28—H28	0.95	O33′—H33′	0.84
C29—C30	1.379 (3)	O34′—C35′	1.397 (2)
C29—H29	0.95	C35′—O36′	1.390 (3)
C30—C31	1.380 (3)	C35′—H35D	0.99
C30—H30	0.95	C35′—H35F	0.99
C31—C32	1.386 (3)	O36′—C37′	1.408 (3)
C31—H31	0.95	C37′—H37D	0.98
C32—H32	0.95	C37′—H37E	0.98
O33—H33	0.84	C37′—H37F	0.98
O34—C35	1.401 (2)	C1P—C2P	1.512 (7)
C35—O36	1.388 (2)	C1P—H1PA	0.98
C35—H35A	0.99	C1P—H1PB	0.98
C35—H35B	0.99	C1P—H1PC	0.98
O36—C37	1.419 (2)	C2P—C3P	1.527 (12)
C37—H37B	0.98	C2P—H2PA	0.99
C37—H37C	0.98	C2P—H2PB	0.99
C37—H37A	0.98	C3P—C4P	1.513 (9)
C1′—O22′	1.468 (2)	C3P—H3PA	0.99
C1′—C14′	1.517 (2)	C3P—H3PB	0.99
C1′—C15′	1.539 (2)	C4P—C5P	1.519 (13)
C1′—C2′	1.548 (2)	C4P—H4PA	0.99
C2′—O20′	1.455 (2)	C4P—H4PB	0.99
C2′—C3′	1.539 (2)	C5P—H5PA	0.98
C2′—H2′	1.0	C5P—H5PB	0.98
C3′—C4′	1.531 (2)	C5P—H5PC	0.98

O22—C1—C14	106.92 (13)	C4′—C3′—H3′	106.8
O22—C1—C15	109.83 (13)	C2′—C3′—H3′	106.8
C14—C1—C15	110.95 (14)	C8′—C3′—H3′	106.8
O22—C1—C2	101.66 (13)	O34′—C4′—C5′	111.30 (14)
C14—C1—C2	115.83 (14)	O34′—C4′—C3′	105.04 (13)
C15—C1—C2	111.06 (13)	C5′—C4′—C3′	111.67 (15)
O20—C2—C3	111.66 (13)	O34′—C4′—H4′	109.6
O20—C2—C1	104.22 (12)	C5′—C4′—H4′	109.6
C3—C2—C1	119.74 (14)	C3′—C4′—H4′	109.6
O20—C2—H2	106.8	C4′—C5′—C6′	110.44 (15)

C3—C2—H2	106.8	C4′—C5′—H5′A	109.6
C1—C2—H2	106.8	C6′—C5′—H5′A	109.6
C4—C3—C2	112.38 (14)	C4′—C5′—H5′B	109.6
C4—C3—C8	114.30 (13)	C6′—C5′—H5′B	109.6
C2—C3—C8	109.19 (13)	H5′A—C5′—H5′B	108.1
C4—C3—H3	106.8	C7′—C6′—C5′	108.79 (15)
C2—C3—H3	106.8	C7′—C6′—H6′A	109.9
C8—C3—H3	106.8	C5′—C6′—H6′A	109.9
O34—C4—C5	110.55 (14)	C7′—C6′—H6′B	109.9
O34—C4—C3	105.85 (13)	C5′—C6′—H6′B	109.9
C5—C4—C3	111.33 (14)	H6′A—C6′—H6′B	108.3
O34—C4—H4	109.7	O24′—C7′—C6′	109.24 (14)
C5—C4—H4	109.7	O24′—C7′—C8′	107.71 (13)
C3—C4—H4	109.7	C6′—C7′—C8′	114.27 (15)
C4—C5—C6	110.58 (14)	O24′—C7′—H7′	108.5
C4—C5—H5A	109.5	C6′—C7′—H7′	108.5
C6—C5—H5A	109.5	C8′—C7′—H7′	108.5
C4—C5—H5B	109.5	C19′—C8′—C7′	109.93 (14)
C6—C5—H5B	109.5	C19′—C8′—C9′	111.03 (14)
H5A—C5—H5B	108.1	C7′—C8′—C9′	105.96 (14)
C7—C6—C5	108.84 (14)	C19′—C8′—C3′	112.37 (14)
C7—C6—H6A	109.9	C7′—C8′—C3′	106.79 (13)
C5—C6—H6A	109.9	C9′—C8′—C3′	110.48 (14)
C7—C6—H6B	109.9	C8′—C9′—C10′	124.69 (14)
C5—C6—H6B	109.9	C8′—C9′—H9′A	106.2
H6A—C6—H6B	108.3	C10′—C9′—H9′A	106.2
O24—C7—C6	110.10 (13)	C8′—C9′—H9′B	106.2
O24—C7—C8	106.75 (13)	C10′—C9′—H9′B	106.2
C6—C7—C8	114.54 (14)	H9′A—C9′—H9′B	106.3
O24—C7—H7	108.4	O33′—C10′—C9′	106.31 (13)
C6—C7—H7	108.4	O33′—C10′—C11′	102.98 (13)
C8—C7—H7	108.4	C9′—C10′—C11′	119.59 (14)
C19—C8—C7	109.82 (14)	O33′—C10′—H10′	109.1
C19—C8—C9	111.08 (14)	C9′—C10′—H10′	109.1
C7—C8—C9	105.73 (13)	C11′—C10′—H10′	109.1
C19—C8—C3	112.58 (14)	C12′—C11′—C15′	110.97 (14)
C7—C8—C3	106.92 (13)	C12′—C11′—C10′	112.90 (14)
C9—C8—C3	110.40 (13)	C15′—C11′—C10′	119.20 (14)
C10—C9—C8	122.76 (14)	C12′—C11′—H11′	104.0
C10—C9—H9B	106.6	C15′—C11′—H11′	104.0
C8—C9—H9B	106.6	C10′—C11′—H11′	104.0
C10—C9—H9A	106.6	C13′—C12′—C18′	122.25 (16)
C8—C9—H9A	106.6	C13′—C12′—C11′	121.39 (16)
H9B—C9—H9A	106.6	C18′—C12′—C11′	116.29 (15)
O33—C10—C9	108.12 (14)	C12′—C13′—C14′	125.10 (16)
O33—C10—C11	103.00 (13)	C12′—C13′—H13′	117.4
C9—C10—C11	118.56 (14)	C14′—C13′—H13′	117.4
O33—C10—H10	108.9	C13′—C14′—C1′	111.79 (14)

C9—C10—H10	108.9	C13′—C14′—H14D	109.3
C11—C10—H10	108.9	C1′—C14′—H14D	109.3
C12—C11—C15	110.87 (14)	C13′—C14′—H14E	109.3
C12—C11—C10	113.25 (14)	C1′—C14′—H14E	109.3
C15—C11—C10	118.18 (14)	H14D—C14′—H14E	107.9
C12—C11—H11	104.3	C16′—C15′—C1′	112.57 (14)
C15—C11—H11	104.3	C16′—C15′—C17′	105.41 (14)
C10—C11—H11	104.3	C1′—C15′—C17′	111.34 (14)
C13—C12—C18	121.45 (16)	C16′—C15′—C11′	113.16 (14)
C13—C12—C11	121.67 (16)	C1′—C15′—C11′	106.73 (13)
C18—C12—C11	116.82 (15)	C17′—C15′—C11′	107.59 (14)
C12—C13—C14	124.68 (16)	C15′—C16′—H16D	109.5
C12—C13—H13	117.7	C15′—C16′—H16E	109.5
C14—C13—H13	117.7	H16D—C16′—H16E	109.5
C13—C14—C1	111.73 (14)	C15′—C16′—H16F	109.5
C13—C14—H14A	109.3	H16D—C16′—H16F	109.5
C1—C14—H14A	109.3	H16E—C16′—H16F	109.5
C13—C14—H14B	109.3	C15′—C17′—H17D	109.5
C1—C14—H14B	109.3	C15′—C17′—H17E	109.5
H14A—C14—H14B	107.9	H17D—C17′—H17E	109.5
C16—C15—C1	112.74 (14)	C15′—C17′—H17F	109.5
C16—C15—C17	105.90 (14)	H17D—C17′—H17F	109.5
C1—C15—C17	110.91 (14)	H17E—C17′—H17F	109.5
C16—C15—C11	112.53 (14)	C12′—C18′—H18D	109.5
C1—C15—C11	106.34 (13)	C12′—C18′—H18E	109.5
C17—C15—C11	108.40 (14)	H18D—C18′—H18E	109.5
C15—C16—H16A	109.5	C12′—C18′—H18F	109.5
C15—C16—H16B	109.5	H18D—C18′—H18F	109.5
H16A—C16—H16B	109.5	H18E—C18′—H18F	109.5
C15—C16—H16C	109.5	C8′—C19′—H19D	109.5
H16A—C16—H16C	109.5	C8′—C19′—H19E	109.5
H16B—C16—H16C	109.5	H19D—C19′—H19E	109.5
C15—C17—H17B	109.5	C8′—C19′—H19F	109.5
C15—C17—H17C	109.5	H19D—C19′—H19F	109.5
H17B—C17—H17C	109.5	H19E—C19′—H19F	109.5
C15—C17—H17A	109.5	C21′—O20′—C2′	110.28 (13)
H17B—C17—H17A	109.5	O23′—C21′—O20′	124.57 (17)
H17C—C17—H17A	109.5	O23′—C21′—O22′	123.78 (16)
C12—C18—H18A	109.5	O20′—C21′—O22′	111.64 (15)
C12—C18—H18B	109.5	C21′—O22′—C1′	111.54 (13)
H18A—C18—H18B	109.5	C25′—O24′—C7′	117.38 (13)
C12—C18—H18C	109.5	O26′—C25′—O24′	123.87 (17)
H18A—C18—H18C	109.5	O26′—C25′—C27′	124.10 (17)
H18B—C18—H18C	109.5	O24′—C25′—C27′	112.03 (15)
C8—C19—H19C	109.5	C28′—C27′—C32′	119.59 (17)
C8—C19—H19A	109.5	C28′—C27′—C25′	117.84 (17)
H19C—C19—H19A	109.5	C32′—C27′—C25′	122.57 (16)
C8—C19—H19B	109.5	C27′—C28′—C29′	120.05 (19)

H19C—C19—H19B	109.5	C27′—C28′—H28′	120.0
H19A—C19—H19B	109.5	C29′—C28′—H28′	120.0
C21—O20—C2	110.60 (13)	C30′—C29′—C28′	120.18 (19)
O23—C21—O20	124.81 (17)	C30′—C29′—H29′	119.9
O23—C21—O22	123.59 (16)	C28′—C29′—H29′	119.9
O20—C21—O22	111.60 (15)	C31′—C30′—C29′	120.00 (19)
C21—O22—C1	111.40 (13)	C31′—C30′—H30′	120.0
C25—O24—C7	116.45 (13)	C29′—C30′—H30′	120.0
O26—C25—O24	123.80 (16)	C30′—C31′—C32′	120.44 (18)
O26—C25—C27	123.31 (16)	C30′—C31′—H31′	119.8
O24—C25—C27	112.90 (15)	C32′—C31′—H31′	119.8
C32—C27—C28	119.41 (16)	C31′—C32′—C27′	119.73 (17)
C32—C27—C25	122.92 (16)	C31′—C32′—H32′	120.1
C28—C27—C25	117.67 (16)	C27′—C32′—H32′	120.1
C29—C28—C27	120.35 (17)	C10′—O33′—H33′	109.5
C29—C28—H28	119.8	C35′—O34′—C4′	115.53 (14)
C27—C28—H28	119.8	O36′—C35′—O34′	114.07 (17)
C28—C29—C30	120.01 (18)	O36′—C35′—H35D	108.7
C28—C29—H29	120.0	O34′—C35′—H35D	108.7
C30—C29—H29	120.0	O36′—C35′—H35F	108.7
C29—C30—C31	120.18 (17)	O34′—C35′—H35F	108.7
C29—C30—H30	119.9	H35D—C35′—H35F	107.6
C31—C30—H30	119.9	C35′—O36′—C37′	112.82 (17)
C30—C31—C32	120.23 (17)	O36′—C37′—H37D	109.5
C30—C31—H31	119.9	O36′—C37′—H37E	109.5
C32—C31—H31	119.9	H37D—C37′—H37E	109.5
C31—C32—C27	119.80 (17)	O36′—C37′—H37F	109.5
C31—C32—H32	120.1	H37D—C37′—H37F	109.5
C27—C32—H32	120.1	H37E—C37′—H37F	109.5
C10—O33—H33	109.5	C2P—C1P—H1PA	109.5
C35—O34—C4	115.66 (13)	C2P—C1P—H1PB	109.5
O36—C35—O34	114.13 (14)	H1PA—C1P—H1PB	109.5
O36—C35—H35A	108.7	C2P—C1P—H1PC	109.5
O34—C35—H35A	108.7	H1PA—C1P—H1PC	109.5
O36—C35—H35B	108.7	H1PB—C1P—H1PC	109.5
O34—C35—H35B	108.7	C1P—C2P—C3P	113.0 (6)
H35A—C35—H35B	107.6	C1P—C2P—H2PA	109.0
C35—O36—C37	112.85 (15)	C3P—C2P—H2PA	109.0
O36—C37—H37B	109.5	C1P—C2P—H2PB	109.0
O36—C37—H37C	109.5	C3P—C2P—H2PB	109.0
H37B—C37—H37C	109.5	H2PA—C2P—H2PB	107.8
O36—C37—H37A	109.5	C4P—C3P—C2P	113.9 (5)
H37B—C37—H37A	109.5	C4P—C3P—H3PA	108.8
H37C—C37—H37A	109.5	C2P—C3P—H3PA	108.8
O22′—C1′—C14′	107.45 (13)	C4P—C3P—H3PB	108.8
O22′—C1′—C15′	109.95 (13)	C2P—C3P—H3PB	108.8
C14′—C1′—C15′	111.00 (14)	H3PA—C3P—H3PB	107.7
O22′—C1′—C2′	101.43 (13)	C3P—C4P—C5P	113.0 (6)

C14′—C1′—C2′	116.01 (14)	C3P—C4P—H4PA	109.0
C15′—C1′—C2′	110.45 (13)	C5P—C4P—H4PA	109.0
O20′—C2′—C3′	111.74 (13)	C3P—C4P—H4PB	109.0
O20′—C2′—C1′	104.56 (13)	C5P—C4P—H4PB	109.0
C3′—C2′—C1′	119.08 (14)	H4PA—C4P—H4PB	107.8
O20′—C2′—H2′	106.9	C4P—C5P—H5PA	109.5
C3′—C2′—H2′	106.9	C4P—C5P—H5PB	109.5
C1′—C2′—H2′	106.9	H5PA—C5P—H5PB	109.5
C4′—C3′—C2′	112.31 (14)	C4P—C5P—H5PC	109.5
C4′—C3′—C8′	115.02 (14)	H5PA—C5P—H5PC	109.5
C2′—C3′—C8′	108.62 (13)	H5PB—C5P—H5PC	109.5

O22—C1—C2—O20	6.62 (15)	C14′—C1′—C2′—O20′	−109.03 (15)
C14—C1—C2—O20	−108.85 (15)	C15′—C1′—C2′—O20′	123.57 (14)
C15—C1—C2—O20	123.41 (14)	O22′—C1′—C2′—C3′	132.64 (15)
O22—C1—C2—C3	132.30 (14)	C14′—C1′—C2′—C3′	16.6 (2)
C14—C1—C2—C3	16.8 (2)	C15′—C1′—C2′—C3′	−110.81 (17)
C15—C1—C2—C3	−110.91 (17)	O20′—C2′—C3′—C4′	13.44 (19)
O20—C2—C3—C4	11.23 (19)	C1′—C2′—C3′—C4′	−108.67 (17)
C1—C2—C3—C4	−110.86 (16)	O20′—C2′—C3′—C8′	−114.93 (15)
O20—C2—C3—C8	−116.69 (14)	C1′—C2′—C3′—C8′	122.97 (16)
C1—C2—C3—C8	121.21 (16)	C2′—C3′—C4′—O34′	62.79 (17)
C2—C3—C4—O34	61.23 (17)	C8′—C3′—C4′—O34′	−172.30 (13)
C8—C3—C4—O34	−173.60 (13)	C2′—C3′—C4′—C5′	−176.44 (15)
C2—C3—C4—C5	−178.60 (14)	C8′—C3′—C4′—C5′	−51.5 (2)
C8—C3—C4—C5	−53.42 (19)	O34′—C4′—C5′—C6′	172.16 (14)
O34—C4—C5—C6	173.67 (14)	C3′—C4′—C5′—C6′	55.1 (2)
C3—C4—C5—C6	56.31 (19)	C4′—C5′—C6′—C7′	−59.6 (2)
C4—C5—C6—C7	−59.01 (19)	C5′—C6′—C7′—O24′	−177.36 (14)
C5—C6—C7—O24	−179.24 (13)	C5′—C6′—C7′—C8′	61.9 (2)
C5—C6—C7—C8	60.47 (19)	O24′—C7′—C8′—C19′	−54.39 (18)
O24—C7—C8—C19	−54.43 (17)	C6′—C7′—C8′—C19′	67.17 (19)
C6—C7—C8—C19	67.71 (18)	O24′—C7′—C8′—C9′	65.65 (16)
O24—C7—C8—C9	65.48 (16)	C6′—C7′—C8′—C9′	−172.78 (14)
C6—C7—C8—C9	−172.38 (14)	O24′—C7′—C8′—C3′	−176.55 (13)
O24—C7—C8—C3	−176.86 (13)	C6′—C7′—C8′—C3′	−54.99 (19)
C6—C7—C8—C3	−54.72 (18)	C4′—C3′—C8′—C19′	−71.54 (18)
C4—C3—C8—C19	−70.39 (18)	C2′—C3′—C8′—C19′	55.28 (18)
C2—C3—C8—C19	56.45 (18)	C4′—C3′—C8′—C7′	49.07 (19)
C4—C3—C8—C7	50.29 (18)	C2′—C3′—C8′—C7′	175.89 (14)
C2—C3—C8—C7	177.14 (13)	C4′—C3′—C8′—C9′	163.86 (14)
C4—C3—C8—C9	164.85 (14)	C2′—C3′—C8′—C9′	−69.33 (17)
C2—C3—C8—C9	−68.31 (17)	C19′—C8′—C9′—C10′	−66.3 (2)
C19—C8—C9—C10	−64.8 (2)	C7′—C8′—C9′—C10′	174.41 (15)
C7—C8—C9—C10	176.12 (14)	C3′—C8′—C9′—C10′	59.1 (2)
C3—C8—C9—C10	60.8 (2)	C8′—C9′—C10′—O33′	161.84 (15)
C8—C9—C10—O33	155.68 (15)	C8′—C9′—C10′—C11′	−82.4 (2)
C8—C9—C10—C11	−87.7 (2)	O33′—C10′—C11′—C12′	89.06 (16)

O33—C10—C11—C12	95.01 (16)	C9′—C10′—C11′—C12′	−28.5 (2)
C9—C10—C11—C12	−24.3 (2)	O33′—C10′—C11′—C15′	−138.00 (15)
O33—C10—C11—C15	−132.92 (15)	C9′—C10′—C11′—C15′	104.46 (18)
C9—C10—C11—C15	107.80 (18)	C15′—C11′—C12′—C13′	−21.8 (2)
C15—C11—C12—C13	−22.0 (2)	C10′—C11′—C12′—C13′	115.02 (18)
C10—C11—C12—C13	113.52 (18)	C15′—C11′—C12′—C18′	155.27 (15)
C15—C11—C12—C18	155.30 (15)	C10′—C11′—C12′—C18′	−67.92 (19)
C10—C11—C12—C18	−69.16 (19)	C18′—C12′—C13′—C14′	−177.07 (16)
C18—C12—C13—C14	−177.24 (16)	C11′—C12′—C13′—C14′	−0.2 (3)
C11—C12—C13—C14	0.0 (3)	C12′—C13′—C14′—C1′	−10.8 (2)
C12—C13—C14—C1	−11.1 (2)	O22′—C1′—C14′—C13′	164.26 (14)
O22—C1—C14—C13	164.41 (13)	C15′—C1′—C14′—C13′	44.01 (19)
C15—C1—C14—C13	44.66 (19)	C2′—C1′—C14′—C13′	−83.12 (18)
C2—C1—C14—C13	−83.13 (18)	O22′—C1′—C15′—C16′	51.20 (18)
O22—C1—C15—C16	52.28 (18)	C14′—C1′—C15′—C16′	169.96 (14)
C14—C1—C15—C16	170.27 (14)	C2′—C1′—C15′—C16′	−59.92 (18)
C2—C1—C15—C16	−59.38 (18)	O22′—C1′—C15′—C17′	−66.92 (18)
O22—C1—C15—C17	−66.29 (17)	C14′—C1′—C15′—C17′	51.83 (18)
C14—C1—C15—C17	51.70 (18)	C2′—C1′—C15′—C17′	−178.04 (14)
C2—C1—C15—C17	−177.95 (14)	O22′—C1′—C15′—C11′	175.93 (13)
O22—C1—C15—C11	176.05 (12)	C14′—C1′—C15′—C11′	−65.31 (17)
C14—C1—C15—C11	−65.96 (17)	C2′—C1′—C15′—C11′	64.81 (17)
C2—C1—C15—C11	64.39 (17)	C12′—C11′—C15′—C16′	176.85 (14)
C12—C11—C15—C16	176.78 (14)	C10′—C11′—C15′—C16′	43.1 (2)
C10—C11—C15—C16	43.7 (2)	C12′—C11′—C15′—C1′	52.47 (17)
C12—C11—C15—C1	52.87 (17)	C10′—C11′—C15′—C1′	−81.28 (18)
C10—C11—C15—C1	−80.24 (17)	C12′—C11′—C15′—C17′	−67.12 (17)
C12—C11—C15—C17	−66.44 (17)	C10′—C11′—C15′—C17′	159.12 (14)
C10—C11—C15—C17	160.45 (14)	C3′—C2′—O20′—C21′	−136.82 (14)
C3—C2—O20—C21	−135.34 (14)	C1′—C2′—O20′—C21′	−6.71 (17)
C1—C2—O20—C21	−4.71 (17)	C2′—O20′—C21′—O23′	−176.50 (16)
C2—O20—C21—O23	−179.45 (17)	C2′—O20′—C21′—O22′	3.45 (19)
C2—O20—C21—O22	0.49 (18)	O23′—C21′—O22′—C1′	−178.39 (16)
O23—C21—O22—C1	−175.69 (16)	O20′—C21′—O22′—C1′	1.65 (19)
O20—C21—O22—C1	4.37 (19)	C14′—C1′—O22′—C21′	116.66 (15)
C14—C1—O22—C21	115.03 (15)	C15′—C1′—O22′—C21′	−122.43 (15)
C15—C1—O22—C21	−124.50 (14)	C2′—C1′—O22′—C21′	−5.52 (17)
C2—C1—O22—C21	−6.83 (17)	C6′—C7′—O24′—C25′	91.63 (18)
C6—C7—O24—C25	88.35 (17)	C8′—C7′—O24′—C25′	−143.73 (15)
C8—C7—O24—C25	−146.75 (14)	C7′—O24′—C25′—O26′	2.1 (3)
C7—O24—C25—O26	−5.8 (2)	C7′—O24′—C25′—C27′	−178.74 (14)
C7—O24—C25—C27	173.98 (13)	O26′—C25′—C27′—C28′	9.8 (3)
O26—C25—C27—C32	−171.09 (17)	O24′—C25′—C27′—C28′	−169.38 (16)
O24—C25—C27—C32	9.1 (2)	O26′—C25′—C27′—C32′	−170.79 (18)
O26—C25—C27—C28	9.3 (3)	O24′—C25′—C27′—C32′	10.0 (2)
O24—C25—C27—C28	−170.50 (15)	C32′—C27′—C28′—C29′	−0.9 (3)
C32—C27—C28—C29	0.5 (3)	C25′—C27′—C28′—C29′	178.55 (18)
C25—C27—C28—C29	−179.89 (16)	C27′—C28′—C29′—C30′	0.7 (3)

C27—C28—C29—C30	0.4 (3)	C28′—C29′—C30′—C31′	0.3 (3)
C28—C29—C30—C31	−0.5 (3)	C29′—C30′—C31′—C32′	−1.2 (3)
C29—C30—C31—C32	−0.4 (3)	C30′—C31′—C32′—C27′	1.0 (3)
C30—C31—C32—C27	1.2 (3)	C28′—C27′—C32′—C31′	0.0 (3)
C28—C27—C32—C31	−1.3 (3)	C25′—C27′—C32′—C31′	−179.37 (16)
C25—C27—C32—C31	179.11 (16)	C5′—C4′—O34′—C35′	83.68 (19)
C5—C4—O34—C35	99.01 (17)	C3′—C4′—O34′—C35′	−155.31 (15)
C3—C4—O34—C35	−140.30 (14)	C4′—O34′—C35′—O36′	−79.7 (2)
C4—O34—C35—O36	−79.12 (18)	O34′—C35′—O36′—C37′	−66.5 (2)
O34—C35—O36—C37	−71.1 (2)	C1P—C2P—C3P—C4P	175.9 (6)
O22′—C1′—C2′—O20′	7.02 (15)	C2P—C3P—C4P—C5P	179.1 (10)

Hydrogen-bond geometry (Å, º)

*Cg*1 is the centroid of the C27–C32 benzene ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C14—H14*A*···O34	0.99	2.47	3.320 (2)	143
C14′—H14*D*···O34′	0.99	2.36	3.221 (2)	145
O33—H33···O22′^{i}	0.84	2.05	2.8563 (17)	160
O33′—H33′···O22^{ii}	0.84	2.05	2.8839 (16)	169
C7—H7···O26^{iii}	1.00	2.28	3.236 (2)	159
C4′—H4′···O36^{iv}	1.00	2.40	3.311 (2)	151
C17′—H17*F*···O33^{i}	0.98	2.48	3.431 (2)	164
C30′—H30′···O23′^{v}	0.95	2.53	3.453 (2)	163
C3*P*—H3*PB*···O33	0.99	2.49	3.369 (9)	149
C4*P*—H4*PA*···O33^{i}	0.99	2.41	3.291 (12)	148
C16′—H16*D*···*Cg*1^{i}	0.98	2.85	3.5315 (19)	127

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

(B) (±)-(1*SR*,5*SR*,6*SR*,7*SR*,10*SR*,11*SR*,13*SR*,14*SR*)-13-Hydroxy-7-methoxymethoxy-11,15,18,18-tetramethyl-3-oxo-2,4-dioxatetracyclo[12.3.1.0^{1,5}.0^{6,11}]octadec-15-en-10-yl benzoate

Crystal data

$C_{29}H_{38}O_8$
M_r = 514.59
Monoclinic, $P2_1/n$
a = 9.3612 (6) Å
b = 19.6336 (15) Å
c = 14.1965 (9) Å
β = 101.762 (2)°
V = 2554.4 (3) Å^3
Z = 4
F(000) = 1104
D_x = 1.338 Mg m^{-3}
Melting point: 489.2 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4716 reflections
θ = 2.4–23.9°
μ = 0.10 mm^{-1}
T = 90 K
Prism, colorless
0.23 × 0.23 × 0.14 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, 2014)
T_{min} = 0.98, T_{max} = 0.99
23252 measured reflections

4480 independent reflections
3212 reflections with $I > 2\sigma(I)$
$R_{int} = 0.058$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.5°$

$h = -11 \rightarrow 11$
$k = -23 \rightarrow 21$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.111$
$S = 1.02$
4480 reflections
340 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
$w = 1/[\sigma^2(F_o^2) + (0.054P)^2 + 1.0752P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.51$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

Special details

Experimental. *M*.p. 488.2–489.2 K (not corrected); IR (film) 3483, 2940, 1799, 1717, 1274, 1099, 1042, 773 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$, at 333 K) δ (p.p.m.) 8.02 (d, J = 7.7 Hz, 2H), 7.58 (t, J = 7.5 Hz, 1H), 7.46 (t, J = 7.7 Hz, 2H), 5.50 (s, 1H), 4.92 (d, J = 4.3 Hz, 1H), 4.91 (dd J = 11.7, 4.9 Hz, 1H), 4.80 (d, J = 6.9 Hz, 1H), 4.64 (d, J = 6.9 Hz, 1H), 4.31 (d, J = 10.9 Hz, 1H), 3.68 (ddd, J = 10.9, 10.6, 4.9 Hz, 1H), 3.39 (s, 3H), 3.19 (d, J = 16.9 Hz, 1H), 2.42 (dd, J = 10.9, 4.3 Hz, 1H), 2.33 (s, 1H), 2.33–2.27 (m, 1H), 2.26 (d, J = 16.9 Hz, 1H), 2.02 (dd, J = 16.0, 10.9 Hz, 1H), 1.95–1.87 (m, 1H), 1.88 (s, 3H), 1.82 (dddd, J = 13.6, 13.6, 13.6, 4.6 Hz, 1H), 1.72 (d, J = 16.0 Hz, 1H), 1.49–1.40 (m, 1H), 1.46 (s, 1H), 1.41 (s, 3H), 1.20 (s, 3H), 1.18 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$, at 333 K) δ (p.p.m.) 166.3 (C), 154.4 (C), 136.3 (C), 133.4 (CH), 130.4 (C), 129.8 (CH), 128.7 (CH), 122.3 (CH), 97.7 (CH_2), 88.5 (C), 79.5 (CH), 75.2 (CH), 74.3 (CH), 70.3 (CH), 60.0 (CH), 56.1 (CH_3), 44.8 (CH_2), 44.2 (CH), 42.0 (C), 39.0 (C), 32.0 (CH_2), 30.7 (CH_2), 28.6 (CH_3), 25.6 (CH_2), 22.9 (CH_3), 20.8 (CH_3), 16.1 (CH_3); LRMS (EI) *m/z* 514 (M^+, 3%), 483 (1), 469 (1), 453 (1), 409 (2), 393 (6), 348 (1), 332 (2), 121 (78), 105 (100), 77 (59); HRMS (EI) *m/z* calcd for $C_{29}H_{38}O_8^+$ $[M]^+$ 514.2567, found 514.2560.

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 one reflection with $|I(\text{obs})-I(\text{calc})|/\sigma W(I)$ greater than 10 (-1 0 1) has 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}
C1	0.7169 (2)	0.18654 (10)	0.68618 (13)	0.0143 (4)
C2	0.6544 (2)	0.17259 (10)	0.77738 (14)	0.0136 (4)
H2	0.6782	0.2123	0.8195	0.016*
C3	0.7047 (2)	0.10935 (10)	0.83870 (13)	0.0131 (4)
H3	0.7884	0.0905	0.8158	0.016*
C4	0.5888 (2)	0.05350 (10)	0.82692 (14)	0.0157 (5)
H4	0.5005	0.0711	0.8454	0.019*
C5	0.6453 (2)	−0.00784 (10)	0.88896 (14)	0.0181 (5)
H5A	0.7279	−0.0275	0.8671	0.022*
H5B	0.5695	−0.0422	0.8827	0.022*
C6	0.6915 (2)	0.01330 (11)	0.99391 (14)	0.0192 (5)

H6A	0.6073	0.0301	1.0169	0.023*
H6B	0.7295	−0.026	1.0325	0.023*
C7	0.8069 (2)	0.06816 (10)	1.00523 (13)	0.0155 (4)
H7	0.8952	0.0491	0.9884	0.019*
C8	0.7615 (2)	0.13268 (10)	0.94372 (14)	0.0135 (4)
C9	0.9051 (2)	0.17425 (10)	0.94939 (14)	0.0164 (5)
H9B	0.9393	0.1848	1.017	0.02*
H9A	0.9752	0.1424	0.9326	0.02*
C10	0.9238 (2)	0.24112 (10)	0.89436 (14)	0.0166 (5)
H10	1.0181	0.259	0.9281	0.02*
C11	0.9370 (2)	0.24049 (10)	0.78491 (14)	0.0172 (5)
H11	1.0036	0.2782	0.7801	0.021*
C12	1.0208 (2)	0.17776 (11)	0.76571 (14)	0.0174 (5)
C13	0.9588 (2)	0.12865 (10)	0.70699 (14)	0.0167 (5)
H13	1.0156	0.0911	0.6989	0.02*
C14	0.8037 (2)	0.12964 (10)	0.65293 (14)	0.0154 (4)
H14A	0.7581	0.0864	0.6617	0.018*
H14B	0.8021	0.1351	0.5848	0.018*
C15	0.8037 (2)	0.25338 (10)	0.69973 (14)	0.0154 (5)
C16	0.7084 (2)	0.31455 (10)	0.71360 (14)	0.0191 (5)
H16B	0.6447	0.3255	0.6535	0.029*
H16C	0.7695	0.353	0.7355	0.029*
H16A	0.6512	0.3035	0.7604	0.029*
C17	0.8650 (2)	0.27030 (11)	0.60997 (15)	0.0209 (5)
H17A	0.9377	0.2373	0.6029	0.031*
H17B	0.908	0.3149	0.6168	0.031*
H17C	0.7874	0.2693	0.5541	0.031*
C18	1.1796 (2)	0.17407 (12)	0.81421 (16)	0.0242 (5)
H18B	1.2193	0.1311	0.7998	0.036*
H18C	1.1887	0.1783	0.8825	0.036*
H18A	1.232	0.2104	0.7912	0.036*
C19	0.6461 (2)	0.17224 (11)	0.98432 (14)	0.0176 (5)
H19C	0.6149	0.2111	0.9444	0.026*
H19A	0.6873	0.1871	1.0485	0.026*
H19B	0.5639	0.1432	0.9854	0.026*
O20	0.49732 (15)	0.17278 (7)	0.74303 (9)	0.0176 (3)
C21	0.4646 (2)	0.18489 (10)	0.64927 (15)	0.0178 (5)
O22	0.58327 (14)	0.19463 (7)	0.61213 (9)	0.0170 (3)
O23	0.34190 (16)	0.18696 (8)	0.60281 (10)	0.0247 (4)
O24	0.84116 (15)	0.09012 (7)	1.10583 (9)	0.0168 (3)
C25	0.9381 (2)	0.05274 (10)	1.16731 (14)	0.0154 (4)
O26	1.00007 (16)	0.00371 (7)	1.14311 (10)	0.0237 (4)
C27	0.9612 (2)	0.07873 (10)	1.26731 (14)	0.0153 (4)
C28	0.8754 (2)	0.13006 (11)	1.29508 (14)	0.0194 (5)
H28	0.7979	0.1484	1.2508	0.023*
C29	0.9062 (2)	0.15358 (12)	1.38896 (15)	0.0252 (5)
H29	0.8491	0.1878	1.4077	0.03*
C30	1.0214 (2)	0.12650 (11)	1.45491 (15)	0.0231 (5)

H30	1.0417	0.1428	1.5178	0.028*
C31	1.1063 (2)	0.07552 (11)	1.42812 (15)	0.0227 (5)
H31	1.1832	0.0571	1.4728	0.027*
C32	1.0765 (2)	0.05194 (10)	1.33430 (14)	0.0187 (5)
H32	1.1342	0.0179	1.316	0.022*
O33	0.82270 (15)	0.29278 (7)	0.90665 (10)	0.0200 (3)
H33	0.8271	0.2993	0.9642	0.03*
O34	0.55698 (14)	0.03544 (7)	0.72690 (9)	0.0188 (3)
C35	0.4129 (2)	0.01081 (12)	0.69395 (15)	0.0232 (5)
H35A	0.3957	−0.0281	0.7322	0.028*
H35B	0.343	0.046	0.7007	0.028*
O36	0.39527 (17)	−0.00779 (8)	0.59939 (11)	0.0289 (4)
C37	0.4715 (3)	−0.06965 (12)	0.58715 (17)	0.0315 (6)
H37C	0.5739	−0.0637	0.6126	0.047*
H37A	0.4566	−0.0805	0.5199	0.047*
H37B	0.4349	−0.1061	0.6207	0.047*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0139 (11)	0.0176 (11)	0.0096 (10)	0.0000 (9)	−0.0017 (8)	0.0003 (8)
C2	0.0092 (10)	0.0182 (11)	0.0126 (10)	0.0005 (8)	0.0003 (8)	−0.0006 (8)
C3	0.0116 (11)	0.0156 (10)	0.0126 (11)	0.0014 (8)	0.0039 (8)	−0.0003 (8)
C4	0.0154 (11)	0.0203 (11)	0.0114 (11)	−0.0014 (9)	0.0026 (9)	−0.0038 (8)
C5	0.0187 (11)	0.0147 (11)	0.0216 (12)	−0.0040 (9)	0.0060 (9)	−0.0018 (9)
C6	0.0218 (12)	0.0190 (11)	0.0174 (11)	−0.0008 (9)	0.0052 (9)	0.0041 (9)
C7	0.0192 (11)	0.0184 (11)	0.0091 (10)	0.0018 (9)	0.0030 (9)	−0.0003 (8)
C8	0.0132 (11)	0.0142 (11)	0.0129 (11)	0.0003 (8)	0.0025 (8)	−0.0007 (8)
C9	0.0167 (11)	0.0174 (11)	0.0133 (11)	0.0010 (9)	−0.0009 (9)	−0.0013 (8)
C10	0.0154 (11)	0.0171 (11)	0.0162 (11)	−0.0002 (9)	0.0010 (9)	−0.0025 (8)
C11	0.0153 (11)	0.0185 (11)	0.0179 (11)	−0.0039 (9)	0.0039 (9)	−0.0010 (9)
C12	0.0160 (11)	0.0205 (11)	0.0170 (11)	−0.0006 (9)	0.0067 (9)	−0.0004 (9)
C13	0.0176 (11)	0.0178 (11)	0.0162 (11)	0.0030 (9)	0.0071 (9)	0.0015 (9)
C14	0.0200 (11)	0.0156 (11)	0.0118 (11)	0.0001 (9)	0.0059 (9)	−0.0001 (8)
C15	0.0170 (11)	0.0153 (11)	0.0141 (11)	0.0000 (9)	0.0040 (9)	0.0005 (8)
C16	0.0217 (12)	0.0188 (11)	0.0152 (11)	0.0028 (9)	0.0003 (9)	0.0027 (8)
C17	0.0248 (12)	0.0187 (11)	0.0207 (12)	−0.0029 (9)	0.0079 (10)	0.0009 (9)
C18	0.0184 (12)	0.0282 (13)	0.0263 (13)	0.0004 (10)	0.0052 (10)	−0.0049 (10)
C19	0.0199 (12)	0.0188 (11)	0.0140 (11)	0.0016 (9)	0.0032 (9)	0.0003 (8)
O20	0.0137 (8)	0.0265 (8)	0.0119 (8)	0.0036 (6)	0.0011 (6)	0.0032 (6)
C21	0.0195 (13)	0.0173 (11)	0.0167 (12)	−0.0003 (9)	0.0041 (10)	0.0018 (9)
O22	0.0152 (8)	0.0225 (8)	0.0124 (7)	0.0000 (6)	0.0007 (6)	0.0024 (6)
O23	0.0157 (9)	0.0375 (10)	0.0189 (8)	0.0007 (7)	−0.0014 (7)	0.0077 (7)
O24	0.0197 (8)	0.0197 (8)	0.0101 (7)	0.0043 (6)	0.0011 (6)	0.0004 (6)
C25	0.0159 (11)	0.0149 (11)	0.0154 (11)	−0.0005 (9)	0.0033 (9)	0.0034 (8)
O26	0.0295 (9)	0.0225 (8)	0.0179 (8)	0.0102 (7)	0.0023 (7)	−0.0004 (6)
C27	0.0173 (11)	0.0139 (10)	0.0152 (11)	−0.0024 (9)	0.0042 (9)	0.0024 (8)
C28	0.0175 (12)	0.0242 (12)	0.0158 (12)	0.0022 (9)	0.0021 (9)	0.0018 (9)

C29	0.0241 (13)	0.0317 (13)	0.0203 (13)	0.0071 (10)	0.0060 (10)	−0.0027 (10)
C30	0.0268 (13)	0.0293 (13)	0.0135 (11)	−0.0049 (10)	0.0045 (10)	−0.0027 (9)
C31	0.0227 (12)	0.0235 (12)	0.0195 (12)	−0.0011 (10)	−0.0010 (10)	0.0050 (9)
C32	0.0221 (12)	0.0155 (11)	0.0181 (11)	0.0006 (9)	0.0031 (9)	0.0010 (9)
O33	0.0251 (8)	0.0187 (8)	0.0162 (8)	0.0041 (6)	0.0046 (7)	−0.0028 (6)
O34	0.0166 (8)	0.0248 (8)	0.0140 (8)	−0.0046 (6)	0.0004 (6)	−0.0050 (6)
C35	0.0183 (12)	0.0304 (13)	0.0196 (12)	−0.0042 (10)	0.0007 (9)	−0.0070 (9)
O36	0.0293 (9)	0.0288 (9)	0.0249 (9)	0.0012 (7)	−0.0031 (7)	−0.0049 (7)
C37	0.0328 (14)	0.0255 (13)	0.0355 (14)	−0.0026 (11)	0.0056 (11)	−0.0103 (11)

Geometric parameters (Å, º)

C1—O22	1.469 (2)	C15—C17	1.536 (3)
C1—C14	1.512 (3)	C16—H16B	0.96
C1—C15	1.535 (3)	C16—H16C	0.96
C1—C2	1.550 (3)	C16—H16A	0.96
C2—O20	1.452 (2)	C17—H17A	0.96
C2—C3	1.534 (3)	C17—H17B	0.96
C2—H2	0.98	C17—H17C	0.96
C3—C4	1.528 (3)	C18—H18B	0.96
C3—C8	1.547 (3)	C18—H18C	0.96
C3—H3	0.98	C18—H18A	0.96
C4—O34	1.435 (2)	C19—H19C	0.96
C4—C5	1.522 (3)	C19—H19A	0.96
C4—H4	0.98	C19—H19B	0.96
C5—C6	1.522 (3)	O20—C21	1.325 (2)
C5—H5A	0.97	C21—O23	1.203 (2)
C5—H5B	0.97	C21—O22	1.337 (2)
C6—C7	1.511 (3)	O24—C25	1.342 (2)
C6—H6A	0.97	C25—O26	1.210 (2)
C6—H6B	0.97	C25—C27	1.482 (3)
C7—O24	1.464 (2)	C27—C32	1.389 (3)
C7—C8	1.548 (3)	C27—C28	1.395 (3)
C7—H7	0.98	C28—C29	1.384 (3)
C8—C19	1.535 (3)	C28—H28	0.93
C8—C9	1.561 (3)	C29—C30	1.382 (3)
C9—C10	1.556 (3)	C29—H29	0.93
C9—H9B	0.97	C30—C31	1.379 (3)
C9—H9A	0.97	C30—H30	0.93
C10—O33	1.422 (2)	C31—C32	1.384 (3)
C10—C11	1.584 (3)	C31—H31	0.93
C10—H10	0.98	C32—H32	0.93
C11—C12	1.515 (3)	O33—H33	0.82
C11—C15	1.571 (3)	O34—C35	1.419 (2)
C11—H11	0.98	C35—O36	1.368 (2)
C12—C13	1.328 (3)	C35—H35A	0.97
C12—C18	1.507 (3)	C35—H35B	0.97
C13—C14	1.498 (3)	O36—C37	1.437 (3)

C13—H13	0.93	C37—H37C	0.96
C14—H14A	0.97	C37—H37A	0.96
C14—H14B	0.97	C37—H37B	0.96
C15—C16	1.533 (3)		

O22—C1—C14	106.84 (15)	C13—C14—H14B	109.2
O22—C1—C15	110.43 (15)	C1—C14—H14B	109.2
C14—C1—C15	111.48 (16)	H14A—C14—H14B	107.9
O22—C1—C2	101.84 (15)	C16—C15—C1	112.24 (16)
C14—C1—C2	116.66 (16)	C16—C15—C17	105.23 (16)
C15—C1—C2	109.08 (15)	C1—C15—C17	111.01 (16)
O20—C2—C3	111.43 (15)	C16—C15—C11	114.78 (16)
O20—C2—C1	104.36 (14)	C1—C15—C11	106.00 (16)
C3—C2—C1	119.49 (16)	C17—C15—C11	107.56 (16)
O20—C2—H2	107.0	C15—C16—H16B	109.5
C3—C2—H2	107.0	C15—C16—H16C	109.5
C1—C2—H2	107.0	H16B—C16—H16C	109.5
C4—C3—C2	112.93 (16)	C15—C16—H16A	109.5
C4—C3—C8	114.52 (15)	H16B—C16—H16A	109.5
C2—C3—C8	108.22 (16)	H16C—C16—H16A	109.5
C4—C3—H3	106.9	C15—C17—H17A	109.5
C2—C3—H3	106.9	C15—C17—H17B	109.5
C8—C3—H3	106.9	H17A—C17—H17B	109.5
O34—C4—C5	110.78 (16)	C15—C17—H17C	109.5
O34—C4—C3	106.57 (15)	H17A—C17—H17C	109.5
C5—C4—C3	110.44 (16)	H17B—C17—H17C	109.5
O34—C4—H4	109.7	C12—C18—H18B	109.5
C5—C4—H4	109.7	C12—C18—H18C	109.5
C3—C4—H4	109.7	H18B—C18—H18C	109.5
C4—C5—C6	110.28 (17)	C12—C18—H18A	109.5
C4—C5—H5A	109.6	H18B—C18—H18A	109.5
C6—C5—H5A	109.6	H18C—C18—H18A	109.5
C4—C5—H5B	109.6	C8—C19—H19C	109.5
C6—C5—H5B	109.6	C8—C19—H19A	109.5
H5A—C5—H5B	108.1	H19C—C19—H19A	109.5
C7—C6—C5	110.65 (16)	C8—C19—H19B	109.5
C7—C6—H6A	109.5	H19C—C19—H19B	109.5
C5—C6—H6A	109.5	H19A—C19—H19B	109.5
C7—C6—H6B	109.5	C21—O20—C2	110.43 (15)
C5—C6—H6B	109.5	O23—C21—O20	123.82 (19)
H6A—C6—H6B	108.1	O23—C21—O22	123.79 (18)
O24—C7—C6	108.86 (15)	O20—C21—O22	112.39 (18)
O24—C7—C8	106.83 (15)	C21—O22—C1	110.93 (15)
C6—C7—C8	114.51 (17)	C25—O24—C7	117.26 (15)
O24—C7—H7	108.8	O26—C25—O24	123.40 (18)
C6—C7—H7	108.8	O26—C25—C27	124.13 (18)
C8—C7—H7	108.8	O24—C25—C27	112.46 (17)
C19—C8—C3	112.46 (16)	C32—C27—C28	119.50 (18)

C19—C8—C7	109.62 (16)	C32—C27—C25	117.84 (18)
C3—C8—C7	107.59 (15)	C28—C27—C25	122.63 (18)
C19—C8—C9	112.39 (16)	C29—C28—C27	119.61 (19)
C3—C8—C9	109.13 (15)	C29—C28—H28	120.2
C7—C8—C9	105.30 (16)	C27—C28—H28	120.2
C10—C9—C8	126.80 (17)	C30—C29—C28	120.3 (2)
C10—C9—H9B	105.6	C30—C29—H29	119.8
C8—C9—H9B	105.6	C28—C29—H29	119.8
C10—C9—H9A	105.6	C31—C30—C29	120.4 (2)
C8—C9—H9A	105.6	C31—C30—H30	119.8
H9B—C9—H9A	106.1	C29—C30—H30	119.8
O33—C10—C9	113.25 (16)	C30—C31—C32	119.6 (2)
O33—C10—C11	108.23 (15)	C30—C31—H31	120.2
C9—C10—C11	121.68 (16)	C32—C31—H31	120.2
O33—C10—H10	103.9	C31—C32—C27	120.6 (2)
C9—C10—H10	103.9	C31—C32—H32	119.7
C11—C10—H10	103.9	C27—C32—H32	119.7
C12—C11—C15	110.59 (16)	C10—O33—H33	109.5
C12—C11—C10	109.26 (16)	C35—O34—C4	113.70 (14)
C15—C11—C10	123.08 (16)	O36—C35—O34	109.31 (16)
C12—C11—H11	104.0	O36—C35—H35A	109.8
C15—C11—H11	104.0	O34—C35—H35A	109.8
C10—C11—H11	104.0	O36—C35—H35B	109.8
C13—C12—C18	121.05 (19)	O34—C35—H35B	109.8
C13—C12—C11	121.81 (19)	H35A—C35—H35B	108.3
C18—C12—C11	117.12 (18)	C35—O36—C37	112.38 (17)
C12—C13—C14	124.47 (19)	O36—C37—H37C	109.5
C12—C13—H13	117.8	O36—C37—H37A	109.5
C14—C13—H13	117.8	H37C—C37—H37A	109.5
C13—C14—C1	112.09 (16)	O36—C37—H37B	109.5
C13—C14—H14A	109.2	H37C—C37—H37B	109.5
C1—C14—H14A	109.2	H37A—C37—H37B	109.5

O22—C1—C2—O20	1.49 (18)	O22—C1—C14—C13	164.37 (15)
C14—C1—C2—O20	−114.39 (18)	C15—C1—C14—C13	43.6 (2)
C15—C1—C2—O20	118.23 (16)	C2—C1—C14—C13	−82.6 (2)
O22—C1—C2—C3	126.81 (17)	O22—C1—C15—C16	50.0 (2)
C14—C1—C2—C3	10.9 (3)	C14—C1—C15—C16	168.56 (16)
C15—C1—C2—C3	−116.45 (19)	C2—C1—C15—C16	−61.2 (2)
O20—C2—C3—C4	15.6 (2)	O22—C1—C15—C17	−67.5 (2)
C1—C2—C3—C4	−106.25 (19)	C14—C1—C15—C17	51.1 (2)
O20—C2—C3—C8	−112.22 (17)	C2—C1—C15—C17	−178.64 (16)
C1—C2—C3—C8	125.90 (18)	O22—C1—C15—C11	176.00 (14)
C2—C3—C4—O34	59.3 (2)	C14—C1—C15—C11	−65.40 (19)
C8—C3—C4—O34	−176.20 (15)	C2—C1—C15—C11	64.86 (19)
C2—C3—C4—C5	179.71 (16)	C12—C11—C15—C16	178.07 (16)
C8—C3—C4—C5	−55.8 (2)	C10—C11—C15—C16	46.4 (3)
O34—C4—C5—C6	174.93 (15)	C12—C11—C15—C1	53.6 (2)

C3—C4—C5—C6	57.1 (2)	C10—C11—C15—C1	−78.1 (2)
C4—C5—C6—C7	−57.8 (2)	C12—C11—C15—C17	−65.2 (2)
C5—C6—C7—O24	176.31 (15)	C10—C11—C15—C17	163.09 (17)
C5—C6—C7—C8	56.8 (2)	C3—C2—O20—C21	−130.71 (17)
C4—C3—C8—C19	−69.6 (2)	C1—C2—O20—C21	−0.4 (2)
C2—C3—C8—C19	57.3 (2)	C2—O20—C21—O23	179.11 (19)
C4—C3—C8—C7	51.2 (2)	C2—O20—C21—O22	−1.0 (2)
C2—C3—C8—C7	178.13 (15)	O23—C21—O22—C1	−178.01 (19)
C4—C3—C8—C9	164.96 (16)	O20—C21—O22—C1	2.1 (2)
C2—C3—C8—C9	−68.11 (19)	C14—C1—O22—C21	120.70 (17)
O24—C7—C8—C19	−49.8 (2)	C15—C1—O22—C21	−117.90 (17)
C6—C7—C8—C19	70.8 (2)	C2—C1—O22—C21	−2.14 (19)
O24—C7—C8—C3	−172.37 (14)	C6—C7—O24—C25	83.5 (2)
C6—C7—C8—C3	−51.8 (2)	C8—C7—O24—C25	−152.31 (16)
O24—C7—C8—C9	71.32 (18)	C7—O24—C25—O26	2.0 (3)
C6—C7—C8—C9	−168.07 (16)	C7—O24—C25—C27	−178.54 (15)
C19—C8—C9—C10	−65.5 (2)	O26—C25—C27—C32	10.3 (3)
C3—C8—C9—C10	60.0 (2)	O24—C25—C27—C32	−169.09 (17)
C7—C8—C9—C10	175.21 (18)	O26—C25—C27—C28	−171.9 (2)
C8—C9—C10—O33	52.8 (3)	O24—C25—C27—C28	8.7 (3)
C8—C9—C10—C11	−78.9 (3)	C32—C27—C28—C29	0.0 (3)
O33—C10—C11—C12	−169.30 (16)	C25—C27—C28—C29	−177.76 (19)
C9—C10—C11—C12	−35.5 (2)	C27—C28—C29—C30	0.1 (3)
O33—C10—C11—C15	−37.1 (2)	C28—C29—C30—C31	−0.4 (3)
C9—C10—C11—C15	96.7 (2)	C29—C30—C31—C32	0.6 (3)
C15—C11—C12—C13	−23.7 (3)	C30—C31—C32—C27	−0.6 (3)
C10—C11—C12—C13	114.8 (2)	C28—C27—C32—C31	0.3 (3)
C15—C11—C12—C18	154.73 (17)	C25—C27—C32—C31	178.12 (18)
C10—C11—C12—C18	−66.8 (2)	C5—C4—O34—C35	88.2 (2)
C18—C12—C13—C14	−177.43 (18)	C3—C4—O34—C35	−151.61 (17)
C11—C12—C13—C14	1.0 (3)	C4—O34—C35—O36	−177.35 (16)
C12—C13—C14—C1	−10.5 (3)	O34—C35—O36—C37	72.7 (2)

Hydrogen-bond geometry (Å, º)

*Cg*2 is the centroid of the C27–C32 benzene ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O33	0.98	2.27	3.200 (2)	157
C14—H14*A*···O34	0.97	2.47	3.293 (2)	142
O33—H33···O23^{i}	0.82	1.96	2.7823 (19)	179
C7—H7···O26^{ii}	0.98	2.50	3.353 (2)	145
C16—H16*A*···*Cg*2^{iii}	0.98	2.93	3.594 (2)	128

Symmetry codes: (i) *x*+1/2, −*y*+1/2, *z*+1/2; (ii) −*x*+2, −*y*, −*z*+2; (iii) *x*−3/2, −*y*−1/2, *z*−3/2.

(C) (±)-(1*SR*,5*SR*,6*SR*,7*SR*,10*SR*,11*SR*,14*SR*)-7-Methoxymethoxy-11,15,18,18-tetramethyl-3,13-dioxo-2,4-dioxatetracyclo[12.3.1.0^{1,5}.0^{6,11}]octadec-15-en-10-yl benzoate

Crystal data

$C_{29}H_{36}O_8$
M_r = 512.59
Monoclinic, $P2_1/c$
a = 13.2416 (8) Å
b = 13.1779 (8) Å
c = 15.2428 (8) Å
β = 109.387 (2)°
V = 2509.0 (3) Å^3
Z = 4
$F(000)$ = 1096
D_x = 1.357 Mg m^{-3}
Melting point: 512.2 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 6186 reflections
θ = 2.8–24.9°
μ = 0.10 mm^{-1}
T = 90 K
Plate, colorless
0.22 × 0.14 × 0.09 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, 2014)
T_{min} = 0.92, T_{max} = 0.99
22512 measured reflections
4395 independent reflections
3050 reflections with $I > 2\sigma(I)$
R_{int} = 0.063
θ_{max} = 25.0°, θ_{min} = 2.4°
h = −15→14
k = −15→15
l = −16→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.105
S = 0.96
4395 reflections
339 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
$w = 1/[\sigma^2(F_o^2) + (0.0391P)^2 + 2.2049P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

Special details

Experimental. *M*.p. 510.7–512.2 K (not corrected); IR (film) 2934, 1804, 1718, 1689, 1668, 1272, 1230, 1108, 1058, 732, 713 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ (p.p.m.) 8.05–8.98 (m, 2H), 7.62–7.56 (m, 1H), 7.49–7.43 (m, 2H), 5.73 (bs, 1H), 4.79 (d, J = 6.9 Hz, 1H), 4.76 (dd, J = 11.0, 4.0 Hz, 1H), 4.60 (d, J = 6.9 Hz, 1H), 4.22 (d, J = 3.4 Hz, 1H), 3.62 (ddd, J = 10.6, 10.3, 5.2 Hz, 1H), 3.37 (s, 3H), 3.00 (bd, J = 17.0 Hz, 1H), 2.61 (d, J = 10.9 Hz, 1H), 2.51 (d, J = 17.0 Hz, 1H), 2.47 (s, 1H), 2.42–2.35 (m, 1H), 2.33 (dd, J = 10.6, 3.4 Hz, 1H), 2.13 (d, J = 10.9 Hz, 1H), 1.95–1.88 (m, 1H), 1.71 (s, 3H), 1.71–1.52 (m, 2H), 1.27 (s, 3H), 1.17 (s, 3H), 1.16 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ (p.p.m.) 211.2 (C), 165.9 (C), 153.7 (C), 135.8 (C), 133.5 (CH), 129.8 (C), 129.7 (CH), 128.7 (CH), 122.6 (CH), 97.8 (CH_2), 87.0 (C), 79.3 (CH), 79.0 (CH), 74.7 (CH), 65.7 (CH), 56.0 (CH_3), 51.2 (CH_2), 48.0 (CH), 47.1 (C), 44.1 (C), 31.9 (CH_2), 31.2 (CH_2), 25.1 (CH_3), 24.6 (CH_3), 24.6 (CH_2), 19.3 (CH_3), 12.3 (CH_3); LRMS (EI) *m/z* 512 (M^+, 5%), 346 (9), 302 (7), 121 (28), 105 (100), 77 (55); HRMS (EI) *m/z* calcd for $C_{29}H_{36}O_8^+$ $[M]^+$ 512.2410, found 514.2408.

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 two reflections with $|I(\text{obs})-I(\text{calc})|/\sigma W(I)$ greater than 10 (1 1 0 and 11 4 3) have been omitted in the final refinement.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.81842 (17)	0.32712 (17)	0.49210 (14)	0.0156 (5)
C2	0.83265 (18)	0.43504 (17)	0.53612 (14)	0.0157 (5)
H2	0.8939	0.4312	0.5959	0.019*
C3	0.73832 (17)	0.48566 (16)	0.55835 (14)	0.0141 (5)
H3	0.6787	0.435	0.5422	0.017*
C4	0.69530 (17)	0.58009 (17)	0.49830 (14)	0.0151 (5)
H4	0.7551	0.629	0.5048	0.018*
C5	0.60735 (18)	0.63170 (17)	0.52421 (14)	0.0186 (5)
H5A	0.543	0.5879	0.5055	0.022*
H5B	0.5884	0.6962	0.4894	0.022*
C6	0.63941 (18)	0.65384 (17)	0.62826 (14)	0.0191 (5)
H6A	0.699	0.7034	0.6466	0.023*
H6B	0.578	0.6834	0.6427	0.023*
C7	0.67390 (18)	0.55614 (17)	0.68165 (13)	0.0152 (5)
H7	0.6122	0.5078	0.6641	0.018*
C8	0.77033 (17)	0.50500 (16)	0.66456 (14)	0.0144 (5)
C9	0.78727 (18)	0.40213 (17)	0.71791 (14)	0.0170 (5)
H9B	0.8022	0.4176	0.7846	0.02*
H9A	0.7185	0.3648	0.6963	0.02*
C10	0.87422 (19)	0.33035 (17)	0.71100 (14)	0.0176 (5)
C11	0.84777 (17)	0.23989 (17)	0.64233 (14)	0.0169 (5)
H11	0.8853	0.1799	0.6791	0.02*
C12	0.72983 (18)	0.21357 (17)	0.60918 (15)	0.0170 (5)
C13	0.66633 (18)	0.23789 (17)	0.52443 (15)	0.0179 (5)
H13	0.5922	0.2228	0.5084	0.021*
C14	0.70379 (17)	0.28774 (17)	0.45229 (14)	0.0175 (5)
H14A	0.6555	0.3451	0.4243	0.021*
H14B	0.6994	0.2383	0.4023	0.021*
C15	0.89166 (17)	0.24989 (17)	0.55936 (14)	0.0177 (5)
C16	1.00975 (18)	0.28215 (18)	0.59015 (16)	0.0209 (5)
H16A	1.0171	0.3507	0.6165	0.031*
H16B	1.0346	0.2816	0.5363	0.031*
H16C	1.0528	0.2347	0.6373	0.031*
C17	0.88509 (19)	0.14530 (17)	0.51289 (16)	0.0221 (6)
H17C	0.9039	0.152	0.4562	0.033*
H17A	0.8121	0.1188	0.497	0.033*
H17B	0.9351	0.0985	0.5559	0.033*
C18	0.69208 (19)	0.15857 (18)	0.67848 (15)	0.0222 (6)

H18B	0.6144	0.1488	0.6528	0.033*
H18C	0.7096	0.1985	0.7358	0.033*
H18A	0.7275	0.0924	0.6922	0.033*
C19	0.87058 (18)	0.57156 (17)	0.70192 (15)	0.0186 (5)
H19A	0.8809	0.5905	0.7665	0.028*
H19B	0.8617	0.633	0.6639	0.028*
H19C	0.9332	0.5337	0.6992	0.028*
O20	0.86805 (11)	0.49630 (11)	0.47260 (9)	0.0165 (4)
C21	0.87791 (17)	0.43902 (18)	0.40303 (15)	0.0169 (5)
O22	0.85523 (12)	0.34137 (12)	0.41269 (10)	0.0187 (4)
O23	0.90357 (12)	0.47131 (12)	0.34036 (10)	0.0224 (4)
O24	0.70651 (11)	0.57550 (11)	0.78110 (9)	0.0170 (4)
C25	0.63110 (18)	0.57567 (16)	0.82216 (15)	0.0146 (5)
O26	0.53686 (12)	0.56418 (12)	0.78106 (10)	0.0203 (4)
C27	0.67949 (18)	0.58868 (16)	0.92408 (14)	0.0150 (5)
C28	0.61586 (19)	0.57355 (17)	0.97941 (15)	0.0212 (5)
H28	0.5422	0.5574	0.9515	0.025*
C29	0.6597 (2)	0.58201 (18)	1.07519 (15)	0.0237 (6)
H29	0.6161	0.5712	1.1128	0.028*
C30	0.7661 (2)	0.60592 (18)	1.11608 (16)	0.0255 (6)
H30	0.796	0.6109	1.1818	0.031*
C31	0.8296 (2)	0.62271 (18)	1.06145 (16)	0.0250 (6)
H31	0.9028	0.6403	1.0896	0.03*
C32	0.78646 (19)	0.61393 (18)	0.96603 (15)	0.0215 (5)
H32	0.8304	0.6253	0.9288	0.026*
O33	0.96560 (13)	0.34029 (12)	0.76362 (10)	0.0236 (4)
O34	0.65380 (12)	0.54447 (11)	0.40409 (9)	0.0182 (4)
C35	0.67590 (19)	0.60542 (18)	0.33721 (15)	0.0214 (6)
H35A	0.7532	0.6218	0.3592	0.026*
H35B	0.6603	0.566	0.2789	0.026*
O36	0.61766 (12)	0.69614 (11)	0.31770 (10)	0.0200 (4)
C37	0.50654 (18)	0.68122 (18)	0.27053 (16)	0.0243 (6)
H37B	0.4746	0.647	0.312	0.036*
H37C	0.4967	0.6393	0.2152	0.036*
H37A	0.4717	0.7471	0.2518	0.036*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0189 (13)	0.0175 (13)	0.0133 (11)	0.0015 (10)	0.0092 (10)	−0.0016 (9)
C2	0.0189 (13)	0.0148 (13)	0.0149 (11)	−0.0009 (10)	0.0079 (10)	0.0026 (9)
C3	0.0149 (12)	0.0126 (12)	0.0152 (11)	−0.0002 (10)	0.0056 (9)	−0.0016 (9)
C4	0.0166 (12)	0.0147 (13)	0.0130 (11)	−0.0004 (10)	0.0037 (10)	−0.0012 (9)
C5	0.0216 (13)	0.0163 (13)	0.0183 (11)	0.0057 (10)	0.0071 (10)	0.0028 (10)
C6	0.0228 (13)	0.0159 (13)	0.0212 (12)	0.0041 (10)	0.0109 (10)	−0.0006 (10)
C7	0.0192 (12)	0.0160 (13)	0.0112 (11)	−0.0035 (10)	0.0061 (10)	−0.0036 (9)
C8	0.0154 (12)	0.0156 (13)	0.0129 (11)	−0.0015 (10)	0.0058 (9)	−0.0010 (9)
C9	0.0199 (13)	0.0170 (13)	0.0150 (11)	0.0001 (10)	0.0070 (10)	−0.0003 (9)

C10	0.0209 (14)	0.0182 (13)	0.0137 (11)	−0.0007 (11)	0.0055 (11)	0.0052 (10)
C11	0.0187 (13)	0.0134 (13)	0.0178 (11)	0.0010 (10)	0.0049 (10)	0.0024 (9)
C12	0.0197 (13)	0.0128 (13)	0.0201 (12)	−0.0010 (10)	0.0089 (10)	−0.0034 (10)
C13	0.0180 (13)	0.0132 (13)	0.0226 (12)	−0.0012 (10)	0.0070 (10)	−0.0040 (10)
C14	0.0192 (13)	0.0151 (13)	0.0154 (11)	0.0009 (10)	0.0021 (10)	−0.0013 (9)
C15	0.0191 (13)	0.0144 (13)	0.0197 (12)	0.0023 (10)	0.0066 (10)	−0.0003 (10)
C16	0.0204 (13)	0.0179 (14)	0.0257 (12)	0.0051 (10)	0.0094 (10)	0.0026 (10)
C17	0.0260 (14)	0.0167 (14)	0.0252 (12)	0.0024 (11)	0.0106 (11)	−0.0014 (10)
C18	0.0242 (14)	0.0209 (14)	0.0229 (12)	−0.0011 (11)	0.0096 (11)	−0.0002 (10)
C19	0.0205 (13)	0.0180 (13)	0.0181 (11)	−0.0001 (10)	0.0074 (10)	−0.0028 (10)
O20	0.0199 (9)	0.0152 (9)	0.0177 (8)	−0.0015 (7)	0.0109 (7)	−0.0013 (7)
C21	0.0127 (12)	0.0184 (14)	0.0175 (12)	0.0024 (10)	0.0022 (10)	−0.0007 (10)
O22	0.0258 (9)	0.0165 (9)	0.0177 (8)	−0.0001 (7)	0.0122 (7)	−0.0014 (7)
O23	0.0254 (9)	0.0267 (10)	0.0201 (8)	−0.0039 (8)	0.0139 (7)	0.0004 (7)
O24	0.0194 (9)	0.0194 (9)	0.0139 (7)	−0.0010 (7)	0.0079 (7)	−0.0032 (6)
C25	0.0171 (13)	0.0079 (12)	0.0222 (12)	0.0014 (10)	0.0109 (11)	0.0004 (9)
O26	0.0175 (10)	0.0225 (10)	0.0211 (8)	−0.0019 (7)	0.0066 (7)	−0.0029 (7)
C27	0.0199 (13)	0.0108 (12)	0.0146 (11)	0.0022 (10)	0.0061 (10)	0.0003 (9)
C28	0.0256 (14)	0.0157 (13)	0.0235 (12)	0.0022 (11)	0.0098 (11)	0.0009 (10)
C29	0.0333 (16)	0.0241 (15)	0.0172 (12)	0.0041 (12)	0.0129 (12)	0.0027 (10)
C30	0.0390 (16)	0.0196 (14)	0.0150 (12)	0.0049 (12)	0.0052 (12)	0.0012 (10)
C31	0.0263 (14)	0.0230 (15)	0.0220 (13)	−0.0033 (11)	0.0029 (11)	−0.0035 (10)
C32	0.0243 (14)	0.0214 (14)	0.0212 (12)	−0.0008 (11)	0.0106 (11)	−0.0026 (10)
O33	0.0205 (10)	0.0264 (10)	0.0200 (8)	0.0017 (8)	0.0016 (8)	−0.0005 (7)
O34	0.0248 (9)	0.0179 (9)	0.0124 (7)	0.0017 (7)	0.0068 (7)	0.0018 (6)
C35	0.0241 (14)	0.0254 (15)	0.0162 (12)	0.0067 (11)	0.0087 (11)	0.0051 (10)
O36	0.0203 (9)	0.0162 (9)	0.0221 (8)	−0.0005 (7)	0.0052 (7)	0.0047 (7)
C37	0.0200 (14)	0.0243 (15)	0.0275 (13)	0.0014 (11)	0.0065 (11)	0.0056 (11)

Geometric parameters (Å, º)

C1—O22	1.459 (2)	C15—C17	1.539 (3)
C1—C14	1.526 (3)	C16—H16A	0.98
C1—C15	1.539 (3)	C16—H16B	0.98
C1—C2	1.557 (3)	C16—H16C	0.98
C2—O20	1.453 (2)	C17—H17C	0.98
C2—C3	1.550 (3)	C17—H17A	0.98
C2—H2	1.0	C17—H17B	0.98
C3—C4	1.538 (3)	C18—H18B	0.98
C3—C8	1.552 (3)	C18—H18C	0.98
C3—H3	1.0	C18—H18A	0.98
C4—O34	1.435 (2)	C19—H19A	0.98
C4—C5	1.510 (3)	C19—H19B	0.98
C4—H4	1.0	C19—H19C	0.98
C5—C6	1.528 (3)	O20—C21	1.343 (3)
C5—H5A	0.99	C21—O23	1.193 (2)
C5—H5B	0.99	C21—O22	1.341 (3)
C6—C7	1.510 (3)	O24—C25	1.343 (2)

C6—H6A	0.99	C25—O26	1.206 (3)
C6—H6B	0.99	C25—C27	1.481 (3)
C7—O24	1.454 (2)	C27—C32	1.387 (3)
C7—C8	1.540 (3)	C27—C28	1.390 (3)
C7—H7	1.0	C28—C29	1.385 (3)
C8—C19	1.534 (3)	C28—H28	0.95
C8—C9	1.558 (3)	C29—C30	1.374 (3)
C9—C10	1.521 (3)	C29—H29	0.95
C9—H9B	0.99	C30—C31	1.384 (3)
C9—H9A	0.99	C30—H30	0.95
C10—O33	1.217 (3)	C31—C32	1.380 (3)
C10—C11	1.548 (3)	C31—H31	0.95
C11—C12	1.514 (3)	C32—H32	0.95
C11—C15	1.563 (3)	O34—C35	1.403 (3)
C11—H11	1.0	C35—O36	1.400 (3)
C12—C13	1.326 (3)	C35—H35A	0.99
C12—C18	1.497 (3)	C35—H35B	0.99
C13—C14	1.500 (3)	O36—C37	1.421 (3)
C13—H13	0.95	C37—H37B	0.98
C14—H14A	0.99	C37—H37C	0.98
C14—H14B	0.99	C37—H37A	0.98
C15—C16	1.536 (3)		

O22—C1—C14	106.01 (16)	C1—C14—H14B	109.1
O22—C1—C15	109.12 (17)	H14A—C14—H14B	107.8
C14—C1—C15	111.33 (18)	C16—C15—C1	111.91 (18)
O22—C1—C2	102.36 (16)	C16—C15—C17	106.55 (18)
C14—C1—C2	116.36 (18)	C1—C15—C17	111.04 (18)
C15—C1—C2	110.96 (17)	C16—C15—C11	112.98 (17)
O20—C2—C3	111.62 (17)	C1—C15—C11	105.87 (17)
O20—C2—C1	103.89 (15)	C17—C15—C11	108.51 (18)
C3—C2—C1	119.76 (18)	C15—C16—H16A	109.5
O20—C2—H2	107.0	C15—C16—H16B	109.5
C3—C2—H2	107.0	H16A—C16—H16B	109.5
C1—C2—H2	107.0	C15—C16—H16C	109.5
C4—C3—C2	112.61 (17)	H16A—C16—H16C	109.5
C4—C3—C8	114.01 (17)	H16B—C16—H16C	109.5
C2—C3—C8	109.74 (17)	C15—C17—H17C	109.5
C4—C3—H3	106.7	C15—C17—H17A	109.5
C2—C3—H3	106.7	H17C—C17—H17A	109.5
C8—C3—H3	106.7	C15—C17—H17B	109.5
O34—C4—C5	109.67 (17)	H17C—C17—H17B	109.5
O34—C4—C3	105.90 (17)	H17A—C17—H17B	109.5
C5—C4—C3	111.98 (17)	C12—C18—H18B	109.5
O34—C4—H4	109.7	C12—C18—H18C	109.5
C5—C4—H4	109.7	H18B—C18—H18C	109.5
C3—C4—H4	109.7	C12—C18—H18A	109.5
C4—C5—C6	112.64 (18)	H18B—C18—H18A	109.5

C4—C5—H5A	109.1	H18C—C18—H18A	109.5
C6—C5—H5A	109.1	C8—C19—H19A	109.5
C4—C5—H5B	109.1	C8—C19—H19B	109.5
C6—C5—H5B	109.1	H19A—C19—H19B	109.5
H5A—C5—H5B	107.8	C8—C19—H19C	109.5
C7—C6—C5	109.07 (18)	H19A—C19—H19C	109.5
C7—C6—H6A	109.9	H19B—C19—H19C	109.5
C5—C6—H6A	109.9	C21—O20—C2	110.57 (17)
C7—C6—H6B	109.9	O23—C21—O22	124.3 (2)
C5—C6—H6B	109.9	O23—C21—O20	124.0 (2)
H6A—C6—H6B	108.3	O22—C21—O20	111.64 (18)
O24—C7—C6	110.13 (17)	C21—O22—C1	111.30 (16)
O24—C7—C8	106.08 (16)	C25—O24—C7	118.46 (17)
C6—C7—C8	113.65 (17)	O26—C25—O24	123.99 (19)
O24—C7—H7	109.0	O26—C25—C27	124.93 (19)
C6—C7—H7	109.0	O24—C25—C27	111.07 (19)
C8—C7—H7	109.0	C32—C27—C28	119.1 (2)
C19—C8—C7	110.53 (18)	C32—C27—C25	122.10 (19)
C19—C8—C3	112.58 (17)	C28—C27—C25	118.8 (2)
C7—C8—C3	107.06 (16)	C29—C28—C27	120.1 (2)
C19—C8—C9	110.29 (17)	C29—C28—H28	120.0
C7—C8—C9	106.03 (17)	C27—C28—H28	120.0
C3—C8—C9	110.09 (17)	C30—C29—C28	120.3 (2)
C10—C9—C8	119.09 (18)	C30—C29—H29	119.8
C10—C9—H9B	107.5	C28—C29—H29	119.8
C8—C9—H9B	107.5	C29—C30—C31	120.0 (2)
C10—C9—H9A	107.5	C29—C30—H30	120.0
C8—C9—H9A	107.5	C31—C30—H30	120.0
H9B—C9—H9A	107.0	C32—C31—C30	120.0 (2)
O33—C10—C9	119.8 (2)	C32—C31—H31	120.0
O33—C10—C11	118.8 (2)	C30—C31—H31	120.0
C9—C10—C11	121.29 (19)	C31—C32—C27	120.5 (2)
C12—C11—C10	112.27 (18)	C31—C32—H32	119.7
C12—C11—C15	111.64 (17)	C27—C32—H32	119.7
C10—C11—C15	114.89 (18)	C35—O34—C4	115.90 (17)
C12—C11—H11	105.7	O36—C35—O34	114.22 (17)
C10—C11—H11	105.7	O36—C35—H35A	108.7
C15—C11—H11	105.7	O34—C35—H35A	108.7
C13—C12—C18	123.5 (2)	O36—C35—H35B	108.7
C13—C12—C11	121.4 (2)	O34—C35—H35B	108.7
C18—C12—C11	115.12 (18)	H35A—C35—H35B	107.6
C12—C13—C14	124.3 (2)	C35—O36—C37	113.22 (18)
C12—C13—H13	117.9	O36—C37—H37B	109.5
C14—C13—H13	117.9	O36—C37—H37C	109.5
C13—C14—C1	112.63 (17)	H37B—C37—H37C	109.5
C13—C14—H14A	109.1	O36—C37—H37A	109.5
C1—C14—H14A	109.1	H37B—C37—H37A	109.5
C13—C14—H14B	109.1	H37C—C37—H37A	109.5

O22—C1—C2—O20	3.79 (19)	O22—C1—C14—C13	161.48 (17)
C14—C1—C2—O20	−111.28 (19)	C15—C1—C14—C13	42.9 (2)
C15—C1—C2—O20	120.08 (18)	C2—C1—C14—C13	−85.5 (2)
O22—C1—C2—C3	129.13 (18)	O22—C1—C15—C16	55.9 (2)
C14—C1—C2—C3	14.1 (3)	C14—C1—C15—C16	172.61 (17)
C15—C1—C2—C3	−114.6 (2)	C2—C1—C15—C16	−56.1 (2)
O20—C2—C3—C4	7.8 (2)	O22—C1—C15—C17	−63.0 (2)
C1—C2—C3—C4	−113.8 (2)	C14—C1—C15—C17	53.7 (2)
O20—C2—C3—C8	−120.39 (18)	C2—C1—C15—C17	−175.02 (17)
C1—C2—C3—C8	118.0 (2)	O22—C1—C15—C11	179.44 (16)
C2—C3—C4—O34	63.5 (2)	C14—C1—C15—C11	−63.9 (2)
C8—C3—C4—O34	−170.62 (16)	C2—C1—C15—C11	67.4 (2)
C2—C3—C4—C5	−177.00 (18)	C12—C11—C15—C16	176.93 (18)
C8—C3—C4—C5	−51.1 (2)	C10—C11—C15—C16	47.6 (3)
O34—C4—C5—C6	169.18 (18)	C12—C11—C15—C1	54.1 (2)
C3—C4—C5—C6	51.9 (3)	C10—C11—C15—C1	−75.2 (2)
C4—C5—C6—C7	−55.8 (2)	C12—C11—C15—C17	−65.1 (2)
C5—C6—C7—O24	179.09 (17)	C10—C11—C15—C17	165.54 (18)
C5—C6—C7—C8	60.2 (2)	C3—C2—O20—C21	−131.96 (18)
O24—C7—C8—C19	−55.8 (2)	C1—C2—O20—C21	−1.6 (2)
C6—C7—C8—C19	65.3 (2)	C2—O20—C21—O23	178.3 (2)
O24—C7—C8—C3	−178.79 (16)	C2—O20—C21—O22	−1.6 (2)
C6—C7—C8—C3	−57.6 (2)	O23—C21—O22—C1	−175.5 (2)
O24—C7—C8—C9	63.7 (2)	O20—C21—O22—C1	4.4 (2)
C6—C7—C8—C9	−175.17 (17)	C14—C1—O22—C21	117.38 (19)
C4—C3—C8—C19	−69.6 (2)	C15—C1—O22—C21	−122.62 (19)
C2—C3—C8—C19	57.8 (2)	C2—C1—O22—C21	−5.0 (2)
C4—C3—C8—C7	52.1 (2)	C6—C7—O24—C25	86.3 (2)
C2—C3—C8—C7	179.44 (17)	C8—C7—O24—C25	−150.29 (18)
C4—C3—C8—C9	166.90 (18)	C7—O24—C25—O26	−2.7 (3)
C2—C3—C8—C9	−65.7 (2)	C7—O24—C25—C27	175.64 (17)
C19—C8—C9—C10	−63.2 (2)	O26—C25—C27—C32	−171.9 (2)
C7—C8—C9—C10	177.06 (18)	O24—C25—C27—C32	9.8 (3)
C3—C8—C9—C10	61.6 (2)	O26—C25—C27—C28	9.1 (3)
C8—C9—C10—O33	86.1 (2)	O24—C25—C27—C28	−169.24 (19)
C8—C9—C10—C11	−98.0 (2)	C32—C27—C28—C29	−1.1 (3)
O33—C10—C11—C12	158.08 (19)	C25—C27—C28—C29	177.9 (2)
C9—C10—C11—C12	−17.9 (3)	C27—C28—C29—C30	0.4 (3)
O33—C10—C11—C15	−72.9 (3)	C28—C29—C30—C31	0.7 (4)
C9—C10—C11—C15	111.1 (2)	C29—C30—C31—C32	−1.0 (4)
C10—C11—C12—C13	104.9 (2)	C30—C31—C32—C27	0.2 (4)
C15—C11—C12—C13	−25.8 (3)	C28—C27—C32—C31	0.8 (3)
C10—C11—C12—C18	−75.4 (2)	C25—C27—C32—C31	−178.2 (2)
C15—C11—C12—C18	153.96 (19)	C5—C4—O34—C35	100.2 (2)
C18—C12—C13—C14	−176.4 (2)	C3—C4—O34—C35	−138.79 (18)
C11—C12—C13—C14	3.3 (3)	C4—O34—C35—O36	−72.6 (2)
C12—C13—C14—C1	−11.5 (3)	O34—C35—O36—C37	−68.7 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C14—H14*A*···O34	0.99	2.65	3.479 (3)	142
C31—H31···O33^{i}	0.95	2.35	3.147 (3)	141
C19—H19*C*···O23^{ii}	0.98	2.43	3.310 (3)	149
C16—H16*A*···O23^{ii}	0.98	2.56	3.491 (3)	158

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