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Crystal structure and Hirshfeld surface analysis of (5a*S*,8a*R*)-3,5a-dimethyl-8-methylidene-2-oxododecahydrooxireno[2′,3′:6,7]naphtho[1,2-*b*]furan-6-yl (*Z*)-2-methylbut-2-enoate extracted from *Ferula persica*

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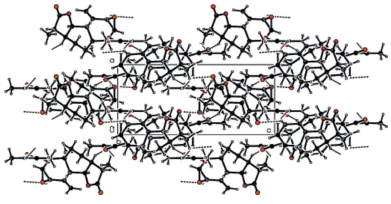
In the title compound, $C_{20}H_{26}O_5$, the two cyclohexane rings adopt boat and half-chair conformations. In the crystal, adjacent molecules are connected by intermolecular C—H···O hydrogen bonds, forming a three-dimensional network. According to a Hirshfeld surface study, H···H interactions are the most significant contributors to the crystal packing (63.0%).

1. Chemical context

Sesquiterpene lactones are a significant group of natural products isolated from the extracts of various parts of medicinal plants. As a medicinal plant, the *Ferula* genus is rich in coumarins, specifically sesquiterpene coumarins. *Ferula* species are found in the Mediterranean region, Central Asia, Siberia, China, Afghanistan, Iran, North Africa and the Caucasus (Mir-Babayev & Houghton, 2002). The members of this genus typically have a heavy fragrance due to the presence of essential oils or oleoresins in their content. This genus is applied for the cure of various organ disorders in folk medicine (Salehi *et al.*, 2019). These herbs have been used for oleo-gum resin, plant extracts, and essential oils. Moreover, the essential oils and extracts of different species of this herb can be used as natural food preservatives due to their antioxidant and antimicrobial activity (Daneshniya *et al.*, 2021).



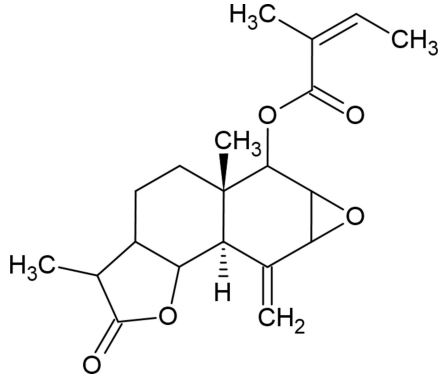


Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C3A—H3A···O4^{i}	1.00	2.45	3.353 (2)	149
C8—H8···O2^{ii}	1.00	2.50	3.176 (2)	124
C14—H14···O10^{iii}	0.95	2.57	3.423 (2)	149

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

Table 2
Summary of short interatomic contacts (Å) in the title compound.

H15A···H16C	2.39	$-\frac{1}{2}+x, \frac{3}{2}-y, 1-z$
O2···H8	2.50	$\frac{1}{2}-x, 1-y, \frac{1}{2}+z$
O4···H3A	2.45	$\frac{3}{2}-x, 1-y, -\frac{1}{2}+z$
H8···H10A	2.48	$1-x, -\frac{1}{2}+y, \frac{3}{2}-z$
H11B···H17A	2.40	$1+x, y, z$

Herein, in the framework of our ongoing structural studies, we report the crystal structure and Hirshfeld surface analysis of the title compound, (5a*S*,8a*R*)-3,5a-dimethyl-8-methylidene-2-oxododecahydrooxireno[2′,3′:6,7]naphtho[1,2-*b*]furan-6-yl (*Z*)-2-methylbut-2-enoate extracted from *Ferula persica*.

2. Structural commentary

A view of the molecular structure of the title compound is shown in Fig. 1. The cyclohexane rings (*A*: C3*A*/C4/C5/C5*A*/C9*A*/C9*B*; *B*: C5*A*/C6–C9/C9*A*) adopt boat and half-chair conformations, respectively. The puckering parameters (Cremer & Pople, 1975) of the *A* and *B* rings are Q_T = 0.7259 (19) Å, θ = 83.29 (15)°, φ = 51.45 (15)°, and Q_T = 0.5337 (18) Å, θ = 52.1 (2)°, φ = 331.7 (3)°, respectively.

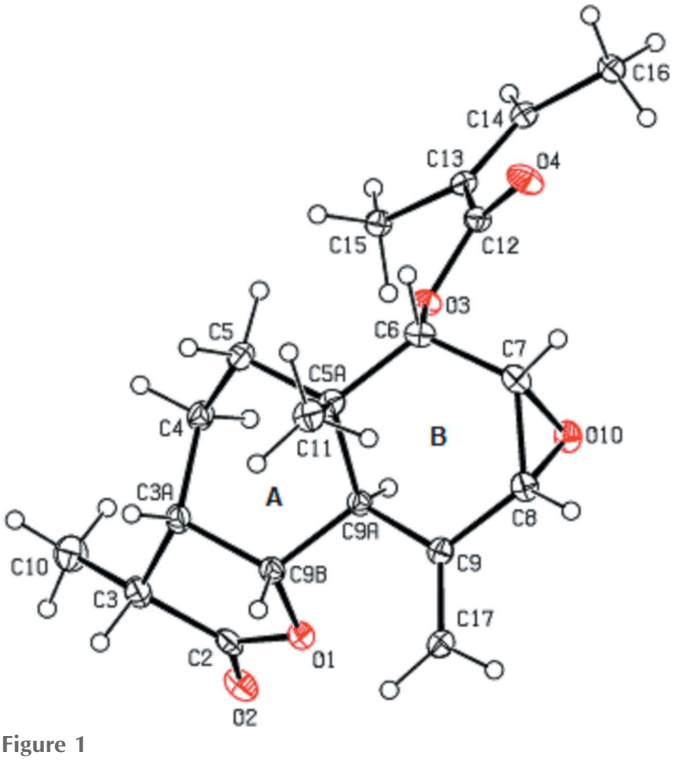


Figure 1
The molecular structure of the title compound, showing the atom labelling and displacement ellipsoids drawn at the 30% probability level.

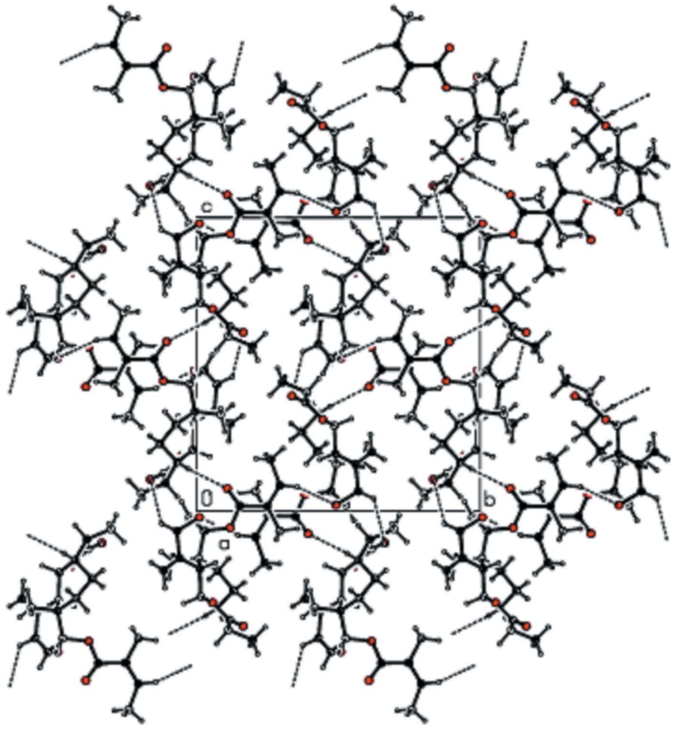


Figure 2
View of the packing of the title compound down the *a* axis.

3. Supramolecular features and Hirshfeld surface analysis

In the crystal of the title compound, adjacent molecules are connected by intermolecular C—H···O hydrogen bonds, forming a three dimensional network (Tables 1 and 2). Figs. 2, 3 and 4 show packing views of the title compound down the *a*, *b* and *c* axes, respectively.

CrystalExplorer17 (Spackman *et al.*, 2021) was used to compute Hirshfeld surfaces of the title molecule. The d_{norm} mappings for the molecule were performed in the range −0.1633 to +1.3364 a.u. The locations of the C—H···O interactions are shown by intense red circles on the d_{norm} surface (Fig. 5*a*,*b*).

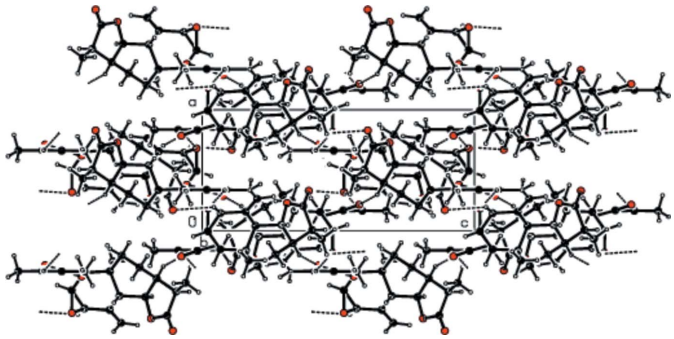


Figure 3
View of the packing of the title compound down the *b* axis.

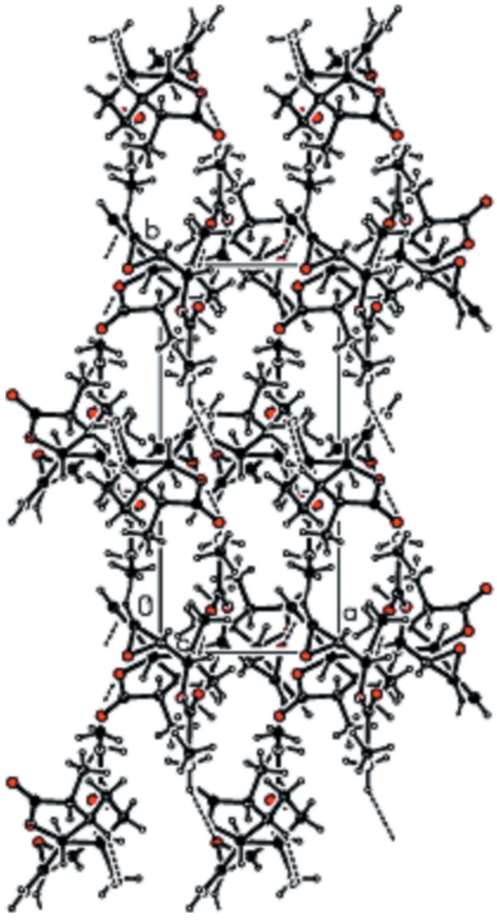


Figure 4
View of the packing of the title compound down the *c* axis.

Fig. 6 shows the full two-dimensional fingerprint plots for the molecule and those delineated into the major contacts. H···H interactions (Fig. 6*b*; 63.0% contribution) are the major contributor to the crystal packing with O···H/H···O (Fig. 6*c*; 28.3%) and C···H/H···C (Fig. 6*d*; 7.5%) interactions representing the next highest contributions. The percentage contributions of comparatively weaker interactions are O···C/C···O (0.5%) , O···O (0.4%) and C···C (0.3%). Relevant short intermolecular atomic contacts are summarized in Table 2.

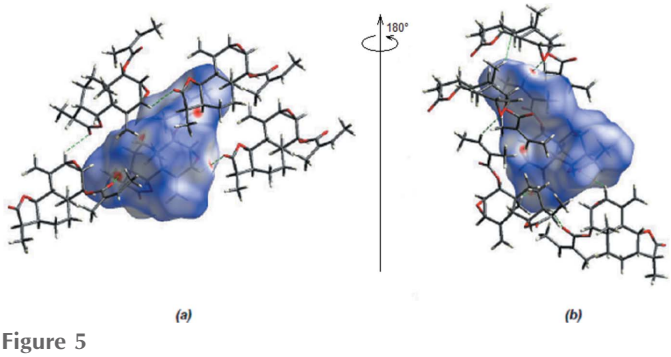


Figure 5
(*a*) Front and (*b*) back sides of the three-dimensional Hirshfeld surface of the title compound mapped over d_{norm}, with a fixed colour scale of −0.1633 to +1.3364 a.u.

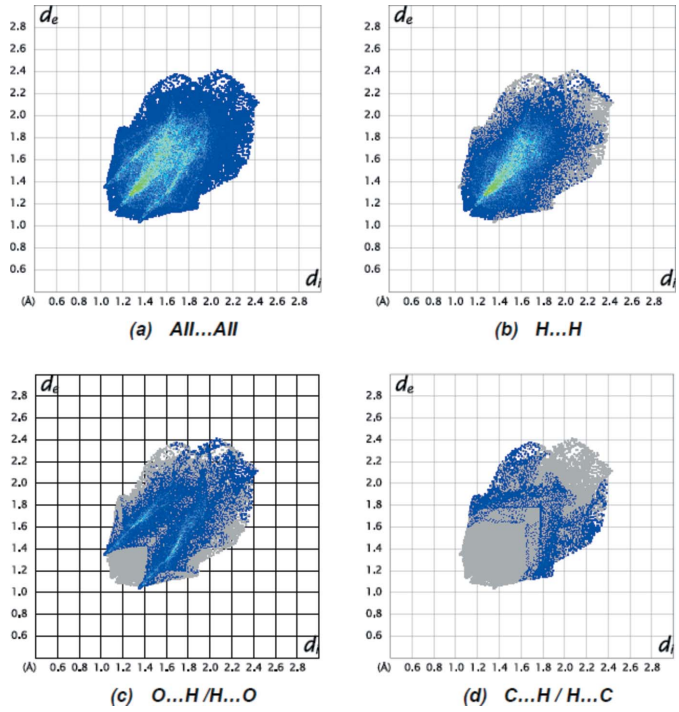


Figure 6
The two-dimensional fingerprint plots of the title compound, showing (*a*) all interactions, and delineated into (*b*) H···H, (*c*) O···H/H···O and (*d*) C···H/H···C interactions. [d_e and d_i represent the distances from a point on the Hirshfeld surface to the nearest atoms outside (external) and inside (internal) the surface, respectively].

4. Database survey

Two closely related compounds are 1β-angeloyloxy-2β,3β-epoxy-5βH,7αH-l0α-methyleudesma-4(15),11(13)-dien-6,12-olide (**I**) (Rychlewska *et al.*, 1992) and 1β-angeloyloxy-5βH,6αH,7αH,11αH-10α-methyleudesma-2,4(15)-dien-6,12-olide (**II**) (Rychlewska *et al.*, 1992).

The largest difference between the two structures (**I** and **II**) lies in the cyclohexane *B* ring, which is of the rigid-chair type in **I** and of the flexible boat type in **II**. In both crystal structures, the molecules are held together mostly by van der Waals forces.

5. Synthesis and crystallization

The title compound has previously been isolated from the roots of the *Ferula oopoda* plant and fully characterized (Serkerov, 1972). The compound used for the current study was isolated from the roots of the *Ferula persica* herb by a similar method.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. H atoms of the –C═CH_2 group were located in a difference-Fourier map and refined freely [C17—H17*A* = 0.94 (2) Å, C17—H17*B* = 0.97 (2) Å]. All other H atoms were placed at calculated positions and refined using a riding model, with C—H = 0.95–1.00 Å, and with

Table 3
Experimental details.

Crystal data	
Chemical formula	$C_{20}H_{26}O_5$
M_r	346.41
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	100
a, b, c (Å)	7.11296 (5), 15.4597 (10), 16.0358 (10)
V (Å^3)	1763.36 (16)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.76
Crystal size (mm)	0.21 × 0.18 × 0.13
Data collection	
Diffractometer	XtaLAB Synergy, Dualflex, HyPix
Absorption correction	Gaussian (*CrysAlis PRO*; Rigaku OD, 2022)
T_{min}, T_{max}	0.674, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	19755, 3751, 3717
R_{int}	0.024
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.634
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.031, 0.082, 1.05
No. of reflections	3751
No. of parameters	238
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.56, −0.16
Absolute structure	Flack x determined using 1576 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons et al, 2013).
Absolute structure parameter	0.01 (4)

Computer programs: *CrysAlis PRO* (Rigaku OD, 2022), SHELXT (Sheldrick, 2015*a*), *SHELXL* (Sheldrick, 2015*b*), *ORTEP-3 for Windows* (Farrugia, 2012), *PLATON* (Spek, 2020).

U_{iso}(H) = 1.2 or 1.5U_{eq}(C). The remaining maximum electron density peak (0.56 e$^-$ Å^{-3}) is 1.41 Å away from C17 and the minimum density peak (–0.16 e Å^{-3}) is 0.92 Å away from C9.

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Authors' contributions are as follows. Conceptualization, ANK and IGM; methodology, ANK, EGK and IGM; investigation, ANK, MA and EGK; writing (original draft), MA and ANK; writing (review and editing of the manuscript), MA and ANK; visualization, MA, ANK and IGM; funding acquisition, VNK, AB and ANK; resources, AB, VNK and MNK; supervision, ANK and MA.

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supporting information

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Computing details

Data collection: *CrysAlis PRO* (Rigaku OD, 2022); cell refinement: *CrysAlis PRO* (Rigaku OD, 2022); data reduction: *CrysAlis PRO* (Rigaku OD, 2022); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015b); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *PLATON* (Spek, 2020).

(5a*S*,8a*R*)-3,5a-Dimethyl-8-methylidene-2-oxododecahydrooxireno[2′,3′:6,7]naphtho[1,2-*b*]furan-6-yl (*Z*)-2-methylbut-2-enoate

Crystal data

$C_{20}H_{26}O_5$
M_r = 346.41
Orthorhombic, $P2_12_12_1$
a = 7.11296 (5) Å
b = 15.4597 (10) Å
c = 16.0358 (10) Å
V = 1763.36 (16) Å^3
Z = 4
$F(000)$ = 744
D_x = 1.305 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 16716 reflections
θ = 3.9–77.3°
μ = 0.76 mm^{-1}
T = 100 K
Prism, yellow
0.21 × 0.18 × 0.13 mm

Data collection

XtaLAB Synergy, Dualflex, HyPix diffractometer
Radiation source: micro-focus sealed X-ray tube
φ and ω scans
Absorption correction: gaussian (CrysAlis PRO; Rigaku OD, 2022)
T_{min} = 0.674, T_{max} = 1.000
19755 measured reflections
3751 independent reflections
3717 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 77.8°, θ_{min} = 4.0°
h = −9→8
k = −19→16
l = −18→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.082
S = 1.05
3751 reflections
238 parameters
0 restraints
Primary atom site location: difference Fourier map

Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0444P)^2 + 0.4585P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.56$ e Å^{-3}
$\Delta\rho_{min} = -0.16$ e Å^{-3}
Absolute structure: Flack *x* determined using 1576 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons et al, 2013).
Absolute structure parameter: 0.01 (4)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.27214 (18)	0.55551 (9)	0.81184 (8)	0.0258 (3)
O2	0.1708 (2)	0.66397 (9)	0.89164 (9)	0.0325 (3)
O3	0.62078 (17)	0.61814 (8)	0.55679 (8)	0.0217 (3)
O4	0.7139 (2)	0.60760 (8)	0.42338 (8)	0.0316 (3)
C2	0.2969 (3)	0.61638 (12)	0.87107 (10)	0.0257 (4)
C3	0.4949 (3)	0.61151 (12)	0.90431 (11)	0.0278 (4)
H3	0.4936	0.5693	0.9516	0.033*
C3A	0.6045 (3)	0.56914 (11)	0.83285 (11)	0.0237 (4)
H3A	0.7026	0.5300	0.8568	0.028*
C4	0.6965 (3)	0.62980 (11)	0.76998 (11)	0.0259 (4)
H4A	0.7893	0.6675	0.7983	0.031*
H4B	0.6003	0.6670	0.7434	0.031*
C5	0.7941 (3)	0.57422 (12)	0.70428 (12)	0.0270 (4)
H5A	0.8994	0.5429	0.7311	0.032*
H5B	0.8483	0.6127	0.6612	0.032*
C5A	0.6636 (2)	0.50725 (11)	0.66081 (11)	0.0220 (3)
C6	0.6543 (3)	0.52614 (11)	0.56680 (11)	0.0226 (3)
H6	0.7798	0.5124	0.5420	0.027*
C7	0.5062 (3)	0.47385 (11)	0.52056 (11)	0.0234 (4)
H7	0.5477	0.4454	0.4677	0.028*
C8	0.3552 (3)	0.42987 (11)	0.56746 (11)	0.0227 (3)
H8	0.3055	0.3750	0.5428	0.027*
C9	0.3431 (2)	0.43826 (11)	0.65931 (11)	0.0217 (3)
C9A	0.4598 (2)	0.51073 (10)	0.69607 (10)	0.0194 (3)
H9A	0.4033	0.5660	0.6756	0.023*
C9B	0.4515 (2)	0.51404 (11)	0.79115 (10)	0.0219 (3)
H9B	0.4543	0.4540	0.8143	0.026*
O10	0.31873 (19)	0.50878 (8)	0.52107 (8)	0.0254 (3)
C10	0.5680 (4)	0.69679 (14)	0.93885 (14)	0.0379 (5)
H10A	0.5624	0.7412	0.8953	0.057*
H10B	0.6984	0.6895	0.9572	0.057*
H10C	0.4902	0.7146	0.9863	0.057*

C11	0.7473 (3)	0.41612 (12)	0.67128 (13)	0.0295 (4)
H11A	0.7450	0.3999	0.7303	0.044*
H11B	0.8774	0.4159	0.6511	0.044*
H11C	0.6729	0.3746	0.6390	0.044*
C12	0.6677 (3)	0.65206 (11)	0.48211 (11)	0.0214 (3)
C13	0.6622 (2)	0.74835 (11)	0.48349 (10)	0.0201 (3)
C14	0.6640 (2)	0.79339 (11)	0.41196 (11)	0.0215 (3)
H14	0.6658	0.8546	0.4177	0.026*
C15	0.6619 (3)	0.79287 (11)	0.56742 (11)	0.0224 (3)
H15A	0.5441	0.7800	0.5966	0.034*
H15B	0.6732	0.8555	0.5594	0.034*
H15C	0.7682	0.7720	0.6007	0.034*
C16	0.6635 (3)	0.75991 (11)	0.32443 (11)	0.0245 (4)
H16A	0.6156	0.8048	0.2868	0.037*
H16B	0.5826	0.7087	0.3210	0.037*
H16C	0.7918	0.7443	0.3081	0.037*
C17	0.2362 (3)	0.38217 (12)	0.70195 (12)	0.0251 (4)
H17A	0.165 (3)	0.3387 (14)	0.6752 (13)	0.018 (5)*
H17B	0.218 (3)	0.3870 (14)	0.7618 (15)	0.028 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0248 (6)	0.0325 (6)	0.0203 (6)	−0.0019 (5)	0.0003 (5)	−0.0013 (5)
O2	0.0399 (8)	0.0341 (7)	0.0234 (6)	0.0069 (6)	0.0046 (6)	0.0021 (5)
O3	0.0268 (6)	0.0167 (5)	0.0218 (6)	0.0001 (5)	0.0052 (5)	0.0007 (5)
O4	0.0485 (8)	0.0216 (6)	0.0247 (6)	−0.0013 (6)	0.0106 (6)	−0.0019 (5)
C2	0.0347 (10)	0.0268 (8)	0.0156 (7)	−0.0025 (8)	0.0019 (7)	0.0039 (6)
C3	0.0355 (10)	0.0264 (9)	0.0214 (8)	−0.0034 (8)	−0.0035 (8)	0.0013 (7)
C3A	0.0269 (9)	0.0224 (8)	0.0219 (8)	−0.0020 (7)	−0.0054 (7)	0.0025 (7)
C4	0.0273 (9)	0.0230 (8)	0.0275 (9)	−0.0057 (7)	−0.0051 (7)	0.0015 (7)
C5	0.0221 (8)	0.0304 (9)	0.0286 (9)	−0.0043 (7)	−0.0033 (7)	0.0025 (7)
C5A	0.0206 (8)	0.0205 (8)	0.0251 (8)	0.0016 (6)	−0.0003 (7)	0.0015 (6)
C6	0.0248 (8)	0.0169 (7)	0.0261 (8)	0.0022 (6)	0.0045 (7)	0.0012 (7)
C7	0.0322 (9)	0.0173 (7)	0.0209 (8)	0.0030 (7)	0.0016 (7)	0.0002 (6)
C8	0.0279 (8)	0.0170 (7)	0.0231 (8)	0.0015 (6)	−0.0018 (7)	0.0004 (6)
C9	0.0216 (8)	0.0207 (8)	0.0230 (8)	0.0005 (7)	−0.0013 (7)	−0.0001 (6)
C9A	0.0211 (8)	0.0184 (7)	0.0188 (7)	0.0001 (6)	−0.0010 (6)	0.0019 (6)
C9B	0.0251 (9)	0.0206 (8)	0.0199 (8)	−0.0018 (7)	−0.0019 (6)	0.0027 (6)
O10	0.0308 (7)	0.0197 (6)	0.0258 (6)	0.0008 (5)	−0.0053 (5)	0.0020 (5)
C10	0.0463 (12)	0.0327 (10)	0.0348 (11)	−0.0077 (9)	0.0000 (10)	−0.0097 (9)
C11	0.0271 (9)	0.0261 (9)	0.0355 (10)	0.0064 (7)	−0.0023 (8)	0.0062 (8)
C12	0.0230 (8)	0.0204 (8)	0.0208 (8)	−0.0017 (7)	0.0022 (7)	−0.0003 (6)
C13	0.0183 (7)	0.0195 (7)	0.0225 (8)	−0.0017 (6)	0.0016 (7)	−0.0004 (6)
C14	0.0196 (8)	0.0200 (7)	0.0248 (8)	−0.0011 (6)	0.0004 (7)	−0.0002 (6)
C15	0.0239 (8)	0.0203 (7)	0.0230 (8)	−0.0013 (7)	0.0010 (7)	−0.0016 (6)
C16	0.0261 (9)	0.0261 (8)	0.0212 (8)	0.0014 (7)	0.0011 (7)	0.0007 (7)
C17	0.0234 (8)	0.0258 (8)	0.0262 (9)	−0.0036 (7)	−0.0017 (7)	−0.0006 (7)

Geometric parameters (Å, º)

O1—C2	1.349 (2)	C8—O10	1.452 (2)
O1—C9B	1.466 (2)	C8—C9	1.481 (2)
O2—C2	1.206 (2)	C8—H8	1.0000
O3—C12	1.349 (2)	C9—C17	1.341 (3)
O3—C6	1.4510 (19)	C9—C9A	1.514 (2)
O4—C12	1.211 (2)	C9A—C9B	1.527 (2)
C2—C3	1.508 (3)	C9A—H9A	1.0000
C3—C10	1.522 (3)	C9B—H9B	1.0000
C3—C3A	1.533 (3)	C10—H10A	0.9800
C3—H3	1.0000	C10—H10B	0.9800
C3A—C4	1.525 (2)	C10—H10C	0.9800
C3A—C9B	1.535 (2)	C11—H11A	0.9800
C3A—H3A	1.0000	C11—H11B	0.9800
C4—C5	1.526 (3)	C11—H11C	0.9800
C4—H4A	0.9900	C12—C13	1.489 (2)
C4—H4B	0.9900	C13—C14	1.342 (2)
C5—C5A	1.556 (2)	C13—C15	1.512 (2)
C5—H5A	0.9900	C14—C16	1.496 (2)
C5—H5B	0.9900	C14—H14	0.9500
C5A—C6	1.537 (2)	C15—H15A	0.9800
C5A—C11	1.539 (2)	C15—H15B	0.9800
C5A—C9A	1.557 (2)	C15—H15C	0.9800
C6—C7	1.521 (3)	C16—H16A	0.9800
C6—H6	1.0000	C16—H16B	0.9800
C7—O10	1.439 (2)	C16—H16C	0.9800
C7—C8	1.477 (3)	C17—H17A	0.94 (2)
C7—H7	1.0000	C17—H17B	0.97 (2)
C2—O1—C9B	110.55 (14)	C17—C9—C8	118.92 (16)
C12—O3—C6	116.01 (13)	C17—C9—C9A	126.23 (16)
O2—C2—O1	121.42 (18)	C8—C9—C9A	114.83 (15)
O2—C2—C3	128.94 (17)	C9—C9A—C9B	113.11 (14)
O1—C2—C3	109.64 (16)	C9—C9A—C5A	110.09 (14)
C2—C3—C10	113.86 (17)	C9B—C9A—C5A	113.57 (14)
C2—C3—C3A	103.43 (14)	C9—C9A—H9A	106.5
C10—C3—C3A	117.94 (17)	C9B—C9A—H9A	106.5
C2—C3—H3	107.0	C5A—C9A—H9A	106.5
C10—C3—H3	107.0	O1—C9B—C9A	105.92 (13)
C3A—C3—H3	107.0	O1—C9B—C3A	106.01 (13)
C4—C3A—C3	116.74 (15)	C9A—C9B—C3A	115.22 (14)
C4—C3A—C9B	110.96 (14)	O1—C9B—H9B	109.8
C3—C3A—C9B	101.65 (15)	C9A—C9B—H9B	109.8
C4—C3A—H3A	109.0	C3A—C9B—H9B	109.8
C3—C3A—H3A	109.0	C7—O10—C8	61.44 (11)
C9B—C3A—H3A	109.0	C3—C10—H10A	109.5
C3A—C4—C5	107.78 (15)	C3—C10—H10B	109.5

C3A—C4—H4A	110.2	H10A—C10—H10B	109.5
C5—C4—H4A	110.2	C3—C10—H10C	109.5
C3A—C4—H4B	110.2	H10A—C10—H10C	109.5
C5—C4—H4B	110.2	H10B—C10—H10C	109.5
H4A—C4—H4B	108.5	C5A—C11—H11A	109.5
C4—C5—C5A	114.37 (16)	C5A—C11—H11B	109.5
C4—C5—H5A	108.7	H11A—C11—H11B	109.5
C5A—C5—H5A	108.7	C5A—C11—H11C	109.5
C4—C5—H5B	108.7	H11A—C11—H11C	109.5
C5A—C5—H5B	108.7	H11B—C11—H11C	109.5
H5A—C5—H5B	107.6	O4—C12—O3	122.45 (16)
C6—C5A—C11	107.32 (15)	O4—C12—C13	125.87 (16)
C6—C5A—C5	109.79 (14)	O3—C12—C13	111.64 (14)
C11—C5A—C5	109.24 (15)	C14—C13—C12	120.38 (15)
C6—C5A—C9A	108.03 (14)	C14—C13—C15	121.65 (15)
C11—C5A—C9A	110.64 (14)	C12—C13—C15	117.93 (15)
C5—C5A—C9A	111.71 (14)	C13—C14—C16	128.49 (15)
O3—C6—C7	110.69 (14)	C13—C14—H14	115.8
O3—C6—C5A	107.57 (13)	C16—C14—H14	115.8
C7—C6—C5A	114.02 (14)	C13—C15—H15A	109.5
O3—C6—H6	108.1	C13—C15—H15B	109.5
C7—C6—H6	108.1	H15A—C15—H15B	109.5
C5A—C6—H6	108.1	C13—C15—H15C	109.5
O10—C7—C8	59.73 (11)	H15A—C15—H15C	109.5
O10—C7—C6	116.08 (13)	H15B—C15—H15C	109.5
C8—C7—C6	120.00 (15)	C14—C16—H16A	109.5
O10—C7—H7	116.3	C14—C16—H16B	109.5
C8—C7—H7	116.3	H16A—C16—H16B	109.5
C6—C7—H7	116.3	C14—C16—H16C	109.5
O10—C8—C7	58.83 (11)	H16A—C16—H16C	109.5
O10—C8—C9	115.17 (14)	H16B—C16—H16C	109.5
C7—C8—C9	120.57 (16)	C9—C17—H17A	122.2 (13)
O10—C8—H8	116.5	C9—C17—H17B	122.1 (14)
C7—C8—H8	116.5	H17A—C17—H17B	115.5 (19)
C9—C8—H8	116.5		

C9B—O1—C2—O2	172.91 (16)	O10—C8—C9—C9A	−52.2 (2)
C9B—O1—C2—C3	−8.26 (18)	C7—C8—C9—C9A	15.0 (2)
O2—C2—C3—C10	−28.0 (3)	C17—C9—C9A—C9B	1.8 (3)
O1—C2—C3—C10	153.32 (16)	C8—C9—C9A—C9B	−176.92 (15)
O2—C2—C3—C3A	−157.18 (18)	C17—C9—C9A—C5A	130.09 (19)
O1—C2—C3—C3A	24.10 (18)	C8—C9—C9A—C5A	−48.67 (19)
C2—C3—C3A—C4	92.11 (18)	C6—C5A—C9A—C9	65.44 (17)
C10—C3—C3A—C4	−34.6 (2)	C11—C5A—C9A—C9	−51.76 (19)
C2—C3—C3A—C9B	−28.74 (17)	C5—C5A—C9A—C9	−173.70 (14)
C10—C3—C3A—C9B	−155.42 (17)	C6—C5A—C9A—C9B	−166.57 (14)
C3—C3A—C4—C5	−179.07 (15)	C11—C5A—C9A—C9B	76.22 (19)
C9B—C3A—C4—C5	−63.29 (19)	C5—C5A—C9A—C9B	−45.71 (19)

C3A—C4—C5—C5A	55.2 (2)	C2—O1—C9B—C9A	−133.99 (14)
C4—C5—C5A—C6	118.63 (17)	C2—O1—C9B—C3A	−11.12 (17)
C4—C5—C5A—C11	−123.94 (17)	C9—C9A—C9B—O1	−78.72 (17)
C4—C5—C5A—C9A	−1.2 (2)	C5A—C9A—C9B—O1	154.87 (13)
C12—O3—C6—C7	−75.56 (18)	C9—C9A—C9B—C3A	164.45 (14)
C12—O3—C6—C5A	159.27 (14)	C5A—C9A—C9B—C3A	38.0 (2)
C11—C5A—C6—O3	−166.03 (14)	C4—C3A—C9B—O1	−99.98 (16)
C5—C5A—C6—O3	−47.40 (18)	C3—C3A—C9B—O1	24.83 (16)
C9A—C5A—C6—O3	74.64 (17)	C4—C3A—C9B—C9A	16.8 (2)
C11—C5A—C6—C7	70.81 (18)	C3—C3A—C9B—C9A	141.61 (15)
C5—C5A—C6—C7	−170.56 (14)	C6—C7—O10—C8	−111.03 (17)
C9A—C5A—C6—C7	−48.52 (18)	C9—C8—O10—C7	111.87 (18)
O3—C6—C7—O10	−36.6 (2)	C6—O3—C12—O4	8.5 (3)
C5A—C6—C7—O10	84.87 (18)	C6—O3—C12—C13	−169.25 (15)
O3—C6—C7—C8	−105.14 (17)	O4—C12—C13—C14	16.9 (3)
C5A—C6—C7—C8	16.3 (2)	O3—C12—C13—C14	−165.42 (16)
C6—C7—C8—O10	104.52 (16)	O4—C12—C13—C15	−160.91 (18)
O10—C7—C8—C9	−102.71 (17)	O3—C12—C13—C15	16.8 (2)
C6—C7—C8—C9	1.8 (2)	C12—C13—C14—C16	2.7 (3)
O10—C8—C9—C17	128.89 (18)	C15—C13—C14—C16	−179.63 (18)
C7—C8—C9—C17	−163.85 (17)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3*A*—H3*A*···O4^{i}	1.00	2.45	3.353 (2)	149
C5—H5*B*···O3	0.99	2.33	2.752 (2)	105
C8—H8···O2^{ii}	1.00	2.50	3.176 (2)	124
C9*A*—H9*A*···O3	1.00	2.58	3.010 (2)	106
C14—H14···O10^{iii}	0.95	2.57	3.423 (2)	149
C16—H16*B*···O4	0.98	2.45	2.862 (2)	105

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