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Crystal structure and Hirshfeld surface analysis of 3,3′-[ethane-1,2-diylbis(oxy)]bis(5,5-dimethylcyclohex-2-en-1-one) including an unknown solvate

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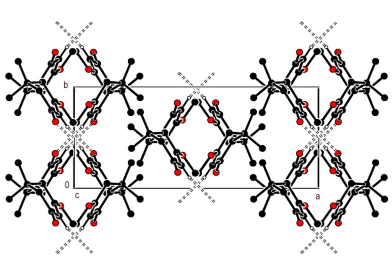
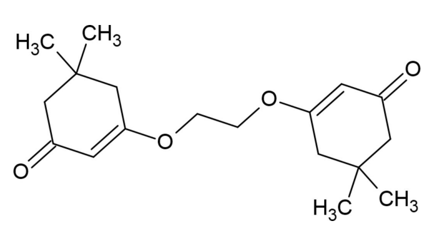
The title molecule, $C_{18}H_{26}O_4$, consists of two symmetrical halves related by the inversion centre at the mid-point of the central –C—C– bond. The hexene ring adopts an envelope conformation. In the crystal, the molecules are connected into dimers by C—H···O hydrogen bonds with $R_2^2(8)$ ring motifs, forming zigzag ribbons along the *b*-axis direction. According to a Hirshfeld surface analysis, H···H (68.2%) and O···H/H···O (25.9%) interactions are the most significant contributors to the crystal packing. The contribution of some disordered solvent to the scattering was removed using the SQUEEZE routine [Spek (2015). *Acta Cryst*. C**71**, 9–18] in *PLATON*. The solvent contribution was not included in the reported molecular weight and density.



1. Chemical context

β-Diketones have been employed as versatile synthetic precursors for the synthesis of new functional materials, such as catalysts, ionophores, heterocycles, organic conductors as well as pharmaceuticals (Abdelhamid *et al.*, 2011; Afkhami *et al.*, 2017; Khalilov *et al.*, 2021; Maharramov *et al.*, 2010; Martins *et al.*, 2017; Safavora *et al.*, 2019). For example, arylhydrazones of β-diketones have been widely used in coordination chemistry for a long time and have recently been the object of increasing attention as constituents of polydentate ligands in metallo-supramolecular chemistry (Gurbanov *et al.*, 2018, 2020; Kopylovich *et al.*, 2012*a*,*b*; Mac Leod *et al.*, 2012; Mahmoudi *et al.*, 2017*a*,*b*, 2019). The reactivity of β-diketones as enols or ketones can also be used as a synthetic strategy to access new organic materials (Yamabe *et al.*, 2004). Moreover, bridging of two β-diketone moieties into one molecule can improve their properties as well as the number of coordination and non-covalent sites (Shixaliyev *et al.*, 2019).



We have bridged two dimedone molecules into 3,3′-[ethane-1,2-diylbis(oxy)]bis(5,5-dimethylcyclohex-2-en-1-one) *via* reaction with dichloroethane, and undertaken a full characterization, including X-ray analysis.

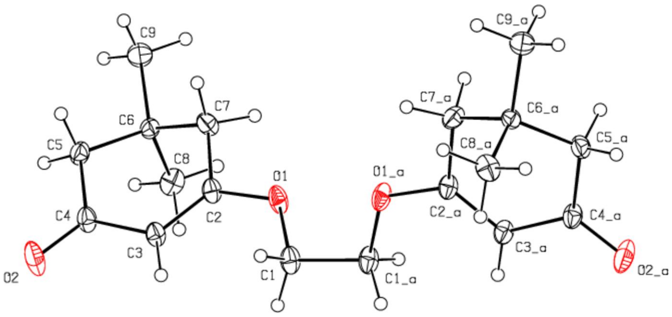


Figure 1
The molecular structure of the title compound. Displacement ellipsoids are drawn at the 30% probability level.

2. Structural commentary

The title compound (Fig. 1) consists of two symmetrical halves related by the inversion centre at the mid-point of the central –C—C– bond. The hexene ring (C2–C7) in the molecule adopts an envelope conformation [the puckering parameters (Cremer & Pople, 1975) are Q_T = 0.4488 (15) Å, θ = 127.49 (19)°, φ = 60.6 (2)°]. The geometric parameters of the title compound are normal and comparable to those of the related compound listed in the *Database survey* section.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O2[i]	0.95	2.46	3.391 (2)	168

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

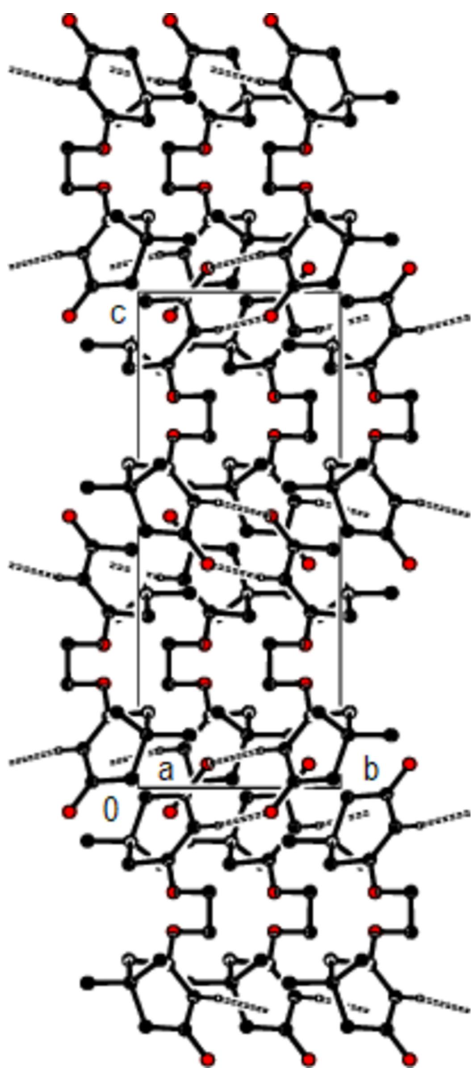


Figure 2
A partial view down the *a* axis of the C—H···O hydrogen bonds (dashed lines) in the title compound.

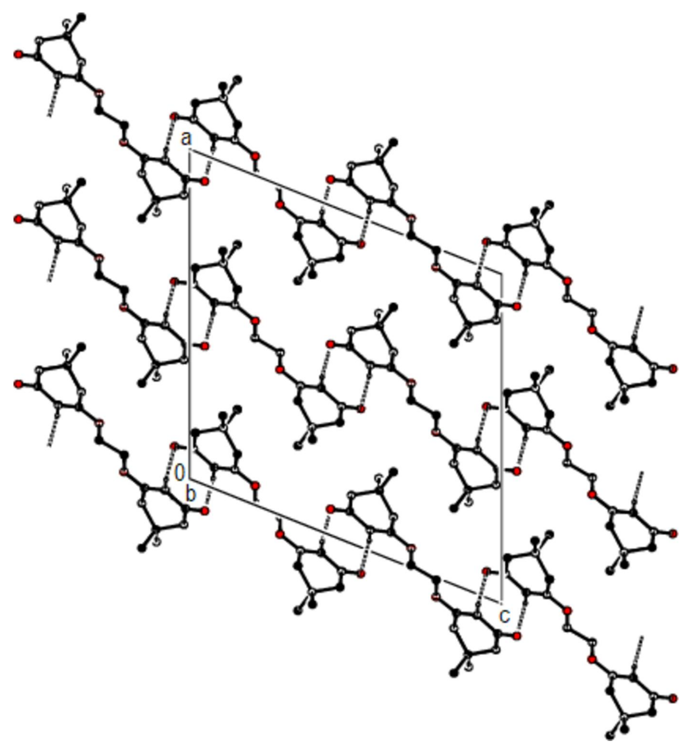


Figure 3
Partial packing of the title compound, viewed down the *b* axis, showing C—H···O hydrogen-bonded inversion-dimers with $R_2^2(8)$ graph-set motifs; H-atoms not involved in hydrogen bonds have been excluded for clarity.

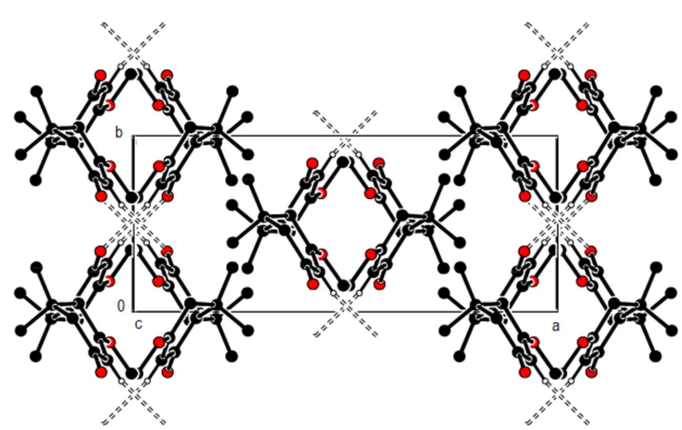


Figure 4
A partial view down the *c* axis of the C—H···O hydrogen bonds (dashed lines) in the title compound.

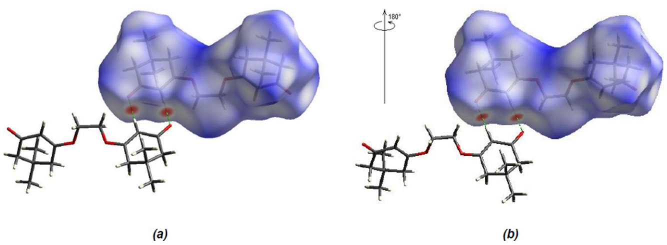


Figure 5
Front and back views of the three-dimensional Hirshfeld surfaces of the title compound.

3. Supramolecular features and Hirshfeld surface analysis

In the crystal, the molecules are connected into dimers by C—H⋯O hydrogen bonds with $R_2^2(8)$ ring motifs, forming zigzag ribbons along the *b*-axis direction (Bernstein *et al.*, 1995; Table 1; Figs. 2, 3 and 4). These ribbons are connected *via* van der Waals interactions, ensuring crystal cohesion.

In order to visualize and quantify the intermolecular interactions, a Hirshfeld surface analysis was performed using *Crystal Explorer 17.5* (Spackman *et al.*, 2021), which was also used to generate the associated two-dimensional fingerprint plots. The Hirshfeld surfaces were mapped over d_{norm} in the range −0.2098 (red) to +1.6767 (blue) a.u. (Fig. 5). The most important interatomic contact is H⋯H as it makes the highest contribution to the crystal packing (68.2%, Fig. 6*b*). The other major contributor is the O⋯H/H⋯O (25.9%, Fig. 6*c*) interaction. Other smaller contributions are made by C⋯H/H⋯C (5.5%) and O⋯O (0.4%) interactions.

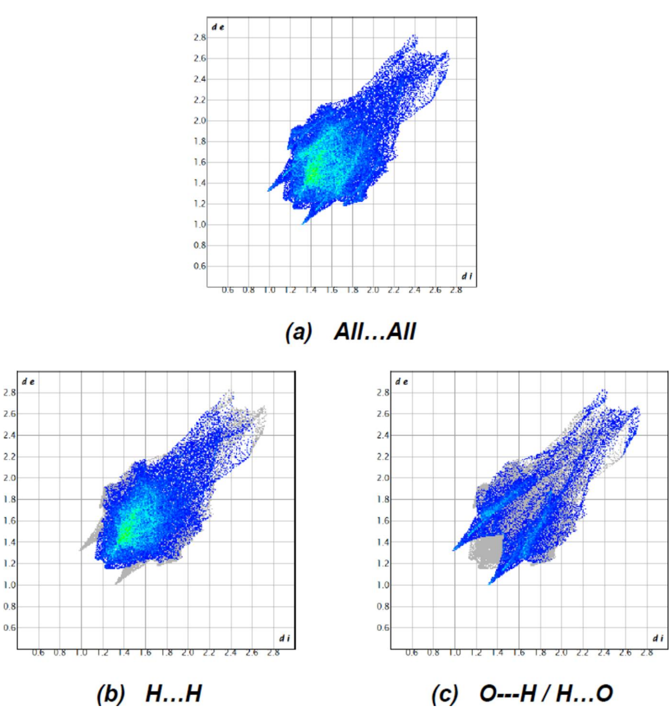


Figure 6
The full two-dimensional fingerprint plots for of the title compound, showing (*a*) all interactions, and delineated into (*b*) H⋯H and (*c*) O⋯H / H⋯O interactions. The d_i and d_e values are the closest internal and external distances (in Å) from given points on the Hirshfeld surface.

4. Database survey

A search of the Cambridge Structural Database (CSD, Version 5.43, last update November 2022; Groom *et al.*, 2016) for the six-membered *cyclohexene* ring yielded nine compounds related to the title compound, *viz*. CSD refcodes WOMWUU (Naghiyev *et al.*, 2024), UPOMOE (Naghiyev *et al.*, 2021), ZOMDUD (Gein *et al.*, 2019), PEWJUZ (Fatahpour *et al.*, 2018), OZUKAX (Tkachenko *et al.*, 2014), IFUDOD (Gein *et al.*, 2007), IWEVOV (Mohan *et al.*, 2003), IWEVUB (Mohan *et al.*, 2003) and HALROB (Ravikumar & Mehdi, 1993).

WOMWUU, UPOMOE and ZOMDUD crystallize in the monoclinic space group $P2_1/c$, with Z = 4, PEWJUZ in $I2/c$ with Z = 4, IFUDOD, HALROB and IWEVUB in $P2_1/n$ with Z = 4, and IWEVOV and OZUKAX in the orthorhombic space group *Pbca* with Z = 8. In WOMWUU, molecules are connected by intermolecular C—H⋯S hydrogen bonds with $R_2^2(10)$ ring motifs, forming ribbons along the *b*-axis direction. C—H⋯π interactions consolidate the ribbon structure while van der Waals forces between the ribbons ensure the cohesion of the crystal structure. In UPOMOE, the central cyclohexane ring adopts a chair conformation. In the crystal, molecules are linked by N—H⋯O, C—H⋯O and C—H⋯N hydrogen bonds, forming molecular layers parallel to the *bc* plane, which are connected by van der Waals interactions between them. In ZOMDUD, molecules are linked by intermolecular N—H⋯O and C—H⋯O hydrogen bonds, forming a three-dimensional network. C—H⋯π interactions are also observed. In PEWJUZ, molecules are linked by intermolecular N—H⋯O and C—H⋯O hydrogen bonds, forming sheets parallel to the *bc* plane. C—H⋯π interactions are also observed. In OZUKAX, molecules are linked by intermolecular N—H⋯O and C—H⋯O hydrogen bonds, forming sheets parallel to the *ac* plane. C—H⋯π interactions are also observed. Intermolecular O—H⋯O hydrogen bonds consolidate the crystal structure. There are no classical hydrogen bonds in the crystal of IFUDOD where intermolecular C—H⋯O contacts and weak C—H⋯π interactions lead to the formation of a three-dimensional network. In the crystal of IWEVOV, the molecules pack such that both carbonyl O atoms participate in hydrogen-bond formation with symmetry-related amide nitrogen atoms present in the carbamoyl substituents, forming N—H⋯O hydrogen bonds in a helical arrangement. In the crystal, the phenyl rings are positioned so as to favour edge-to-edge aromatic stacking. When the crystal packing is viewed normal to the *ac* plane, it reveals a 'wire-mesh' type hydrogen-bond network. In the crystal of IWEVUB, unlike in IWEVOV where both carbonyl O atoms participate in hydrogen bonding, only one of the carbonyl oxygen atoms participates in intermolecular N—H⋯O hydrogen bonding while the other carbonyl oxygen participates in a weak C—H⋯O interaction. In addition, one of the amide nitrogen atoms participates in N—H⋯O

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{18}H_{26}O_4$
M_r	306.39
Crystal system, space group	Monoclinic, *C*2/*c*
Temperature (K)	150
a, *b*, *c* (Å)	16.9184 (13), 6.5230 (5), 17.2645 (11)
β (°)	111.822 (4)
V (Å^3)	1768.8 (2)
Z	4
Radiation type	Mo *K*α
μ (mm^{-1})	0.08
Crystal size (mm)	0.33 × 0.29 × 0.18
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.966, 0.980
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10346, 2111, 1559
R_{int}	0.046
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.659
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.045, 0.118, 1.04
No. of reflections	2111
No. of parameters	102
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.26, −0.20

Computer programs: *APEX4* and *SAINT* (Bruker, 2018), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2018* (Sheldrick, 2015*b*), *ORTEP-3 for Windows* (Farrugia, 2012) and *PLATON* (Spek, 2020).

hydrogen bonding with the hydroxyl oxygen atom, linking the molecules in a helical arrangement, which is similar to that in the structure of IWEVOV. As observed in the structure of IWEVOV, the packing of the molecules viewed normal to the *ab* plane resembles a 'wire-mesh' arrangement of the molecules. In the crystal of HALROB, the amide carbonyl groups are oriented in different directions with respect to the cyclohexanone ring. These orientations of the carboxamide groups facilitate the formation of an intramolecular O—H···O hydrogen bond. The molecules are packed such that chains are formed along the *b*-axis direction. These chains are held together by N—H···O hydrogen bonds.

5. Synthesis and crystallization

0.12 mol of dichloroethane were added drop by drop to a mixture of 0.12 mol of dimedone and 0.25 mol of K_2CO_3 in 50 mL of DMSO. The reaction mixture was held for 12 h at 353 K then cooled to room temperature, water added and extracted with ethyl ether. The extract was dried with $MgSO_4$, the solvent was distilled off, and the residue was distilled under vacuum. Crystals suitable for X-ray analysis were obtained by evaporation of a dimethylformamide solution. Colourless solid (65%); m.p. 416–418 K. Analysis calculated for $C_{18}H_{26}O_4$ (M = 306.40): C 70.56, H 8.55; found: C 70.52, H 8.49%. ^{1}H NMR (300 MHz, DMSO-d_6) δ 0.99 (12H, $4CH_3$), 2.12 and 2.30 (8H, $4CH_2$), 4.16 (4H, $2CH_2$) and 5.36 (2H, 2CH). ^{13}C NMR (75 MHz, DMSO-d_6) δ 27.72 ($4CH_3$), 32.12 ($2C_{ipso}$), 41.78 ($2CH_2$), 50.25 ($2CH_2$), 66.37 ($2CH_2$), 101.44 (2CH), 175.18 (2C—O) and 197.89 (2C=O).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All H atoms were placed in calculated positions (C—H = 0.95–0.99 Å) and allowed to ride on their carrier atoms, with U_{iso}= 1.2 or $1.5U_{eq}$(C). The residual electron density was difficult to model and therefore the SQUEEZE routine (Spek, 2015) in *PLATON* (Spek, 2020) was used to remove the contribution of the electron density in the solvent region from the intensity data and the solvent-free model was employed for the final refinement. The solvent formula mass and unit-cell characteristics were not taken into account during refinement. The cavity of volume *ca* 77 Å^3 (*ca* 4.4% of the unit-cell volume) contains approximately 11 electrons. A suitable solvent with this electron number may be about four dimethylformamide molecules per unit cell.

Acknowledgements

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Crystal structure and Hirshfeld surface analysis of 3,3′-[ethane-1,2-diylbis(-oxy)]bis(5,5-dimethylcyclohex-2-en-1-one) including an unknown solvate

Nurlana D. Sadikhova, Mehmet Akkurt, Valeh M. Ismayilov, Niftali N. Yusubov, Khudayar I. Hasanov and Ajaya Bhattarai

Computing details

3,3′-[Ethane-1,2-diylbis(oxy)]bis(5,5-dimethylcyclohex-2-en-1-one)

Crystal data

$C_{18}H_{26}O_4$
M_r = 306.39
Monoclinic, $C2/c$
a = 16.9184 (13) Å
b = 6.5230 (5) Å
c = 17.2645 (11) Å
β = 111.822 (4)°
V = 1768.8 (2) Å^3
Z = 4
$F(000)$ = 664
D_x = 1.151 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2235 reflections
θ = 2.5–27.7°
μ = 0.08 mm^{-1}
T = 150 K
Prism, colourless
0.33 × 0.29 × 0.18 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.966, T_{max} = 0.980
10346 measured reflections
2111 independent reflections
1559 reflections with $I > 2\sigma(I)$
R_{int} = 0.046
θ_{max} = 27.9°, θ_{min} = 2.5°
h = −22→22
k = −8→8
l = −22→22

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.118
S = 1.04
2111 reflections
102 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
$w = 1/[\sigma^2(F_o^2) + (0.0456P)^2 + 1.1303P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ < 0.001
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
O1	0.44150 (6)	0.32631 (17)	0.28986 (6)	0.0333 (3)
O2	0.42054 (9)	0.1572 (2)	0.54805 (7)	0.0545 (4)
C6	0.30515 (9)	0.5396 (2)	0.39219 (8)	0.0252 (3)
C2	0.41732 (8)	0.3534 (2)	0.35542 (8)	0.0264 (3)
C3	0.43558 (8)	0.2261 (2)	0.42109 (8)	0.0276 (3)
H3	0.468878	0.106836	0.424213	0.033*
C7	0.36773 (9)	0.5477 (2)	0.34683 (9)	0.0311 (3)
H7A	0.335662	0.575102	0.286885	0.037*
H7B	0.407869	0.662711	0.369541	0.037*
C5	0.35360 (9)	0.4640 (2)	0.48148 (8)	0.0296 (3)
H5A	0.392750	0.573764	0.513085	0.036*
H5B	0.312185	0.439501	0.508610	0.036*
C4	0.40447 (9)	0.2706 (2)	0.48729 (8)	0.0299 (3)
C1	0.49293 (9)	0.1495 (3)	0.29054 (9)	0.0316 (4)
H1A	0.463346	0.022376	0.295832	0.038*
H1B	0.548105	0.157036	0.338214	0.038*
C8	0.23173 (9)	0.3930 (3)	0.34703 (10)	0.0350 (4)
H8A	0.201336	0.441546	0.289903	0.052*
H8B	0.192439	0.388104	0.376769	0.052*
H8C	0.254452	0.255480	0.345518	0.052*
C9	0.26825 (12)	0.7527 (3)	0.39343 (10)	0.0429 (4)
H9A	0.314317	0.847356	0.423877	0.064*
H9B	0.227197	0.745890	0.421145	0.064*
H9C	0.239567	0.801562	0.336078	0.064*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0318 (5)	0.0481 (7)	0.0280 (5)	0.0114 (5)	0.0202 (4)	0.0110 (5)
O2	0.0716 (9)	0.0690 (9)	0.0366 (6)	0.0442 (7)	0.0362 (6)	0.0280 (6)
C6	0.0289 (7)	0.0247 (7)	0.0238 (7)	0.0061 (6)	0.0121 (6)	0.0007 (5)
C2	0.0206 (6)	0.0374 (8)	0.0251 (7)	0.0023 (6)	0.0130 (5)	0.0031 (6)
C3	0.0255 (7)	0.0352 (8)	0.0259 (7)	0.0101 (6)	0.0141 (6)	0.0058 (6)
C7	0.0341 (8)	0.0309 (8)	0.0319 (7)	0.0035 (6)	0.0163 (6)	0.0082 (6)
C5	0.0342 (8)	0.0323 (8)	0.0244 (7)	0.0065 (6)	0.0134 (6)	−0.0016 (6)
C4	0.0291 (7)	0.0393 (8)	0.0237 (7)	0.0107 (6)	0.0124 (6)	0.0056 (6)
C1	0.0259 (7)	0.0468 (9)	0.0274 (7)	0.0067 (6)	0.0160 (6)	0.0036 (6)
C8	0.0247 (7)	0.0441 (9)	0.0355 (8)	0.0049 (6)	0.0106 (6)	−0.0037 (7)
C9	0.0584 (11)	0.0332 (9)	0.0406 (9)	0.0173 (8)	0.0223 (8)	0.0045 (7)

Geometric parameters (Å, º)

O1—C2	1.3507 (16)	C5—C4	1.5096 (19)
O1—C1	1.4423 (17)	C5—H5A	0.9900
O2—C4	1.2284 (17)	C5—H5B	0.9900
C6—C9	1.527 (2)	C1—C1^{i}	1.505 (3)
C6—C8	1.532 (2)	C1—H1A	0.9900
C6—C5	1.5336 (19)	C1—H1B	0.9900
C6—C7	1.5340 (19)	C8—H8A	0.9800
C2—C3	1.3454 (19)	C8—H8B	0.9800
C2—C7	1.496 (2)	C8—H8C	0.9800
C3—C4	1.4543 (18)	C9—H9A	0.9800
C3—H3	0.9500	C9—H9B	0.9800
C7—H7A	0.9900	C9—H9C	0.9800
C7—H7B	0.9900		
C2—O1—C1	117.88 (10)	C6—C5—H5B	108.6
C9—C6—C8	108.50 (12)	H5A—C5—H5B	107.6
C9—C6—C5	110.31 (11)	O2—C4—C3	121.41 (13)
C8—C6—C5	109.89 (12)	O2—C4—C5	119.97 (12)
C9—C6—C7	109.84 (12)	C3—C4—C5	118.59 (12)
C8—C6—C7	110.15 (11)	O1—C1—C1^{i}	107.24 (10)
C5—C6—C7	108.15 (11)	O1—C1—H1A	110.3
C3—C2—O1	125.39 (13)	C1^{i}—C1—H1A	110.3
C3—C2—C7	123.44 (12)	O1—C1—H1B	110.3
O1—C2—C7	111.17 (11)	C1^{i}—C1—H1B	110.3
C2—C3—C4	120.14 (13)	H1A—C1—H1B	108.5
C2—C3—H3	119.9	C6—C8—H8A	109.5
C4—C3—H3	119.9	C6—C8—H8B	109.5
C2—C7—C6	112.86 (11)	H8A—C8—H8B	109.5
C2—C7—H7A	109.0	C6—C8—H8C	109.5
C6—C7—H7A	109.0	H8A—C8—H8C	109.5
C2—C7—H7B	109.0	H8B—C8—H8C	109.5
C6—C7—H7B	109.0	C6—C9—H9A	109.5
H7A—C7—H7B	107.8	C6—C9—H9B	109.5
C4—C5—C6	114.46 (11)	H9A—C9—H9B	109.5
C4—C5—H5A	108.6	C6—C9—H9C	109.5
C6—C5—H5A	108.6	H9A—C9—H9C	109.5
C4—C5—H5B	108.6	H9B—C9—H9C	109.5
C1—O1—C2—C3	−1.6 (2)	C9—C6—C5—C4	−170.57 (13)
C1—O1—C2—C7	177.57 (12)	C8—C6—C5—C4	69.86 (16)
O1—C2—C3—C4	−178.91 (13)	C7—C6—C5—C4	−50.42 (16)
C7—C2—C3—C4	2.0 (2)	C2—C3—C4—O2	−179.70 (15)
C3—C2—C7—C6	−28.0 (2)	C2—C3—C4—C5	−1.5 (2)
O1—C2—C7—C6	152.85 (12)	C6—C5—C4—O2	−154.38 (15)
C9—C6—C7—C2	170.39 (13)	C6—C5—C4—C3	27.4 (2)

C8—C6—C7—C2	−70.16 (15)	C2—O1—C1—C1^{i}	177.15 (12)
C5—C6—C7—C2	49.95 (16)		

Symmetry code: (i) −*x*+1, *y*, −*z*+1/2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O2^{ii}	0.95	2.46	3.391 (2)	168

Symmetry code: (ii) −*x*+1, −*y*, −*z*+1.