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Crystal structure and Hirshfeld surface analysis of ethyl 2-[9-(2-hydroxyphenyl)-3,3,6,6-tetramethyl-1,8-dioxo-2,3,4,4a,5,6,7,8a,9,9a,10,10a-dodecahydroacridin-10-yl]acetate

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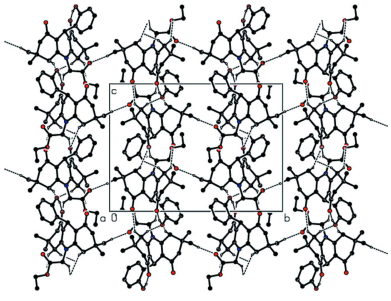
In the title compound, $C_{27}H_{33}NO_5$, a 3,3,6,6-tetramethyltetrahydroacridine-1,8-dione ring system carries an ethyl acetate substituent on the acridine N atom and an *o*-hydroxyphenyl ring on the central methine C atom of the dihydropyridine ring. The benzene ring is inclined to the acridine ring system at an angle of 80.45 (7)° and this conformation is stabilized by an intramolecular O—H···O hydrogen bond between the hydroxy substituent on the benzene ring and one of the carbonyl groups of the acridinedione unit. The ester C═O oxygen atom is disordered over major and minor orientations in a 0.777 (9):0.223 (9) ratio and the terminal $-CH_3$ unit of the ethyl side chain is disordered over two sets of sites in a 0.725 (5): 0.275 (5) ratio. In the crystal, C—H···O hydrogen bonds combine to link the molecules into a three-dimensional network. van der Waals H···H contacts contribute the most to the Hirshfeld surface (66.9%) followed by O···H/H···O (22.1%) contacts associated with weak hydrogen bonds.

1. Chemical context

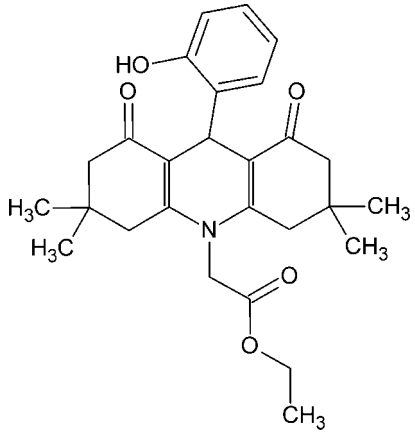
Acridine derivatives occur in a number of compounds of importance in medicinal chemistry such as bucricaine, which used for surface anesthesia of the eye and given by injection for infiltration anesthesia, peripheral nerve block and spinal anesthesia (Ramesh *et al.*, 2012). Quinacrine, also known as mepacrine, is used as a gametocytocide and acts as an antimalarial agent (Valdés, 2011). Proflavin is also found to be active as a bacteriostatic agent (Patel *et al.*, 2010) and nitracrine is as anticancer agent (Cholewinski *et al.*, 2011). Acriflavin is used as an antiseptic for skin and mucous membranes (Ramesh *et al.*, 2012). As part of our studies in this area, we report herein the synthesis and crystal structure of the title compound, $C_{27}H_{33}NO_5$.



2. Structural commentary

As shown in Fig.1, the 3,3,6,6-tetramethyltetrahydroacridine-1,8-dione ring system carries an ethyl acetate substituent on the acridine N1 atom and an *o*-hydroxyphenyl ring on the central methine C7 atom of the C1/C6–C8/C13/N1 dihydropyridine ring. The acridinedione ring system deviates signifi-

cantly from planarity with an r.m.s. deviation of 0.404 Å for the thirteen C atoms and one N atom of the acridine unit. The benzene ring is inclined to the acridine ring system at a dihedral angle of 80.45 (7)°.



The outer C1–C6 and C8–C13 cyclohexenone rings both adopt flattened chair conformations with the C4 and C11 atoms displaced in the same direction, by 0.308 (2) and 0.338 (2) Å, respectively, from the best-fit planes through the remaining five C atoms. In contrast, the central C13/N1/C1/C6–C8 ring can best be described as a flattened boat with N1 and C7 displaced by 0.146 (1) and 0.191 (14) Å, respectively, from the remaining four C atoms. The bond lengths and angles in the title molecule agree reasonably well with those found in closely related molecules (Abdelhamid *et al.*, 2011, 2014; Khalilov *et al.*, 2011).

The molecular conformation of the title compound is stabilized by an intramolecular O5—H5···O1 hydrogen bond between the hydroxy substituent on the benzene ring and one of the carbonyl groups of the acridinedione unit (Table 1; Fig. 1). Atom O3 is disordered over major and minor orientations in a 0.777 (9):0.223 (9) ratio and the terminal C17 methyl group is disordered over two sets of sites in a 0.725 (5):0.275 (5) ratio.

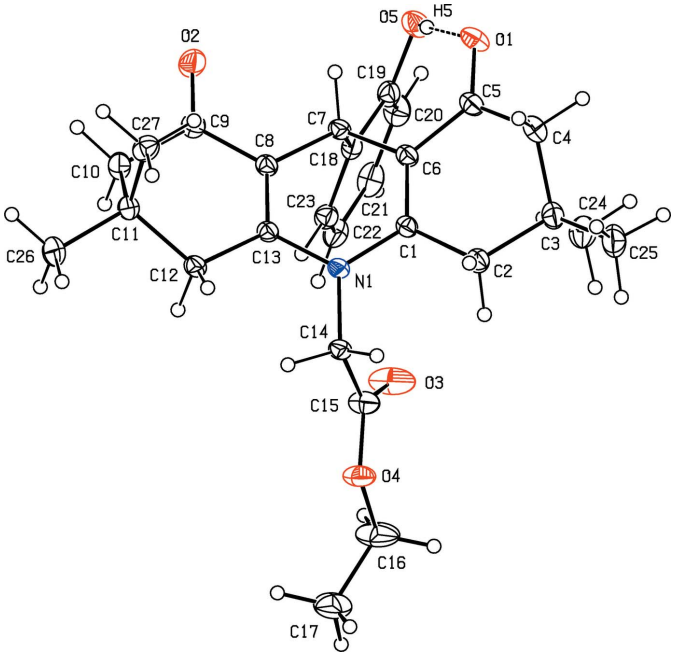


Figure 1
The title molecule with displacement ellipsoids drawn at the 30% probability level. Only the major disorder components for O3 and C17 are shown.

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

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5···O1	0.84	1.81	2.6319 (17)	166
C2—H2A···O2^{i}	0.99	2.52	3.1663 (19)	123
C2—H2B···O5^{i}	0.99	2.53	3.457 (2)	157
C12—H12B···O1^{ii}	0.99	2.52	3.2708 (17)	133
C14—H14A···O1^{ii}	0.99	2.53	3.3299 (18)	138
C14—H14B···O2^{i}	0.99	2.66	3.473 (2)	140
C27—H27B···O3^{iii}	0.98	2.51	3.452 (3)	161

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

3. Supramolecular features

In the crystal, a number of C—H···O hydrogen bonds link the molecules into a three-dimensional network (Table 1; Fig. 2); all the oxygen atoms in the molecule except O4 accept at least one of these bonds.

4. Hirshfeld surface analysis

The *CrystalExplorer* software (Wolff *et al.*, 2012) was used to produce the d_{norm}-mapped Hirshfeld surfaces and the electrostatic potential for the title compound. The contact distances, d_i and d_e, from the Hirshfeld surface to the nearest atom, inside and outside, respectively, enable the analysis of the intermolecular interactions through the mapping of d_{norm}.

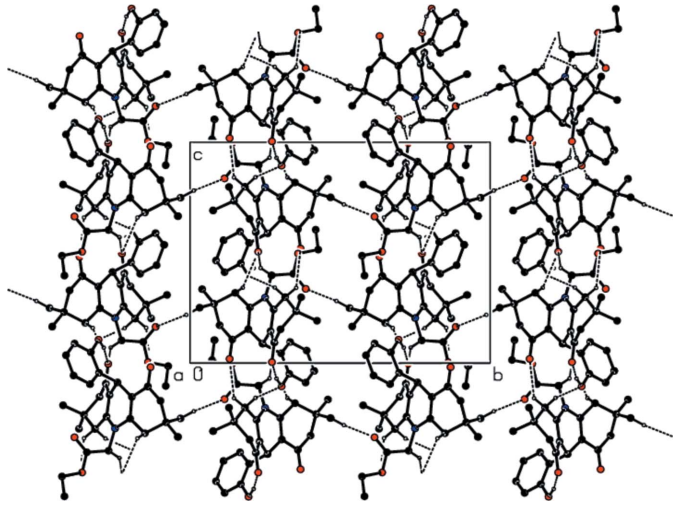


Figure 2
The molecular packing, viewed down the *a*-axis direction, showing hydrogen bonds as dashed lines.

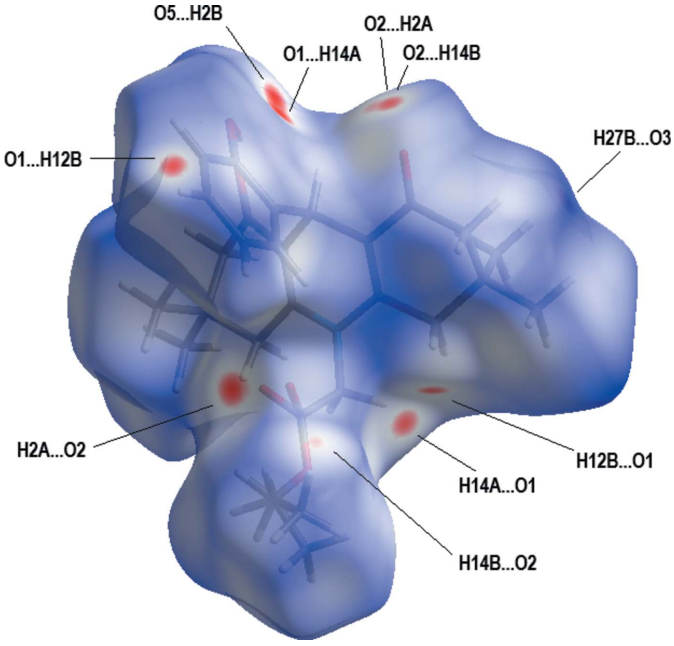


Figure 3
A view of the three-dimensional Hirshfeld surface for the title compound, plotted over d_{norm} in the range −0.14 to 1.68 a.u.

An illustration of the inter-molecular contacts in the crystal is given by two-dimensional fingerprint plots.

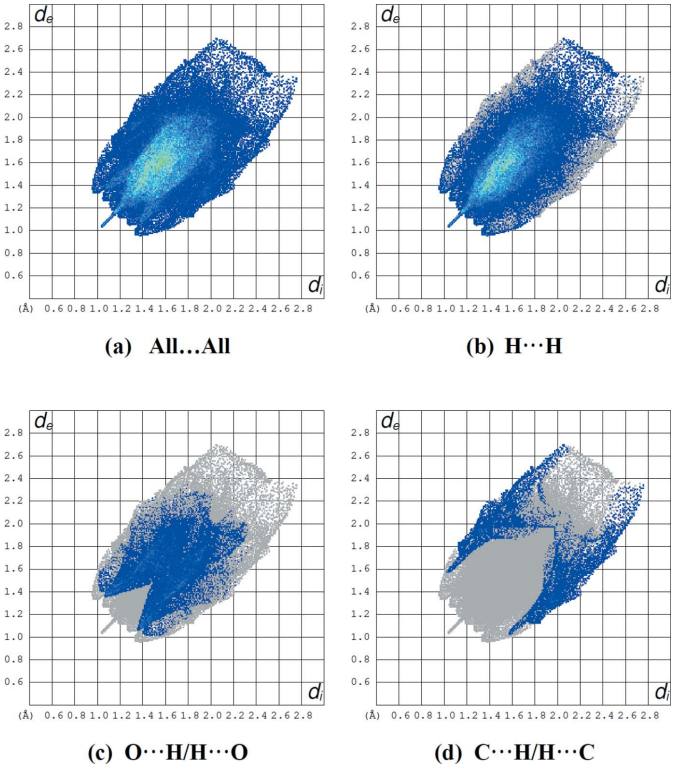


Figure 4
A view of the two-dimensional fingerprint plots for the title compound, showing (*a*) all interactions, and delineated into (*b*) H···H, (*c*) O···H/H···O and (*d*) C···H/H···C interactions. The d_i and d_e values are the closest internal and external distances (in Å) from given points on the Hirshfeld surface.

Table 2
Short H···H interatomic contacts (Å) in the title compound.

Contact	Distance	Symmetry operation
H21···H27*A*	2.26	$-\frac{1}{2}+x, \frac{1}{2}-y, -\frac{1}{2}+z$
H22···H27*A*	2.46	$\frac{1}{2}-x, -\frac{1}{2}+y, \frac{3}{2}-z$
H22···H4*B*	2.43	$-1+x, y, z$

The bright-red spots on the Hirshfeld surface mapped over d_{norm} (Fig. 3), with labels H27*B*, H12*B*, H14*A*, H14*B*, H2*A* and H2*B* on the surface represent donors for potential C—H···O hydrogen bonds (see Table 1); the corresponding acceptors on the surface appear as bright-red spots at atoms O1, O2 and O5. Short H···H contacts are given in Table 2.

The overall two-dimensional fingerprint plot is illustrated in Fig. 4*a*, and those delineated into H···H, O···H/H···O and C···H/H···C in Fig. 4*b–d*, respectively. The greatest contribution to the overall Hirshfeld surface, *i.e.* 66.9%, is due to H···H contacts (Fig. 4*b*). The relative contributions of the other interactions in descending order are: O···H/H···O (22.1%), C···H/H···C (9.2%), O···O (1.3%), N···H/H···N (0.2%) and N···C/C···N (0.2%). This illustrates that the C—H···O interactions contribute significantly to the crystal packing.

5. Database survey

Compounds similar to the title compound with a octahydroacridin moiety are [9-(2-hydroxyphenyl)-1,8-dioxo-2,3,4,5,6,7,8,9-octahydroacridin-10(1*H*)-yl]acetic acid [Cambridge Structural Database (Groom *et al.*, 2016)] refcode DABSAD; Akkurt *et al.*, 2015), ethyl [9-(5-bromo-2-hydroxyphenyl)-3,3,6,6-tetramethyl-1,8-dioxo-2,3,4,5,6,7,8,9-octahydroacridin-10(1*H*)-yl]acetate (VANBUK; Mohamed *et al.*, 2017), 9-(3-bromo-5-chloro-2-hydroxyphenyl)-10-(2-hydroxyethyl)-3,6-diphenyl-3,4,6,7,9,10-hexahydroacridine-1,8(2*H*,5*H*)-dione (SILBIB; Abdelhamid *et al.*, 2018) and 10-benzyl-9-(3,4-dimethoxyphenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2*H*,5*H*)-dione (PUSJEU; Sureshbabu *et al.*, 2015).

The DABSAD compound crystallizes with two molecules in the asymmetric unit. In each molecule, the central 1,4-dihydropyridine ring adopts a shallow sofa conformation (with the C atom bearing the phenol ring as the flap), whereas the pendant cyclohexene rings both have twisted-boat conformations. Each molecule features an intramolecular O—H···O hydrogen bond, which closes an *S*(8) ring. In the crystal, the molecules are linked by O—H···O, C—H···O and C—H···π interactions, forming a three-dimensional network. In VANBUK, the central 1,4-dihydropyridine ring adopts a shallow sofa conformation (with the C atom bearing the bromophenol ring as the flap), whereas the pendant cyclohexene rings both have twisted-boat conformations. The molecule features an intramolecular O—H···O hydrogen bond, which closes an *S*(8) ring. In the crystal, molecules are linked by C—H···O interactions, forming *C*(12) chains propagating along the *c*-axis direction. In the crystal of

Table 3
Experimental details.

Crystal data	
Chemical formula	$C_{27}H_{33}NO_5$
M_r	451.54
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	173
a, b, c (Å)	9.5289 (2), 18.6653 (5), 13.8046 (3)
β (°)	96.410 (2)
V (Å^3)	2439.93 (10)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.68
Crystal size (mm)	0.48 × 0.22 × 0.08
Data collection	
Diffractometer	Rigaku Oxford Diffraction EOS
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2015)
T_{min}, T_{max}	0.861, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	9527, 4648, 3949
R_{int}	0.029
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.614
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.045, 0.126, 1.04
No. of reflections	4648
No. of parameters	313
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.27, −0.23

Computer programs: *CrysAlis PRO* (Rigaku OD, 2015), *SHELXT* (Sheldrick, 2015b), *SHELXL* (Sheldrick, 2015a) and *OLEX2* (Dolomanov *et al.*, 2009).

SILBIB, O—H···O, C—H···O and C—H···π(ring) hydrogen bonds combine with an Br—O and unusual C—Br···π(ring) halogen bonds to generate a three dimensional network with molecules stacked along the *a*-axis direction. In the acridinedione moiety of PUSJEU, the central dihydropyridine ring adopts a flattened-boat conformation, with the N atom and the methine C atom displaced from the mean plane of the other four atoms by 0.0513 (14) and 0.1828 (18) Å, respectively. The two cyclohexenone rings adopt envelope conformations, with the tetrasubsituted C atoms as the flap atoms. In the crystal, molecules are linked *via* a pair of C—H···O hydrogen bonds, forming inversion dimers, which are, in turn, linked by C—H···O hydrogen bonds, forming slabs lying parallel to (001).

6. Synthesis and crystallization

To a mixture of dimedone (1.12 g, 0.008 mol), ethyl glycinate hydrochloride (0.56 g, 0.004 mol) and salicaldehyde (0.43 ml, 0.004 mol) in ethanol (20 ml), triethyl amine (1.12 ml, 0.008 mol) was added. The reaction mixture was heated under reflux for 5 h at 353–358 K then left to cool. The separated solid was filtered off, dried and recrystallized from ethanol solution as yellow plates of the title compound, yield 68%, m.p. 497 K.

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. All H atoms were placed in idealized locations and and refined using a riding model with C—H = 0.9–1.00 Å U_{iso}(H) = 1.2U_{eq} (C) and O—H = 0.84 Å, U_{iso}(H) = 1.5U_{eq} (O). Atom O3 of the oxo group and terminal methyl group (C17) of the ethyl acetate substituent are disordered over two sites in 0.777 (9):0.223 (9) (for O3 and O3*A*) and 0.725 (5):0.275 (5) (for C17 and C17*A*) ratios, respectively.

Funding information

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supporting information

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Crystal structure and Hirshfeld surface analysis of ethyl 2-[9-(2-hydroxyphenyl)-3,3,6,6-tetramethyl-1,8-dioxo-2,3,4,4a,5,6,7,8a,9,9a,10,10a-dodecahydroacridin-10-yl]acetate

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Computing details

Data collection: *CrysAlis PRO* (Rigaku OD, 2015); cell refinement: *CrysAlis PRO* (Rigaku OD, 2015); data reduction: *CrysAlis PRO* (Rigaku OD, 2015); program(s) used to solve structure: ShelXT (Sheldrick, 2015b); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015a); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009).

Ethyl 2-[9-(2-hydroxyphenyl)-3,3,6,6-tetramethyl-1,8-dioxo-2,3,4,4a,5,6,7,8a,9,9a,10,10a-dodecahydroacridin-10-yl]acetate

Crystal data

$C_{27}H_{33}NO_5$
M_r = 451.54
Monoclinic, $P2_1/n$
a = 9.5289 (2) Å
b = 18.6653 (5) Å
c = 13.8046 (3) Å
β = 96.410 (2)°
V = 2439.93 (10) Å^3
Z = 4
$F(000)$ = 968
D_x = 1.229 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 3784 reflections
θ = 4.0–71.4°
μ = 0.68 mm^{-1}
T = 173 K
Plate, yellow
0.48 × 0.22 × 0.08 mm

Data collection

Rigaku Oxford Diffraction EOS diffractometer
Radiation source: fine-focus sealed X-ray tube, Enhance (Cu) X-ray Source
Graphite monochromator
Detector resolution: 16.0416 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysalisPro; Rigaku OD, 2015)
T_{min} = 0.861, T_{max} = 1.000
9527 measured reflections
4648 independent reflections
3949 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 71.2°, θ_{min} = 4.0°
h = −11→11
k = −22→14
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.126
S = 1.04
4648 reflections
313 parameters
0 restraints

Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0643P)^2 + 0.6477P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ 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}	Occ. (<1)
O1	0.65518 (12)	0.22713 (7)	0.50403 (8)	0.0373 (3)	
O2	0.21715 (14)	0.36821 (7)	0.51857 (8)	0.0426 (3)	
O3	0.3445 (5)	0.11473 (19)	0.83632 (16)	0.0636 (11)	0.777 (9)
O3A	0.2813 (17)	0.1388 (6)	0.8394 (7)	0.0636 (11)	0.223 (9)
O4	0.35606 (14)	0.14006 (6)	0.99586 (9)	0.0406 (3)	
O5	0.41985 (13)	0.19489 (7)	0.39430 (8)	0.0389 (3)	
H5	0.4887	0.2122	0.4300	0.058*	
N1	0.44568 (13)	0.24991 (6)	0.79603 (8)	0.0236 (2)	
C1	0.54686 (14)	0.22154 (7)	0.74277 (10)	0.0228 (3)	
C2	0.66871 (16)	0.18305 (8)	0.79873 (11)	0.0292 (3)	
H2A	0.6309	0.1464	0.8407	0.035*	
H2B	0.7234	0.2179	0.8419	0.035*	
C3	0.76892 (16)	0.14645 (9)	0.73461 (12)	0.0328 (3)	
C4	0.79512 (16)	0.19849 (9)	0.65285 (12)	0.0356 (4)	
H4A	0.8424	0.2421	0.6814	0.043*	
H4B	0.8587	0.1758	0.6098	0.043*	
C5	0.65908 (16)	0.21917 (8)	0.59366 (11)	0.0286 (3)	
C6	0.53718 (15)	0.23162 (8)	0.64433 (10)	0.0245 (3)	
C7	0.40195 (15)	0.25806 (8)	0.58758 (10)	0.0244 (3)	
H7	0.4283	0.2880	0.5323	0.029*	
C8	0.32755 (14)	0.30588 (7)	0.65354 (10)	0.0237 (3)	
C9	0.22950 (16)	0.35939 (8)	0.60657 (11)	0.0291 (3)	
C10	0.14223 (16)	0.40152 (9)	0.67124 (12)	0.0323 (3)	
H10A	0.0581	0.3731	0.6833	0.039*	
H10B	0.1091	0.4463	0.6376	0.039*	
C11	0.22671 (16)	0.42010 (8)	0.76873 (11)	0.0280 (3)	
C12	0.27846 (15)	0.34965 (8)	0.81805 (10)	0.0263 (3)	
H12A	0.3461	0.3608	0.8758	0.032*	
H12B	0.1970	0.3245	0.8411	0.032*	
C13	0.34886 (14)	0.30040 (7)	0.75185 (10)	0.0222 (3)	
C14	0.43949 (15)	0.22835 (8)	0.89738 (10)	0.0256 (3)	
H14A	0.3858	0.2646	0.9304	0.031*	
H14B	0.5365	0.2265	0.9315	0.031*	
C15	0.37019 (19)	0.15605 (9)	0.90423 (12)	0.0368 (4)	

C16	0.2856 (3)	0.07304 (12)	1.01388 (17)	0.0641 (7)	
H16A	0.3553	0.0335	1.0218	0.077*	0.725 (5)
H16B	0.2146	0.0615	0.9581	0.077*	0.725 (5)
H16C	0.2872	0.0420	0.9559	0.077*	0.275 (5)
H16D	0.3405	0.0485	1.0693	0.077*	0.275 (5)
C17	0.2157 (4)	0.08124 (19)	1.1040 (3)	0.0665 (9)	0.725 (5)
H17A	0.2875	0.0880	1.1598	0.100*	0.725 (5)
H17B	0.1604	0.0381	1.1141	0.100*	0.725 (5)
H17C	0.1530	0.1230	1.0976	0.100*	0.725 (5)
C17A	0.1551 (11)	0.0790 (5)	1.0331 (8)	0.0665 (9)	0.275 (5)
H17D	0.1499	0.1142	1.0853	0.100*	0.275 (5)
H17E	0.1217	0.0324	1.0541	0.100*	0.275 (5)
H17F	0.0956	0.0947	0.9745	0.100*	0.275 (5)
C18	0.30828 (15)	0.19663 (8)	0.54418 (10)	0.0258 (3)	
C19	0.32270 (16)	0.16932 (9)	0.45105 (11)	0.0307 (3)	
C20	0.23375 (19)	0.11504 (10)	0.41156 (13)	0.0405 (4)	
H20	0.2428	0.0976	0.3479	0.049*	
C21	0.13242 (18)	0.08647 (9)	0.46441 (14)	0.0410 (4)	
H21	0.0723	0.0493	0.4371	0.049*	
C22	0.11833 (17)	0.11164 (9)	0.55659 (13)	0.0356 (4)	
H22	0.0494	0.0916	0.5933	0.043*	
C23	0.20564 (16)	0.16652 (8)	0.59556 (11)	0.0295 (3)	
H23	0.1949	0.1839	0.6590	0.035*	
C24	0.7045 (2)	0.07682 (10)	0.69143 (15)	0.0469 (4)	
H24A	0.6894	0.0436	0.7443	0.070*	
H24B	0.7688	0.0550	0.6493	0.070*	
H24C	0.6139	0.0872	0.6531	0.070*	
C25	0.9082 (2)	0.12973 (11)	0.79789 (14)	0.0468 (4)	
H25A	0.9533	0.1746	0.8214	0.070*	
H25B	0.9713	0.1036	0.7590	0.070*	
H25C	0.8887	0.1003	0.8537	0.070*	
C26	0.13320 (19)	0.45952 (9)	0.83469 (13)	0.0405 (4)	
H26A	0.0495	0.4305	0.8424	0.061*	
H26B	0.1038	0.5057	0.8052	0.061*	
H26C	0.1864	0.4677	0.8987	0.061*	
C27	0.35163 (17)	0.46822 (8)	0.75087 (12)	0.0340 (3)	
H27A	0.4061	0.4799	0.8133	0.051*	
H27B	0.3163	0.5125	0.7187	0.051*	
H27C	0.4123	0.4431	0.7092	0.051*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0375 (6)	0.0503 (7)	0.0266 (6)	−0.0016 (5)	0.0144 (4)	−0.0032 (5)
O2	0.0548 (7)	0.0469 (7)	0.0249 (6)	0.0155 (6)	−0.0006 (5)	0.0006 (5)
O3	0.114 (3)	0.0437 (16)	0.0360 (8)	−0.0364 (17)	0.0192 (12)	−0.0122 (10)
O3A	0.114 (3)	0.0437 (16)	0.0360 (8)	−0.0364 (17)	0.0192 (12)	−0.0122 (10)
O4	0.0571 (7)	0.0352 (6)	0.0317 (6)	−0.0098 (5)	0.0158 (5)	0.0055 (5)

O5	0.0448 (6)	0.0485 (7)	0.0245 (5)	0.0025 (5)	0.0080 (5)	−0.0075 (5)
N1	0.0288 (6)	0.0242 (6)	0.0189 (5)	0.0020 (5)	0.0069 (4)	−0.0005 (4)
C1	0.0248 (6)	0.0203 (6)	0.0241 (7)	−0.0020 (5)	0.0064 (5)	−0.0023 (5)
C2	0.0305 (7)	0.0316 (7)	0.0257 (7)	0.0041 (6)	0.0035 (6)	−0.0014 (6)
C3	0.0324 (8)	0.0323 (8)	0.0344 (8)	0.0071 (6)	0.0065 (6)	−0.0027 (6)
C4	0.0290 (8)	0.0417 (9)	0.0382 (9)	0.0041 (6)	0.0125 (6)	−0.0014 (7)
C5	0.0309 (7)	0.0280 (7)	0.0287 (7)	−0.0025 (6)	0.0109 (6)	−0.0038 (6)
C6	0.0269 (7)	0.0244 (7)	0.0233 (7)	−0.0005 (5)	0.0073 (5)	−0.0025 (5)
C7	0.0303 (7)	0.0246 (7)	0.0190 (6)	0.0010 (6)	0.0065 (5)	−0.0012 (5)
C8	0.0260 (6)	0.0226 (7)	0.0233 (7)	−0.0010 (5)	0.0059 (5)	−0.0021 (5)
C9	0.0312 (7)	0.0294 (7)	0.0261 (7)	0.0006 (6)	0.0010 (6)	−0.0025 (6)
C10	0.0304 (7)	0.0318 (8)	0.0345 (8)	0.0063 (6)	0.0028 (6)	−0.0016 (6)
C11	0.0327 (7)	0.0243 (7)	0.0277 (7)	0.0043 (6)	0.0072 (6)	−0.0017 (6)
C12	0.0323 (7)	0.0241 (7)	0.0236 (7)	0.0020 (6)	0.0088 (5)	−0.0016 (5)
C13	0.0245 (6)	0.0195 (6)	0.0232 (7)	−0.0018 (5)	0.0064 (5)	−0.0010 (5)
C14	0.0325 (7)	0.0267 (7)	0.0187 (6)	0.0019 (6)	0.0068 (5)	0.0003 (5)
C15	0.0492 (9)	0.0353 (8)	0.0269 (8)	−0.0090 (7)	0.0085 (6)	0.0001 (6)
C16	0.0987 (18)	0.0460 (12)	0.0520 (12)	−0.0294 (12)	0.0280 (12)	0.0052 (9)
C17	0.082 (2)	0.0543 (16)	0.070 (2)	−0.0088 (15)	0.0384 (17)	0.0188 (17)
C17A	0.082 (2)	0.0543 (16)	0.070 (2)	−0.0088 (15)	0.0384 (17)	0.0188 (17)
C18	0.0286 (7)	0.0242 (7)	0.0239 (7)	0.0051 (5)	−0.0003 (5)	−0.0014 (5)
C19	0.0338 (8)	0.0323 (8)	0.0256 (7)	0.0080 (6)	0.0015 (6)	−0.0029 (6)
C20	0.0479 (9)	0.0391 (9)	0.0326 (8)	0.0063 (7)	−0.0039 (7)	−0.0137 (7)
C21	0.0388 (9)	0.0307 (8)	0.0503 (10)	0.0005 (7)	−0.0097 (7)	−0.0093 (7)
C22	0.0344 (8)	0.0281 (8)	0.0434 (9)	0.0008 (6)	−0.0001 (7)	0.0019 (7)
C23	0.0328 (7)	0.0274 (7)	0.0280 (7)	0.0026 (6)	0.0021 (6)	−0.0002 (6)
C24	0.0588 (11)	0.0312 (9)	0.0514 (11)	0.0062 (8)	0.0094 (8)	−0.0059 (8)
C25	0.0405 (10)	0.0533 (11)	0.0463 (10)	0.0185 (8)	0.0037 (8)	−0.0019 (8)
C26	0.0469 (10)	0.0344 (8)	0.0418 (9)	0.0118 (7)	0.0124 (7)	−0.0053 (7)
C27	0.0409 (8)	0.0247 (7)	0.0363 (8)	−0.0019 (6)	0.0034 (6)	0.0005 (6)

Geometric parameters (Å, º)

O1—C5	1.2426 (19)	C12—C13	1.5057 (18)
O2—C9	1.2185 (19)	C14—H14A	0.9900
O3—C15	1.217 (3)	C14—H14B	0.9900
O3A—C15	1.205 (11)	C14—C15	1.510 (2)
O4—C15	1.321 (2)	C16—H16A	0.9900
O4—C16	1.454 (2)	C16—H16B	0.9900
O5—H5	0.8400	C16—H16C	0.9900
O5—C19	1.364 (2)	C16—H16D	0.9900
N1—C1	1.3816 (18)	C16—C17	1.483 (4)
N1—C13	1.4091 (18)	C16—C17A	1.305 (9)
N1—C14	1.4633 (17)	C17—H17A	0.9800
C1—C2	1.504 (2)	C17—H17B	0.9800
C1—C6	1.365 (2)	C17—H17C	0.9800
C2—H2A	0.9900	C17A—H17D	0.9800
C2—H2B	0.9900	C17A—H17E	0.9800

C2—C3	1.533 (2)	C17A—H17F	0.9800
C3—C4	1.531 (2)	C18—C19	1.404 (2)
C3—C24	1.530 (2)	C18—C23	1.389 (2)
C3—C25	1.537 (2)	C19—C20	1.392 (2)
C4—H4A	0.9900	C20—H20	0.9500
C4—H4B	0.9900	C20—C21	1.381 (3)
C4—C5	1.504 (2)	C21—H21	0.9500
C5—C6	1.4401 (19)	C21—C22	1.377 (3)
C6—C7	1.514 (2)	C22—H22	0.9500
C7—H7	1.0000	C22—C23	1.390 (2)
C7—C8	1.5074 (18)	C23—H23	0.9500
C7—C18	1.5334 (19)	C24—H24A	0.9800
C8—C9	1.468 (2)	C24—H24B	0.9800
C8—C13	1.354 (2)	C24—H24C	0.9800
C9—C10	1.507 (2)	C25—H25A	0.9800
C10—H10A	0.9900	C25—H25B	0.9800
C10—H10B	0.9900	C25—H25C	0.9800
C10—C11	1.529 (2)	C26—H26A	0.9800
C11—C12	1.537 (2)	C26—H26B	0.9800
C11—C26	1.532 (2)	C26—H26C	0.9800
C11—C27	1.533 (2)	C27—H27A	0.9800
C12—H12A	0.9900	C27—H27B	0.9800
C12—H12B	0.9900	C27—H27C	0.9800

C15—O4—C16	117.16 (15)	O3—C15—O4	124.13 (18)
C19—O5—H5	109.5	O3—C15—C14	124.71 (17)
C1—N1—C13	119.22 (11)	O3A—C15—O4	120.8 (5)
C1—N1—C14	120.56 (12)	O3A—C15—C14	117.9 (5)
C13—N1—C14	120.23 (11)	O4—C15—C14	110.73 (13)
N1—C1—C2	117.03 (12)	O4—C16—H16A	110.0
C6—C1—N1	120.23 (13)	O4—C16—H16B	110.0
C6—C1—C2	122.68 (13)	O4—C16—H16C	108.4
C1—C2—H2A	108.7	O4—C16—H16D	108.4
C1—C2—H2B	108.7	O4—C16—C17	108.3 (2)
C1—C2—C3	114.28 (12)	H16A—C16—H16B	108.4
H2A—C2—H2B	107.6	H16C—C16—H16D	107.5
C3—C2—H2A	108.7	C17—C16—H16A	110.0
C3—C2—H2B	108.7	C17—C16—H16B	110.0
C2—C3—C25	108.42 (13)	C17A—C16—O4	115.5 (4)
C4—C3—C2	107.84 (13)	C17A—C16—H16C	108.4
C4—C3—C25	110.27 (14)	C17A—C16—H16D	108.4
C24—C3—C2	110.77 (14)	C16—C17—H17A	109.5
C24—C3—C4	110.08 (14)	C16—C17—H17B	109.5
C24—C3—C25	109.43 (15)	C16—C17—H17C	109.5
C3—C4—H4A	109.4	H17A—C17—H17B	109.5
C3—C4—H4B	109.4	H17A—C17—H17C	109.5
H4A—C4—H4B	108.0	H17B—C17—H17C	109.5
C5—C4—C3	111.22 (13)	C16—C17A—H17D	109.5

C5—C4—H4A	109.4	C16—C17A—H17E	109.5
C5—C4—H4B	109.4	C16—C17A—H17F	109.5
O1—C5—C4	119.99 (13)	H17D—C17A—H17E	109.5
O1—C5—C6	121.89 (14)	H17D—C17A—H17F	109.5
C6—C5—C4	118.08 (13)	H17E—C17A—H17F	109.5
C1—C6—C5	119.53 (13)	C19—C18—C7	121.14 (13)
C1—C6—C7	121.28 (12)	C23—C18—C7	121.01 (13)
C5—C6—C7	119.16 (12)	C23—C18—C19	117.85 (14)
C6—C7—H7	107.8	O5—C19—C18	122.76 (14)
C6—C7—C18	112.53 (11)	O5—C19—C20	116.89 (14)
C8—C7—C6	108.12 (11)	C20—C19—C18	120.34 (15)
C8—C7—H7	107.8	C19—C20—H20	119.8
C8—C7—C18	112.73 (11)	C21—C20—C19	120.35 (15)
C18—C7—H7	107.8	C21—C20—H20	119.8
C9—C8—C7	117.07 (12)	C20—C21—H21	119.9
C13—C8—C7	122.19 (13)	C22—C21—C20	120.17 (15)
C13—C8—C9	120.73 (13)	C22—C21—H21	119.9
O2—C9—C8	121.20 (14)	C21—C22—H22	120.2
O2—C9—C10	121.44 (14)	C21—C22—C23	119.56 (16)
C8—C9—C10	117.34 (13)	C23—C22—H22	120.2
C9—C10—H10A	109.3	C18—C23—C22	121.71 (14)
C9—C10—H10B	109.3	C18—C23—H23	119.1
C9—C10—C11	111.62 (12)	C22—C23—H23	119.1
H10A—C10—H10B	108.0	C3—C24—H24A	109.5
C11—C10—H10A	109.3	C3—C24—H24B	109.5
C11—C10—H10B	109.3	C3—C24—H24C	109.5
C10—C11—C12	107.90 (12)	H24A—C24—H24B	109.5
C10—C11—C26	110.38 (13)	H24A—C24—H24C	109.5
C10—C11—C27	109.47 (13)	H24B—C24—H24C	109.5
C26—C11—C12	109.08 (12)	C3—C25—H25A	109.5
C26—C11—C27	109.14 (13)	C3—C25—H25B	109.5
C27—C11—C12	110.87 (12)	C3—C25—H25C	109.5
C11—C12—H12A	108.9	H25A—C25—H25B	109.5
C11—C12—H12B	108.9	H25A—C25—H25C	109.5
H12A—C12—H12B	107.7	H25B—C25—H25C	109.5
C13—C12—C11	113.34 (12)	C11—C26—H26A	109.5
C13—C12—H12A	108.9	C11—C26—H26B	109.5
C13—C12—H12B	108.9	C11—C26—H26C	109.5
N1—C13—C12	117.39 (12)	H26A—C26—H26B	109.5
C8—C13—N1	120.15 (12)	H26A—C26—H26C	109.5
C8—C13—C12	122.36 (13)	H26B—C26—H26C	109.5
N1—C14—H14A	109.3	C11—C27—H27A	109.5
N1—C14—H14B	109.3	C11—C27—H27B	109.5
N1—C14—C15	111.74 (12)	C11—C27—H27C	109.5
H14A—C14—H14B	107.9	H27A—C27—H27B	109.5
C15—C14—H14A	109.3	H27A—C27—H27C	109.5
C15—C14—H14B	109.3	H27B—C27—H27C	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5···O1	0.84	1.81	2.6319 (17)	166
C2—H2*A*···O2^{i}	0.99	2.52	3.1663 (19)	123
C2—H2*B*···O5^{i}	0.99	2.53	3.457 (2)	157
C12—H12*B*···O1^{ii}	0.99	2.52	3.2708 (17)	133
C14—H14*A*···O1^{ii}	0.99	2.53	3.3299 (18)	138
C14—H14*B*···O2^{i}	0.99	2.66	3.473 (2)	140
C27—H27*B*···O3^{iii}	0.98	2.51	3.452 (3)	161

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