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Crystal structure and Hirshfeld surface analysis of 2-(dimethoxymethyl)phenyl (3a*RS*,6*SR*,7*RS*,7a*SR*)-1-oxo-2-phenyl-1,2,3,6,7,7a-hexahydro-3a,6-epoxyisoindole-7-carboxylate

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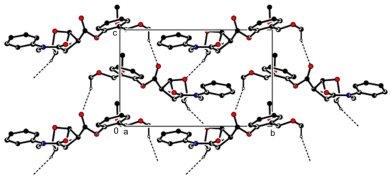
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The molecular structure of the title compound, $C_{24}H_{23}NO_6$, exhibits stability due to four intramolecular C—H···O interactions that form rings with *S*(6), *S*(9), *S*(5), and *S*(5) motifs. In the 7-oxabicyclo[2.2.1]hept-2-ene unit, the five-membered rings and the pyrrolidine ring all adopt envelope conformations. In the crystal, the molecules are connected by C—H···O interactions, creating a three-dimensional framework. The molecules are further connected by C—H···π interactions, forming layers parallel to the $(10\bar{1})$ plane. Important intermolecular interactions highlighted by Hirshfeld surface analysis are H···H (54.6%), C···H/H···C (21.6%) and O···H/H···O (21.3%) contacts.

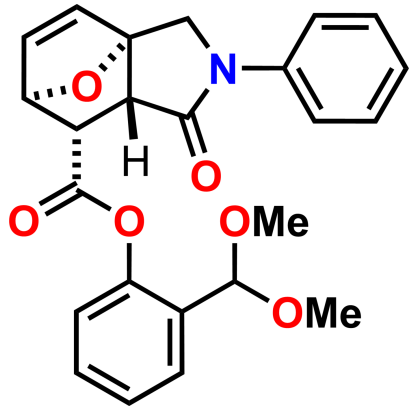
1. Chemical context

3a,6-Epoxyisoindole carboxylates are of interest as rigid polycyclic N,O-containing heterocyclic systems. Their value is related not only to their spatial structure, but also to the possibility of further functionalization: the presence of a carboxyl group makes it possible to obtain esters and introduce various aromatic or biologically significant fragments into the molecule (Varlamov *et al.*, 2004; Zubkov *et al.*, 2011). 3a,6-Epoxyisoindole carboxylic acids, their esters, and formylphenyl derivatives have already been described in the literature, which were then used for the synthesis of azomethines (Dikusar *et al.*, 2024; Mamedov *et al.*, 2024). This shows that the epoxyisoindole framework can be considered as a convenient basis for building more complex molecules, rather than just as a separate synthetic product. Salicylaldehyde is also an important and widely used fragment in organic chemistry. Its derivatives readily condense with amines to form Schiff bases, which are actively studied as ligands for metal complexes, fluorescent sensors, and compounds with potential biological activity (Afrin *et al.*, 2023; Ceramella *et al.*, 2022; Javadzade *et al.*, 2026; Li *et al.*, 2025). Therefore, the combination of the 3a,6-epoxyisoindole framework with the protected salicylaldehyde fragment in the form of acetal is justified: the epoxyisoindole sets a rigid spatial structure and increases molecular complexity, and the salicylaldehyde fragment retains the possibility of further transformations after



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removing the protection. Thus, the resulting ester can be considered as a hybrid of two synthetically valuable fragments, which expands the range of derivatives of 3a,6-epoxyisoindoles and may be useful for further preparation of functionalized heterocyclic compounds.



2. Structural commentary

The molecular structure of the title compound exhibits stability due to four intramolecular C—H···O interactions that form rings with *S*(6), *S*(9), *S*(5), and *S*(5) motifs (Fig. 1, Table 1; Bernstein *et al.*, 1995). The pyrrolidine ring (N2/C1/C7*A*/C3*A*/C3) makes the dihedral angles of 21.14 (11) and 21.56 (10)°, respectively, with the phenyl ring (C8–C13) and the benzene ring (C15–C20), while these rings subtend a dihedral angle of 13.69 (10)° with each other. In the 7-oxabicyclo[2.2.1]hept-2-ene unit of the ten-membered fused 1,2,3,6,7,7a-hexahydro-3a,6-epoxyisoindole ring system, the (O8/C3*A*/C4/C5/C6) and (O8/C3*A*/C7*A*/C7/C6) rings adopt envelope conformations with the puckering parameters (Cremer & Pople, 1975) $Q(2)$ = 0.511 (2) Å, $\varphi(2)$ = 181.3 (3)° and $Q(2)$ = 0.599 (2) Å, $\varphi(2)$ = 3.6 (2)°, respectively, and atom O8 as the flap in both cases. The pyrrolidine ring (N2/C1/C7*A*/C3*A*/C3) adopts an envelope conformation on atom C3*A* as the flap with puckering parameters $Q(2)$ = 0.317 (2) Å, $\varphi(2)$ = 282.4 (4)°.

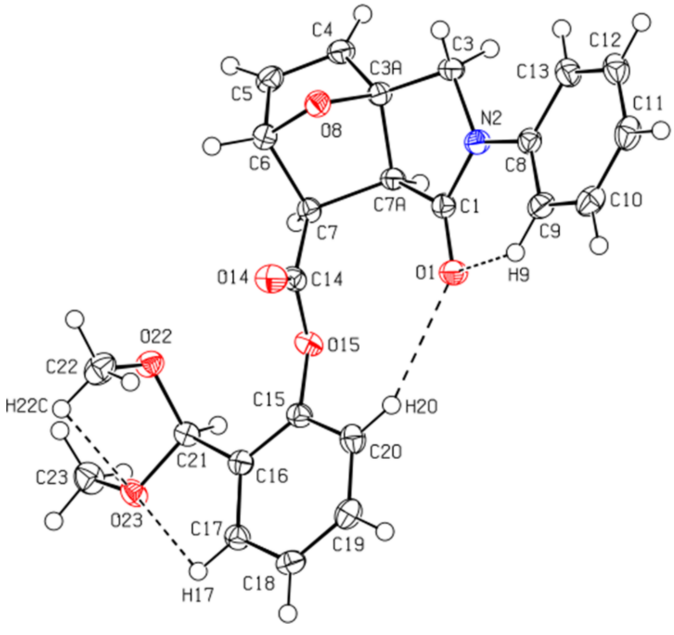


Figure 1
Molecular structure of the title compound showing the atom labeling and ring system with displacement ellipsoids at 50% probability level. The intramolecular C9—H9···O1, C20—H20···O1, C17—H17···O23 and C22—H22*C*···O23 interactions, shown as dashed lines, enclose *S*(6), *S*(9), *S*(5) and *S*(5) motifs, respectively.

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

*Cg*5 and *Cg*6 are the centroids of the C8—C13 phenyl ring and the C15–C20 benzene ring, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···O8^{i}	0.95	2.42	3.314 (3)	156
C5—H5···O1^{ii}	0.95	2.53	3.314 (3)	140
C9—H9···O1	0.95	2.24	2.863 (3)	123
C17—H17···O23	0.95	2.36	2.702 (3)	101
C20—H20···O1	0.95	2.58	3.387 (3)	143
C22—H22*C*···O23	0.98	2.49	2.823 (3)	100
C23—H23*B*···O14^{iii}	0.98	2.57	3.492 (3)	157
C7*A*—H7*A*···*Cg*5^{i}	1.00	2.68	3.564 (2)	147
C21—H21···*Cg*6^{iii}	1.00	2.96	3.912 (2)	159

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

3. Supramolecular features and Hirshfeld surface analysis

There were no classical hydrogen bonds in the crystal. The molecules are linked by C—H···O interactions, forming a three-dimensional network (Figs. 2, 3 and 4, Table 1). The molecules are further linked by C—H···π interactions, forming layers that are oriented with the (10$\bar{1}$) plane (Figs. 5 and 6, Table 1). No π–π interactions were observed.

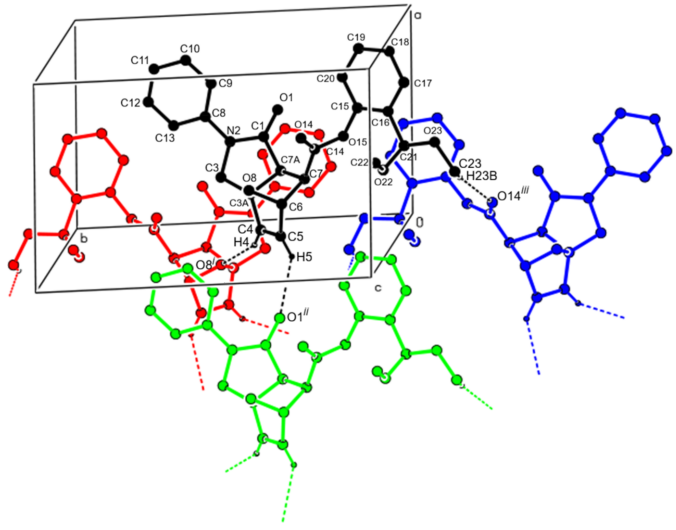


Figure 2
Partial packing view showing the C4—H4···O8^{i}, C5—H5···O1^{ii} and C23—H23*B*···O14^{iii} interactions in the unit cell. Symmetry codes: (i) $x-\frac{1}{2}, -y+1, z-\frac{1}{2}$; (ii) $x-1, y, z$; (iii) $x-\frac{1}{2}, -y, z-\frac{1}{2}$.

Table 2
Summary of short interatomic contacts (Å).

Contact	Distance	Symmetry operation
H20···H5	2.34	$1 + x, y, z$
O8···H4	2.42	$\frac{1}{2} + x, 1 - y, \frac{1}{2} + z$
H22*B*···H21	2.45	$\frac{1}{2} + x, -y, \frac{1}{2} + z$
C12···H6	2.87	$-\frac{1}{2} + x, 1 - y, -\frac{1}{2} + z$
H12···C17	2.95	$x, 1 + y, z$
C19···H22*C*	2.92	$\frac{1}{2} + x, -y, -\frac{1}{2} + z$
H23*C*···H11	2.57	$-1 + x, -1 + y, z$

CrystalExplorer 21 (Spackman *et al.*, 2021) was used to construct Hirshfeld surfaces and generate the related two-dimensional fingerprint plots to illustrate the intermolecular interactions for the molecules of the title compound. The d_{norm} mappings were conducted in the range −0.2256 to +1.3633 a.u. Bright-red circles on the d_{norm} surfaces (Fig. 7) represent O—H···O and C—H···O interaction zones (Table 1 and 2).

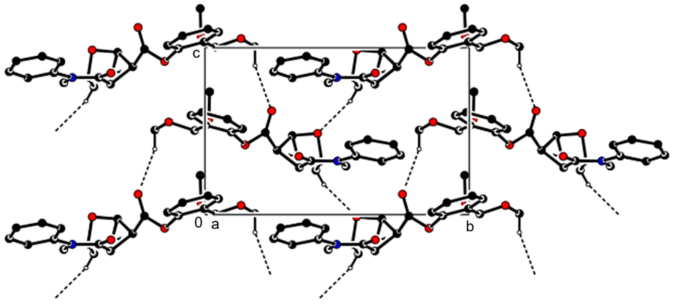


Figure 3
A view of the C—H···O interactions along the *a*-axis direction.

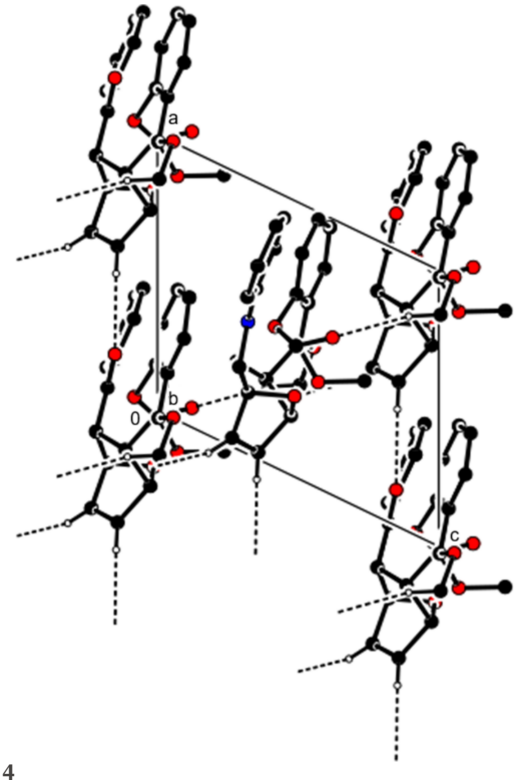


Figure 4
A view of the C—H···O interactions along the *b*-axis direction.

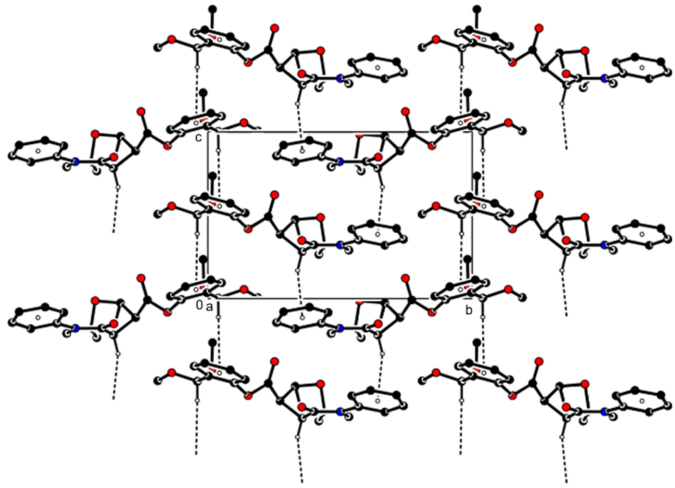


Figure 5
A view of the C—H···π interactions along the *a*-axis direction.

Two-dimensional fingerprint plots of the title compound together with their percentage contributions are shown in Fig. 8. The crystal packing is dominated by H···H contacts, representing van der Waals interactions (54.6% contribution to the overall surface), followed by C···H/H···C and O···H/H···O interactions, which contribute to 21.6% and 21.3%,

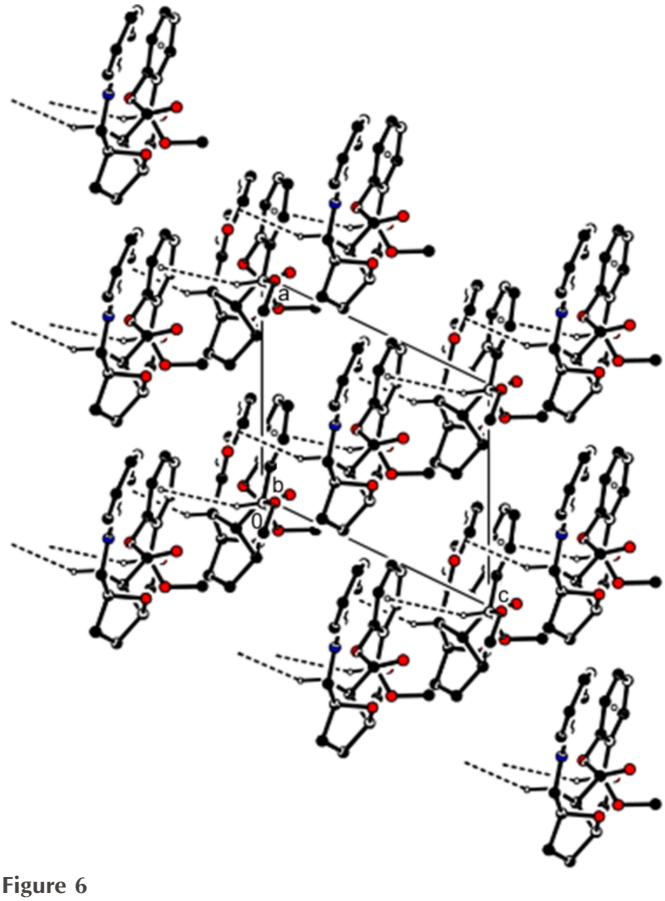


Figure 6
A view of the C—H···π interactions along the *b*-axis direction.

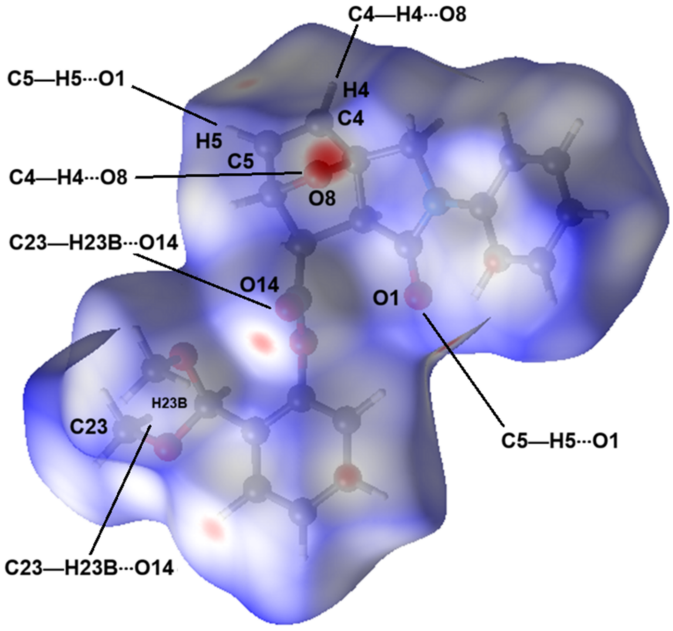


Figure 7
View of the three-dimensional Hirshfeld surface of the title compound mapped over d_{norm} in the range −0.2256 to 1.3633 a.u.

respectively. The other contacts [N···H/H···N (1.3%), O···C/C···O (0.6%), C···C (0.5%) and N···C/C···N (0.1%)] only make a minor contribution to the crystal packing.

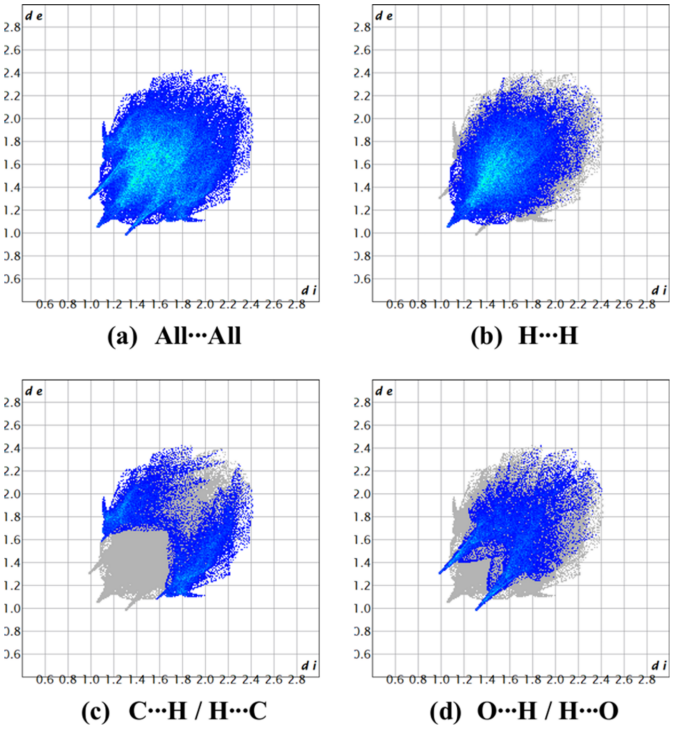


Figure 8
The two-dimensional fingerprint plots of the title compound, showing (*a*) all interactions, and those delineated into (*b*) H···H, (*c*) C···H/H···C, (*d*) O···H/H···O interactions. The d_i and d_e values are the closest internal and external distances (in Å) from given points on the Hirshfeld surface.

4. Database survey

A search of the Cambridge Structural Database (CSD version 6.01, updated February 2026; Groom *et al.*, 2016) for the 1,2,3,6,7,7a-hexahydro-3a,6-epoxyisoindole ring system returned five matches. These are CSD refcodes ERIVIL (Temel *et al.*, 2011), OMUTAU (Mertsalov *et al.*, 2021), UPAQEI (Koşar *et al.*, 2011), TIJMIK (Demircan *et al.*, 2013), and YAXCIL (Temel *et al.*, 2012).

ERIVIL and OMUTAU crystallize in the triclinic space group $P\overline{1}$ while UPAQEI, TIJMIK and YAXCIL crystallize in the monoclinic space group $P2_1/c$,

In ERIVIL, the two tetrahydrofuran rings adopt envelope conformations, the pyrrolidine ring adopts a half-chair conformation and the six-membered ring is in a boat conformation. In the crystal, weak intermolecular C—H···O hydrogen bonds link the molecules into $R_2^2(8)$ and $R_2^2(14)$ rings along the *b*-axis direction.

In OMUTAU, the tetrahydrofuran rings of both molecules (*A* and *B*) in the asymmetric unit, adopt envelope conformations, the pyrrolidine rings adopt a twisted-envelope conformation and the six-membered ring is in a boat conformation. In the crystal, strong intermolecular O—H···O hydrogen bonds and weak intermolecular C—H···O contacts link the molecules, forming a three-dimensional network. In addition weak π–π stacking interactions are observed of the *A* molecules.

In UPAQEI, the boat form of the six-membered ring is almost symmetrical with respect to the epoxy bridge. The two five-membered rings generated by the epoxy bridge of the six-membered ring adopt envelope conformations, whereas the N-containing five-membered ring adopts a twisted conformation. In the crystal, molecules are linked by C—H···O hydrogen bonds.

In TIJMIK, the tetrahydrofuran ring adopts an envelope conformation. The pyrrolidine ring adopts an envelope conformation. In the crystal, two types of C—H···O hydrogen bonds generate $R_2^2(20)$ and $R_4^4(26)$ rings, with adjacent rings running parallel to *ac* plane. Further C—H···O hydrogen bonds form a $C(6)$ chain, linking the molecules in the *b*-axis direction.

In YAXCIL, the six-membered ring has a boat conformation. The two five-membered rings with the bridging O atom adopt envelope conformations, whereas the N-containing five-membered ring adopts a twisted conformation. In the crystal, C—H···O hydrogen bonds link the molecules into a three-dimensional network.

5. Synthesis and crystallization

A solution of the 2-formylphenyl (3a*RS*,6*SR*,7*RS*,7a*SR*)-1-oxo-2-phenyl-1,2,3,6,7,7a-hexahydro-3a,6-epoxyisoindole-7-carboxylate (0.1 g, 0.27 mmol, 1.0 equiv) in methanol (3 mL) was treated with methanolic hydrogen chloride (1.25 *M* solution in MeOH, 22 µL, 0.027 mmol, 0.1 equiv). The reaction mixture was stirred at room temperature for 7 days (TLC

Table 3
Experimental details.

Crystal data	
Chemical formula	$C_{24}H_{23}NO_6$
M_r	421.43
Crystal system, space group	Monoclinic, *Pn*
Temperature (K)	100
a, *b*, *c* (Å)	8.4620 (1), 13.6810 (17), 9.5690 (13)
β (°)	115.731 (6)
V (Å^3)	997.94 (19)
Z	2
Radiation type	Synchrotron, λ = 0.75170 Å
μ (mm^{-1})	0.11
Crystal size (mm)	0.12 × 0.08 × 0.06
Data collection	
Diffractometer	Rayonix SX165 CCD
Absorption correction	Multi-scan (*SCALA*; Evans, 2006)
T_{min}, T_{max}	0.963, 0.987
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	11428, 5347, 4834
R_{int}	0.034
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.688
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.035, 0.084, 1.04
No. of reflections	5347
No. of parameters	282
No. of restraints	2
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.18, −0.20
Absolute structure	Flack *x* determined using 2152 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	0.2 (3)

Computer programs: *Marccd* (Doyle, 2011), *iMosflm* (Battye *et al.*, 2011), *SHELXT* (Sheldrick, 2015*a*), *SHELXL* (Sheldrick, 2015*b*), *ORTEP-3 for Windows* (Farrugia, 2012) and *PLATON* (Spek, 2020).

control, hexane–EtOAc, 1:2). After completion of the reaction, the reaction mixture was poured into water (50 mL). The precipitate was filtered off, washed with water (3 × 5 mL), dried under vacuum, and air-dried to afford the corresponding acetal as a colorless powder, yield 25%, 28 mg (0.07 mmol), m.p. 437–440 K. Single crystals of the 2-(dimethoxymethyl) phenyl (3a*RS*,6*SR*,7*RS*,7a*SR*)-1-oxo-2-phenyl-1,2,3,6,7,7a-hexahydro-3a,6-epoxyisoindole-7-carboxylate were grown from the 1:1 *v*/*v* mixture of a $CHCl_3$/EtOH at 281 K. ^{1}H NMR (700.2 MHz, $CDCl_3$), (*J*, Hz) 7.61–7.60 (*m*, 3 H, H-Ar), 7.48 (*d*, *J* = 8.1 Hz, 1 H, H-Ar), 7.41–7.36 (*m*, 3 H, H-Ar), 7.25 (*t*, *J* = 7.6 Hz, 1 H, H-Ar), 7.16 (*t*, *J* = 8.1 Hz, 1 H, H-Ar), 6.65 (*d*, *J* = 5.7 Hz, 1 H, H-4), 6.59 (*dd*, *J* = 5.7, 1.4 Hz, 1 H, H-5), 5.65 (*s*, 1 H, C*H*$(OCH_3)_2$), 5.43 (*d*, *J* = 1.4 Hz, 1 H, H-6), 4.50 and 4.24 (*two d*, *J* = 11.4 Hz, 2 H, H-3), 3.30 (*s*, 3 H, CH_3), 3.26 (*s*, 3 H, CH_3), 3.15 and 3.05 (*two d*, *J* = 9.1 Hz, 2 H, H-7,7a). ^{13}C NMR (176.1 MHz, $CDCl_3$) δ 170.0, 169.9, 148.8, 139.1, 137.3, 135.5, 129.8, 129.6, 128.9 (2C), 127.8, 125.5, 125.0, 123.4, 120.5 (2C), 98.7, 87.8, 81.9, 52.8, 52.4, 52.2, 50.2, 45.8. MS (ESI) *m*/*z*: $[M]^+$ 422.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. H atoms were placed in idealized positions and allowed to ride on their parent atoms, with C—H = 0.95, 0.98, 0.99 and 1.00 Å for aromatic, methyl, methylene and methine H respectively, and U_{iso}(H) = 1.5U_{eq}(C) for methyl H and U_{iso}(H) = 1.2U_{eq}(C) for all other H atoms.

Acknowledgements

The authors' contributions are as follows; conceptualization MMW and MA; synthesis, VSM and DDS; X-ray analysis VNK; writing (review and editing of the manuscript) MA, IHM and KIH; supervision MA and KIH.

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supporting information

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Crystal structure and Hirshfeld surface analysis of 2-(dimethoxymethyl)phenyl (3a*RS*,6*SR*,7*RS*,7a*SR*)-1-oxo-2-phenyl-1,2,3,6,7,7a-hexahydro-3a,6-epoxyisoindole-7-carboxylate

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Computing details

2-(Dimethoxymethyl)phenyl (3a*RS*,6*SR*,7*RS*,7a*SR*)-1-oxo-2-phenyl-1,2,3,6,7,7a-hexahydro-3a,6-epoxyisoindole-7-carboxylate

Crystal data

$C_{24}H_{23}NO_6$
M_r = 421.43
Monoclinic, *Pn*
a = 8.4620 (1) Å
b = 13.6810 (17) Å
c = 9.5690 (13) Å
β = 115.731 (6)°
V = 997.94 (19) Å^3
Z = 2
F(000) = 444
D_x = 1.403 Mg m^{-3}
Synchrotron radiation, *λ* = 0.75170 Å
Cell parameters from 1000 reflections
θ = 3.0–30.0°
μ = 0.11 mm^{-1}
T = 100 K
Prism, colourless
0.12 × 0.08 × 0.06 mm

Data collection

Rayonix SX165 CCD diffractometer
/f scan
Absorption correction: multi-scan (*SCALA*; Evans, 2006)
T_{min} = 0.963, T_{max} = 0.987
11428 measured reflections
5347 independent reflections
4834 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 31.1°, θ_{min} = 3.0°
h = −11→11
k = −18→18
l = −13→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.084
S = 1.04
5347 reflections
282 parameters
2 restraints
Primary atom site location: difference Fourier map
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0432P)^2 + 0.1076P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Absolute structure: Flack *x* determined using 2152 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.2 (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}
C1	0.4961 (3)	0.40137 (15)	0.3223 (2)	0.0190 (4)
O1	0.6301 (2)	0.35560 (11)	0.34868 (19)	0.0247 (3)
N2	0.4799 (2)	0.50154 (13)	0.3215 (2)	0.0201 (3)
C3	0.3012 (3)	0.53354 (15)	0.2908 (3)	0.0212 (4)
H3A	0.234768	0.555430	0.182043	0.025*
H3B	0.304050	0.587019	0.361521	0.025*
C3A	0.2241 (3)	0.44210 (15)	0.3220 (2)	0.0190 (4)
C4	0.0320 (3)	0.41977 (16)	0.2651 (2)	0.0224 (4)
H4	−0.063836	0.449515	0.181188	0.027*
C5	0.0260 (3)	0.34939 (17)	0.3580 (3)	0.0246 (4)
H5	−0.075822	0.317177	0.352920	0.029*
C6	0.2148 (3)	0.33061 (16)	0.4743 (2)	0.0216 (4)
H6	0.229673	0.300789	0.574637	0.026*
C7	0.3029 (3)	0.27320 (15)	0.3857 (2)	0.0186 (4)
H7	0.221508	0.221351	0.319527	0.022*
C7A	0.3171 (3)	0.35687 (15)	0.2817 (2)	0.0181 (4)
H7A	0.255702	0.339462	0.169199	0.022*
O8	0.2907 (2)	0.42620 (11)	0.48675 (16)	0.0214 (3)
C8	0.6140 (3)	0.57109 (15)	0.3512 (2)	0.0197 (4)
C9	0.7912 (3)	0.54460 (16)	0.4169 (2)	0.0235 (4)
H9	0.824033	0.477938	0.438949	0.028*
C10	0.9183 (3)	0.61563 (18)	0.4496 (3)	0.0269 (5)
H10	1.038297	0.596990	0.492347	0.032*
C11	0.8743 (3)	0.71356 (18)	0.4212 (3)	0.0277 (5)
H11	0.962897	0.761937	0.445983	0.033*
C12	0.6989 (3)	0.73968 (17)	0.3563 (3)	0.0266 (5)
H12	0.667261	0.806645	0.336615	0.032*
C13	0.5684 (3)	0.66946 (16)	0.3194 (2)	0.0234 (4)
H13	0.448485	0.688281	0.272662	0.028*
C14	0.4759 (3)	0.22845 (14)	0.4920 (2)	0.0191 (4)
O14	0.5702 (2)	0.25257 (12)	0.62243 (17)	0.0266 (3)
O15	0.50935 (19)	0.15215 (11)	0.41812 (16)	0.0206 (3)
C15	0.6653 (3)	0.09970 (15)	0.4948 (2)	0.0191 (4)
C16	0.6535 (3)	0.00321 (15)	0.5349 (2)	0.0187 (4)
C17	0.8069 (3)	−0.05195 (15)	0.5946 (2)	0.0203 (4)
H17	0.803329	−0.118128	0.623109	0.024*
C18	0.9650 (3)	−0.01146 (16)	0.6129 (2)	0.0226 (4)
H18	1.068604	−0.050075	0.653582	0.027*
C19	0.9727 (3)	0.08477 (17)	0.5722 (2)	0.0243 (4)

H19	1.081235	0.111944	0.584366	0.029*
C20	0.8222 (3)	0.14171 (16)	0.5137 (2)	0.0235 (4)
H20	0.826585	0.208218	0.487071	0.028*
C21	0.4759 (3)	−0.03941 (15)	0.5039 (2)	0.0189 (4)
H21	0.402850	−0.039989	0.389011	0.023*
O22	0.3843 (2)	0.01482 (12)	0.57138 (17)	0.0231 (3)
C22	0.4688 (3)	0.0178 (2)	0.7361 (3)	0.0304 (5)
H22A	0.395144	0.053485	0.774374	0.046*
H22B	0.582221	0.050938	0.770393	0.046*
H22C	0.487505	−0.049026	0.777041	0.046*
O23	0.5021 (2)	−0.13678 (11)	0.55526 (18)	0.0230 (3)
C23	0.3417 (3)	−0.18961 (18)	0.5065 (3)	0.0287 (5)
H23A	0.366870	−0.257094	0.544044	0.043*
H23B	0.280366	−0.189553	0.392951	0.043*
H23C	0.267661	−0.158497	0.548995	0.043*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0200 (9)	0.0184 (9)	0.0198 (9)	−0.0005 (8)	0.0097 (7)	0.0002 (7)
O1	0.0216 (7)	0.0192 (7)	0.0356 (8)	0.0010 (6)	0.0148 (6)	0.0017 (6)
N2	0.0191 (8)	0.0178 (8)	0.0251 (8)	−0.0011 (7)	0.0110 (7)	−0.0009 (6)
C3	0.0201 (9)	0.0183 (10)	0.0269 (10)	0.0031 (8)	0.0119 (8)	0.0019 (8)
C3A	0.0194 (9)	0.0197 (9)	0.0185 (9)	0.0024 (8)	0.0090 (7)	0.0010 (7)
C4	0.0202 (10)	0.0211 (10)	0.0269 (10)	0.0020 (8)	0.0113 (8)	−0.0002 (8)
C5	0.0210 (10)	0.0259 (11)	0.0306 (11)	0.0003 (9)	0.0148 (8)	−0.0001 (8)
C6	0.0241 (10)	0.0207 (10)	0.0236 (9)	0.0003 (8)	0.0135 (8)	0.0009 (7)
C7	0.0186 (9)	0.0182 (9)	0.0188 (8)	−0.0010 (7)	0.0079 (7)	0.0007 (7)
C7A	0.0172 (9)	0.0169 (9)	0.0204 (8)	0.0023 (7)	0.0084 (7)	0.0007 (7)
O8	0.0243 (7)	0.0212 (7)	0.0200 (7)	0.0002 (6)	0.0108 (6)	−0.0007 (5)
C8	0.0228 (10)	0.0188 (9)	0.0199 (9)	−0.0015 (8)	0.0115 (8)	−0.0014 (7)
C9	0.0243 (11)	0.0238 (11)	0.0262 (10)	0.0004 (9)	0.0145 (9)	−0.0020 (8)
C10	0.0242 (10)	0.0296 (12)	0.0308 (11)	−0.0048 (9)	0.0156 (9)	−0.0056 (8)
C11	0.0330 (12)	0.0259 (11)	0.0294 (11)	−0.0103 (10)	0.0183 (9)	−0.0055 (9)
C12	0.0356 (12)	0.0207 (10)	0.0259 (11)	−0.0045 (9)	0.0156 (9)	−0.0012 (8)
C13	0.0280 (10)	0.0204 (10)	0.0227 (9)	−0.0015 (9)	0.0118 (8)	0.0005 (8)
C14	0.0204 (9)	0.0154 (9)	0.0213 (9)	−0.0009 (7)	0.0088 (7)	0.0009 (7)
O14	0.0265 (8)	0.0283 (8)	0.0209 (7)	0.0030 (7)	0.0065 (6)	−0.0030 (6)
O15	0.0202 (7)	0.0178 (7)	0.0202 (6)	0.0016 (6)	0.0053 (5)	0.0002 (5)
C15	0.0192 (9)	0.0195 (9)	0.0168 (8)	0.0017 (8)	0.0062 (7)	0.0000 (7)
C16	0.0203 (9)	0.0201 (9)	0.0146 (8)	0.0001 (8)	0.0065 (7)	−0.0021 (7)
C17	0.0221 (9)	0.0199 (9)	0.0181 (9)	0.0014 (8)	0.0080 (7)	0.0004 (7)
C18	0.0209 (10)	0.0263 (10)	0.0197 (9)	0.0049 (8)	0.0079 (7)	−0.0021 (7)
C19	0.0215 (10)	0.0293 (11)	0.0230 (9)	−0.0045 (9)	0.0106 (8)	−0.0037 (8)
C20	0.0254 (11)	0.0222 (10)	0.0228 (9)	−0.0036 (9)	0.0106 (8)	−0.0003 (8)
C21	0.0187 (9)	0.0178 (9)	0.0190 (8)	0.0005 (7)	0.0070 (7)	−0.0002 (7)
O22	0.0199 (7)	0.0266 (8)	0.0224 (7)	0.0021 (6)	0.0090 (6)	−0.0002 (6)
C22	0.0289 (11)	0.0405 (13)	0.0224 (10)	0.0014 (10)	0.0116 (9)	−0.0040 (9)

O23	0.0211 (7)	0.0182 (7)	0.0272 (7)	−0.0014 (6)	0.0082 (6)	0.0033 (6)
C23	0.0262 (11)	0.0242 (11)	0.0332 (12)	−0.0061 (9)	0.0104 (9)	−0.0007 (9)

Geometric parameters (Å, º)

C1—O1	1.222 (3)	C11—H11	0.9500
C1—N2	1.377 (3)	C12—C13	1.388 (3)
C1—C7A	1.518 (3)	C12—H12	0.9500
N2—C8	1.412 (3)	C13—H13	0.9500
N2—C3	1.476 (3)	C14—O14	1.200 (3)
C3—C3A	1.499 (3)	C14—O15	1.357 (2)
C3—H3A	0.9900	O15—C15	1.398 (2)
C3—H3B	0.9900	C15—C20	1.385 (3)
C3A—O8	1.442 (2)	C15—C16	1.390 (3)
C3A—C4	1.505 (3)	C16—C17	1.392 (3)
C3A—C7A	1.546 (3)	C16—C21	1.516 (3)
C4—C5	1.326 (3)	C17—C18	1.387 (3)
C4—H4	0.9500	C17—H17	0.9500
C5—C6	1.521 (3)	C18—C19	1.382 (3)
C5—H5	0.9500	C18—H18	0.9500
C6—O8	1.439 (3)	C19—C20	1.387 (3)
C6—C7	1.562 (3)	C19—H19	0.9500
C6—H6	1.0000	C20—H20	0.9500
C7—C14	1.503 (3)	C21—O23	1.404 (2)
C7—C7A	1.555 (3)	C21—O22	1.416 (2)
C7—H7	1.0000	C21—H21	1.0000
C7A—H7A	1.0000	O22—C22	1.421 (3)
C8—C13	1.396 (3)	C22—H22A	0.9800
C8—C9	1.399 (3)	C22—H22B	0.9800
C9—C10	1.381 (3)	C22—H22C	0.9800
C9—H9	0.9500	O23—C23	1.425 (3)
C10—C11	1.386 (4)	C23—H23A	0.9800
C10—H10	0.9500	C23—H23B	0.9800
C11—C12	1.384 (4)	C23—H23C	0.9800
O1—C1—N2	126.32 (19)	C12—C11—C10	118.9 (2)
O1—C1—C7A	125.50 (19)	C12—C11—H11	120.6
N2—C1—C7A	108.17 (17)	C10—C11—H11	120.6
C1—N2—C8	126.88 (18)	C11—C12—C13	121.0 (2)
C1—N2—C3	112.78 (17)	C11—C12—H12	119.5
C8—N2—C3	120.30 (18)	C13—C12—H12	119.5
N2—C3—C3A	102.07 (16)	C12—C13—C8	119.9 (2)
N2—C3—H3A	111.4	C12—C13—H13	120.1
C3A—C3—H3A	111.4	C8—C13—H13	120.1
N2—C3—H3B	111.4	O14—C14—O15	123.97 (19)
C3A—C3—H3B	111.4	O14—C14—C7	127.16 (19)
H3A—C3—H3B	109.2	O15—C14—C7	108.84 (16)
O8—C3A—C3	110.27 (16)	C14—O15—C15	118.94 (15)

O8—C3A—C4	102.26 (16)	C20—C15—C16	122.51 (19)
C3—C3A—C4	126.31 (18)	C20—C15—O15	119.22 (19)
O8—C3A—C7A	99.17 (15)	C16—C15—O15	117.93 (18)
C3—C3A—C7A	105.52 (16)	C15—C16—C17	117.55 (19)
C4—C3A—C7A	110.09 (17)	C15—C16—C21	119.63 (18)
C5—C4—C3A	104.76 (18)	C17—C16—C21	122.72 (18)
C5—C4—H4	127.6	C18—C17—C16	120.80 (19)
C3A—C4—H4	127.6	C18—C17—H17	119.6
C4—C5—C6	106.21 (19)	C16—C17—H17	119.6
C4—C5—H5	126.9	C19—C18—C17	120.4 (2)
C6—C5—H5	126.9	C19—C18—H18	119.8
O8—C6—C5	101.32 (17)	C17—C18—H18	119.8
O8—C6—C7	101.62 (16)	C18—C19—C20	120.1 (2)
C5—C6—C7	106.58 (16)	C18—C19—H19	120.0
O8—C6—H6	115.2	C20—C19—H19	120.0
C5—C6—H6	115.2	C15—C20—C19	118.7 (2)
C7—C6—H6	115.2	C15—C20—H20	120.7
C14—C7—C7A	114.19 (16)	C19—C20—H20	120.7
C14—C7—C6	113.07 (16)	O23—C21—O22	111.82 (17)
C7A—C7—C6	99.47 (16)	O23—C21—C16	107.70 (16)
C14—C7—H7	109.9	O22—C21—C16	113.67 (17)
C7A—C7—H7	109.9	O23—C21—H21	107.8
C6—C7—H7	109.9	O22—C21—H21	107.8
C1—C7A—C3A	101.11 (16)	C16—C21—H21	107.8
C1—C7A—C7	118.48 (16)	C21—O22—C22	114.17 (17)
C3A—C7A—C7	101.94 (15)	O22—C22—H22A	109.5
C1—C7A—H7A	111.4	O22—C22—H22B	109.5
C3A—C7A—H7A	111.4	H22A—C22—H22B	109.5
C7—C7A—H7A	111.4	O22—C22—H22C	109.5
C6—O8—C3A	95.62 (14)	H22A—C22—H22C	109.5
C13—C8—C9	119.2 (2)	H22B—C22—H22C	109.5
C13—C8—N2	118.94 (19)	C21—O23—C23	112.55 (16)
C9—C8—N2	121.80 (19)	O23—C23—H23A	109.5
C10—C9—C8	119.8 (2)	O23—C23—H23B	109.5
C10—C9—H9	120.1	H23A—C23—H23B	109.5
C8—C9—H9	120.1	O23—C23—H23C	109.5
C9—C10—C11	121.3 (2)	H23A—C23—H23C	109.5
C9—C10—H10	119.4	H23B—C23—H23C	109.5
C11—C10—H10	119.4		

O1—C1—N2—C8	−0.2 (3)	C3—N2—C8—C13	−13.3 (3)
C7A—C1—N2—C8	−179.18 (18)	C1—N2—C8—C9	−13.4 (3)
O1—C1—N2—C3	−178.0 (2)	C3—N2—C8—C9	164.23 (19)
C7A—C1—N2—C3	3.1 (2)	C13—C8—C9—C10	0.1 (3)
C1—N2—C3—C3A	17.1 (2)	N2—C8—C9—C10	−177.4 (2)
C8—N2—C3—C3A	−160.84 (17)	C8—C9—C10—C11	1.2 (3)
N2—C3—C3A—O8	76.45 (19)	C9—C10—C11—C12	−1.2 (3)
N2—C3—C3A—C4	−160.02 (18)	C10—C11—C12—C13	−0.2 (3)

N2—C3—C3A—C7A	−29.7 (2)	C11—C12—C13—C8	1.4 (3)
O8—C3A—C4—C5	−33.7 (2)	C9—C8—C13—C12	−1.4 (3)
C3—C3A—C4—C5	−160.6 (2)	N2—C8—C13—C12	176.19 (19)
C7A—C3A—C4—C5	70.9 (2)	C7A—C7—C14—O14	91.2 (3)
C3A—C4—C5—C6	1.4 (2)	C6—C7—C14—O14	−21.6 (3)
C4—C5—C6—O8	31.3 (2)	C7A—C7—C14—O15	−90.62 (19)
C4—C5—C6—C7	−74.6 (2)	C6—C7—C14—O15	156.59 (17)
O8—C6—C7—C14	87.89 (19)	O14—C14—O15—C15	−2.3 (3)
C5—C6—C7—C14	−166.43 (17)	C7—C14—O15—C15	179.40 (17)
O8—C6—C7—C7A	−33.62 (18)	C14—O15—C15—C20	−73.4 (2)
C5—C6—C7—C7A	72.07 (19)	C14—O15—C15—C16	113.2 (2)
O1—C1—C7A—C3A	159.9 (2)	C20—C15—C16—C17	−0.7 (3)
N2—C1—C7A—C3A	−21.1 (2)	O15—C15—C16—C17	172.60 (17)
O1—C1—C7A—C7	49.6 (3)	C20—C15—C16—C21	−177.05 (18)
N2—C1—C7A—C7	−131.42 (18)	O15—C15—C16—C21	−3.8 (3)
O8—C3A—C7A—C1	−82.73 (16)	C15—C16—C17—C18	0.0 (3)
C3—C3A—C7A—C1	31.42 (19)	C21—C16—C17—C18	176.26 (18)
C4—C3A—C7A—C1	170.53 (16)	C16—C17—C18—C19	0.1 (3)
O8—C3A—C7A—C7	39.83 (18)	C17—C18—C19—C20	0.4 (3)
C3—C3A—C7A—C7	153.98 (15)	C16—C15—C20—C19	1.2 (3)
C4—C3A—C7A—C7	−66.91 (19)	O15—C15—C20—C19	−171.98 (18)
C14—C7—C7A—C1	−14.7 (3)	C18—C19—C20—C15	−1.1 (3)
C6—C7—C7A—C1	106.01 (19)	C15—C16—C21—O23	−179.92 (17)
C14—C7—C7A—C3A	−124.49 (18)	C17—C16—C21—O23	3.9 (3)
C6—C7—C7A—C3A	−3.79 (19)	C15—C16—C21—O22	−55.5 (2)
C5—C6—O8—C3A	−49.64 (17)	C17—C16—C21—O22	128.4 (2)
C7—C6—O8—C3A	60.13 (17)	O23—C21—O22—C22	58.6 (2)
C3—C3A—O8—C6	−172.14 (17)	C16—C21—O22—C22	−63.6 (2)
C4—C3A—O8—C6	51.28 (17)	O22—C21—O23—C23	65.2 (2)
C7A—C3A—O8—C6	−61.75 (17)	C16—C21—O23—C23	−169.22 (17)
C1—N2—C8—C13	169.08 (19)		

Hydrogen-bond geometry (Å, º)

*Cg*5 and *Cg*6 are the centroids of the C8—C23 phenyl ring and the C15–C20 benzene ring, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···O8^{i}	0.95	2.42	3.314 (3)	156
C5—H5···O1^{ii}	0.95	2.53	3.314 (3)	140
C9—H9···O1	0.95	2.24	2.863 (3)	123
C17—H17···O23	0.95	2.36	2.702 (3)	101
C20—H20···O1	0.95	2.58	3.387 (3)	143
C22—H22*C*···O23	0.98	2.49	2.823 (3)	100
C23—H23*B*···O14^{iii}	0.98	2.57	3.492 (3)	157
C7*A*—H7*A*···Cg5^{i}	1.00	2.68	3.564 (2)	147
C21—H21···Cg6^{iii}	1.00	2.96	3.912 (2)	159

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