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Crystal structure and Hirshfeld surface analysis of (3a*RS*,4*RS*,10*SR*,10a*SR*)-2-(3,5-dimethylphenyl)-4-hydroxy-10-methyl-1-oxo-2,3,3a,4,10,10a-hexahydro-1*H*-[1]benzofuro[2,3-*f*]isoindole-10-carboxylic acid dimethylformamide monosolvate

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The molecular conformation of the title compound, C₂₄H₂₃NO₅·C₃H₇NO, is consolidated by intramolecular C—H···O—H···O hydrogen bonds, forming an *S*(6) ring motif. In the crystal, the molecules are connected by C—H···O hydrogen bonds, forming layers parallel to the (101) plane. Furthermore, the molecules form layers parallel to the (10 $\bar{2}$) plane by C—H··· π interactions. Important intermolecular interactions highlighted by Hirshfeld surface analysis are H···H (54.7%), O···H/H···O (23.0%), and C···H/H···C (19.9%) contacts.

1. Chemical context

The IMDAV reaction (Intra-Molecular Diels–Alder in Vinylheteroarenes) is a useful tool for the one-step synthesis of benzofurans, indoles, benzothiophenes, and pyrrolopyridines annulated with other carbocycles and heterocycles (Horak *et al.*, 2017; Krishna *et al.*, 2022; Nadirova *et al.*, 2020; Shelukho *et al.*, 2025; Yakovleva *et al.*, 2024; Zaytsev *et al.*, 2023, 2025; Zubkov *et al.*, 2016). In a continuation of our research on the properties of vinylheteroarene systems previously obtained *via* tandem acylation/[4 + 2] cycloaddition between 3-(heteroaryl)allyl amines and maleic anhydrides, an example of an IMDAV reaction, we present here the second instance of spontaneous slow oxidation of adduct **1** (Fig. 1) in DMSO under aerobic conditions. Previous studies have shown that benzothienoisindolones of type **1** undergo oxidation when stored for a long time in DMSO at room temperature (Mammadova *et al.*, 2023). Presumably, the DMSO acts as a

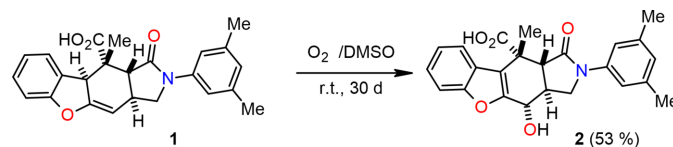
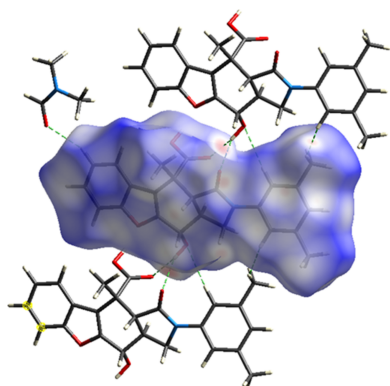
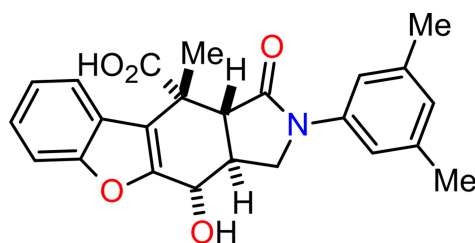


Figure 1
Synthesis of (3a*RS*,4*RS*,10*SR*,10a*SR*)-2-(3,5-dimethylphenyl)-4-hydroxy-10-methyl-1-oxo-2,3,3a,4,10,10a-hexahydro-1*H*-[1]benzofuro[2,3-*f*]isoindole-10-carboxylic acid (**2**).

mild oxidant, as observed in several other oxidation reactions, including the Pfitzner–Moffatt, Corey–Kim, Swern, and Kornblum oxidations (Epstein *et al.*, 1967).

Slow oxidation of (3*aRS*,9*bRS*,10*RS*,10*aSR*)-2-(3,5-dimethylphenyl)-10-methyl-1-oxo-2,3,3*a*,9*b*,10,10*a*-hexahydro-1*H*-[1]benzofuro[2,3-*f*]isoindole-10-carboxylic acid (**1**) occurs when the solution is stirred in dimethyl sulfoxide (DMSO) for one month at r.t. The title compound **2** was isolated in a 53% yield after standard treatment of the reaction mixture followed by recrystallization from an EtOH/DMF mixture. As in the previous case (Mammadova *et al.*, 2023), the reaction does not stop at the formation of an alcohol. This leads to the formation of the aromatic product **2** as a result of proton migration.



2. Structural commentary

The molecular conformation of the title compound is consolidated by intramolecular C—H...O hydrogen bonds and intramolecular O—H...O hydrogen bonds, forming an *S*(6) ring motif (Fig. 2; Table 1; Bernstein *et al.*, 1995). The main molecule of the title compound is planar, with a mean deviation of 0.002 Å from the least-squares plane defined by the 53 atoms (excluding H atoms). The deviations of some atoms

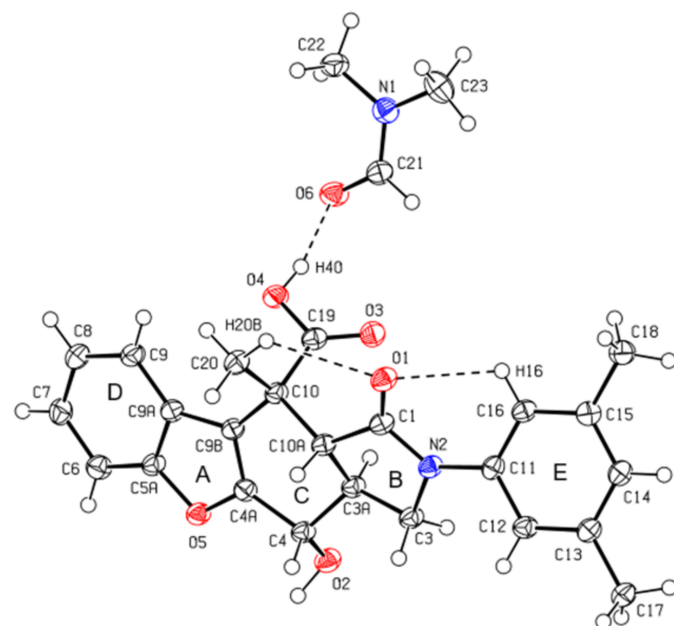


Figure 2

View of the title molecule. Displacement ellipsoids are drawn at the 50% probability level.

Table 1

Hydrogen-bond geometry (Å, °).

Cg4 and *Cg5* are the centroids of the C5*A*/C6–C9/C9*A* and C11–C16 rings, respectively.

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O2—H2O...O1 ⁱ	0.99 (5)	2.23 (4)	3.013 (2)	135 (3)
O2—H2O...O3 ⁱ	0.99 (5)	2.32 (4)	3.152 (3)	141 (3)
O4—H4O...O6	0.84 (4)	1.74 (4)	2.560 (3)	165 (4)
C3—H3A...O1 ⁱⁱ	0.99	2.54	3.452 (3)	152
C8—H8...O6 ⁱⁱⁱ	0.95	2.44	3.344 (3)	159
C16—H16...O1	0.95	2.44	2.970 (3)	115
C20—H20B...O1	0.98	2.59	3.229 (3)	123
C10 <i>A</i> —H10 <i>A</i> ... <i>Cg5</i> ⁱⁱ	1.00	2.88	3.865 (2)	169
C22—H22 <i>A</i> ... <i>Cg5</i> ^{iv}	0.98	2.96	3.401 (3)	109
C22—H22C... <i>Cg4</i> ^v	0.98	2.68	3.594 (3)	155

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

from the least-squares plane are 1.141 (2) Å for O1, −1.083 (2) Å for O2, −1.414 (2) Å for O3, −1.224 (2) Å for O4, 0.734 (2) Å for C1, 0.718 (2) Å for C10*A*, −0.631 (2) Å for C18, −0.841 (2) Å for C19 and 1.585 (2) Å for C20. The five-membered *B* (N2/C1/C10*A*/C3*A*/C3) ring adopts an envelope conformation, as indicated by the puckering parameters (Cremer & Pople, 1975) $Q(2) = 0.337$ (2) Å, $\varphi(2) = 287.7$ (4)°, with the C3*A* atom −0.212 (2) Å out of the plane defined by the other atoms of the main molecule. The six-membered *C* (C3*A*/C4/C4*A*/C9*B*/C10/C10*A*) ring has a half-chair conformation [the puckering parameters are $Q_T = 0.533$ (2) Å, $\theta = 127.0$ (2)°, and $\varphi = 152.1$ (3)°]. The dihedral angles between the least-squares planes of the rings in the molecule are *A*/*B* = 19.28 (12), *A*/*C* = 6.72 (11), *A*/*D* = 2.10 (11), *A*/*E* = 19.19 (11),

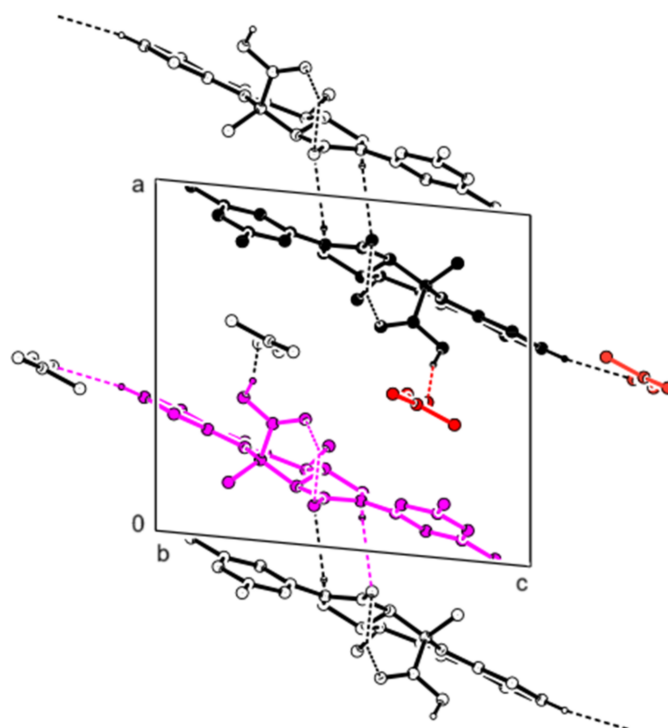


Figure 3

A partial view of the molecular packing along the *b* axis, showing the O—H...O and C—H...O interactions.

$B/C = 12.57$ (11), $B/D = 20.45$ (12), $B/E = 35.55$ (11), $C/D = 8.10$ (10), $C/E = 24.24$ (10) and $D/E = 19.75$ (10) $^\circ$. There is one stereogenic center in the title molecule and the chirality about atom C23 is *S* in the chosen asymmetric unit. The geometric properties of the title compound are normal and consistent with those of related compounds listed in the *Database survey* section.

3. Supramolecular features and Hirshfeld surface analysis

In the crystal, the molecules are connected by C—H $\cdots$ O hydrogen bonds, forming layers parallel to the (101) plane (Table 1; Fig. 3). Furthermore, the molecules form layers parallel to the (10 $\bar{2}$) plane by C—H $\cdots\pi$ interactions (Table 1; Fig. 4). No π – π interactions were observed.

CrystalExplorer 17.5 (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 of the title compound were conducted in the range -0.7845 to $+1.3229$ a.u. Bright-red circles on the d_{norm} surfaces (Fig. 5) represent H $\cdots$ H, O—H $\cdots$ O and C—H $\cdots$ O interaction zones (Tables 1 and 2).

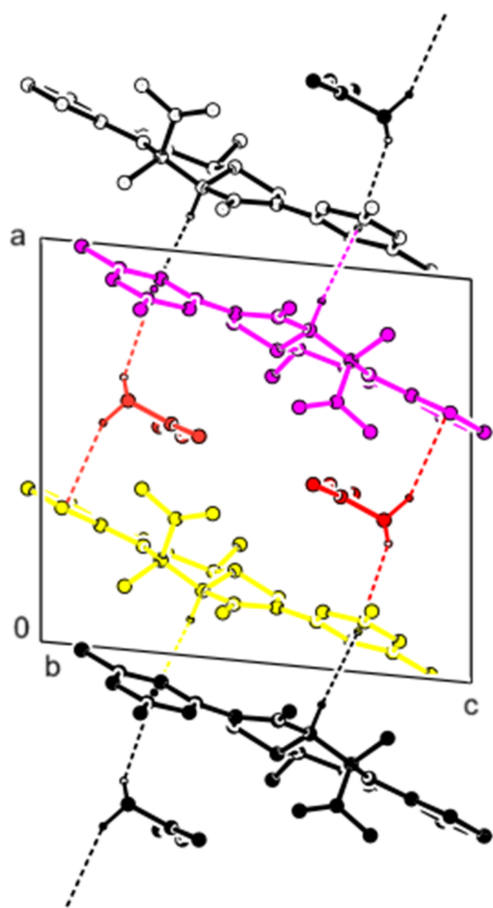


Figure 4

A partial view of the molecular packing along the *b* axis, showing the C—H $\cdots\pi$ interactions.

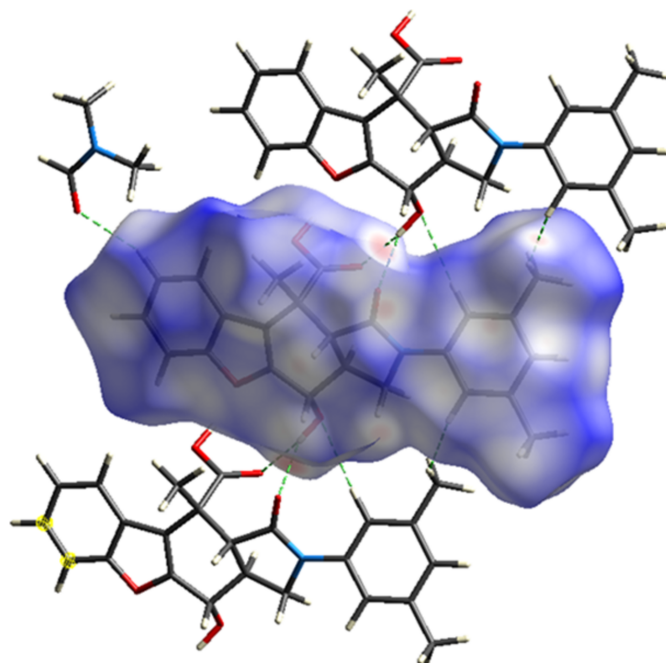


Figure 5

Hirshfeld surface of the title compound mapped with d_{norm} .

Two-dimensional fingerprint plots together with their percentage contributions are shown in Fig. 6. The crystal packing is dominated by H $\cdots$ H contacts, representing van der

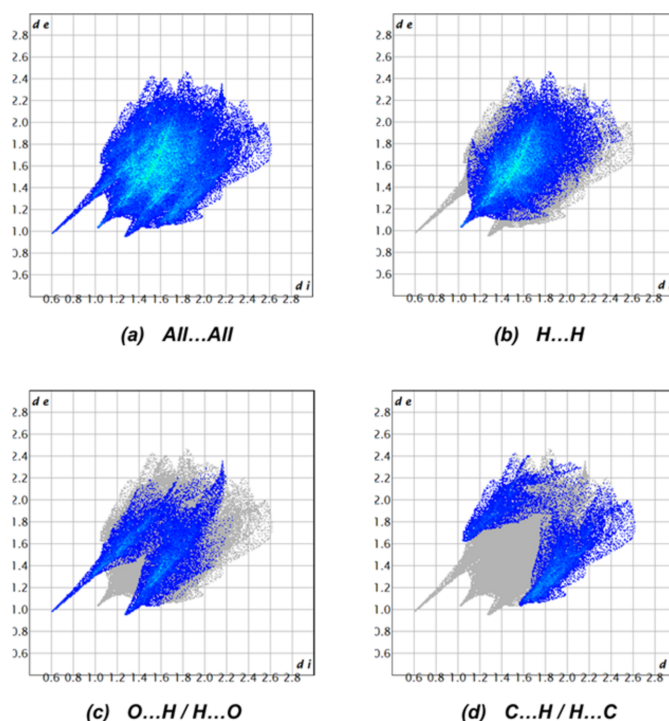


Figure 6

The two-dimensional fingerprint plots for the compound showing (a) all interactions, and delineated into (b) H $\cdots$ H (54.7%), (c) O $\cdots$ H/H $\cdots$ O (23.0%) and (d) C $\cdots$ H/H $\cdots$ C (19.9%) 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

Summary of short interatomic contacts (Å).

Contact	Distance	Symmetry operation
O1...H2O	2.23	$x, 1 + y, z$
O1...H3A	2.54	$2 - x, \frac{1}{2} + y, 1 - z$
O4...H23A	2.60	$x, -1 + y, z$
H2O...H23B	2.55	$1 - x, -\frac{3}{2} + y, 1 - z$
H4O...O6	1.74	x, y, z
H16...H23B	2.58	$1 - x, -\frac{1}{2} + y, 1 - z$
C7...H18B	3.04	$x, -1 + y, 1 + z$
H8...O6	2.44	$1 - x, -\frac{1}{2} + y, 2 - z$
H7...H22B	2.52	$1 - x, -\frac{3}{2} + y, 2 - z$

Waals interactions (54.7% contribution to the overall surface), followed by O...H/H...O and C...H/H...C interactions, which contribute to 23.0% and 19.9%, respectively. The other contacts (N...H/H...N 0.7%, O...C/C...O 0.6%, C...C 0.4%, O...O 0.3%, N...C/C...N 0.2%, O...N/N...O 0.1% and N...N 0.1%) only make a minor contribution to the crystal packing.

4. Database survey

A search of the Cambridge Structural Database (CSD, version 6.00, update April 2025; Groom *et al.*, 2016) for the *octahydro-1H-isoindol-1-one* unit gave 467 hits. The five related compound CSD reference codes are ANAMUZ (Mariaule *et al.*, 2016), BAFYAL (Zhong *et al.*, 2017), NAMROK (Chou & Wu, 2012), TODKEF (Elliott & Booker-Milburn, 2019) and YOPXIL (Paddon-Row *et al.*, 2009).

ANAMUZ crystallizes in the monoclinic $P2_1/c$ space group, BAFYAL in the orthorhombic $Pna2_1$ space group, NAMROK in the monoclinic $P2_1/n$ space group, TODKEF in the monoclinic $C2/c$ space group, and YOPXIL in the monoclinic $P2_1$ like the title compound.

In the structure of ANAMUZ, the molecules are linked by C—H...O and O—H...O intermolecular hydrogen bonds, forming a three-dimensional network. Weak π – π interactions are also observed. In BAFYAL, the molecules are linked by C—H...O interactions, forming layers parallel to the (002) plane. π – π interactions are also present. In NAMROK, pairs of molecules are linked by C—H...O interactions. π – π and C—H... π interactions are not observed. In TODKEF, the molecules are linked by intermolecular C—H...O and O—H...O hydrogen bonds, forming a three-dimensional network. C—H... π interactions are also observed. In YOPXIL, the molecules are linked by intermolecular C—H...O hydrogen bonds, forming chains along the *b*-axis direction. No π – π or C—H... π interactions are observed.

5. Synthesis and crystallization

A solution of (3aRS,9bRS,10RS,10aSR)-2-(3,5-dimethylphenyl)-10-methyl-1-oxo-2,3,3a,9b,10,10a-hexahydro-1H-[1]benzofuro[2,3-*f*]isoindole-10-carboxylic acid **1** (39.0 mg, 0.1 mmol) in 0.5 mL of DMSO was stirred for 30 d in an open flask. The reaction mixture was concentrated, recrystallized from a mixture of EtOH/DMF. The solid was filtered off, washed with

Table 3

Experimental details.

Crystal data	
Chemical formula	C ₂₄ H ₂₃ NO ₅ ·C ₃ H ₇ NO
M_r	478.53
Crystal system, space group	Monoclinic, $P2_1$
Temperature (K)	100
a, b, c (Å)	11.88334 (13), 7.80196 (10), 12.71675 (15)
β (°)	95.5166 (10)
V (Å ³)	1173.55 (2)
Z	2
Radiation type	Cu $K\alpha$
μ (mm ^{−1})	0.79
Crystal size (mm)	0.32 × 0.18 × 0.04
Data collection	
Diffraction	Rigaku XtaLAB Synergy-S, HyPix-6000HE area-detector
Absorption correction	Multi-scan (CrysAlis PRO; Rigaku OD, 2021)
$T_{\min}, T_{\max}$	0.840, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	16579, 4632, 4504
R_{int}	0.039
$(\sin \theta/\lambda)_{\text{max}}$ (Å ^{−1})	0.639
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.039, 0.104, 1.05
No. of reflections	4632
No. of parameters	327
No. of restraints	1
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å ^{−3})	0.29, −0.20
Absolute structure	Flack x determined using 1816 quotients $[(I^+) - (I^-)] / [(I^+) + (I^-)]$ (Parsons <i>et al.</i> , 2013)
Absolute structure parameter	0.26 (7)

Computer programs: CrysAlis PRO (Rigaku OD, 2021), SHELXT (Sheldrick, 2015a), SHELXL (Sheldrick, 2015b), ORTEP-3 for Windows (Farrugia, 2012) and PLATON (Spek, 2020).

Et₂O (3 × 1 mL), and air dried. The title compound was obtained as a colorless plates, yield 53%, 21.5 mg; m.p. > 523 K (with decomp.). IR (KBr), ν (cm^{−1}): 3047 (OH), 1744 (CO₂), 1683 (N—C=O). ¹H NMR (700.2 MHz, DMSO-*d*₆): δ (J, Hz) 12.87 (*s*, 1H, CO₂H), 7.30–7.22 (*m*, 4H, H Ar), 7.09–7.06 (*m*, 2H, H Ar), 6.76 (*br.s*, 1H, H Ar), 5.69 (*br.s*, 1H, OH), 4.31 (*br.s*, 1H, H-4) 4.02 (*t*, $J = 8.6$, 1H, H-3A), 3.69 (*t*, $J = 8.6$, 1H, H-3B), 3.03–2.98 (*m*, 1H, H-3a), 2.26 (*s*, 6H, CH₃), 2.14 (*d*, $J = 12.6$, 1H, H-10a), 0.99 (*s*, 3H, CH₃) ppm. ¹³C{¹H} NMR (176.1 MHz, DMSO-*d*₆): δ 177.3, 172.6, 158.8, 156.9, 140.2, 138.2 (2C), 129.4, 126.4, 125.8, 125.4, 122.8, 117.6 (2C), 110.6, 98.8, 58.9, 50.3, 49.7, 42.9, 35.9, 22.8, 21.7 (2C) ppm. MS (ESI) m/z : $[M + H]^+$ 406. Elemental analysis calculated (%) for C₂₄H₂₃NO₅·C₃H₇NO: C 67.77, H 6.32, N 5.85; found: C 68.04, H 6.49, N 6.01.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. The hydroxyl H atoms were found in the difference Fourier maps [O2—H₂O = 0.99 (5) and O4—H₄O = 0.84 (4) Å] and refined with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$.

All C-bound H atoms were positioned geometrically (C—H = 0.95 and 1.00 Å) and refined using a riding model with $U_{\text{iso}}(\text{H}) = 1.2$ or $1.5U_{\text{eq}}(\text{C})$. Owing to poor agreement between observed and calculated intensities, two outliers ($\sim 13 \sim 5$ and $14 \sim 3$) were omitted in the final cycles of refinement.

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supporting information

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Manahelohe**

Computing details

(3a*RS*,4*RS*,10*SR*,10a*SR*)-2-(3,5-Dimethylphenyl)-4-hydroxy-10-methyl-1-oxo-2,3,3a,4,10,10a-
hexahydro-1*H*-[1]benzofuro[2,3-*f*]isoindole-10-carboxylic acid dimethylformamide monosolvate

Crystal data

$C_{24}H_{23}NO_5 \cdot C_3H_7NO$
 $M_r = 478.53$
Monoclinic, $P2_1$
 $a = 11.88334$ (13) Å
 $b = 7.80196$ (10) Å
 $c = 12.71675$ (15) Å
 $\beta = 95.5166$ (10)°
 $V = 1173.55$ (2) Å³
 $Z = 2$

$F(000) = 508$
 $D_x = 1.354$ Mg m⁻³
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 11292 reflections
 $\theta = 3.5\text{--}79.3^\circ$
 $\mu = 0.79$ mm⁻¹
 $T = 100$ K
Plate, colourless
 $0.32 \times 0.18 \times 0.04$ mm

Data collection

Rigaku XtaLAB Synergy-S, HyPix-6000HE
area-detector
diffractometer
Radiation source: micro-focus sealed X-ray tube
 φ and ω scans
Absorption correction: multi-scan
(CrysAlisPro; Rigaku OD, 2021)
 $T_{\min} = 0.840$, $T_{\max} = 1.000$

16579 measured reflections
4632 independent reflections
4504 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.039$
 $\theta_{\max} = 80.0^\circ$, $\theta_{\min} = 3.5^\circ$
 $h = -15 \rightarrow 14$
 $k = -9 \rightarrow 9$
 $l = -16 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.104$
 $S = 1.05$
4632 reflections
327 parameters

1 restraint
Hydrogen site location: mixed
H atoms treated by a mixture of independent
and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0664P)^2 + 0.2084P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$

$$\Delta\rho_{\max} = 0.29 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.20 \text{ e } \text{\AA}^{-3}$$

Absolute structure: Flack x determined using
1816 quotients $[(F^+)-(F^-)]/[(F^+)+(F^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.26 (7)

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 ($\text{\AA}^2$)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.88530 (14)	0.6262 (2)	0.57739 (13)	0.0271 (3)
O2	0.71225 (14)	−0.0932 (2)	0.53774 (13)	0.0284 (3)
H2O	0.730 (3)	−0.211 (6)	0.563 (3)	0.043*
O3	0.64231 (13)	0.5337 (3)	0.60067 (13)	0.0305 (4)
O4	0.59786 (13)	0.4918 (2)	0.76539 (13)	0.0273 (3)
H4O	0.548 (3)	0.562 (6)	0.741 (3)	0.041*
C1	0.86110 (16)	0.4789 (3)	0.55174 (17)	0.0224 (4)
N2	0.85953 (15)	0.4116 (3)	0.45216 (14)	0.0226 (4)
C3	0.83569 (18)	0.2262 (3)	0.44817 (17)	0.0238 (4)
H3A	0.906192	0.158378	0.448925	0.029*
H3B	0.784816	0.195658	0.384677	0.029*
C3A	0.77808 (17)	0.1988 (3)	0.54918 (17)	0.0227 (4)
H3C	0.696408	0.229798	0.534790	0.027*
C4	0.78611 (17)	0.0212 (3)	0.59804 (17)	0.0224 (4)
H4	0.865679	−0.021613	0.601253	0.027*
C4A	0.75155 (16)	0.0443 (3)	0.70768 (16)	0.0222 (4)
O5	0.72355 (13)	−0.0995 (2)	0.76165 (13)	0.0249 (3)
C5A	0.69017 (17)	−0.0398 (3)	0.85613 (17)	0.0235 (4)
C6	0.65086 (19)	−0.1429 (3)	0.93339 (19)	0.0274 (5)
H6	0.646651	−0.264079	0.926576	0.033*
C7	0.6179 (2)	−0.0579 (3)	1.02159 (19)	0.0290 (5)
H7	0.590681	−0.122544	1.077164	0.035*
C8	0.6241 (2)	0.1209 (4)	1.03023 (19)	0.0293 (5)
H8	0.600598	0.174756	1.091446	0.035*
C9	0.66374 (19)	0.2215 (3)	0.95132 (18)	0.0266 (4)
H9	0.667526	0.342649	0.957983	0.032*
C9A	0.69807 (17)	0.1389 (3)	0.86142 (17)	0.0227 (4)
C9B	0.73912 (17)	0.1912 (3)	0.76232 (16)	0.0215 (4)
C10	0.76988 (17)	0.3675 (3)	0.72104 (17)	0.0217 (4)
C10A	0.83541 (16)	0.3314 (3)	0.62441 (17)	0.0221 (4)
H10A	0.909918	0.281612	0.652289	0.027*
C11	0.88384 (16)	0.5005 (3)	0.35935 (16)	0.0225 (4)
C12	0.92752 (18)	0.4062 (3)	0.27861 (18)	0.0246 (4)
H12	0.946899	0.289074	0.289792	0.029*
C13	0.94276 (17)	0.4833 (3)	0.18188 (17)	0.0247 (4)

C14	0.91430 (18)	0.6547 (3)	0.16710 (18)	0.0254 (4)
H14	0.922624	0.706973	0.100812	0.030*
C15	0.87377 (18)	0.7520 (3)	0.24734 (18)	0.0248 (4)
C16	0.85946 (17)	0.6739 (3)	0.34459 (17)	0.0231 (4)
H16	0.833172	0.739347	0.400308	0.028*
C17	0.9891 (2)	0.3806 (3)	0.09481 (19)	0.0310 (5)
H17A	1.067808	0.349135	0.116298	0.047*
H17B	0.985651	0.449642	0.030209	0.047*
H17C	0.943878	0.276417	0.081523	0.047*
C18	0.8463 (2)	0.9381 (3)	0.2310 (2)	0.0306 (5)
H18A	0.772745	0.962733	0.256594	0.046*
H18B	0.843368	0.965518	0.155612	0.046*
H18C	0.904707	1.007900	0.270236	0.046*
C19	0.66366 (17)	0.4730 (3)	0.68735 (17)	0.0235 (4)
C20	0.84293 (18)	0.4711 (3)	0.80565 (18)	0.0261 (4)
H20A	0.800078	0.490770	0.866655	0.039*
H20B	0.863104	0.581564	0.775894	0.039*
H20C	0.911934	0.406868	0.828098	0.039*
O6	0.43899 (15)	0.7105 (3)	0.72511 (14)	0.0318 (4)
N1	0.43032 (15)	0.9966 (3)	0.69699 (16)	0.0285 (4)
C21	0.45729 (18)	0.8385 (3)	0.67122 (18)	0.0273 (5)
H21	0.492584	0.821996	0.608054	0.033*
C22	0.3827 (2)	1.0261 (4)	0.7974 (2)	0.0337 (5)
H22A	0.325742	0.938282	0.807445	0.051*
H22B	0.347375	1.139676	0.796540	0.051*
H22C	0.443070	1.020126	0.855480	0.051*
C23	0.4538 (2)	1.1426 (4)	0.6328 (2)	0.0374 (6)
H23A	0.504688	1.221382	0.674296	0.056*
H23B	0.382962	1.201749	0.609764	0.056*
H23C	0.489733	1.103704	0.570820	0.056*

Atomic displacement parameters ($\text{\AA}^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0306 (7)	0.0246 (8)	0.0275 (8)	−0.0040 (6)	0.0093 (6)	−0.0023 (6)
O2	0.0342 (8)	0.0240 (8)	0.0278 (7)	−0.0030 (7)	0.0073 (6)	−0.0033 (7)
O3	0.0278 (7)	0.0373 (9)	0.0275 (8)	0.0075 (7)	0.0080 (6)	0.0036 (7)
O4	0.0257 (7)	0.0295 (8)	0.0284 (8)	0.0054 (6)	0.0117 (6)	0.0017 (7)
C1	0.0180 (8)	0.0254 (10)	0.0247 (9)	−0.0014 (7)	0.0067 (7)	−0.0023 (8)
N2	0.0234 (8)	0.0222 (9)	0.0235 (8)	−0.0025 (7)	0.0089 (6)	−0.0006 (7)
C3	0.0259 (10)	0.0221 (11)	0.0247 (9)	−0.0005 (8)	0.0090 (7)	−0.0014 (8)
C3A	0.0207 (9)	0.0244 (10)	0.0240 (9)	0.0004 (8)	0.0072 (7)	−0.0010 (8)
C4	0.0207 (8)	0.0225 (10)	0.0250 (9)	0.0007 (8)	0.0079 (7)	−0.0012 (8)
C4A	0.0196 (8)	0.0231 (10)	0.0248 (10)	0.0009 (8)	0.0060 (7)	0.0020 (8)
O5	0.0270 (7)	0.0233 (8)	0.0259 (7)	0.0006 (6)	0.0093 (5)	0.0000 (6)
C5A	0.0213 (9)	0.0273 (11)	0.0228 (10)	0.0018 (8)	0.0064 (7)	−0.0008 (8)
C6	0.0269 (10)	0.0260 (11)	0.0301 (11)	0.0006 (8)	0.0073 (8)	0.0014 (9)
C7	0.0309 (11)	0.0319 (13)	0.0253 (10)	0.0015 (9)	0.0086 (8)	0.0053 (9)

C8	0.0316 (10)	0.0338 (13)	0.0240 (10)	0.0025 (9)	0.0106 (8)	0.0008 (9)
C9	0.0296 (10)	0.0263 (11)	0.0250 (10)	0.0023 (9)	0.0090 (8)	−0.0006 (9)
C9A	0.0195 (8)	0.0259 (11)	0.0234 (10)	0.0020 (8)	0.0059 (7)	0.0011 (8)
C9B	0.0199 (8)	0.0234 (10)	0.0219 (9)	0.0018 (7)	0.0052 (7)	−0.0005 (8)
C10	0.0214 (9)	0.0222 (10)	0.0227 (9)	0.0004 (7)	0.0080 (7)	−0.0001 (8)
C10A	0.0189 (8)	0.0238 (10)	0.0244 (9)	0.0000 (8)	0.0061 (7)	−0.0014 (8)
C11	0.0192 (8)	0.0279 (11)	0.0212 (9)	−0.0020 (8)	0.0060 (7)	−0.0001 (8)
C12	0.0233 (9)	0.0242 (11)	0.0271 (10)	0.0010 (8)	0.0076 (7)	−0.0008 (9)
C13	0.0219 (9)	0.0285 (11)	0.0245 (10)	−0.0013 (8)	0.0070 (7)	−0.0004 (9)
C14	0.0237 (9)	0.0286 (12)	0.0246 (10)	−0.0031 (8)	0.0065 (7)	0.0003 (8)
C15	0.0229 (9)	0.0243 (11)	0.0275 (10)	−0.0026 (8)	0.0043 (7)	−0.0004 (9)
C16	0.0207 (9)	0.0248 (11)	0.0247 (9)	−0.0013 (7)	0.0073 (7)	−0.0021 (8)
C17	0.0372 (12)	0.0322 (13)	0.0253 (10)	0.0051 (10)	0.0119 (8)	0.0004 (9)
C18	0.0354 (11)	0.0266 (12)	0.0307 (11)	0.0019 (9)	0.0079 (8)	0.0002 (9)
C19	0.0220 (9)	0.0227 (10)	0.0267 (10)	−0.0020 (8)	0.0073 (7)	−0.0020 (8)
C20	0.0267 (10)	0.0274 (11)	0.0250 (10)	−0.0017 (8)	0.0059 (7)	−0.0023 (9)
O6	0.0307 (8)	0.0297 (9)	0.0369 (8)	0.0032 (7)	0.0135 (6)	0.0011 (7)
N1	0.0236 (8)	0.0310 (11)	0.0305 (9)	0.0014 (8)	0.0015 (7)	−0.0003 (8)
C21	0.0250 (10)	0.0289 (12)	0.0287 (10)	0.0006 (9)	0.0059 (8)	−0.0017 (9)
C22	0.0318 (11)	0.0363 (14)	0.0328 (11)	0.0102 (10)	0.0023 (8)	−0.0057 (10)
C23	0.0305 (11)	0.0341 (13)	0.0463 (14)	−0.0025 (10)	−0.0030 (10)	0.0077 (11)

Geometric parameters (Å, °)

O1—C1	1.221 (3)	C10—C10A	1.543 (3)
O2—C4	1.423 (3)	C10—C20	1.545 (3)
O2—H2O	0.99 (4)	C10A—H10A	1.0000
O3—C19	1.204 (3)	C11—C16	1.393 (3)
O4—C19	1.329 (3)	C11—C12	1.402 (3)
O4—H4O	0.85 (4)	C12—C13	1.397 (3)
C1—N2	1.369 (3)	C12—H12	0.9500
C1—C10A	1.525 (3)	C13—C14	1.388 (4)
N2—C11	1.422 (3)	C13—C17	1.513 (3)
N2—C3	1.475 (3)	C14—C15	1.394 (3)
C3—C3A	1.528 (3)	C14—H14	0.9500
C3—H3A	0.9900	C15—C16	1.404 (3)
C3—H3B	0.9900	C15—C18	1.498 (3)
C3A—C4	1.517 (3)	C16—H16	0.9500
C3A—C10A	1.525 (3)	C17—H17A	0.9800
C3A—H3C	1.0000	C17—H17B	0.9800
C4—C4A	1.502 (3)	C17—H17C	0.9800
C4—H4	1.0000	C18—H18A	0.9800
C4A—C9B	1.356 (3)	C18—H18B	0.9800
C4A—O5	1.373 (3)	C18—H18C	0.9800
O5—C5A	1.382 (3)	C20—H20A	0.9800
C5A—C6	1.385 (3)	C20—H20B	0.9800
C5A—C9A	1.398 (3)	C20—H20C	0.9800
C6—C7	1.391 (3)	O6—C21	1.242 (3)

C6—H6	0.9500	N1—C21	1.324 (3)
C7—C8	1.401 (4)	N1—C23	1.444 (4)
C7—H7	0.9500	N1—C22	1.464 (3)
C8—C9	1.391 (3)	C21—H21	0.9500
C8—H8	0.9500	C22—H22A	0.9800
C9—C9A	1.407 (3)	C22—H22B	0.9800
C9—H9	0.9500	C22—H22C	0.9800
C9A—C9B	1.453 (3)	C23—H23A	0.9800
C9B—C10	1.529 (3)	C23—H23B	0.9800
C10—C19	1.533 (3)	C23—H23C	0.9800
C4—O2—H2O	108 (2)	C3A—C10A—H10A	106.6
C19—O4—H4O	104 (3)	C1—C10A—H10A	106.6
O1—C1—N2	126.1 (2)	C10—C10A—H10A	106.6
O1—C1—C10A	127.2 (2)	C16—C11—C12	119.8 (2)
N2—C1—C10A	106.56 (19)	C16—C11—N2	121.94 (19)
C1—N2—C11	126.5 (2)	C12—C11—N2	118.1 (2)
C1—N2—C3	113.19 (18)	C13—C12—C11	120.5 (2)
C11—N2—C3	120.22 (18)	C13—C12—H12	119.7
N2—C3—C3A	102.03 (17)	C11—C12—H12	119.7
N2—C3—H3A	111.4	C14—C13—C12	118.9 (2)
C3A—C3—H3A	111.4	C14—C13—C17	120.8 (2)
N2—C3—H3B	111.4	C12—C13—C17	120.2 (2)
C3A—C3—H3B	111.4	C13—C14—C15	121.5 (2)
H3A—C3—H3B	109.2	C13—C14—H14	119.3
C4—C3A—C10A	110.85 (18)	C15—C14—H14	119.3
C4—C3A—C3	117.16 (18)	C14—C15—C16	119.2 (2)
C10A—C3A—C3	102.90 (17)	C14—C15—C18	120.8 (2)
C4—C3A—H3C	108.5	C16—C15—C18	119.9 (2)
C10A—C3A—H3C	108.5	C11—C16—C15	119.9 (2)
C3—C3A—H3C	108.5	C11—C16—H16	120.0
O2—C4—C4A	111.45 (17)	C15—C16—H16	120.0
O2—C4—C3A	109.92 (17)	C13—C17—H17A	109.5
C4A—C4—C3A	105.01 (18)	C13—C17—H17B	109.5
O2—C4—H4	110.1	H17A—C17—H17B	109.5
C4A—C4—H4	110.1	C13—C17—H17C	109.5
C3A—C4—H4	110.1	H17A—C17—H17C	109.5
C9B—C4A—O5	113.02 (18)	H17B—C17—H17C	109.5
C9B—C4A—C4	129.1 (2)	C15—C18—H18A	109.5
O5—C4A—C4	117.87 (19)	C15—C18—H18B	109.5
C4A—O5—C5A	105.24 (17)	H18A—C18—H18B	109.5
O5—C5A—C6	124.4 (2)	C15—C18—H18C	109.5
O5—C5A—C9A	110.75 (19)	H18A—C18—H18C	109.5
C6—C5A—C9A	124.8 (2)	H18B—C18—H18C	109.5
C5A—C6—C7	115.8 (2)	O3—C19—O4	123.6 (2)
C5A—C6—H6	122.1	O3—C19—C10	124.22 (19)
C7—C6—H6	122.1	O4—C19—C10	112.22 (18)
C6—C7—C8	121.4 (2)	C10—C20—H20A	109.5

C6—C7—H7	119.3	C10—C20—H20B	109.5
C8—C7—H7	119.3	H20A—C20—H20B	109.5
C9—C8—C7	121.6 (2)	C10—C20—H20C	109.5
C9—C8—H8	119.2	H20A—C20—H20C	109.5
C7—C8—H8	119.2	H20B—C20—H20C	109.5
C8—C9—C9A	118.2 (2)	C21—N1—C23	122.0 (2)
C8—C9—H9	120.9	C21—N1—C22	119.1 (2)
C9A—C9—H9	120.9	C23—N1—C22	118.7 (2)
C5A—C9A—C9	118.2 (2)	O6—C21—N1	123.6 (2)
C5A—C9A—C9B	105.37 (19)	O6—C21—H21	118.2
C9—C9A—C9B	136.4 (2)	N1—C21—H21	118.2
C4A—C9B—C9A	105.6 (2)	N1—C22—H22A	109.5
C4A—C9B—C10	122.89 (18)	N1—C22—H22B	109.5
C9A—C9B—C10	131.4 (2)	H22A—C22—H22B	109.5
C9B—C10—C19	111.18 (16)	N1—C22—H22C	109.5
C9B—C10—C10A	105.37 (18)	H22A—C22—H22C	109.5
C19—C10—C10A	109.91 (17)	H22B—C22—H22C	109.5
C9B—C10—C20	111.64 (18)	N1—C23—H23A	109.5
C19—C10—C20	107.81 (18)	N1—C23—H23B	109.5
C10A—C10—C20	110.95 (17)	H23A—C23—H23B	109.5
C3A—C10A—C1	103.62 (17)	N1—C23—H23C	109.5
C3A—C10A—C10	113.22 (17)	H23A—C23—H23C	109.5
C1—C10A—C10	119.38 (19)	H23B—C23—H23C	109.5
O1—C1—N2—C11	−1.1 (3)	C9A—C9B—C10—C10A	166.1 (2)
C10A—C1—N2—C11	−177.07 (19)	C4A—C9B—C10—C20	−131.6 (2)
O1—C1—N2—C3	175.4 (2)	C9A—C9B—C10—C20	45.5 (3)
C10A—C1—N2—C3	−0.6 (2)	C4—C3A—C10A—C1	158.47 (16)
C1—N2—C3—C3A	21.1 (2)	C3—C3A—C10A—C1	32.4 (2)
C11—N2—C3—C3A	−162.14 (17)	C4—C3A—C10A—C10	−70.7 (2)
N2—C3—C3A—C4	−154.02 (18)	C3—C3A—C10A—C10	163.22 (18)
N2—C3—C3A—C10A	−32.2 (2)	O1—C1—C10A—C3A	163.7 (2)
C10A—C3A—C4—O2	167.45 (16)	N2—C1—C10A—C3A	−20.4 (2)
C3—C3A—C4—O2	−74.9 (2)	O1—C1—C10A—C10	36.7 (3)
C10A—C3A—C4—C4A	47.5 (2)	N2—C1—C10A—C10	−147.41 (18)
C3—C3A—C4—C4A	165.10 (17)	C9B—C10—C10A—C3A	46.8 (2)
O2—C4—C4A—C9B	−132.0 (2)	C19—C10—C10A—C3A	−73.1 (2)
C3A—C4—C4A—C9B	−13.1 (3)	C20—C10—C10A—C3A	167.77 (19)
O2—C4—C4A—O5	45.1 (2)	C9B—C10—C10A—C1	169.18 (17)
C3A—C4—C4A—O5	164.06 (17)	C19—C10—C10A—C1	49.3 (2)
C9B—C4A—O5—C5A	0.6 (2)	C20—C10—C10A—C1	−69.8 (2)
C4—C4A—O5—C5A	−176.98 (17)	C1—N2—C11—C16	−32.0 (3)
C4A—O5—C5A—C6	177.6 (2)	C3—N2—C11—C16	151.7 (2)
C4A—O5—C5A—C9A	−0.5 (2)	C1—N2—C11—C12	151.8 (2)
O5—C5A—C6—C7	−178.00 (19)	C3—N2—C11—C12	−24.5 (3)
C9A—C5A—C6—C7	−0.2 (3)	C16—C11—C12—C13	−2.5 (3)
C5A—C6—C7—C8	0.3 (3)	N2—C11—C12—C13	173.85 (19)
C6—C7—C8—C9	−0.3 (4)	C11—C12—C13—C14	0.2 (3)

C7—C8—C9—C9A	0.0 (4)	C11—C12—C13—C17	−179.54 (19)
O5—C5A—C9A—C9	178.00 (17)	C12—C13—C14—C15	1.6 (3)
C6—C5A—C9A—C9	−0.1 (3)	C17—C13—C14—C15	−178.6 (2)
O5—C5A—C9A—C9B	0.2 (2)	C13—C14—C15—C16	−1.2 (3)
C6—C5A—C9A—C9B	−177.9 (2)	C13—C14—C15—C18	178.4 (2)
C8—C9—C9A—C5A	0.2 (3)	C12—C11—C16—C15	2.9 (3)
C8—C9—C9A—C9B	177.2 (2)	N2—C11—C16—C15	−173.26 (19)
O5—C4A—C9B—C9A	−0.5 (2)	C14—C15—C16—C11	−1.1 (3)
C4—C4A—C9B—C9A	176.74 (19)	C18—C15—C16—C11	179.3 (2)
O5—C4A—C9B—C10	177.23 (18)	C9B—C10—C19—O3	−123.8 (2)
C4—C4A—C9B—C10	−5.5 (3)	C10A—C10—C19—O3	−7.5 (3)
C5A—C9A—C9B—C4A	0.2 (2)	C20—C10—C19—O3	113.6 (2)
C9—C9A—C9B—C4A	−177.0 (2)	C9B—C10—C19—O4	56.9 (2)
C5A—C9A—C9B—C10	−177.3 (2)	C10A—C10—C19—O4	173.20 (19)
C9—C9A—C9B—C10	5.5 (4)	C20—C10—C19—O4	−65.7 (2)
C4A—C9B—C10—C19	108.0 (2)	C23—N1—C21—O6	179.5 (2)
C9A—C9B—C10—C19	−74.9 (3)	C22—N1—C21—O6	4.0 (3)
C4A—C9B—C10—C10A	−11.0 (3)		

Hydrogen-bond geometry ($\text{\AA}$, $^\circ$)

Cg4 and Cg5 are the centroids of the C5A/C6—C9/C9A and C11—C16 rings, respectively.

$D\cdots H\cdots A$	$D\cdots H$	$H\cdots A$	$D\cdots A$	$D\cdots H\cdots A$
O2—H2O $\cdots$ O1 ⁱ	0.99 (5)	2.23 (4)	3.013 (2)	135 (3)
O2—H2O $\cdots$ O3 ⁱ	0.99 (5)	2.32 (4)	3.152 (3)	141 (3)
O4—H4O $\cdots$ O6	0.84 (4)	1.74 (4)	2.560 (3)	165 (4)
C3—H3A $\cdots$ O1 ⁱⁱ	0.99	2.54	3.452 (3)	152
C8—H8 $\cdots$ O6 ⁱⁱⁱ	0.95	2.44	3.344 (3)	159
C16—H16 $\cdots$ O1	0.95	2.44	2.970 (3)	115
C20—H20B $\cdots$ O1	0.98	2.59	3.229 (3)	123
C10A—H10A $\cdots$ Cg5 ⁱⁱ	1.00	2.88	3.865 (2)	169
C22—H22A $\cdots$ Cg5 ^{iv}	0.98	2.96	3.401 (3)	109
C22—H22C $\cdots$ Cg4 ^v	0.98	2.68	3.594 (3)	155

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