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Crystal structures of the dimethyl sulfoxide solvate of 3,6-bis(indol-3-yl)-1,4-dimethylpiperazine-2,5-dione and of the dimethyl sulfoxide and tetrahydrofuran solvates of 1,4-dimethyl-3,6-bis(2-methylindol-3-yl)piperazine-2,5-dione

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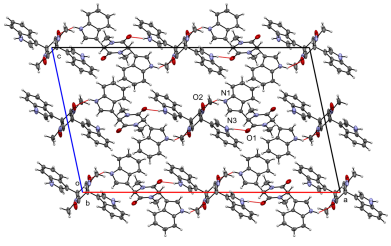
The syntheses and structures of the dimethyl sulfoxide (DMSO) solvate of 3,6-bis(indol-3-yl)-1,4-dimethylpiperazine-2,5-dione, $C_{11}H_{10}N_2O$ (**I**), and of the dimethyl sulfoxide and tetrahydrofuran (THF) solvates of 1,4-dimethyl-3,6-bis(2-methylindol-3-yl)piperazine-2,5-dione, $C_{12}H_{12}N_2O$, (**II**) and (**III**), respectively, are reported. The asymmetric units of (**I**) and (**II**) each contain two crystallographically independent half-molecules that are completed by inversion symmetry, whereas (**III**) contains one independent half-molecule. In all three structures, the piperazine-2,5-dione core is essentially planar and the overall molecular non-planarity arises from rotations of the indole substituents: ranging between 58 and 63° in (**I**), approximately 72° for both independent molecules in (**II**) and approximately 62° in (**III**). In the crystal of (**I**), molecules are linked by two N—H···O hydrogen bonds to form *C*(18) chains; (**II**) features a single N—H···O contact giving *C*(8) chains; and (**III**) exhibits N—H···O interactions that generate *C*(7) chains assembling into sheets lying parallel to (100). No significant π–π stacking is present in any of these structures. All three structures contain regions of disordered solvent (DMSO or THF) that were treated with a solvent mask during refinement.

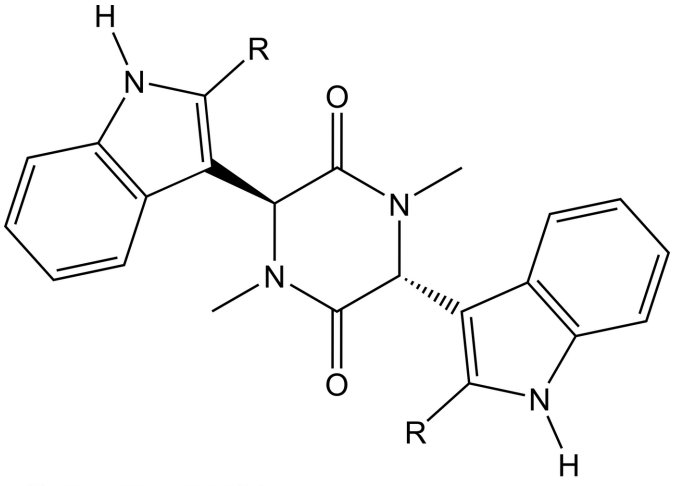
1. Chemical context

The bisindolyl piperazine-2,5-dione motif has attracted considerable interest as a precursor to the dragmacidin family of marine natural products (Garg *et al.*, 2002). These alkaloids, isolated from deep-sea sponges and tunicates, are distinguished by a piperazine core bearing indole units at the 3- and 6-positions (Kawasaki *et al.*, 2002, 2003). Members of the dragmacidin series have been reported to display anticancer, antiviral, anti-inflammatory and antibacterial activity (Cutignano *et al.*, 2000; Feldman & Ngernmeesri, 2011; Morris & Andersen, 1990; Wright *et al.*, 1992). Notably, dragmacidin, the first reported member, exhibits *in vitro* cytotoxic activity in A-549 (human lung), HCT-8 (human colon), P388 (murine leukemia) and MDA-MB (human mammary) cell lines (Kohmoto *et al.*, 1988).

3,6-Bis(indol-3-yl)-1,4-dimethylpiperazine-2,5-dione and 1,4-dimethyl-3,6-bis(2-methylindol-3-yl)piperazine-2,5-dione were prepared during efforts to access new dragmacidin derivatives (Crooke & Whitlock, 2012). In addition to a one-pot route, a reproducible two-step procedure was used:



sarcosine anhydride was brominated, and the resulting precipitate was reacted with the appropriate indole in dimethylformamide (DMF) to afford the bisindolyl products. As part of our work in this area, we now describe the syntheses and structures of the title compounds, each of which contain disordered solvent regions.



(I), R = H, + DMSO
(II), R = CH_3, + DMSO
(III) R = CH_3, + THF

2. Structural commentary

Compound (**I**) crystallizes in the monoclinic space group *C*2/c and its molecular structure is shown in Fig. 1. The asymmetric unit contains two independent molecular halves with each complete $C_{22}H_{20}N_4O_2$ molecule generated by crystallographic inversion symmetry. Each indole ring shows an r.m.s. deviation of 0.004 Å. The indole ring containing N1 is rotated by 57.9 (2)° (C10—C9—C7—C6) relative to the piperazine-2,5-dione ring (r.m.s. deviation = 0.037 Å). Similarly, the indole ring containing N3 is rotated by 63.3 (3)° (C21—C20—C18—C17) relative to the piperazine-2,5-dione ring (r.m.s. deviation = 0.072 Å). These rotations are the principal contributors to the overall nonplanarity of the molecule. The carbonyl C=O distances are normal at 1.234 (2) Å for C10=O1 and 1.230 (4) Å for C21=O2.

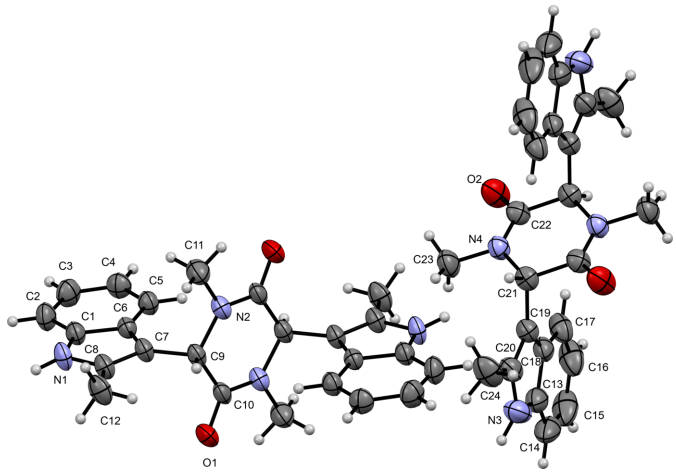


Figure 2
The molecular structure of (**II**) with displacement ellipsoids drawn at the 50% probability level. The unlabelled atoms in the C1 molecule are generated by the symmetry operation $-x+2, -y+1, -z+1$ and those in the C13 molecule by $-x, -y, -z$.

The molecular structure of compound (**II**) (triclinic, space group $P\overline{1}$) is shown in Fig. 2. The asymmetric unit also contains two independent molecular halves completed by inversion symmetry to form $C_{24}H_{24}N_4O_2$ molecules. The indole rings show r.m.s. deviations of 0.019 and 0.021 Å for the rings containing N1 and N3, respectively. The indole ring containing N1 is rotated by 71.4 (2)° (C10—C9—C7—C6) relative to the piperazine-2,5-dione ring (r.m.s. deviation = 0.037 Å). Similarly, the indole ring containing N3 is rotated by −72.7 (3)° (C22—C21—C19—C18) relative to the piperazine-2,5-dione ring (r.m.s. deviation = 0.046 Å). These rotations are the principal contributors to the overall nonplanarity of the molecule. The carbonyl C=O distances are 1.232 (3) Å (C10=O1) and 1.226 (3) Å (C22=O2).

Compound (**III**) crystallizes in the monoclinic space group $P2_1/c$ and its molecular structure is shown in Fig. 3. The

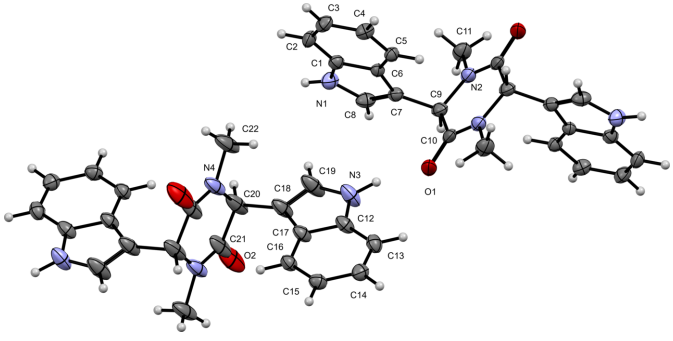


Figure 1
The molecular structure of (**I**) with displacement ellipsoids drawn at the 50% probability level. The unlabelled atoms in the C1 molecule are generated by the symmetry operation $-x+\frac{3}{2}, -y+\frac{3}{2}, -z+1$ and those in the C12 molecule by $-x+1, -y, -z+1$.

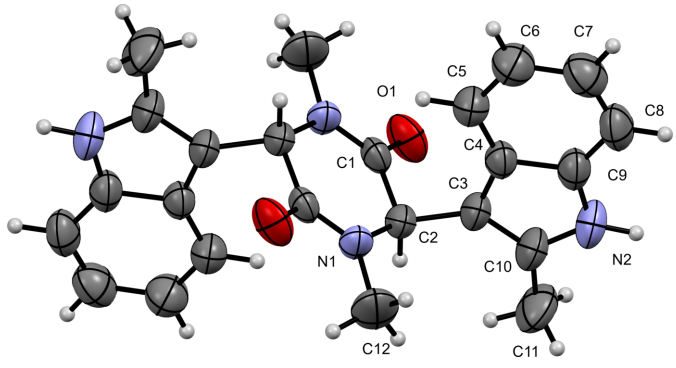


Figure 3
The molecular structure of (**III**) with displacement ellipsoids drawn at the 50% probability level. The unlabelled atoms are generated by the symmetry operation $-x+1, -y+1, -z+1$.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2^{i}	0.89 (3)	2.10 (3)	2.828 (2)	138 (2)
N3—H3A···O1	0.95 (3)	1.86 (3)	2.730 (2)	151 (3)

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

Table 2
Hydrogen-bond geometry (Å, °) for (**II**).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.85 (3)	2.11 (3)	2.950 (2)	168 (3)

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

Table 3
Hydrogen-bond geometry (Å, °) for (**III**).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1^{i}	0.91 (3)	1.89 (3)	2.787 (2)	168 (3)

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

asymmetric unit contains a half molecule, which is completed by inversion symmetry to generate a $C_{24}H_{24}N_4O_2$ molecule. The indole ring shows an r.m.s. deviation of 0.010 Å. The indole ring is rotated by −62.4 (3)° (C1—C2—C3—C4) relative to the piperazine-2,5-dione ring (r.m.s. deviation = 0.031 Å) and the carbonyl C1═O1 distance is 1.231 (2) Å.

All three structures contain disordered solvent regions that were masked (see *Refinement*).

3. Supramolecular features

In the crystal of (**I**), the molecules are linked by two N—H···O hydrogen bonds (Table 1, Fig. 4): N1—H1···O2, which generates chains parallel to [1$\bar{1}$0] with graph-set motif $C_2^2(18)$, and N3—H3···O1, forming chains along [130] with graph-set motif $C_2^2(18)$. Together, the hydrogen bonds generate (001) sheets. No significant aromatic π–π stacking is observed.

In the crystal of (**II**), molecules are linked by an N1—H1···O2 hydrogen bond (Table 2, Fig. 5), which generates chains running parallel to [100] with graph-set motif $C_1^1(8)$. The second molecule (containing N3) is not involved in hydrogen bonding. No significant π–π stacking is observed.

In the crystal of (**III**), molecules are linked by an N2—H2···O1 hydrogen bond (Table 3, Fig. 6), which generates chains of N—H···O hydrogen bonds [graph-set motif $C_1^1(7)$] that form sheets parallel to the (100) plane. No significant π–π stacking is observed.

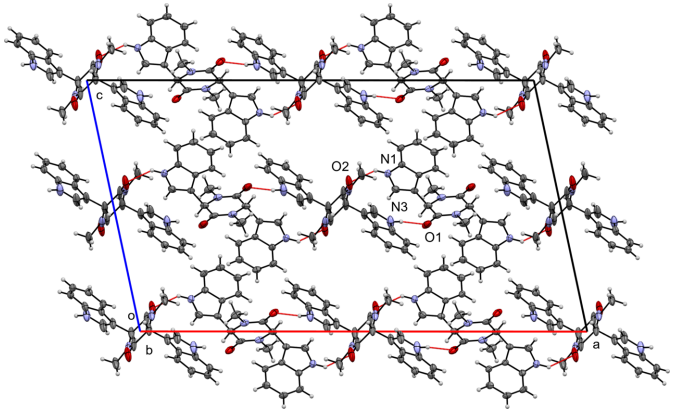


Figure 4
A view along the *b*-axis direction of the crystal packing of (**I**) with close contacts shown as red dashed lines.

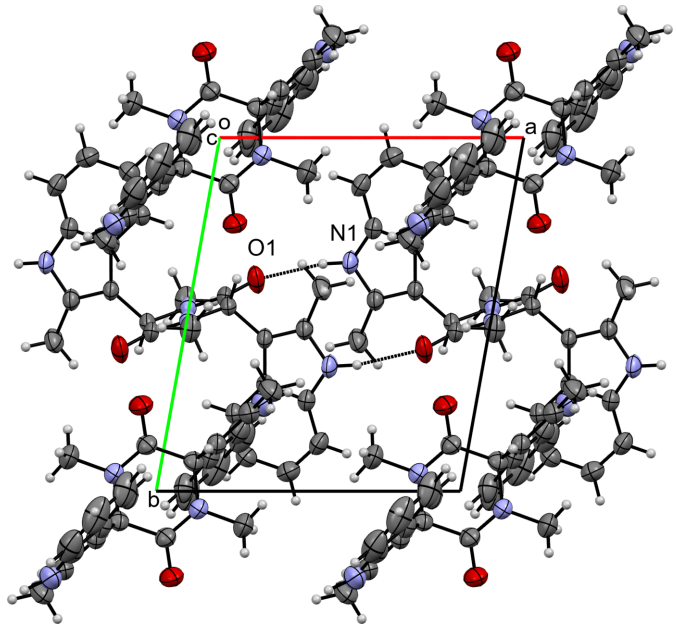


Figure 5
A view along the *c*-axis direction of the crystal packing of (**II**) with close contacts shown as red dashed lines.

4. Database survey

A search of the Cambridge Structural Database ((CSD; website, accessed on August, 2025; Groom *et al.*, 2016) for 1,4-dimethyl-3,6-dioxopiperazine (diketopiperazine) frameworks bearing 2,6-substitution returned 13 structures. Seven entries feature simple alkyl groups; methyl (CSD refcode

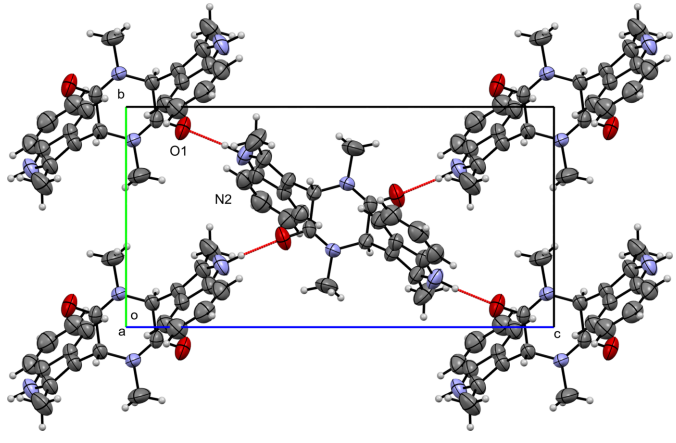


Figure 6
A view along the *a*-axis direction of the crystal packing of (**III**) with close contacts shown as red dashed lines.

Table 4
Experimental details.

	(**I**)	(**II**)	(**III**)
Crystal data			
Chemical formula	$C_{22}H_{20}N_4O_2{\cdot}0.5C_2H_6OS$	$C_{24}H_{24}N_4O_2{\cdot}C_2H_6SO$	$C_{24}H_{24}N_4O_2{\cdot}C_4H_8O$
M_r	411.48	478.60	472.57
Crystal system, space group	Monoclinic, *C*2/*c*	Triclinic, $P\overline{1}$	Monoclinic, $P2_1/c$
Temperature (K)	100	298	293
a, *b*, *c* (Å)	31.2994 (4), 7.3881 (1), 17.9817 (2)	9.3137 (2), 12.3332 (2), 12.3873 (2)	8.5276 (9), 8.9357 (7), 17.4883 (13)
α, β, γ (°)	90, 102.023 (1), 90	117.110 (2), 93.520 (1), 97.399 (2)	90, 96.731 (8), 90
V (Å^3)	4066.93 (9)	1244.35 (4)	1323.4 (2)
Z	8	2	2
Radiation type	Cu *K*α	Cu *K*α	Mo *K*α
μ (mm^{-1})	1.18	1.44	0.08
Crystal size (mm)	0.10 × 0.06 × 0.04	0.36 × 0.11 × 0.07	0.4 × 0.4 × 0.3
Data collection			
Diffractometer	XtaLAB Synergy, Single source at home/near, HyPix3000	XtaLAB Synergy, Single source at home/near, HyPix3000	XtaLAB Mini (ROW)
Absorption correction	Gaussian (*CrysAlis PRO*; Rigaku OD, 2023)	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2023)	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2023)
T_{min}, T_{max}	0.927, 1.000	0.436, 1.000	0.968, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	12151, 3716, 3134	25135, 4560, 4029	6423, 2420, 1519
R_{int}	0.027	0.039	0.031
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.602	0.602	0.602
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.046, 0.120, 1.05	0.058, 0.177, 1.06	0.053, 0.178, 1.02
No. of reflections	3716	4560	2420
No. of parameters	263	283	143
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.21, −0.36	0.60, −0.21	0.26, −0.15

Computer programs: *CrysAlis PRO* (Rigaku OD, 2023), *SHELXT2018/2* (Sheldrick, 2015*a*), *SHELXL2018/3* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

RMNALA10; Benedetti *et al.*, 1976) and isopropyl (NMLVAL10; Benedetti *et al.*, 1976), a mixed methyl/isopropyl pair (MOJTUB; Wang *et al.*, 2008), and 1,1,1-trifluoroisopropyl (LAKGAF; Su *et al.*, 1993). Five entries carry benzylic/aromatic groups, including benzyl (NMLPHE11; Ge *et al.*, 2019) and (4-hydroxyphenyl)methyl (NIPBIB; Croft *et al.*, 2004). One structure bears a carboxylate protecting group, *tert*-butoxycarbonyl (EZESIO; Yang, 2021).

5. Synthesis and crystallization

To prepare compound (**I**), 0.0117 g of 3,6-bis(indol-3-yl)-1,4-dimethylpiperazine-2,5-dione (Miles & Whitlock, 2009) were dissolved in ∼10 ml of dimethylsulfoxide (DMSO) and heated to ∼423 K in a 50 ml beaker. The beaker was placed in a fume hood to allow slow evaporation for approximately 1 week, after which X-ray quality crystals began to form.

Compound (**II**): a 0.0079-g sample of 1,4-dimethyl-3,6-bis(2-methylindol-3-yl)piperazine-2,5-dione (Miles & Whitlock, 2009) was dissolved in ∼5 ml of DMSO and heated to near boiling (∼423 K) in a 50 ml beaker. The beaker was placed in a fume hood to allow slow evaporation for approximately 1 week, after which X-ray quality crystals began to form.

Compound (**III**) was prepared by the same procedure as (**II**) except that 0.050 g of 1,4-dimethyl-3,6-bis(2-methylindol-3-yl)piperazine-2,5-dione (0.05 g) (Miles & Whitlock, 2009) was dissolved in ∼50 ml of tetrahydrofuran (THF) in a 100 ml beaker. The solution was allowed to slowly evaporate for approximately 1 week, after which X-ray quality crystals began to form.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 4. In all three structures, disordered solvent regions were treated using the built-in solvent-mask routine in *OLEX2*. Solvent-accessible voids were located in each unit cell with the following characteristics: for compound (**I**), a total cavity volume of 520 Å^3 per unit cell containing 172 electrons (consistent with one C_2H_6OS molecule per asymmetric unit, 168 electrons per cell); for compound (**II**), a 312 Å^3 void with 85 electrons (one C_2H_6OS per asymmetric unit, 84 electrons per cell); and for compound (**III**), a 404 Å^3 cavity holding 90 electrons (one C_4H_8O per asymmetric unit, 80 electrons per cell). All disordered solvent electron density was subsequently removed *via* the solvent-mask procedure.

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supporting information

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Crystal structures of the dimethyl sulfoxide solvate of 3,6-bis(indol-3-yl)-1,4-dimethylpiperazine-2,5-dione and of the dimethyl sulfoxide and tetrahydrofuran solvates of 1,4-dimethyl-3,6-bis(2-methylindol-3-yl)piperazine-2,5-dione

Clifford W. Padgett, Will E. Lynch, Stephen N. Crooke and Christine R. Whitlock

Computing details

3,6-Bis(indol-3-yl)-1,4-dimethylpiperazine-2,5-dione dimethyl sulfoxide hemihydrate (I)

Crystal data

$C_{22}H_{20}N_4O_2 \cdot 0.5C_2H_6OS$
$M_r = 411.48$
Monoclinic, $C2/c$
$a = 31.2994$ (4) Å
$b = 7.3881$ (1) Å
$c = 17.9817$ (2) Å
$\beta = 102.023$ (1)°
$V = 4066.93$ (9) Å^3
$Z = 8$
$F(000) = 1736$
$D_x = 1.344$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 6717 reflections
$\theta = 2.9$–$69.6°$
$\mu = 1.18$ mm^{-1}
$T = 100$ K
Block, clear colourless
0.10 × 0.06 × 0.04 mm

Data collection

XtaLAB Synergy, Single source at home/near, HyPix3000 diffractometer
Radiation source: micro-focus sealed X-ray tube, PhotonJet (Cu) X-ray Source
Mirror monochromator
Detector resolution: 10.0000 pixels mm^{-1}
ω scans
Absorption correction: gaussian (CrysAlisPro; Rigaku OD, 2023)
$T_{min} = 0.927$, $T_{max} = 1.000$
12151 measured reflections
3716 independent reflections
3134 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 68.3°$, $\theta_{min} = 2.9°$
$h = -37 \rightarrow 37$
$k = -8 \rightarrow 8$
$l = -21 \rightarrow 19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.120$
$S = 1.05$
3716 reflections
263 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0451P)^2 + 5.8441P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.36$ 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	0.69198 (5)	0.5128 (2)	0.42587 (8)	0.0444 (4)
N2	0.76249 (5)	0.5922 (2)	0.46638 (8)	0.0308 (4)
N1	0.87085 (5)	0.8346 (3)	0.37106 (9)	0.0344 (4)
O2	0.53832 (5)	−0.2656 (3)	0.59204 (13)	0.0703 (6)
C6	0.80197 (6)	0.9116 (2)	0.37659 (9)	0.0220 (4)
N3	0.61216 (6)	0.3788 (3)	0.43637 (13)	0.0474 (5)
N4	0.53359 (5)	0.0338 (3)	0.56374 (12)	0.0489 (5)
C1	0.83537 (6)	0.9333 (3)	0.33524 (10)	0.0264 (4)
C5	0.76208 (6)	1.0008 (2)	0.35093 (9)	0.0250 (4)
H5	0.739130	0.989127	0.377837	0.030*
C7	0.81931 (6)	0.7946 (3)	0.43927 (9)	0.0258 (4)
C9	0.79628 (6)	0.7240 (3)	0.49886 (10)	0.0296 (4)
H9	0.818533	0.658316	0.537430	0.035*
C10	0.71973 (7)	0.6176 (3)	0.46083 (10)	0.0314 (4)
C4	0.75654 (6)	1.1058 (3)	0.28604 (10)	0.0299 (4)
H4	0.729678	1.167259	0.268447	0.036*
C16	0.56976 (6)	−0.0656 (3)	0.39021 (11)	0.0303 (4)
H16	0.546853	−0.137952	0.401555	0.036*
C8	0.86087 (6)	0.7530 (3)	0.43377 (10)	0.0329 (4)
H8	0.880200	0.678246	0.468420	0.040*
C3	0.79030 (7)	1.1224 (3)	0.24595 (10)	0.0325 (4)
H3	0.785603	1.193938	0.201093	0.039*
C17	0.57703 (6)	0.1098 (3)	0.42017 (12)	0.0334 (4)
C15	0.59634 (6)	−0.1316 (3)	0.34402 (11)	0.0315 (4)
H15	0.591717	−0.250734	0.324046	0.038*
C2	0.82979 (6)	1.0385 (3)	0.26952 (10)	0.0315 (4)
H2	0.852523	1.051494	0.242183	0.038*
C14	0.63015 (6)	−0.0256 (3)	0.32604 (11)	0.0324 (4)
H14	0.647679	−0.074018	0.293596	0.039*
C12	0.61152 (6)	0.2127 (3)	0.40155 (12)	0.0346 (5)
C13	0.63833 (6)	0.1467 (3)	0.35452 (11)	0.0338 (4)
H13	0.661286	0.217965	0.342668	0.041*
C11	0.77761 (8)	0.4289 (3)	0.43354 (12)	0.0408 (5)
H11A	0.783541	0.457243	0.383422	0.061*
H11B	0.755010	0.335199	0.428275	0.061*
H11C	0.804409	0.384838	0.466972	0.061*
C18	0.55721 (6)	0.2240 (3)	0.46845 (14)	0.0447 (6)
C21	0.52059 (7)	−0.1388 (4)	0.55313 (16)	0.0543 (7)
C20	0.52044 (7)	0.1779 (4)	0.50718 (15)	0.0515 (7)

H20	0.514382	0.288344	0.535425	0.062*
C19	0.57983 (7)	0.3831 (3)	0.47672 (16)	0.0540 (7)
H19	0.573892	0.482684	0.506405	0.065*
C22	0.56984 (7)	0.0801 (4)	0.62727 (16)	0.0614 (8)
H22A	0.570409	−0.005583	0.669088	0.092*
H22B	0.565694	0.203079	0.644882	0.092*
H22C	0.597536	0.073426	0.610121	0.092*
H1	0.8970 (9)	0.837 (4)	0.3587 (14)	0.054 (7)*
H3A	0.6357 (10)	0.461 (4)	0.4377 (17)	0.076 (9)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0531 (9)	0.0501 (9)	0.0362 (7)	−0.0279 (8)	0.0232 (7)	−0.0200 (7)
N2	0.0444 (10)	0.0248 (8)	0.0255 (8)	−0.0095 (7)	0.0124 (7)	−0.0039 (6)
N1	0.0252 (8)	0.0467 (10)	0.0321 (8)	0.0051 (8)	0.0080 (7)	−0.0045 (8)
O2	0.0217 (8)	0.0698 (13)	0.1167 (17)	0.0058 (9)	0.0079 (9)	−0.0323 (12)
C6	0.0252 (8)	0.0206 (8)	0.0209 (8)	−0.0030 (7)	0.0067 (6)	−0.0032 (7)
N3	0.0293 (9)	0.0349 (10)	0.0782 (14)	−0.0068 (8)	0.0118 (9)	−0.0178 (10)
N4	0.0218 (8)	0.0628 (14)	0.0636 (12)	−0.0095 (9)	0.0121 (8)	−0.0367 (11)
C1	0.0258 (9)	0.0288 (10)	0.0253 (9)	−0.0001 (8)	0.0065 (7)	−0.0057 (7)
C5	0.0263 (9)	0.0259 (9)	0.0243 (8)	−0.0006 (7)	0.0088 (7)	−0.0022 (7)
C7	0.0292 (9)	0.0256 (9)	0.0218 (8)	−0.0011 (8)	0.0037 (7)	−0.0022 (7)
C9	0.0392 (10)	0.0282 (10)	0.0203 (8)	−0.0052 (8)	0.0039 (7)	0.0005 (7)
C10	0.0474 (11)	0.0303 (10)	0.0194 (8)	−0.0143 (9)	0.0139 (8)	−0.0034 (7)
C4	0.0338 (10)	0.0279 (10)	0.0276 (9)	0.0063 (8)	0.0056 (7)	−0.0005 (8)
C16	0.0225 (9)	0.0314 (10)	0.0364 (10)	−0.0024 (8)	0.0052 (7)	−0.0047 (8)
C8	0.0328 (10)	0.0354 (11)	0.0282 (9)	0.0062 (9)	0.0006 (7)	−0.0025 (8)
C3	0.0440 (11)	0.0303 (10)	0.0250 (9)	0.0000 (9)	0.0108 (8)	0.0038 (8)
C17	0.0186 (8)	0.0348 (11)	0.0453 (11)	−0.0003 (8)	0.0031 (8)	−0.0109 (9)
C15	0.0301 (9)	0.0308 (10)	0.0325 (10)	0.0011 (8)	0.0042 (8)	−0.0048 (8)
C2	0.0358 (10)	0.0354 (11)	0.0275 (9)	−0.0046 (9)	0.0164 (8)	−0.0011 (8)
C14	0.0265 (9)	0.0398 (11)	0.0309 (9)	0.0049 (8)	0.0058 (7)	−0.0010 (9)
C12	0.0220 (9)	0.0316 (11)	0.0478 (11)	−0.0011 (8)	0.0015 (8)	−0.0056 (9)
C13	0.0230 (9)	0.0381 (11)	0.0397 (10)	−0.0015 (8)	0.0048 (8)	0.0036 (9)
C11	0.0601 (14)	0.0277 (11)	0.0393 (11)	−0.0066 (10)	0.0208 (10)	−0.0081 (9)
C18	0.0201 (9)	0.0458 (13)	0.0677 (15)	−0.0031 (9)	0.0082 (9)	−0.0293 (12)
C21	0.0170 (10)	0.0634 (17)	0.0854 (18)	−0.0041 (11)	0.0175 (11)	−0.0408 (15)
C20	0.0218 (10)	0.0555 (15)	0.0787 (17)	−0.0048 (10)	0.0141 (10)	−0.0425 (14)
C19	0.0269 (10)	0.0444 (14)	0.0899 (19)	−0.0028 (10)	0.0104 (11)	−0.0347 (13)
C22	0.0233 (10)	0.081 (2)	0.0773 (17)	−0.0086 (12)	0.0056 (11)	−0.0446 (16)

Geometric parameters (Å, º)

O1—C10	1.234 (2)	C16—H16	0.9500
N2—C9	1.466 (2)	C16—C17	1.404 (3)
N2—C10	1.335 (3)	C16—C15	1.381 (3)
N2—C11	1.465 (3)	C8—H8	0.9500

N1—C1	1.372 (2)	C3—H3	0.9500
N1—C8	1.371 (3)	C3—C2	1.369 (3)
N1—H1	0.89 (3)	C17—C12	1.417 (3)
O2—C21	1.230 (4)	C17—C18	1.440 (3)
C6—C1	1.413 (2)	C15—H15	0.9500
C6—C5	1.402 (2)	C15—C14	1.407 (3)
C6—C7	1.434 (2)	C2—H2	0.9500
N3—C12	1.376 (3)	C14—H14	0.9500
N3—C19	1.363 (3)	C14—C13	1.377 (3)
N3—H3A	0.95 (3)	C12—C13	1.397 (3)
N4—C21	1.340 (3)	C13—H13	0.9500
N4—C20	1.471 (4)	C11—H11A	0.9800
N4—C22	1.473 (3)	C11—H11B	0.9800
C1—C2	1.395 (3)	C11—H11C	0.9800
C5—H5	0.9500	C18—C20	1.503 (3)
C5—C4	1.382 (3)	C18—C19	1.364 (3)
C7—C9	1.504 (2)	C21—C20^{ii}	1.526 (3)
C7—C8	1.360 (3)	C20—H20	1.0000
C9—H9	1.0000	C19—H19	0.9500
C9—C10^{i}	1.516 (3)	C22—H22A	0.9800
C4—H4	0.9500	C22—H22B	0.9800
C4—C3	1.403 (3)	C22—H22C	0.9800

C10—N2—C9	124.44 (17)	C16—C17—C12	118.38 (18)
C10—N2—C11	119.27 (17)	C16—C17—C18	135.76 (19)
C11—N2—C9	116.15 (17)	C12—C17—C18	105.86 (18)
C1—N1—H1	124.6 (17)	C16—C15—H15	119.4
C8—N1—C1	108.59 (16)	C16—C15—C14	121.16 (19)
C8—N1—H1	126.3 (17)	C14—C15—H15	119.4
C1—C6—C7	106.35 (15)	C1—C2—H2	121.3
C5—C6—C1	118.65 (16)	C3—C2—C1	117.39 (17)
C5—C6—C7	135.01 (16)	C3—C2—H2	121.3
C12—N3—H3A	121.3 (19)	C15—C14—H14	119.3
C19—N3—C12	108.71 (19)	C13—C14—C15	121.33 (18)
C19—N3—H3A	129.0 (19)	C13—C14—H14	119.3
C21—N4—C20	123.9 (2)	N3—C12—C17	108.09 (18)
C21—N4—C22	119.5 (3)	N3—C12—C13	129.3 (2)
C20—N4—C22	115.3 (2)	C13—C12—C17	122.63 (19)
N1—C1—C6	107.95 (16)	C14—C13—C12	117.34 (18)
N1—C1—C2	129.81 (17)	C14—C13—H13	121.3
C2—C1—C6	122.24 (17)	C12—C13—H13	121.3
C6—C5—H5	120.4	N2—C11—H11A	109.5
C4—C5—C6	119.23 (16)	N2—C11—H11B	109.5
C4—C5—H5	120.4	N2—C11—H11C	109.5
C6—C7—C9	127.58 (16)	H11A—C11—H11B	109.5
C8—C7—C6	106.87 (16)	H11A—C11—H11C	109.5
C8—C7—C9	125.51 (17)	H11B—C11—H11C	109.5
N2—C9—C7	111.08 (14)	C17—C18—C20	127.9 (2)

N2—C9—H9	107.1	C19—C18—C17	106.9 (2)
N2—C9—C10^{i}	114.85 (16)	C19—C18—C20	125.1 (2)
C7—C9—H9	107.1	O2—C21—N4	123.8 (2)
C7—C9—C10^{i}	109.13 (15)	O2—C21—C20^{ii}	118.3 (2)
C10^{i}—C9—H9	107.1	N4—C21—C20^{ii}	117.8 (3)
O1—C10—N2	122.59 (19)	N4—C20—C18	110.70 (18)
O1—C10—C9^{i}	117.43 (18)	N4—C20—C21^{ii}	115.3 (2)
N2—C10—C9^{i}	119.93 (17)	N4—C20—H20	107.1
C5—C4—H4	119.8	C18—C20—C21^{ii}	109.0 (2)
C5—C4—C3	120.49 (17)	C18—C20—H20	107.1
C3—C4—H4	119.8	C21^{ii}—C20—H20	107.1
C17—C16—H16	120.4	N3—C19—C18	110.4 (2)
C15—C16—H16	120.4	N3—C19—H19	124.8
C15—C16—C17	119.16 (18)	C18—C19—H19	124.8
N1—C8—H8	124.9	N4—C22—H22A	109.5
C7—C8—N1	110.24 (17)	N4—C22—H22B	109.5
C7—C8—H8	124.9	N4—C22—H22C	109.5
C4—C3—H3	119.0	H22A—C22—H22B	109.5
C2—C3—C4	121.99 (17)	H22A—C22—H22C	109.5
C2—C3—H3	119.0	H22B—C22—H22C	109.5

N1—C1—C2—C3	179.52 (19)	C8—C7—C9—N2	−107.9 (2)
C6—C1—C2—C3	0.2 (3)	C8—C7—C9—C10^{i}	124.5 (2)
C6—C5—C4—C3	0.4 (3)	C17—C16—C15—C14	−0.6 (3)
C6—C7—C9—N2	69.8 (2)	C17—C12—C13—C14	−0.1 (3)
C6—C7—C9—C10^{i}	−57.9 (2)	C17—C18—C20—N4	64.5 (3)
C6—C7—C8—N1	−0.7 (2)	C17—C18—C20—C21^{ii}	−63.3 (3)
N3—C12—C13—C14	−179.1 (2)	C17—C18—C19—N3	0.8 (3)
C1—N1—C8—C7	0.7 (2)	C15—C16—C17—C12	0.1 (3)
C1—C6—C5—C4	0.4 (3)	C15—C16—C17—C18	179.7 (2)
C1—C6—C7—C9	−177.64 (17)	C15—C14—C13—C12	−0.4 (3)
C1—C6—C7—C8	0.4 (2)	C12—N3—C19—C18	−1.0 (3)
C5—C6—C1—N1	179.87 (16)	C12—C17—C18—C20	−176.9 (2)
C5—C6—C1—C2	−0.7 (3)	C12—C17—C18—C19	−0.3 (3)
C5—C6—C7—C9	2.6 (3)	C11—N2—C9—C7	61.3 (2)
C5—C6—C7—C8	−179.4 (2)	C11—N2—C9—C10^{i}	−174.29 (16)
C5—C4—C3—C2	−0.9 (3)	C11—N2—C10—O1	−3.5 (3)
C7—C6—C1—N1	0.1 (2)	C11—N2—C10—C9^{i}	173.93 (16)
C7—C6—C1—C2	179.48 (17)	C18—C17—C12—N3	−0.2 (2)
C7—C6—C5—C4	−179.87 (19)	C18—C17—C12—C13	−179.45 (19)
C9—N2—C10—O1	171.94 (17)	C21—N4—C20—C18	−104.3 (2)
C9—N2—C10—C9^{i}	−10.6 (3)	C21—N4—C20—C21^{ii}	20.0 (3)
C9—C7—C8—N1	177.38 (17)	C20—N4—C21—O2	163.8 (2)
C10—N2—C9—C7	−114.30 (19)	C20—N4—C21—C20^{ii}	−20.5 (3)
C10—N2—C9—C10^{i}	10.1 (3)	C20—C18—C19—N3	177.5 (2)
C4—C3—C2—C1	0.6 (3)	C19—N3—C12—C17	0.7 (3)
C16—C17—C12—N3	179.44 (18)	C19—N3—C12—C13	179.9 (2)
C16—C17—C12—C13	0.2 (3)	C19—C18—C20—N4	−111.5 (3)

C16—C17—C18—C20	3.5 (4)	C19—C18—C20—C21^{ii}	120.6 (3)
C16—C17—C18—C19	−179.9 (2)	C22—N4—C21—O2	−2.9 (3)
C16—C15—C14—C13	0.8 (3)	C22—N4—C21—C20^{ii}	172.8 (2)
C8—N1—C1—C6	−0.5 (2)	C22—N4—C20—C18	62.9 (2)
C8—N1—C1—C2	−179.8 (2)	C22—N4—C20—C21^{ii}	−172.75 (18)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2^{iii}	0.89 (3)	2.10 (3)	2.828 (2)	138 (2)
N3—H3*A*···O1	0.95 (3)	1.86 (3)	2.730 (2)	151 (3)

Symmetry code: (iii) −*x*+3/2, −*y*+1/2, −*z*+1.

1,4-Dimethyl-3,6-bis(2-methylindol-3-yl)piperazine-2,5-dione dimethyl sulfoxide monosolvate (II)

Crystal data

$C_{24}H_{24}N_4O_2·C_2H_6SO$
M_r = 478.60
Triclinic, $P\overline{1}$
a = 9.3137 (2) Å
b = 12.3332 (2) Å
c = 12.3873 (2) Å
α = 117.110 (2)°
β = 93.520 (1)°
γ = 97.399 (2)°
V = 1244.35 (4) Å^3
Z = 2
F(000) = 508
D_x = 1.277 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 17902 reflections
θ = 4.0–69.7°
μ = 1.44 mm^{-1}
T = 298 K
Block, clear colourless
0.36 × 0.11 × 0.07 mm

Data collection

XtaLAB Synergy, Single source at home/near, HyPix3000 diffractometer
Radiation source: micro-focus sealed X-ray tube, PhotonJet (Cu) X-ray Source
Mirror monochromator
Detector resolution: 10.0000 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2023)
T_{min} = 0.436, T_{max} = 1.000
25135 measured reflections
4560 independent reflections
4029 reflections with $I > 2\sigma(I)$
R_{int} = 0.039
θ_{max} = 68.2°, θ_{min} = 4.1°
h = −11→11
k = −14→14
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.177
S = 1.06
4560 reflections
283 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0886P)^2 + 0.6913P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ < 0.001
$\Delta\rho_{max}$ = 0.60 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 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	1.20329 (17)	0.40135 (17)	0.56994 (18)	0.0588 (5)
O2	0.1008 (2)	0.24272 (15)	0.12756 (17)	0.0649 (5)
N2	0.99719 (17)	0.48117 (16)	0.60324 (16)	0.0397 (4)
N4	0.14924 (19)	0.05031 (16)	0.02277 (16)	0.0428 (4)
N1	0.50383 (19)	0.35296 (18)	0.53767 (18)	0.0472 (5)
C6	0.7058 (2)	0.30236 (18)	0.44973 (18)	0.0358 (4)
C10	1.1103 (2)	0.44957 (18)	0.5413 (2)	0.0398 (5)
C7	0.7330 (2)	0.43181 (18)	0.53668 (18)	0.0372 (4)
N3	0.2971 (3)	−0.2443 (2)	0.1043 (2)	0.0638 (6)
C9	0.87168 (19)	0.52304 (18)	0.56525 (19)	0.0373 (4)
H9	0.859990	0.600449	0.635377	0.045*
C1	0.5593 (2)	0.2567 (2)	0.4515 (2)	0.0411 (5)
C8	0.6080 (2)	0.4585 (2)	0.58912 (19)	0.0416 (5)
C5	0.7883 (2)	0.21910 (19)	0.37191 (19)	0.0419 (5)
H5	0.885399	0.245795	0.369211	0.050*
C19	0.1706 (2)	−0.1339 (2)	0.04553 (19)	0.0421 (5)
C22	0.0582 (2)	0.13100 (19)	0.06521 (19)	0.0430 (5)
C18	0.1568 (2)	−0.0974 (2)	0.1717 (2)	0.0465 (5)
C21	0.1057 (2)	−0.08458 (19)	−0.03270 (18)	0.0403 (4)
H21	0.147604	−0.119704	−0.109616	0.048*
C4	0.7237 (2)	0.0973 (2)	0.2995 (2)	0.0486 (5)
H4	0.778644	0.041646	0.248834	0.058*
C20	0.2590 (2)	−0.2212 (2)	0.0089 (2)	0.0511 (5)
C2	0.4933 (2)	0.1344 (2)	0.3766 (2)	0.0519 (6)
H2	0.395996	0.106783	0.377810	0.062*
C3	0.5770 (3)	0.0556 (2)	0.3007 (2)	0.0546 (6)
H3	0.535511	−0.026734	0.249262	0.065*
C13	0.2377 (3)	−0.1687 (2)	0.2053 (3)	0.0569 (6)
C23	0.3070 (2)	0.0969 (2)	0.0516 (3)	0.0577 (6)
H23A	0.327020	0.174278	0.049641	0.087*
H23B	0.356563	0.038075	−0.007708	0.087*
H23C	0.340683	0.108909	0.131721	0.087*
C12	0.5754 (3)	0.5746 (2)	0.6888 (2)	0.0556 (6)
H12A	0.565830	0.564851	0.760764	0.083*
H12B	0.485780	0.592162	0.663120	0.083*
H12C	0.653563	0.641771	0.706880	0.083*
C17	0.0903 (3)	−0.0086 (3)	0.2608 (2)	0.0586 (6)
H17	0.037260	0.040576	0.241326	0.070*
C11	0.9833 (3)	0.4598 (3)	0.7088 (2)	0.0563 (6)

H11A	1.078464	0.462640	0.745463	0.084*
H11B	0.924690	0.380024	0.683006	0.084*
H11C	0.937461	0.522671	0.767542	0.084*
C16	0.1048 (3)	0.0049 (4)	0.3782 (3)	0.0756 (9)
H16	0.061535	0.064059	0.438113	0.091*
C24	0.3164 (3)	−0.2864 (3)	−0.1101 (3)	0.0691 (8)
H24A	0.409581	−0.306274	−0.095669	0.104*
H24B	0.327194	−0.233579	−0.147959	0.104*
H24C	0.249199	−0.361111	−0.163052	0.104*
C14	0.2508 (3)	−0.1543 (3)	0.3241 (3)	0.0760 (9)
H14	0.304350	−0.202074	0.345346	0.091*
C15	0.1829 (4)	−0.0684 (4)	0.4080 (3)	0.0858 (11)
H15	0.189089	−0.058611	0.487213	0.103*
H1	0.414 (4)	0.357 (3)	0.548 (3)	0.073 (9)*
H3A	0.375 (4)	−0.280 (3)	0.111 (3)	0.095 (11)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0336 (8)	0.0773 (12)	0.0873 (12)	0.0219 (8)	0.0136 (8)	0.0533 (10)
O2	0.0573 (10)	0.0392 (9)	0.0720 (12)	0.0033 (7)	−0.0119 (9)	0.0077 (8)
N2	0.0262 (8)	0.0467 (9)	0.0451 (9)	0.0066 (7)	0.0039 (7)	0.0205 (8)
N4	0.0352 (9)	0.0410 (9)	0.0461 (10)	0.0036 (7)	0.0004 (7)	0.0165 (8)
N1	0.0245 (8)	0.0544 (11)	0.0559 (11)	0.0062 (7)	0.0117 (7)	0.0194 (9)
C6	0.0272 (9)	0.0402 (10)	0.0395 (10)	0.0052 (7)	0.0041 (7)	0.0185 (8)
C10	0.0227 (9)	0.0392 (10)	0.0544 (12)	0.0045 (7)	0.0017 (8)	0.0200 (9)
C7	0.0243 (9)	0.0418 (10)	0.0408 (10)	0.0060 (7)	0.0050 (7)	0.0153 (9)
N3	0.0533 (12)	0.0568 (13)	0.0852 (16)	0.0139 (10)	−0.0035 (11)	0.0370 (12)
C9	0.0247 (9)	0.0357 (10)	0.0441 (10)	0.0069 (7)	0.0048 (7)	0.0121 (8)
C1	0.0293 (9)	0.0456 (11)	0.0474 (11)	0.0055 (8)	0.0061 (8)	0.0212 (9)
C8	0.0287 (9)	0.0475 (11)	0.0436 (11)	0.0082 (8)	0.0068 (8)	0.0165 (9)
C5	0.0325 (10)	0.0434 (11)	0.0480 (12)	0.0080 (8)	0.0093 (8)	0.0189 (9)
C19	0.0359 (10)	0.0442 (11)	0.0437 (11)	0.0075 (8)	0.0044 (8)	0.0185 (9)
C22	0.0432 (11)	0.0401 (11)	0.0371 (10)	0.0048 (9)	−0.0028 (8)	0.0123 (9)
C18	0.0331 (10)	0.0582 (13)	0.0500 (12)	0.0017 (9)	0.0023 (9)	0.0289 (11)
C21	0.0404 (11)	0.0399 (11)	0.0342 (10)	0.0091 (8)	0.0059 (8)	0.0112 (8)
C4	0.0473 (12)	0.0426 (11)	0.0514 (13)	0.0120 (9)	0.0115 (10)	0.0166 (10)
C20	0.0419 (12)	0.0436 (12)	0.0617 (14)	0.0084 (9)	0.0021 (10)	0.0197 (11)
C2	0.0343 (11)	0.0515 (13)	0.0618 (14)	−0.0041 (9)	0.0031 (10)	0.0229 (11)
C3	0.0509 (13)	0.0411 (12)	0.0599 (14)	−0.0003 (10)	0.0036 (11)	0.0163 (11)
C13	0.0442 (12)	0.0642 (15)	0.0688 (16)	−0.0034 (11)	−0.0046 (11)	0.0415 (13)
C23	0.0367 (12)	0.0583 (14)	0.0704 (16)	0.0011 (10)	−0.0013 (11)	0.0266 (13)
C12	0.0387 (11)	0.0606 (14)	0.0535 (13)	0.0165 (10)	0.0133 (10)	0.0121 (11)
C17	0.0415 (12)	0.0866 (18)	0.0497 (13)	0.0120 (12)	0.0113 (10)	0.0327 (13)
C11	0.0424 (12)	0.0770 (17)	0.0587 (14)	0.0101 (11)	0.0062 (10)	0.0399 (13)
C16	0.0521 (15)	0.123 (3)	0.0509 (15)	0.0039 (16)	0.0127 (12)	0.0420 (17)
C24	0.0587 (15)	0.0563 (15)	0.0728 (18)	0.0226 (12)	0.0116 (13)	0.0102 (13)
C14	0.0594 (16)	0.101 (2)	0.087 (2)	−0.0105 (16)	−0.0146 (15)	0.071 (2)

C15	0.0646 (18)	0.140 (3)	0.0644 (18)	−0.014 (2)	0.0007 (15)	0.067 (2)

Geometric parameters (Å, º)

O1—C10	1.232 (3)	C18—C13	1.407 (3)
O2—C22	1.226 (3)	C18—C17	1.400 (4)
N2—C10	1.336 (3)	C21—H21	0.9800
N2—C9	1.471 (2)	C4—H4	0.9300
N2—C11	1.457 (3)	C4—C3	1.400 (3)
N4—C22	1.334 (3)	C20—C24	1.494 (4)
N4—C21	1.468 (3)	C2—H2	0.9300
N4—C23	1.465 (3)	C2—C3	1.374 (3)
N1—C1	1.373 (3)	C3—H3	0.9300
N1—C8	1.373 (3)	C13—C14	1.395 (4)
N1—H1	0.85 (3)	C23—H23A	0.9600
C6—C7	1.438 (3)	C23—H23B	0.9600
C6—C1	1.414 (3)	C23—H23C	0.9600
C6—C5	1.402 (3)	C12—H12A	0.9600
C10—C9^{i}	1.517 (3)	C12—H12B	0.9600
C7—C9	1.506 (3)	C12—H12C	0.9600
C7—C8	1.371 (3)	C17—H17	0.9300
N3—C20	1.372 (3)	C17—C16	1.382 (4)
N3—C13	1.377 (4)	C11—H11A	0.9600
N3—H3A	0.92 (4)	C11—H11B	0.9600
C9—H9	0.9800	C11—H11C	0.9600
C1—C2	1.389 (3)	C16—H16	0.9300
C8—C12	1.488 (3)	C16—C15	1.387 (5)
C5—H5	0.9300	C24—H24A	0.9600
C5—C4	1.377 (3)	C24—H24B	0.9600
C19—C18	1.435 (3)	C24—H24C	0.9600
C19—C21	1.495 (3)	C14—H14	0.9300
C19—C20	1.368 (3)	C14—C15	1.361 (5)
C22—C21^{ii}	1.520 (3)	C15—H15	0.9300
C10—N2—C9	125.04 (18)	C5—C4—H4	119.3
C10—N2—C11	119.54 (18)	C5—C4—C3	121.4 (2)
C11—N2—C9	114.98 (17)	C3—C4—H4	119.3
C22—N4—C21	124.73 (18)	N3—C20—C24	120.2 (2)
C22—N4—C23	118.72 (19)	C19—C20—N3	109.0 (2)
C23—N4—C21	115.63 (18)	C19—C20—C24	130.7 (2)
C1—N1—C8	109.87 (17)	C1—C2—H2	121.2
C1—N1—H1	128 (2)	C3—C2—C1	117.7 (2)
C8—N1—H1	121 (2)	C3—C2—H2	121.2
C1—C6—C7	106.30 (17)	C4—C3—H3	119.5
C5—C6—C7	135.52 (18)	C2—C3—C4	121.0 (2)
C5—C6—C1	118.17 (18)	C2—C3—H3	119.5
O1—C10—N2	121.9 (2)	N3—C13—C18	107.6 (2)
O1—C10—C9^{i}	118.61 (18)	N3—C13—C14	130.6 (3)

N2—C10—C9^{i}	119.49 (17)	C14—C13—C18	121.7 (3)
C6—C7—C9	126.94 (17)	N4—C23—H23A	109.5
C8—C7—C6	107.46 (17)	N4—C23—H23B	109.5
C8—C7—C9	125.59 (18)	N4—C23—H23C	109.5
C20—N3—C13	109.4 (2)	H23A—C23—H23B	109.5
C20—N3—H3A	125 (2)	H23A—C23—H23C	109.5
C13—N3—H3A	122 (2)	H23B—C23—H23C	109.5
N2—C9—C10^{i}	114.68 (16)	C8—C12—H12A	109.5
N2—C9—C7	110.94 (16)	C8—C12—H12B	109.5
N2—C9—H9	107.0	C8—C12—H12C	109.5
C10^{i}—C9—H9	107.0	H12A—C12—H12B	109.5
C7—C9—C10^{i}	109.76 (16)	H12A—C12—H12C	109.5
C7—C9—H9	107.0	H12B—C12—H12C	109.5
N1—C1—C6	107.51 (18)	C18—C17—H17	120.6
N1—C1—C2	129.92 (19)	C16—C17—C18	118.9 (3)
C2—C1—C6	122.56 (19)	C16—C17—H17	120.6
N1—C8—C12	119.88 (18)	N2—C11—H11A	109.5
C7—C8—N1	108.82 (18)	N2—C11—H11B	109.5
C7—C8—C12	131.2 (2)	N2—C11—H11C	109.5
C6—C5—H5	120.4	H11A—C11—H11B	109.5
C4—C5—C6	119.18 (19)	H11A—C11—H11C	109.5
C4—C5—H5	120.4	H11B—C11—H11C	109.5
C18—C19—C21	127.36 (19)	C17—C16—H16	119.5
C20—C19—C18	107.4 (2)	C17—C16—C15	121.0 (3)
C20—C19—C21	125.2 (2)	C15—C16—H16	119.5
O2—C22—N4	122.9 (2)	C20—C24—H24A	109.5
O2—C22—C21^{ii}	117.74 (19)	C20—C24—H24B	109.5
N4—C22—C21^{ii}	119.35 (18)	C20—C24—H24C	109.5
C13—C18—C19	106.5 (2)	H24A—C24—H24B	109.5
C17—C18—C19	134.7 (2)	H24A—C24—H24C	109.5
C17—C18—C13	118.8 (2)	H24B—C24—H24C	109.5
N4—C21—C19	111.20 (17)	C13—C14—H14	121.0
N4—C21—C22^{ii}	114.68 (17)	C15—C14—C13	118.0 (3)
N4—C21—H21	106.7	C15—C14—H14	121.0
C19—C21—C22^{ii}	110.40 (17)	C16—C15—H15	119.2
C19—C21—H21	106.7	C14—C15—C16	121.6 (3)
C22^{ii}—C21—H21	106.7	C14—C15—H15	119.2

N1—C1—C2—C3	−177.7 (2)	C22—N4—C21—C19	−113.3 (2)
C6—C7—C9—N2	−56.4 (3)	C22—N4—C21—C22^{ii}	12.9 (3)
C6—C7—C9—C10^{i}	71.4 (2)	C18—C19—C21—N4	55.7 (3)
C6—C7—C8—N1	−1.1 (2)	C18—C19—C21—C22^{ii}	−72.7 (3)
C6—C7—C8—C12	176.2 (2)	C18—C19—C20—N3	2.1 (3)
C6—C1—C2—C3	1.6 (4)	C18—C19—C20—C24	−176.6 (2)
C6—C5—C4—C3	1.1 (3)	C18—C13—C14—C15	−0.1 (4)
C10—N2—C9—C10^{i}	−10.2 (3)	C18—C17—C16—C15	−0.4 (4)
C10—N2—C9—C7	114.8 (2)	C21—N4—C22—O2	168.4 (2)
C7—C6—C1—N1	−1.8 (2)	C21—N4—C22—C21^{ii}	−13.4 (3)

C7—C6—C1—C2	178.7 (2)	C21—C19—C18—C13	−179.5 (2)
C7—C6—C5—C4	179.5 (2)	C21—C19—C18—C17	−2.8 (4)
N3—C13—C14—C15	177.8 (3)	C21—C19—C20—N3	−179.7 (2)
C9—N2—C10—O1	−171.18 (19)	C21—C19—C20—C24	1.6 (4)
C9—N2—C10—C9^{i}	10.7 (3)	C20—N3—C13—C18	1.3 (3)
C9—C7—C8—N1	178.31 (19)	C20—N3—C13—C14	−176.9 (3)
C9—C7—C8—C12	−4.3 (4)	C20—C19—C18—C13	−1.3 (2)
C1—N1—C8—C7	0.0 (3)	C20—C19—C18—C17	175.3 (3)
C1—N1—C8—C12	−177.7 (2)	C20—C19—C21—N4	−122.1 (2)
C1—C6—C7—C9	−177.62 (19)	C20—C19—C21—C22^{ii}	109.4 (2)
C1—C6—C7—C8	1.8 (2)	C13—N3—C20—C19	−2.1 (3)
C1—C6—C5—C4	0.8 (3)	C13—N3—C20—C24	176.7 (2)
C1—C2—C3—C4	0.3 (4)	C13—C18—C17—C16	−0.8 (4)
C8—N1—C1—C6	1.2 (2)	C13—C14—C15—C16	−1.1 (4)
C8—N1—C1—C2	−179.4 (2)	C23—N4—C22—O2	−0.1 (3)
C8—C7—C9—N2	124.3 (2)	C23—N4—C22—C21^{ii}	178.1 (2)
C8—C7—C9—C10^{i}	−108.0 (2)	C23—N4—C21—C19	55.6 (2)
C5—C6—C7—C9	3.5 (4)	C23—N4—C21—C22^{ii}	−178.29 (19)
C5—C6—C7—C8	−177.0 (2)	C17—C18—C13—N3	−177.2 (2)
C5—C6—C1—N1	177.29 (19)	C17—C18—C13—C14	1.1 (4)
C5—C6—C1—C2	−2.2 (3)	C17—C16—C15—C14	1.4 (5)
C5—C4—C3—C2	−1.6 (4)	C11—N2—C10—O1	0.8 (3)
C19—C18—C13—N3	0.0 (2)	C11—N2—C10—C9^{i}	−177.35 (19)
C19—C18—C13—C14	178.4 (2)	C11—N2—C9—C10^{i}	177.48 (18)
C19—C18—C17—C16	−177.1 (3)	C11—N2—C9—C7	−57.5 (2)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{iii}	0.85 (3)	2.11 (3)	2.950 (2)	168 (3)

Symmetry code: (iii) *x*−1, *y*, *z*.

1,4-Dimethyl-3,6-bis(2-methylindol-3-yl)piperazine-2,5-dione tetrahydrofuran monosolvate (III)

Crystal data

$C_{24}H_{24}N_4O_2·C_4H_8O$
M_r = 472.57
Monoclinic, $P2_1/c$
a = 8.5276 (9) Å
b = 8.9357 (7) Å
c = 17.4883 (13) Å
β = 96.731 (8)°
V = 1323.4 (2) Å^3
Z = 2
$F(000)$ = 504
D_x = 1.186 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1124 reflections
θ = 2.4–21.3°
μ = 0.08 mm^{-1}
T = 293 K
Irregular, clear light red
0.4 × 0.4 × 0.3 mm

Data collection

XtaLAB Mini (ROW) diffractometer
Radiation source: fine-focus sealed X-ray tube, Rigaku (Mo) X-ray Source
Graphite monochromator
ω scans
Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2023)
T_{min} = 0.968, T_{max} = 1.000
6423 measured reflections
2420 independent reflections
1519 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 25.4°, θ_{min} = 2.4°
h = −9→10
k = −10→9
l = −21→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.178
S = 1.02
2420 reflections
143 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0989P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}
Extinction correction: SHELXL-2018/3 (Sheldrick 2015b), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.019 (4)

Special details

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.6513 (2)	0.59725 (19)	0.63282 (8)	0.0738 (6)
N1	0.5240 (2)	0.65140 (17)	0.51655 (9)	0.0552 (5)
N2	0.6285 (3)	0.7814 (2)	0.27788 (12)	0.0705 (6)
C1	0.5781 (3)	0.5565 (2)	0.57146 (10)	0.0493 (6)
C4	0.7086 (3)	0.6138 (2)	0.36953 (11)	0.0516 (6)
C2	0.4530 (3)	0.6105 (2)	0.43922 (10)	0.0499 (6)
H2A	0.350921	0.661728	0.430019	0.060*
C3	0.5519 (3)	0.6631 (2)	0.37871 (11)	0.0550 (6)
C9	0.7526 (3)	0.6901 (2)	0.30495 (11)	0.0587 (6)
C10	0.5065 (3)	0.7634 (3)	0.32147 (12)	0.0648 (7)
C5	0.8164 (3)	0.5138 (3)	0.40716 (12)	0.0623 (6)
H5	0.791463	0.462054	0.450270	0.075*
C8	0.8974 (3)	0.6686 (3)	0.27802 (14)	0.0700 (7)
H8	0.924270	0.720385	0.235309	0.084*
C7	0.9994 (3)	0.5686 (3)	0.31634 (15)	0.0779 (8)
H7	1.097093	0.551645	0.299209	0.094*
C6	0.9599 (3)	0.4919 (3)	0.38035 (15)	0.0759 (7)
H6	1.031507	0.424606	0.405495	0.091*

C12	0.5442 (5)	0.8121 (3)	0.53101 (16)	0.0991 (11)
H12A	0.465945	0.866466	0.498128	0.149*
H12B	0.647594	0.842222	0.520525	0.149*
H12C	0.532183	0.833018	0.583857	0.149*
C11	0.3572 (4)	0.8489 (3)	0.30298 (16)	0.0923 (10)
H11A	0.379818	0.954165	0.304557	0.138*
H11B	0.286236	0.825603	0.340035	0.138*
H11C	0.309282	0.822071	0.252427	0.138*
H2	0.622 (3)	0.825 (3)	0.2308 (16)	0.092 (9)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0805 (12)	0.0974 (13)	0.0430 (8)	−0.0108 (10)	0.0060 (8)	−0.0230 (8)
N1	0.0905 (15)	0.0337 (9)	0.0431 (9)	−0.0042 (9)	0.0150 (9)	−0.0044 (7)
N2	0.0864 (16)	0.0763 (13)	0.0498 (11)	−0.0047 (12)	0.0129 (11)	0.0245 (10)
C1	0.0623 (14)	0.0549 (12)	0.0330 (10)	−0.0011 (10)	0.0157 (9)	−0.0048 (9)
C4	0.0645 (15)	0.0517 (12)	0.0394 (11)	−0.0034 (10)	0.0100 (10)	0.0024 (9)
C2	0.0643 (14)	0.0462 (12)	0.0406 (11)	0.0089 (10)	0.0117 (10)	0.0061 (9)
C3	0.0722 (16)	0.0516 (12)	0.0423 (11)	0.0043 (11)	0.0115 (10)	0.0119 (9)
C9	0.0748 (17)	0.0593 (13)	0.0430 (11)	−0.0088 (12)	0.0111 (11)	0.0041 (10)
C10	0.0811 (18)	0.0644 (14)	0.0491 (12)	0.0045 (13)	0.0083 (12)	0.0189 (10)
C5	0.0703 (17)	0.0625 (14)	0.0560 (12)	0.0033 (12)	0.0144 (12)	0.0089 (11)
C8	0.0795 (19)	0.0754 (16)	0.0594 (14)	−0.0175 (14)	0.0261 (13)	0.0032 (12)
C7	0.0702 (18)	0.0842 (18)	0.0833 (18)	−0.0055 (15)	0.0254 (15)	−0.0084 (15)
C6	0.0714 (19)	0.0778 (17)	0.0798 (16)	0.0058 (14)	0.0143 (14)	0.0063 (14)
C12	0.168 (3)	0.0423 (13)	0.093 (2)	−0.0147 (16)	0.038 (2)	−0.0148 (13)
C11	0.098 (2)	0.092 (2)	0.0856 (19)	0.0211 (17)	0.0080 (17)	0.0409 (16)

Geometric parameters (Å, º)

O1—C1	1.231 (2)	C10—C11	1.487 (4)
N1—C1	1.323 (3)	C5—H5	0.9300
N1—C2	1.462 (3)	C5—C6	1.375 (3)
N1—C12	1.465 (3)	C8—H8	0.9300
N2—C9	1.376 (3)	C8—C7	1.366 (4)
N2—C10	1.369 (3)	C7—H7	0.9300
N2—H2	0.91 (3)	C7—C6	1.387 (3)
C1—C2^{i}	1.524 (3)	C6—H6	0.9300
C4—C3	1.434 (3)	C12—H12A	0.9600
C4—C9	1.408 (3)	C12—H12B	0.9600
C4—C5	1.391 (3)	C12—H12C	0.9600
C2—H2A	0.9800	C11—H11A	0.9600
C2—C3	1.504 (3)	C11—H11B	0.9600
C3—C10	1.365 (3)	C11—H11C	0.9600
C9—C8	1.386 (4)		
C1—N1—C2	125.65 (16)	C3—C10—C11	131.2 (2)

C1—N1—C12	118.62 (19)	C4—C5—H5	120.2
C2—N1—C12	115.62 (18)	C6—C5—C4	119.7 (2)
C9—N2—H2	122.0 (18)	C6—C5—H5	120.2
C10—N2—C9	110.06 (18)	C9—C8—H8	121.1
C10—N2—H2	125.8 (18)	C7—C8—C9	117.8 (2)
O1—C1—N1	122.78 (19)	C7—C8—H8	121.1
O1—C1—C2^{i}	117.35 (19)	C8—C7—H7	119.4
N1—C1—C2^{i}	119.85 (18)	C8—C7—C6	121.2 (2)
C9—C4—C3	106.31 (19)	C6—C7—H7	119.4
C5—C4—C3	135.82 (19)	C5—C6—C7	121.0 (3)
C5—C4—C9	117.9 (2)	C5—C6—H6	119.5
N1—C2—C1^{i}	113.94 (15)	C7—C6—H6	119.5
N1—C2—H2A	107.4	N1—C12—H12A	109.5
N1—C2—C3	111.53 (18)	N1—C12—H12B	109.5
C1^{i}—C2—H2A	107.4	N1—C12—H12C	109.5
C3—C2—C1^{i}	108.90 (15)	H12A—C12—H12B	109.5
C3—C2—H2A	107.4	H12A—C12—H12C	109.5
C4—C3—C2	126.14 (17)	H12B—C12—H12C	109.5
C10—C3—C4	107.89 (19)	C10—C11—H11A	109.5
C10—C3—C2	126.0 (2)	C10—C11—H11B	109.5
N2—C9—C4	107.2 (2)	C10—C11—H11C	109.5
N2—C9—C8	130.2 (2)	H11A—C11—H11B	109.5
C8—C9—C4	122.5 (2)	H11A—C11—H11C	109.5
N2—C10—C11	120.36 (19)	H11B—C11—H11C	109.5
C3—C10—N2	108.5 (2)		

N1—C2—C3—C4	64.2 (3)	C9—N2—C10—C3	1.6 (3)
N1—C2—C3—C10	−117.7 (2)	C9—N2—C10—C11	−179.2 (2)
N2—C9—C8—C7	−179.5 (2)	C9—C4—C3—C2	178.71 (19)
C1—N1—C2—C1^{i}	8.7 (3)	C9—C4—C3—C10	0.3 (2)
C1—N1—C2—C3	−115.1 (2)	C9—C4—C5—C6	−0.5 (3)
C1^{i}—C2—C3—C4	−62.4 (3)	C9—C8—C7—C6	−0.4 (4)
C1^{i}—C2—C3—C10	115.7 (2)	C10—N2—C9—C4	−1.4 (3)
C4—C3—C10—N2	−1.2 (3)	C10—N2—C9—C8	178.3 (2)
C4—C3—C10—C11	179.8 (3)	C5—C4—C3—C2	−0.4 (4)
C4—C9—C8—C7	0.2 (4)	C5—C4—C3—C10	−178.8 (2)
C4—C5—C6—C7	0.3 (4)	C5—C4—C9—N2	180.0 (2)
C2—N1—C1—O1	172.4 (2)	C5—C4—C9—C8	0.3 (3)
C2—N1—C1—C2^{i}	−9.1 (4)	C8—C7—C6—C5	0.2 (4)
C2—C3—C10—N2	−179.6 (2)	C12—N1—C1—O1	−3.5 (3)
C2—C3—C10—C11	1.4 (4)	C12—N1—C1—C2^{i}	174.9 (2)
C3—C4—C9—N2	0.7 (2)	C12—N1—C2—C1^{i}	−175.3 (2)
C3—C4—C9—C8	−179.1 (2)	C12—N1—C2—C3	60.9 (3)
C3—C4—C5—C6	178.6 (2)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O1^{ii}	0.91 (3)	1.89 (3)	2.787 (2)	168 (3)

Symmetry code: (ii) x, $-y+3/2$, $z-1/2$.