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Opipramol

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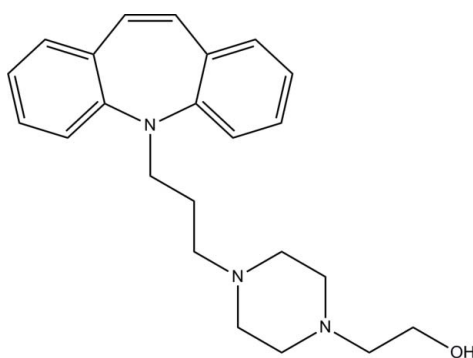
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.001$ Å;
R factor = 0.042; wR factor = 0.117; data-to-parameter ratio = 32.1.

In the title compound (systematic name: 2-[4-[3-(5*H*-dibenz[*b,f*]azepin-5-yl)propyl]piperazin-1-yl]ethanol), $\text{C}_{23}\text{H}_{29}\text{N}_3\text{O}$, the 5*H*-dibenz[*b,f*]azepine and piperazine rings adopt boat and chair conformations, respectively, and the overall shape of the fused ring part of the molecule is a butterfly. In the crystal, $\text{O}-\text{H}\cdots\text{N}$ and $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds link the molecules into a layer parallel to the *bc* plane.

Related literature

For the application of opipramol, see: Moller *et al.* (2001). For related structures, see: Jasinski *et al.* (2010); Nagaraj *et al.* (2005). For ring conformations, see: Cremer & Pople (1975). For bond-length data, see: Allen *et al.* (1987). For the stability of the temperature controller used for the data collection, see: Cosier & Glazer (1986).



Experimental

Crystal data

$\text{C}_{23}\text{H}_{29}\text{N}_3\text{O}$
 $M_r = 363.49$

Monoclinic, $P2_1/c$
 $a = 12.6215$ (2) Å

$b = 10.5929$ (2) Å
 $c = 14.3629$ (2) Å
 $\beta = 90.966$ (1)°
 $V = 1920.02$ (5) Å³
 $Z = 4$

Mo $K\alpha$ radiation
 $\mu = 0.08$ mm⁻¹
 $T = 100$ K
 $0.59 \times 0.36 \times 0.30$ mm

Data collection

Bruker SMART APEXII CCD
area-detector diffractometer
Absorption correction: multi-scan
(*SADABS*; Bruker, 2009)
 $T_{\min} = 0.955$, $T_{\max} = 0.977$

30830 measured reflections
7949 independent reflections
6682 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.026$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.117$
 $S = 1.03$
7949 reflections
248 parameters

H atoms treated by a mixture of
independent and constrained
refinement
 $\Delta\rho_{\text{max}} = 0.56$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.25$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|--|------------|-------------|-------------|---------------|
| $\text{O1}-\text{H1O1}\cdots\text{N2}^i$ | 0.896 (16) | 1.999 (16) | 2.8822 (9) | 168.3 (14) |
| $\text{C5}-\text{H5A}\cdots\text{O1}^{ii}$ | 0.95 | 2.41 | 3.3478 (11) | 167 |

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2726).

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Jasinski, J. P., Pek, A. E., Siddaraju, B. P., Yathirajan, H. S. & Narayana, B. (2010). *Acta Cryst.* **E66**, o1979–o1980.
Moller, H. J., Volz, H. P., Reimann, I. W. & Stoll, K. D. (2001). *J. Clin. Psychopharmacol.* **21**, 59–65.
Nagaraj, B., Yathirajan, H. S. & Lynch, D. E. (2005). *Acta Cryst.* **E61**, o1757–o1759.
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.

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§ Thomson Reuters ResearcherID: C-7581-2009.

supporting information

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Opipramol

Hoong-Kun Fun, Wan-Sin Loh, M. S. Siddegowda, H. S. Yathirajan and B. Narayana

S1. Comment

Opipramol [systematic IUPAC name: 4-[3-(5*H*-dibenz[*b,f*]azepin-5-yl)propyl]-1-piperazinethanol] is an antidepressant and anxiolytic typically used in the treatment of generalized anxiety disorder (Moller *et al.*, 2001). Opipramol is a tricyclic compound with no reuptake-inhibiting properties. However, it has pronounced D2-, 5-HT2-, and H1-blocking potential and high affinity to sigma receptors (sigma-1 and sigma-2). The crystal structure studies of opipramol dipicrate is reported (Jasinski *et al.*, 2010). In view of the importance of the title compound, C₂₃H₂₉N₃O, the crystal structure is reported.

In the title compound (Fig. 1), the seven-membered, 5*H*-dibenz[*b,f*]azepine ring (N1/C1/C6–C9/C14) adopts a boat conformation (Cremer & Pople, 1975) and the overall molecular shape is that of a butterfly (Nagaraj *et al.*, 2005) whereas the piperazine ring (N2/C18/C19/N3/C20/C21) adopts a chair conformation (Cremer & Pople, 1975) with the puckering parameters, $Q = 0.5934(8) \text{ \AA}$, $\Theta = 177.94(8)^\circ$, $\varphi = 305.8(18)^\circ$. The torsion angle between the 5*H*-dibenz[*b,f*]azepine and the piperazine rings, N1–C15–C16–C17 is $61.73(8)^\circ$. Bond lengths (Allen *et al.*, 1987) and angles are within the normal ranges.

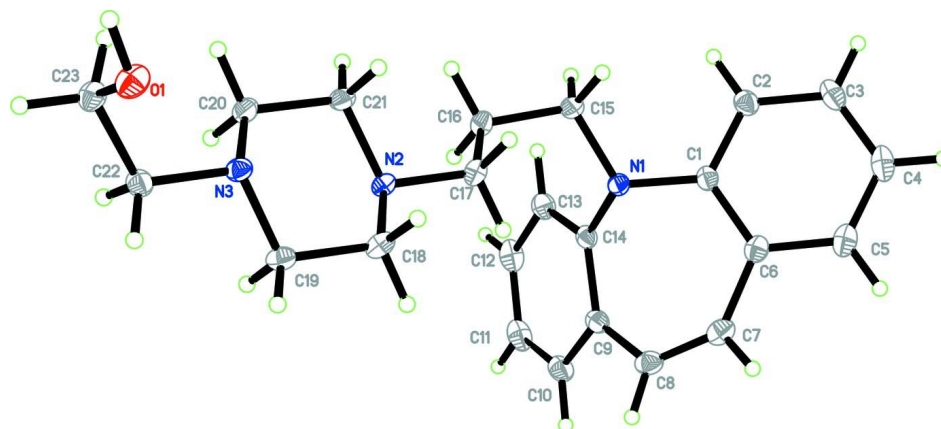
In the crystal packing (Fig. 2), intermolecular O1—H1O1 \cdots N2 and C5—H5A \cdots O1 hydrogen bonds link the molecules into layers parallel to the *bc* plane.

S2. Experimental

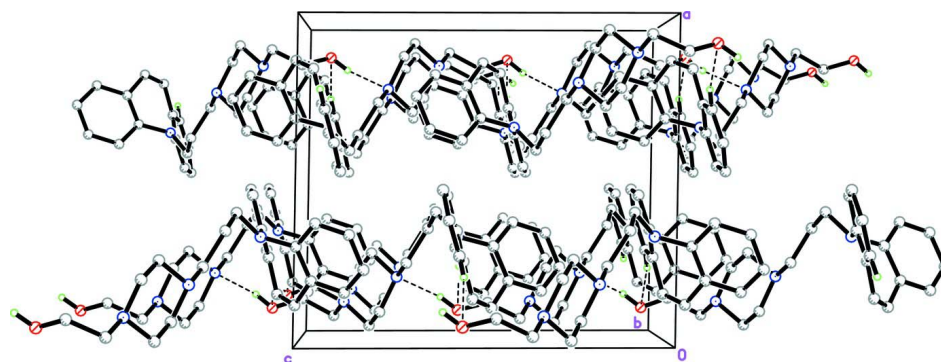
The title compound was obtained as a gift sample from *R. L. Fine Chem. Ltd.*, Bangalore, India. The compound was recrystallized from acetone (*m. p.* 373–374 K).

S3. Refinement

H1O1 was located from a difference map and was refined freely [O—H = $0.896(16) \text{ \AA}$]. The remaining H atoms were positioned geometrically (C—H = 0.95 or 0.99 \AA) and refined with a riding model with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

**Figure 1**

The molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme.

**Figure 2**

The crystal packing of the title compound, viewed along the *b* axis. H atoms not involved in the intermolecular interactions (dashed lines) have been omitted for clarity.

2-[4-[3-(5*H*-dibenz[*b,f*]azepin-5-yl)propyl]piperazin-1-yl]ethanol

Crystal data

$C_{23}H_{29}N_3O$

$M_r = 363.49$

Monoclinic, $P2_1/c$

Hall symbol: $-P 2_1/c$

$a = 12.6215 (2) \text{ \AA}$

$b = 10.5929 (2) \text{ \AA}$

$c = 14.3629 (2) \text{ \AA}$

$\beta = 90.966 (1)^\circ$

$V = 1920.02 (5) \text{ \AA}^3$

$Z = 4$

$F(000) = 784$

$D_x = 1.257 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 9885 reflections

$\theta = 2.9\text{--}34.2^\circ$

$\mu = 0.08 \text{ mm}^{-1}$

$T = 100 \text{ K}$

Block, colourless

$0.59 \times 0.36 \times 0.30 \text{ mm}$

Data collection

Bruker SMART APEXII CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2009)

$T_{\min} = 0.955$, $T_{\max} = 0.977$

30830 measured reflections
 7949 independent reflections
 6682 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.026$

$\theta_{\text{max}} = 34.3^\circ$, $\theta_{\text{min}} = 2.4^\circ$
 $h = -16 \rightarrow 19$
 $k = -13 \rightarrow 16$
 $l = -22 \rightarrow 20$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.117$
 $S = 1.03$
 7949 reflections
 248 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H atoms treated by a mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0611P)^2 + 0.4097P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.56 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.25 \text{ e } \text{\AA}^{-3}$

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

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

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|-------------|-------------|-------------|----------------------------------|
| O1 | 0.10636 (5) | 1.33255 (6) | 1.06904 (4) | 0.01965 (12) |
| N1 | 0.33738 (5) | 0.74218 (6) | 0.57588 (4) | 0.01418 (11) |
| N2 | 0.21096 (5) | 1.06560 (6) | 0.73239 (4) | 0.01326 (11) |
| N3 | 0.12890 (5) | 1.23458 (6) | 0.87387 (4) | 0.01380 (11) |
| C1 | 0.35654 (6) | 0.60944 (7) | 0.58110 (5) | 0.01411 (12) |
| C2 | 0.45637 (6) | 0.56231 (8) | 0.60674 (5) | 0.01763 (13) |
| H2A | 0.5136 | 0.6192 | 0.6165 | 0.021* |
| C3 | 0.47316 (7) | 0.43319 (8) | 0.61812 (5) | 0.02086 (15) |
| H3A | 0.5412 | 0.4027 | 0.6363 | 0.025* |
| C4 | 0.39006 (8) | 0.34908 (8) | 0.60285 (6) | 0.02318 (16) |
| H4A | 0.4011 | 0.2610 | 0.6106 | 0.028* |
| C5 | 0.29077 (7) | 0.39456 (8) | 0.57628 (6) | 0.02090 (15) |
| H5A | 0.2344 | 0.3367 | 0.5656 | 0.025* |
| C6 | 0.27213 (6) | 0.52452 (7) | 0.56485 (5) | 0.01608 (13) |
| C7 | 0.16454 (6) | 0.56539 (8) | 0.53991 (5) | 0.01885 (14) |
| H7A | 0.1084 | 0.5136 | 0.5611 | 0.023* |
| C8 | 0.13636 (6) | 0.66812 (8) | 0.49038 (5) | 0.01869 (14) |
| H8A | 0.0627 | 0.6846 | 0.4831 | 0.022* |

| | | | | |
|------|-------------|-------------|-------------|--------------|
| C9 | 0.20930 (6) | 0.75680 (7) | 0.44664 (5) | 0.01590 (13) |
| C10 | 0.17851 (7) | 0.81058 (8) | 0.36112 (5) | 0.02081 (15) |
| H10A | 0.1099 | 0.7931 | 0.3362 | 0.025* |
| C11 | 0.24589 (8) | 0.88867 (8) | 0.31209 (5) | 0.02329 (16) |
| H11A | 0.2239 | 0.9232 | 0.2539 | 0.028* |
| C12 | 0.34570 (7) | 0.91589 (8) | 0.34884 (6) | 0.02238 (16) |
| H12A | 0.3926 | 0.9685 | 0.3153 | 0.027* |
| C13 | 0.37721 (6) | 0.86616 (8) | 0.43480 (5) | 0.01829 (14) |
| H13A | 0.4454 | 0.8858 | 0.4597 | 0.022* |
| C14 | 0.30944 (6) | 0.78756 (7) | 0.48477 (5) | 0.01414 (12) |
| C15 | 0.40721 (6) | 0.82280 (7) | 0.63287 (5) | 0.01655 (13) |
| H15A | 0.4750 | 0.8347 | 0.6004 | 0.020* |
| H15B | 0.4229 | 0.7799 | 0.6928 | 0.020* |
| C16 | 0.35831 (6) | 0.95205 (7) | 0.65234 (5) | 0.01594 (13) |
| H16A | 0.4100 | 1.0040 | 0.6881 | 0.019* |
| H16B | 0.3434 | 0.9954 | 0.5925 | 0.019* |
| C17 | 0.25587 (6) | 0.94200 (7) | 0.70697 (5) | 0.01620 (13) |
| H17A | 0.2028 | 0.8949 | 0.6691 | 0.019* |
| H17B | 0.2700 | 0.8929 | 0.7645 | 0.019* |
| C18 | 0.10673 (6) | 1.04721 (7) | 0.77555 (5) | 0.01661 (13) |
| H18A | 0.1148 | 0.9920 | 0.8308 | 0.020* |
| H18B | 0.0580 | 1.0051 | 0.7307 | 0.020* |
| C19 | 0.06022 (6) | 1.17294 (8) | 0.80442 (5) | 0.01601 (13) |
| H19A | 0.0522 | 1.2281 | 0.7491 | 0.019* |
| H19B | -0.0109 | 1.1594 | 0.8307 | 0.019* |
| C20 | 0.23213 (6) | 1.25503 (7) | 0.83151 (5) | 0.01593 (13) |
| H20A | 0.2805 | 1.2962 | 0.8772 | 0.019* |
| H20B | 0.2237 | 1.3121 | 0.7773 | 0.019* |
| C21 | 0.28023 (6) | 1.13121 (7) | 0.80016 (5) | 0.01553 (13) |
| H21A | 0.3497 | 1.1479 | 0.7716 | 0.019* |
| H21B | 0.2924 | 1.0762 | 0.8550 | 0.019* |
| C22 | 0.08179 (6) | 1.35398 (7) | 0.90363 (5) | 0.01556 (13) |
| H22A | 0.0054 | 1.3403 | 0.9144 | 0.019* |
| H22B | 0.0881 | 1.4162 | 0.8526 | 0.019* |
| C23 | 0.13187 (6) | 1.40919 (7) | 0.99127 (5) | 0.01650 (13) |
| H23A | 0.2097 | 1.4132 | 0.9847 | 0.020* |
| H23B | 0.1053 | 1.4960 | 1.0010 | 0.020* |
| H10I | 0.1474 (12) | 1.3609 (15) | 1.1160 (11) | 0.047 (4)* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|----|------------|------------|------------|-------------|---------------|---------------|
| O1 | 0.0255 (3) | 0.0212 (3) | 0.0123 (2) | -0.0037 (2) | -0.0003 (2) | -0.00024 (19) |
| N1 | 0.0171 (3) | 0.0124 (3) | 0.0129 (2) | 0.0007 (2) | -0.00349 (19) | -0.00178 (19) |
| N2 | 0.0140 (2) | 0.0135 (3) | 0.0123 (2) | -0.0022 (2) | -0.00079 (19) | -0.00190 (19) |
| N3 | 0.0138 (2) | 0.0157 (3) | 0.0119 (2) | -0.0006 (2) | -0.00102 (19) | -0.00231 (19) |
| C1 | 0.0180 (3) | 0.0130 (3) | 0.0113 (3) | 0.0014 (2) | -0.0002 (2) | -0.0009 (2) |
| C2 | 0.0203 (3) | 0.0175 (3) | 0.0150 (3) | 0.0033 (3) | -0.0019 (2) | -0.0009 (2) |

| | | | | | | |
|-----|------------|------------|------------|-------------|-------------|-------------|
| C3 | 0.0276 (4) | 0.0191 (3) | 0.0159 (3) | 0.0078 (3) | -0.0006 (3) | 0.0010 (2) |
| C4 | 0.0352 (4) | 0.0149 (3) | 0.0196 (3) | 0.0043 (3) | 0.0037 (3) | 0.0018 (3) |
| C5 | 0.0289 (4) | 0.0155 (3) | 0.0183 (3) | -0.0023 (3) | 0.0036 (3) | -0.0002 (3) |
| C6 | 0.0199 (3) | 0.0153 (3) | 0.0131 (3) | -0.0010 (2) | 0.0018 (2) | -0.0017 (2) |
| C7 | 0.0178 (3) | 0.0205 (4) | 0.0184 (3) | -0.0035 (3) | 0.0021 (2) | -0.0039 (3) |
| C8 | 0.0157 (3) | 0.0219 (4) | 0.0184 (3) | 0.0004 (3) | -0.0010 (2) | -0.0059 (3) |
| C9 | 0.0172 (3) | 0.0165 (3) | 0.0139 (3) | 0.0036 (2) | -0.0019 (2) | -0.0036 (2) |
| C10 | 0.0251 (4) | 0.0211 (4) | 0.0160 (3) | 0.0063 (3) | -0.0062 (3) | -0.0038 (3) |
| C11 | 0.0349 (4) | 0.0213 (4) | 0.0136 (3) | 0.0087 (3) | -0.0013 (3) | -0.0003 (3) |
| C12 | 0.0306 (4) | 0.0196 (4) | 0.0171 (3) | 0.0047 (3) | 0.0058 (3) | 0.0021 (3) |
| C13 | 0.0194 (3) | 0.0177 (3) | 0.0177 (3) | 0.0023 (3) | 0.0023 (2) | -0.0002 (2) |
| C14 | 0.0163 (3) | 0.0136 (3) | 0.0125 (3) | 0.0029 (2) | -0.0007 (2) | -0.0020 (2) |
| C15 | 0.0172 (3) | 0.0151 (3) | 0.0172 (3) | 0.0009 (2) | -0.0041 (2) | -0.0034 (2) |
| C16 | 0.0179 (3) | 0.0134 (3) | 0.0165 (3) | -0.0006 (2) | -0.0003 (2) | -0.0025 (2) |
| C17 | 0.0190 (3) | 0.0126 (3) | 0.0170 (3) | -0.0016 (2) | 0.0007 (2) | -0.0017 (2) |
| C18 | 0.0168 (3) | 0.0181 (3) | 0.0150 (3) | -0.0054 (2) | 0.0012 (2) | -0.0026 (2) |
| C19 | 0.0135 (3) | 0.0210 (3) | 0.0135 (3) | -0.0019 (2) | -0.0009 (2) | -0.0027 (2) |
| C20 | 0.0142 (3) | 0.0159 (3) | 0.0177 (3) | -0.0019 (2) | 0.0001 (2) | -0.0039 (2) |
| C21 | 0.0144 (3) | 0.0169 (3) | 0.0152 (3) | -0.0003 (2) | -0.0026 (2) | -0.0037 (2) |
| C22 | 0.0169 (3) | 0.0163 (3) | 0.0135 (3) | 0.0020 (2) | -0.0008 (2) | -0.0005 (2) |
| C23 | 0.0198 (3) | 0.0161 (3) | 0.0136 (3) | -0.0008 (2) | 0.0009 (2) | -0.0015 (2) |

Geometric parameters (Å, °)

| | | | |
|---------|-------------|----------|-------------|
| O1—C23 | 1.4221 (9) | C11—C12 | 1.3879 (13) |
| O1—H10I | 0.896 (16) | C11—H11A | 0.9500 |
| N1—C1 | 1.4284 (10) | C12—C13 | 1.3941 (11) |
| N1—C14 | 1.4327 (9) | C12—H12A | 0.9500 |
| N1—C15 | 1.4669 (9) | C13—C14 | 1.4002 (11) |
| N2—C21 | 1.4719 (9) | C13—H13A | 0.9500 |
| N2—C17 | 1.4750 (10) | C15—C16 | 1.5295 (10) |
| N2—C18 | 1.4766 (9) | C15—H15A | 0.9900 |
| N3—C20 | 1.4633 (9) | C15—H15B | 0.9900 |
| N3—C19 | 1.4641 (9) | C16—C17 | 1.5275 (11) |
| N3—C22 | 1.4644 (10) | C16—H16A | 0.9900 |
| C1—C2 | 1.3988 (10) | C16—H16B | 0.9900 |
| C1—C6 | 1.4109 (11) | C17—H17A | 0.9900 |
| C2—C3 | 1.3932 (11) | C17—H17B | 0.9900 |
| C2—H2A | 0.9500 | C18—C19 | 1.5160 (11) |
| C3—C4 | 1.3908 (13) | C18—H18A | 0.9900 |
| C3—H3A | 0.9500 | C18—H18B | 0.9900 |
| C4—C5 | 1.3900 (13) | C19—H19A | 0.9900 |
| C4—H4A | 0.9500 | C19—H19B | 0.9900 |
| C5—C6 | 1.4057 (11) | C20—C21 | 1.5168 (11) |
| C5—H5A | 0.9500 | C20—H20A | 0.9900 |
| C6—C7 | 1.4641 (11) | C20—H20B | 0.9900 |
| C7—C8 | 1.3446 (12) | C21—H21A | 0.9900 |
| C7—H7A | 0.9500 | C21—H21B | 0.9900 |

| | | | |
|--------------|-------------|---------------|-------------|
| C8—C9 | 1.4644 (12) | C22—C23 | 1.5163 (10) |
| C8—H8A | 0.9500 | C22—H22A | 0.9900 |
| C9—C10 | 1.4029 (11) | C22—H22B | 0.9900 |
| C9—C14 | 1.4072 (10) | C23—H23A | 0.9900 |
| C10—C11 | 1.3870 (13) | C23—H23B | 0.9900 |
| C10—H10A | 0.9500 | | |
| | | | |
| C23—O1—H10I | 105.3 (10) | N1—C15—H15A | 109.1 |
| C1—N1—C14 | 114.65 (6) | C16—C15—H15A | 109.1 |
| C1—N1—C15 | 116.37 (6) | N1—C15—H15B | 109.1 |
| C14—N1—C15 | 116.79 (6) | C16—C15—H15B | 109.1 |
| C21—N2—C17 | 110.88 (6) | H15A—C15—H15B | 107.8 |
| C21—N2—C18 | 107.94 (6) | C17—C16—C15 | 112.28 (6) |
| C17—N2—C18 | 109.61 (6) | C17—C16—H16A | 109.1 |
| C20—N3—C19 | 107.69 (5) | C15—C16—H16A | 109.1 |
| C20—N3—C22 | 111.24 (6) | C17—C16—H16B | 109.1 |
| C19—N3—C22 | 110.21 (6) | C15—C16—H16B | 109.1 |
| C2—C1—C6 | 119.36 (7) | H16A—C16—H16B | 107.9 |
| C2—C1—N1 | 121.07 (7) | N2—C17—C16 | 113.42 (6) |
| C6—C1—N1 | 119.49 (6) | N2—C17—H17A | 108.9 |
| C3—C2—C1 | 121.06 (8) | C16—C17—H17A | 108.9 |
| C3—C2—H2A | 119.5 | N2—C17—H17B | 108.9 |
| C1—C2—H2A | 119.5 | C16—C17—H17B | 108.9 |
| C4—C3—C2 | 119.84 (8) | H17A—C17—H17B | 107.7 |
| C4—C3—H3A | 120.1 | N2—C18—C19 | 110.53 (6) |
| C2—C3—H3A | 120.1 | N2—C18—H18A | 109.5 |
| C5—C4—C3 | 119.69 (8) | C19—C18—H18A | 109.5 |
| C5—C4—H4A | 120.2 | N2—C18—H18B | 109.5 |
| C3—C4—H4A | 120.2 | C19—C18—H18B | 109.5 |
| C4—C5—C6 | 121.32 (8) | H18A—C18—H18B | 108.1 |
| C4—C5—H5A | 119.3 | N3—C19—C18 | 110.52 (6) |
| C6—C5—H5A | 119.3 | N3—C19—H19A | 109.5 |
| C5—C6—C1 | 118.73 (7) | C18—C19—H19A | 109.5 |
| C5—C6—C7 | 118.10 (7) | N3—C19—H19B | 109.5 |
| C1—C6—C7 | 123.14 (7) | C18—C19—H19B | 109.5 |
| C8—C7—C6 | 127.21 (7) | H19A—C19—H19B | 108.1 |
| C8—C7—H7A | 116.4 | N3—C20—C21 | 111.04 (6) |
| C6—C7—H7A | 116.4 | N3—C20—H20A | 109.4 |
| C7—C8—C9 | 125.70 (7) | C21—C20—H20A | 109.4 |
| C7—C8—H8A | 117.1 | N3—C20—H20B | 109.4 |
| C9—C8—H8A | 117.1 | C21—C20—H20B | 109.4 |
| C10—C9—C14 | 118.72 (7) | H20A—C20—H20B | 108.0 |
| C10—C9—C8 | 117.98 (7) | N2—C21—C20 | 111.61 (6) |
| C14—C9—C8 | 123.27 (7) | N2—C21—H21A | 109.3 |
| C11—C10—C9 | 121.56 (8) | C20—C21—H21A | 109.3 |
| C11—C10—H10A | 119.2 | N2—C21—H21B | 109.3 |
| C9—C10—H10A | 119.2 | C20—C21—H21B | 109.3 |
| C10—C11—C12 | 119.42 (7) | H21A—C21—H21B | 108.0 |

| | | | |
|-----------------|-------------|----------------|-------------|
| C10—C11—H11A | 120.3 | N3—C22—C23 | 114.20 (6) |
| C12—C11—H11A | 120.3 | N3—C22—H22A | 108.7 |
| C11—C12—C13 | 120.12 (8) | C23—C22—H22A | 108.7 |
| C11—C12—H12A | 119.9 | N3—C22—H22B | 108.7 |
| C13—C12—H12A | 119.9 | C23—C22—H22B | 108.7 |
| C12—C13—C14 | 120.73 (8) | H22A—C22—H22B | 107.6 |
| C12—C13—H13A | 119.6 | O1—C23—C22 | 109.58 (6) |
| C14—C13—H13A | 119.6 | O1—C23—H23A | 109.8 |
| C13—C14—C9 | 119.38 (7) | C22—C23—H23A | 109.8 |
| C13—C14—N1 | 121.65 (7) | O1—C23—H23B | 109.8 |
| C9—C14—N1 | 118.91 (7) | C22—C23—H23B | 109.8 |
| N1—C15—C16 | 112.50 (6) | H23A—C23—H23B | 108.2 |
| | | | |
| C14—N1—C1—C2 | 116.85 (7) | C10—C9—C14—C13 | -2.75 (11) |
| C15—N1—C1—C2 | -24.50 (10) | C8—C9—C14—C13 | 175.33 (7) |
| C14—N1—C1—C6 | -66.45 (9) | C10—C9—C14—N1 | 174.38 (7) |
| C15—N1—C1—C6 | 152.20 (7) | C8—C9—C14—N1 | -7.54 (11) |
| C6—C1—C2—C3 | -1.21 (11) | C1—N1—C14—C13 | -111.94 (8) |
| N1—C1—C2—C3 | 175.49 (7) | C15—N1—C14—C13 | 29.25 (10) |
| C1—C2—C3—C4 | 0.74 (12) | C1—N1—C14—C9 | 71.01 (8) |
| C2—C3—C4—C5 | 0.03 (12) | C15—N1—C14—C9 | -147.81 (7) |
| C3—C4—C5—C6 | -0.32 (12) | C1—N1—C15—C16 | -158.48 (6) |
| C4—C5—C6—C1 | -0.14 (11) | C14—N1—C15—C16 | 61.01 (8) |
| C4—C5—C6—C7 | -177.90 (7) | N1—C15—C16—C17 | 61.73 (8) |
| C2—C1—C6—C5 | 0.90 (10) | C21—N2—C17—C16 | -67.12 (8) |
| N1—C1—C6—C5 | -175.86 (7) | C18—N2—C17—C16 | 173.81 (6) |
| C2—C1—C6—C7 | 178.53 (7) | C15—C16—C17—N2 | 175.98 (6) |
| N1—C1—C6—C7 | 1.78 (10) | C21—N2—C18—C19 | 57.76 (8) |
| C5—C6—C7—C8 | -150.54 (8) | C17—N2—C18—C19 | 178.62 (6) |
| C1—C6—C7—C8 | 31.80 (12) | C20—N3—C19—C18 | 60.33 (8) |
| C6—C7—C8—C9 | 3.66 (13) | C22—N3—C19—C18 | -178.17 (6) |
| C7—C8—C9—C10 | 144.64 (8) | N2—C18—C19—N3 | -61.60 (8) |
| C7—C8—C9—C14 | -33.45 (12) | C19—N3—C20—C21 | -58.73 (8) |
| C14—C9—C10—C11 | 2.62 (12) | C22—N3—C20—C21 | -179.59 (6) |
| C8—C9—C10—C11 | -175.56 (7) | C17—N2—C21—C20 | -176.59 (6) |
| C9—C10—C11—C12 | -0.88 (13) | C18—N2—C21—C20 | -56.52 (8) |
| C10—C11—C12—C13 | -0.73 (12) | N3—C20—C21—N2 | 58.74 (8) |
| C11—C12—C13—C14 | 0.55 (12) | C20—N3—C22—C23 | -74.86 (8) |
| C12—C13—C14—C9 | 1.22 (11) | C19—N3—C22—C23 | 165.77 (6) |
| C12—C13—C14—N1 | -175.83 (7) | N3—C22—C23—O1 | -69.38 (8) |

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

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|----------------------------------|------------|-------------|-------------|---------------|
| O1—H1O1 \cdots N2 ⁱ | 0.896 (16) | 1.999 (16) | 2.8822 (9) | 168.3 (14) |
| C5—H5A \cdots O1 ⁱⁱ | 0.95 | 2.41 | 3.3478 (11) | 167 |

Symmetry codes: (i) $x, -y+5/2, z+1/2$; (ii) $x, -y+3/2, z-1/2$.