# 14a-Hydroxy-12-methyl-10-(4-methyl-phenyl)-8,9,9a,10,12,13,14,14a-octa-hydro-10a,14-methano-5*H*-indeno-[2′,1′:4,5]azepino[3,4-*b*]pyrrolizine-5,15(7*H*,11*H*)-dione

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In the title compound, $C_{27}H_{28}N_2O_3$, each of the pyrrolidine rings adopts a twisted conformation, as does the cyclopentane ring. The indane ring has an r.m.s deviation of 0.0693 Å. The dihedral angle between the mean plane of the pyrrolizine ring and indane system is 82.58 (1)°. The piperidine ring has the methyl substituent in an equatorial position and adopts a twisted chair conformation. The molecular structure is stabilized by a weak intramolecular O—H···N interaction.

## Related literature

For the importance of pyrrazole derivatives, see: Mahajan *et al.* (1991); Katayama & Oshiyama (1997); Baraldi *et al.* (1998). For additional conformation analysis, see: Cremer & Pople (1975).
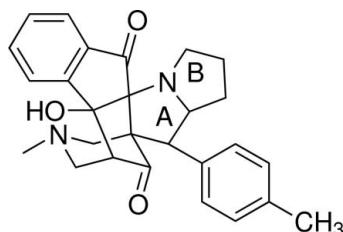


## Experimental

### Crystal data

| | |
|---|---|
| $C_{27}H_{28}N_2O_3$ | $V = 2244.25$ (14) Å$^3$ |
| $M_r = 428.51$ | $Z = 4$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation |
| $a = 16.8684$ (6) Å | $\mu = 0.08$ mm$^{-1}$ |
| $b = 8.3754$ (3) Å | $T = 293$ K |
| $c = 15.9930$ (6) Å | $0.21 \times 0.19 \times 0.18$ mm |
| $\beta = 96.658$ (1)° | |

### Data collection

| | |
|---|---|
| Bruker Kappa APEXII diffractometer | 28459 measured reflections |
| Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996) | 6850 independent reflections |
| | 4500 reflections with $I > 2\sigma(I)$ |
| $T_{min} = 0.967$, $T_{max} = 0.974$ | $R_{int} = 0.030$ |

### Refinement

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)] = 0.053$ | 291 parameters |
| $wR(F^2) = 0.161$ | H-atom parameters constrained |
| $S = 1.02$ | $\Delta\rho_{max} = 0.29$ e Å$^{-3}$ |
| 6850 reflections | $\Delta\rho_{min} = -0.18$ e Å$^{-3}$ |

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O1—H1···N2 | 0.82 | 2.13 | 2.6497 (16) | 121 |

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

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## supporting information

# 14a-Hydroxy-12-methyl-10-(4-methylphenyl)-8,9,9a,10,12,13,14,14a-octa-hydro-10a,14-methano-5*H*-indeno[2′,1′:4,5]azepino[3,4-*b*]pyrrolizine-5,15(7*H*,11*H*)-dione

**R. A. Nagalakshmi, J. Suresh, K. Malathi, R. Ranjith Kumar and P. L. Nilantha Lakshman**

### S1. Comment

Pyrazole derivatives in general are well known nitrogen-containing heterocyclic compounds that have been the subject of enormous research due to their importance in various applications and their widespread potential biological and pharmacological activities such as anti-microbial (Mahajan *et al.*, 1991), anti-viral (Baraldi *et al.*, 1998), anti-tumor (Katayama & Oshiyama, 1997) and anti-fungal (Baraldi *et al.*, 1998). In view of its medicinal importance we report the crystal structure of the title compound.

In the title compound (Fig. 1), each of the pyrrolidine rings A and B adopts twisted conformation with the puckering parameters (Cremer & Pople, 1975): $q_2 = 0.4201$ (14) Å, $\Phi_2 = 10.43$ (19)° and $q_2 = 0.4547$ (16) Å, $\Phi_2 = 196.1$ (2)° respectively. The sum of bond angles around N1 (335.69 (2)°) and N2 (340.55 (2)°) indicates $sp^3$ hybridization. The N2—C11 = 1.462 (12) Å and N2—C8 = 1.463 (17) Å bonds are approximately equivalent and both are longer than the N2—C12 = 1.453 (16) Å bond. The indane (C12—C20) fused-ring system is nearly planar with a r.m.s deviation 0.0693 Å. The plane through the two fused rings of the indane system is slightly folded around the C14—C15 bond, as indicated by the dihedral angle between them of 5.20 (1)°. The dihedral angle between the mean plane of the pyrrolizine and indane system is 82.58 (1)°, indicating that they are nearly perpendicular to each other. The cyclopentane ring adopts a twisted conformation with puckering parameters: $q_2 = 0.4839$ (16) Å and $\Phi_2 = 17.98$ (17)°. The dihedral angle between the mean planes of the pyrrolizine ring system and cyclopentane ring is 58.90 (1)°, indicating the significant curvature between the pyrrolizine and cyclopentene ring systems. The hydroxyl oxygen (O1) attached to the cyclopentane ring deviates by 1.1990 (10) Å from the mean plane of the ring. The piperidine ring with the methyl substituent in an equatorial position, adopts a twisted chair conformation with atoms C2 and C5 deviating by -0.6623 (10) Å and 0.8578 (10) Å respectively, from the mean plane defined by other atoms. In the structure, the aryl ring is in equatorial position of the attached pyrrolidine ring, as indicated by the torsion angle C7—C21—C26—C27 = -178.5 (15)°. The structure features a weak intramolecular O—H⋯N interaction.

### S2. Experimental

A mixture of 1-methyl-3-[*E*-(4-methylphenyl)methylidene]tetrahydro-2(1*H*)-pyridinone (1 mmol), ninhydrin (1 mmol) and proline (1 mmol) in methanol was refluxed for 3–4 h. After completion of the reaction, as indicated by TLC, the reaction mixture was poured into cold water. The solid precipitate obtained was filtered and dried. The product was purified by column chromatography using petroleum ether:ethylacetate mixture (90:10 *v*/v). Suitable crystals for the single-crystal-X-ray studies were obtained by recrystallizing the product from methanol. Yield: 63%, Melting point: 464–466 K.

## S3. Refinement

H atoms were placed at calculated positions and allowed to ride on their carrier atoms with O—H = 0.82 Å and C—H = 0.93–0.98 Å, and with $U_{iso} = 1.2U_{eq}(C)$ for CH$_2$ and CH groups, and $U_{iso} = 1.5U_{eq}(O, C)$ for OH and CH$_3$ groups.
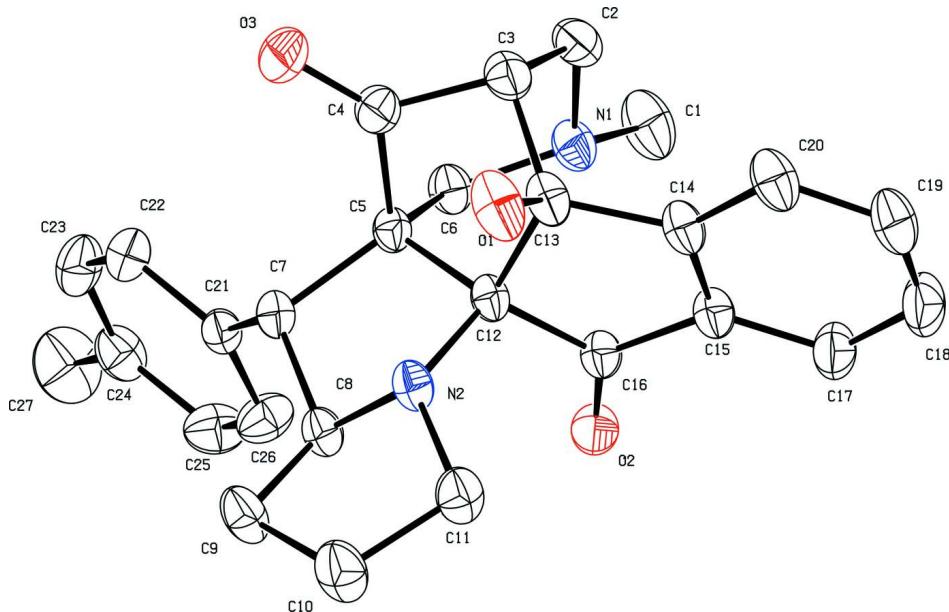


**Figure 1**

The molecular structure of (I), showing 20% probability displacement ellipsoids and the atom-numbering scheme. H-atoms are omitted for clarity.

**14a-Hydroxy-12-methyl-10-(4-methylphenyl)-8,9,9a,10,12,13,14,14a-octahydro-10a,14-methano-5*H*-indeno[2′,1′:4,5]azepino[3,4-*b*]pyrrolizine-5,15(7*H*,11*H*)-dione**

*Crystal data*

| | |
|---|---|
| C$_{27}$H$_{28}$N$_2$O$_3$ | $F(000) = 912$ |
| $M_r = 428.51$ | $D_x = 1.268$ Mg m$^{-3}$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| Hall symbol: -P 2ybc | Cell parameters from 2000 reflections |
| $a = 16.8684$ (6) Å | $\theta = 2$–31° |
| $b = 8.3754$ (3) Å | $\mu = 0.08$ mm$^{-1}$ |
| $c = 15.9930$ (6) Å | $T = 293$ K |
| $\beta = 96.658$ (1)° | Block, colourless |
| $V = 2244.25$ (14) Å$^3$ | $0.21 \times 0.19 \times 0.18$ mm |
| $Z = 4$ | |

*Data collection*

| | |
|---|---|
| Bruker Kappa APEXII diffractometer | 28459 measured reflections |
| Radiation source: fine-focus sealed tube | 6850 independent reflections |
| Graphite monochromator | 4500 reflections with $I > 2\sigma(I)$ |
| Detector resolution: 0 pixels mm$^{-1}$ | $R_{int} = 0.030$ |
| $\omega$ and $\varphi$ scans | $\theta_{max} = 30.6°$, $\theta_{min} = 2.4°$ |
| Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996) | $h = -24 \rightarrow 13$ |
| | $k = -11 \rightarrow 11$ |
| $T_{min} = 0.967$, $T_{max} = 0.974$ | $l = -22 \rightarrow 22$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | |
| $R[F^2 > 2\sigma(F^2)] = 0.053$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.161$ | |
| $S = 1.02$ | H-atom parameters constrained |
| 6850 reflections | $w = 1/[\sigma^2(F_o^2) + (0.0784P)^2 + 0.3451P]$ |
| 291 parameters | where $P = (F_o^2 + 2F_c^2)/3$ |
| 0 restraints | $(\Delta/\sigma)_{max} < 0.001$ |
| Primary atom site location: structure-invariant direct methods | $\Delta\rho_{max} = 0.29$ e Å$^{-3}$ |
| | $\Delta\rho_{min} = -0.18$ e Å$^{-3}$ |

*Special details*

**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 (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.16397 (13) | −0.0312 (2) | 0.12713 (14) | 0.0736 (6) |
| H1A | 0.1240 | −0.0997 | 0.0986 | 0.110* |
| H1B | 0.1529 | −0.0146 | 0.1840 | 0.110* |
| H1C | 0.2155 | −0.0800 | 0.1274 | 0.110* |
| C2 | 0.08361 (9) | 0.1896 (2) | 0.06440 (12) | 0.0555 (4) |
| H2A | 0.0618 | 0.2147 | 0.1164 | 0.067* |
| H2B | 0.0488 | 0.1119 | 0.0338 | 0.067* |
| C3 | 0.08668 (8) | 0.34102 (18) | 0.01156 (11) | 0.0523 (4) |
| H3 | 0.0337 | 0.3886 | −0.0022 | 0.063* |
| C4 | 0.14397 (9) | 0.45411 (17) | 0.06160 (10) | 0.0476 (3) |
| C5 | 0.22549 (7) | 0.37463 (15) | 0.06215 (8) | 0.0365 (3) |
| C6 | 0.22196 (9) | 0.23342 (17) | 0.12322 (9) | 0.0436 (3) |
| H6A | 0.2738 | 0.1825 | 0.1337 | 0.052* |
| H6B | 0.2064 | 0.2702 | 0.1765 | 0.052* |
| C7 | 0.29914 (8) | 0.48839 (15) | 0.07586 (8) | 0.0381 (3) |
| H7 | 0.2795 | 0.5984 | 0.0765 | 0.046* |
| C8 | 0.33710 (8) | 0.46387 (17) | −0.00551 (9) | 0.0409 (3) |
| H8 | 0.3715 | 0.3694 | 0.0007 | 0.049* |
| C9 | 0.37893 (11) | 0.5938 (2) | −0.04996 (10) | 0.0579 (4) |
| H9A | 0.4353 | 0.5990 | −0.0294 | 0.069* |
| H9B | 0.3548 | 0.6974 | −0.0429 | 0.069* |
| C10 | 0.36594 (13) | 0.5389 (2) | −0.14257 (11) | 0.0689 (5) |
| H10A | 0.3475 | 0.6271 | −0.1790 | 0.083* |
| H10B | 0.4153 | 0.4983 | −0.1600 | 0.083* |
| C11 | 0.30260 (10) | 0.4065 (2) | −0.14693 (10) | 0.0544 (4) |

| | | | |
|---|---|---|---|
| H11A | 0.2626 | 0.4204 | −0.1951 | 0.065* |
| H11B | 0.3265 | 0.3016 | −0.1495 | 0.065* |
| C12 | 0.22046 (7) | 0.31215 (15) | −0.03006 (8) | 0.0360 (3) |
| C13 | 0.13045 (8) | 0.31994 (16) | −0.06716 (10) | 0.0452 (3) |
| C14 | 0.11636 (8) | 0.16665 (17) | −0.11540 (9) | 0.0448 (3) |
| C15 | 0.17990 (8) | 0.06209 (17) | −0.09945 (9) | 0.0438 (3) |
| C16 | 0.24466 (8) | 0.13717 (16) | −0.04209 (9) | 0.0396 (3) |
| C17 | 0.17844 (10) | −0.0870 (2) | −0.13690 (12) | 0.0576 (4) |
| H17 | 0.2205 | −0.1583 | −0.1247 | 0.069* |
| C18 | 0.11302 (11) | −0.1270 (2) | −0.19287 (13) | 0.0681 (5) |
| H18 | 0.1113 | −0.2261 | −0.2193 | 0.082* |
| C19 | 0.05035 (11) | −0.0227 (2) | −0.21015 (12) | 0.0659 (5) |
| H19 | 0.0073 | −0.0514 | −0.2489 | 0.079* |
| C20 | 0.05024 (10) | 0.1241 (2) | −0.17081 (11) | 0.0572 (4) |
| H20 | 0.0069 | 0.1928 | −0.1812 | 0.069* |
| C21 | 0.35334 (8) | 0.46023 (16) | 0.15661 (8) | 0.0386 (3) |
| C22 | 0.34229 (10) | 0.54611 (18) | 0.22797 (9) | 0.0492 (4) |
| H22 | 0.3025 | 0.6233 | 0.2253 | 0.059* |
| C23 | 0.38944 (12) | 0.5195 (2) | 0.30366 (10) | 0.0588 (4) |
| H23 | 0.3801 | 0.5786 | 0.3508 | 0.071* |
| C24 | 0.44955 (10) | 0.4081 (2) | 0.31081 (10) | 0.0544 (4) |
| C25 | 0.46025 (10) | 0.3211 (2) | 0.24003 (11) | 0.0595 (4) |
| H25 | 0.5002 | 0.2440 | 0.2430 | 0.071* |
| C26 | 0.41302 (10) | 0.3457 (2) | 0.16452 (10) | 0.0554 (4) |
| H26 | 0.4215 | 0.2841 | 0.1180 | 0.066* |
| C27 | 0.50107 (14) | 0.3797 (3) | 0.39303 (13) | 0.0907 (7) |
| H27A | 0.5197 | 0.2713 | 0.3952 | 0.136* |
| H27B | 0.4704 | 0.3987 | 0.4390 | 0.136* |
| H27C | 0.5459 | 0.4510 | 0.3973 | 0.136* |
| N1 | 0.16329 (7) | 0.12170 (14) | 0.08371 (8) | 0.0466 (3) |
| N2 | 0.26863 (7) | 0.42998 (14) | −0.06780 (7) | 0.0412 (3) |
| O1 | 0.11417 (7) | 0.45503 (13) | −0.11974 (9) | 0.0661 (4) |
| H1 | 0.1531 | 0.5144 | −0.1152 | 0.099* |
| O2 | 0.30850 (6) | 0.07901 (13) | −0.01635 (7) | 0.0495 (3) |
| O3 | 0.12969 (7) | 0.57851 (14) | 0.09413 (9) | 0.0683 (4) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0835 (13) | 0.0447 (9) | 0.0899 (14) | −0.0161 (9) | −0.0015 (11) | 0.0194 (9) |
| C2 | 0.0445 (8) | 0.0507 (9) | 0.0722 (11) | −0.0076 (7) | 0.0108 (7) | 0.0000 (8) |
| C3 | 0.0346 (6) | 0.0406 (8) | 0.0802 (11) | 0.0045 (6) | 0.0001 (7) | −0.0030 (7) |
| C4 | 0.0462 (7) | 0.0355 (7) | 0.0611 (9) | 0.0035 (6) | 0.0065 (7) | 0.0001 (6) |
| C5 | 0.0383 (6) | 0.0290 (6) | 0.0413 (7) | −0.0008 (5) | 0.0010 (5) | −0.0004 (5) |
| C6 | 0.0487 (7) | 0.0373 (7) | 0.0440 (7) | −0.0039 (6) | 0.0026 (6) | 0.0042 (6) |
| C7 | 0.0417 (6) | 0.0314 (6) | 0.0398 (7) | −0.0031 (5) | −0.0011 (5) | −0.0006 (5) |
| C8 | 0.0429 (7) | 0.0407 (7) | 0.0374 (7) | −0.0081 (5) | −0.0024 (5) | 0.0013 (5) |
| C9 | 0.0632 (10) | 0.0599 (10) | 0.0499 (9) | −0.0257 (8) | 0.0036 (7) | 0.0054 (7) |

| C10 | 0.0879 (13) | 0.0713 (12) | 0.0489 (10) | −0.0298 (10) | 0.0138 (9) | 0.0033 (8) |
| C11 | 0.0668 (10) | 0.0559 (9) | 0.0396 (8) | −0.0119 (8) | 0.0028 (7) | 0.0005 (7) |
| C12 | 0.0364 (6) | 0.0301 (6) | 0.0396 (7) | −0.0022 (5) | −0.0043 (5) | 0.0025 (5) |
| C13 | 0.0396 (7) | 0.0326 (6) | 0.0592 (9) | 0.0005 (5) | −0.0119 (6) | 0.0076 (6) |
| C14 | 0.0448 (7) | 0.0394 (7) | 0.0471 (8) | −0.0074 (6) | −0.0087 (6) | 0.0053 (6) |
| C15 | 0.0438 (7) | 0.0394 (7) | 0.0462 (8) | −0.0046 (6) | −0.0037 (6) | −0.0021 (6) |
| C16 | 0.0408 (7) | 0.0359 (7) | 0.0405 (7) | −0.0012 (5) | −0.0019 (5) | −0.0007 (5) |
| C17 | 0.0548 (9) | 0.0479 (9) | 0.0674 (11) | −0.0029 (7) | −0.0046 (8) | −0.0152 (8) |
| C18 | 0.0704 (11) | 0.0599 (11) | 0.0704 (12) | −0.0127 (9) | −0.0077 (9) | −0.0229 (9) |
| C19 | 0.0642 (10) | 0.0682 (12) | 0.0593 (10) | −0.0210 (9) | −0.0177 (8) | −0.0066 (9) |
| C20 | 0.0521 (8) | 0.0527 (9) | 0.0608 (10) | −0.0091 (7) | −0.0187 (7) | 0.0091 (8) |
| C21 | 0.0394 (6) | 0.0376 (7) | 0.0382 (7) | −0.0056 (5) | 0.0023 (5) | −0.0020 (5) |
| C22 | 0.0598 (9) | 0.0427 (8) | 0.0446 (8) | 0.0016 (7) | 0.0035 (7) | −0.0055 (6) |
| C23 | 0.0833 (12) | 0.0543 (9) | 0.0377 (8) | −0.0053 (9) | 0.0018 (8) | −0.0079 (7) |
| C24 | 0.0524 (8) | 0.0644 (10) | 0.0436 (8) | −0.0118 (8) | −0.0058 (7) | 0.0055 (7) |
| C25 | 0.0476 (8) | 0.0773 (12) | 0.0526 (9) | 0.0129 (8) | 0.0014 (7) | 0.0074 (8) |
| C26 | 0.0552 (9) | 0.0691 (11) | 0.0412 (8) | 0.0156 (8) | 0.0027 (7) | −0.0045 (7) |
| C27 | 0.0929 (15) | 0.1150 (19) | 0.0560 (12) | −0.0076 (14) | −0.0256 (11) | 0.0093 (12) |
| N1 | 0.0495 (7) | 0.0351 (6) | 0.0547 (7) | −0.0066 (5) | 0.0038 (6) | 0.0059 (5) |
| N2 | 0.0464 (6) | 0.0390 (6) | 0.0364 (6) | −0.0089 (5) | −0.0027 (5) | 0.0026 (5) |
| O1 | 0.0616 (7) | 0.0435 (6) | 0.0851 (9) | −0.0024 (5) | −0.0266 (6) | 0.0229 (6) |
| O2 | 0.0404 (5) | 0.0462 (6) | 0.0591 (6) | 0.0073 (4) | −0.0057 (5) | −0.0043 (5) |
| O3 | 0.0640 (7) | 0.0453 (6) | 0.0966 (10) | 0.0087 (5) | 0.0137 (7) | −0.0168 (6) |

## Geometric parameters (Å, º)

| | | | |
|---|---|---|---|
| C1—N1 | 1.456 (2) | C11—H11B | 0.9700 |
| C1—H1A | 0.9600 | C12—N2 | 1.4533 (17) |
| C1—H1B | 0.9600 | C12—C16 | 1.5394 (18) |
| C1—H1C | 0.9600 | C12—C13 | 1.5661 (18) |
| C2—N1 | 1.459 (2) | C13—O1 | 1.4174 (17) |
| C2—C3 | 1.528 (2) | C13—C14 | 1.503 (2) |
| C2—H2A | 0.9700 | C14—C15 | 1.385 (2) |
| C2—H2B | 0.9700 | C14—C20 | 1.3881 (19) |
| C3—C4 | 1.514 (2) | C15—C17 | 1.384 (2) |
| C3—C13 | 1.542 (2) | C15—C16 | 1.4825 (19) |
| C3—H3 | 0.9800 | C16—O2 | 1.2098 (16) |
| C4—O3 | 1.2014 (18) | C17—C18 | 1.379 (2) |
| C4—C5 | 1.5269 (19) | C17—H17 | 0.9300 |
| C5—C6 | 1.5394 (18) | C18—C19 | 1.375 (3) |
| C5—C12 | 1.5578 (18) | C18—H18 | 0.9300 |
| C5—C7 | 1.5608 (17) | C19—C20 | 1.382 (3) |
| C6—N1 | 1.4524 (18) | C19—H19 | 0.9300 |
| C6—H6A | 0.9700 | C20—H20 | 0.9300 |
| C6—H6B | 0.9700 | C21—C22 | 1.380 (2) |
| C7—C21 | 1.5118 (18) | C21—C26 | 1.385 (2) |
| C7—C8 | 1.5293 (19) | C22—C23 | 1.387 (2) |
| C7—H7 | 0.9800 | C22—H22 | 0.9300 |

| | | | |
|---|---|---|---|
| C8—N2 | 1.4629 (17) | C23—C24 | 1.373 (3) |
| C8—C9 | 1.518 (2) | C23—H23 | 0.9300 |
| C8—H8 | 0.9800 | C24—C25 | 1.375 (2) |
| C9—C10 | 1.542 (2) | C24—C27 | 1.509 (2) |
| C9—H9A | 0.9700 | C25—C26 | 1.383 (2) |
| C9—H9B | 0.9700 | C25—H25 | 0.9300 |
| C10—C11 | 1.536 (2) | C26—H26 | 0.9300 |
| C10—H10A | 0.9700 | C27—H27A | 0.9600 |
| C10—H10B | 0.9700 | C27—H27B | 0.9600 |
| C11—N2 | 1.462 (2) | C27—H27C | 0.9600 |
| C11—H11A | 0.9700 | O1—H1 | 0.8200 |
| | | | |
| N1—C1—H1A | 109.5 | N2—C12—C5 | 101.15 (10) |
| N1—C1—H1B | 109.5 | C16—C12—C5 | 116.87 (11) |
| H1A—C1—H1B | 109.5 | N2—C12—C13 | 112.24 (10) |
| N1—C1—H1C | 109.5 | C16—C12—C13 | 104.58 (10) |
| H1A—C1—H1C | 109.5 | C5—C12—C13 | 106.78 (11) |
| H1B—C1—H1C | 109.5 | O1—C13—C14 | 111.67 (12) |
| N1—C2—C3 | 110.55 (12) | O1—C13—C3 | 108.35 (12) |
| N1—C2—H2A | 109.5 | C14—C13—C3 | 117.05 (12) |
| C3—C2—H2A | 109.5 | O1—C13—C12 | 111.76 (11) |
| N1—C2—H2B | 109.5 | C14—C13—C12 | 104.34 (11) |
| C3—C2—H2B | 109.5 | C3—C13—C12 | 103.32 (11) |
| H2A—C2—H2B | 108.1 | C15—C14—C20 | 120.18 (14) |
| C4—C3—C2 | 106.70 (13) | C15—C14—C13 | 111.89 (11) |
| C4—C3—C13 | 99.58 (12) | C20—C14—C13 | 127.93 (14) |
| C2—C3—C13 | 113.99 (12) | C17—C15—C14 | 121.09 (13) |
| C4—C3—H3 | 111.9 | C17—C15—C16 | 128.71 (14) |
| C2—C3—H3 | 111.9 | C14—C15—C16 | 110.17 (12) |
| C13—C3—H3 | 111.9 | O2—C16—C15 | 127.06 (13) |
| O3—C4—C3 | 128.58 (14) | O2—C16—C12 | 125.40 (12) |
| O3—C4—C5 | 126.97 (14) | C15—C16—C12 | 107.10 (11) |
| C3—C4—C5 | 104.45 (11) | C18—C17—C15 | 118.22 (16) |
| C4—C5—C6 | 103.74 (11) | C18—C17—H17 | 120.9 |
| C4—C5—C12 | 101.00 (11) | C15—C17—H17 | 120.9 |
| C6—C5—C12 | 109.92 (10) | C19—C18—C17 | 120.98 (17) |
| C4—C5—C7 | 115.87 (11) | C19—C18—H18 | 119.5 |
| C6—C5—C7 | 117.94 (11) | C17—C18—H18 | 119.5 |
| C12—C5—C7 | 107.05 (10) | C18—C19—C20 | 121.05 (15) |
| N1—C6—C5 | 107.06 (11) | C18—C19—H19 | 119.5 |
| N1—C6—H6A | 110.3 | C20—C19—H19 | 119.5 |
| C5—C6—H6A | 110.3 | C19—C20—C14 | 118.41 (16) |
| N1—C6—H6B | 110.3 | C19—C20—H20 | 120.8 |
| C5—C6—H6B | 110.3 | C14—C20—H20 | 120.8 |
| H6A—C6—H6B | 108.6 | C22—C21—C26 | 116.85 (13) |
| C21—C7—C8 | 115.76 (11) | C22—C21—C7 | 119.93 (13) |
| C21—C7—C5 | 114.67 (11) | C26—C21—C7 | 123.17 (13) |
| C8—C7—C5 | 101.74 (10) | C21—C22—C23 | 121.27 (15) |

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| C21—C7—H7 | 108.1 | C21—C22—H22 | 119.4 |
| C8—C7—H7 | 108.1 | C23—C22—H22 | 119.4 |
| C5—C7—H7 | 108.1 | C24—C23—C22 | 121.68 (15) |
| N2—C8—C9 | 101.15 (11) | C24—C23—H23 | 119.2 |
| N2—C8—C7 | 103.31 (11) | C22—C23—H23 | 119.2 |
| C9—C8—C7 | 124.43 (13) | C23—C24—C25 | 117.22 (15) |
| N2—C8—H8 | 108.9 | C23—C24—C27 | 121.66 (18) |
| C9—C8—H8 | 108.9 | C25—C24—C27 | 121.11 (18) |
| C7—C8—H8 | 108.9 | C24—C25—C26 | 121.50 (16) |
| C8—C9—C10 | 102.34 (12) | C24—C25—H25 | 119.3 |
| C8—C9—H9A | 111.3 | C26—C25—H25 | 119.3 |
| C10—C9—H9A | 111.3 | C25—C26—C21 | 121.46 (15) |
| C8—C9—H9B | 111.3 | C25—C26—H26 | 119.3 |
| C10—C9—H9B | 111.3 | C21—C26—H26 | 119.3 |
| H9A—C9—H9B | 109.2 | C24—C27—H27A | 109.5 |
| C11—C10—C9 | 106.25 (13) | C24—C27—H27B | 109.5 |
| C11—C10—H10A | 110.5 | H27A—C27—H27B | 109.5 |
| C9—C10—H10A | 110.5 | C24—C27—H27C | 109.5 |
| C11—C10—H10B | 110.5 | H27A—C27—H27C | 109.5 |
| C9—C10—H10B | 110.5 | H27B—C27—H27C | 109.5 |
| H10A—C10—H10B | 108.7 | C6—N1—C1 | 113.06 (13) |
| N2—C11—C10 | 101.77 (12) | C6—N1—C2 | 113.93 (12) |
| N2—C11—H11A | 111.4 | C1—N1—C2 | 113.56 (13) |
| C10—C11—H11A | 111.4 | C12—N2—C11 | 124.22 (11) |
| N2—C11—H11B | 111.4 | C12—N2—C8 | 106.51 (10) |
| C10—C11—H11B | 111.4 | C11—N2—C8 | 104.95 (11) |
| H11A—C11—H11B | 109.3 | C13—O1—H1 | 109.5 |
| N2—C12—C16 | 115.18 (11) | | |
| | | | |
| N1—C2—C3—C4 | −57.27 (17) | C12—C13—C14—C15 | 10.41 (16) |
| N1—C2—C3—C13 | 51.62 (18) | O1—C13—C14—C20 | −48.4 (2) |
| C2—C3—C4—O3 | −111.93 (19) | C3—C13—C14—C20 | 77.3 (2) |
| C13—C3—C4—O3 | 129.31 (18) | C12—C13—C14—C20 | −169.26 (15) |
| C2—C3—C4—C5 | 67.92 (15) | C20—C14—C15—C17 | −1.1 (2) |
| C13—C3—C4—C5 | −50.84 (14) | C13—C14—C15—C17 | 179.19 (15) |
| O3—C4—C5—C6 | 106.79 (18) | C20—C14—C15—C16 | 177.07 (14) |
| C3—C4—C5—C6 | −73.07 (14) | C13—C14—C15—C16 | −2.63 (18) |
| O3—C4—C5—C12 | −139.33 (17) | C17—C15—C16—O2 | −1.3 (3) |
| C3—C4—C5—C12 | 40.81 (14) | C14—C15—C16—O2 | −179.28 (14) |
| O3—C4—C5—C7 | −24.1 (2) | C17—C15—C16—C12 | 171.45 (16) |
| C3—C4—C5—C7 | 156.03 (12) | C14—C15—C16—C12 | −6.55 (16) |
| C4—C5—C6—N1 | 67.25 (14) | N2—C12—C16—O2 | 61.65 (19) |
| C12—C5—C6—N1 | −40.06 (14) | C5—C12—C16—O2 | −56.89 (19) |
| C7—C5—C6—N1 | −163.10 (11) | C13—C12—C16—O2 | −174.68 (14) |
| C4—C5—C7—C21 | 115.03 (14) | N2—C12—C16—C15 | −111.23 (12) |
| C6—C5—C7—C21 | −8.76 (17) | C5—C12—C16—C15 | 130.23 (12) |
| C12—C5—C7—C21 | −133.23 (11) | C13—C12—C16—C15 | 12.44 (14) |
| C4—C5—C7—C8 | −119.25 (13) | C14—C15—C17—C18 | 2.1 (3) |

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| C6—C5—C7—C8 | 116.97 (13) | C16—C15—C17—C18 | −175.70 (16) |
| C12—C5—C7—C8 | −7.51 (13) | C15—C17—C18—C19 | −0.9 (3) |
| C21—C7—C8—N2 | 156.36 (11) | C17—C18—C19—C20 | −1.3 (3) |
| C5—C7—C8—N2 | 31.36 (12) | C18—C19—C20—C14 | 2.3 (3) |
| C21—C7—C8—C9 | −89.87 (17) | C15—C14—C20—C19 | −1.1 (2) |
| C5—C7—C8—C9 | 145.12 (13) | C13—C14—C20—C19 | 178.57 (16) |
| N2—C8—C9—C10 | −36.92 (17) | C8—C7—C21—C22 | 148.72 (13) |
| C7—C8—C9—C10 | −151.73 (15) | C5—C7—C21—C22 | −93.23 (15) |
| C8—C9—C10—C11 | 12.7 (2) | C8—C7—C21—C26 | −34.09 (19) |
| C9—C10—C11—N2 | 16.2 (2) | C5—C7—C21—C26 | 83.96 (17) |
| C4—C5—C12—N2 | 102.67 (11) | C26—C21—C22—C23 | 0.6 (2) |
| C6—C5—C12—N2 | −148.20 (11) | C7—C21—C22—C23 | 177.97 (14) |
| C7—C5—C12—N2 | −18.97 (12) | C21—C22—C23—C24 | 0.7 (3) |
| C4—C5—C12—C16 | −131.46 (12) | C22—C23—C24—C25 | −1.3 (3) |
| C6—C5—C12—C16 | −22.32 (15) | C22—C23—C24—C27 | 179.71 (18) |
| C7—C5—C12—C16 | 106.91 (12) | C23—C24—C25—C26 | 0.6 (3) |
| C4—C5—C12—C13 | −14.88 (12) | C27—C24—C25—C26 | 179.64 (19) |
| C6—C5—C12—C13 | 94.26 (12) | C24—C25—C26—C21 | 0.7 (3) |
| C7—C5—C12—C13 | −136.51 (10) | C22—C21—C26—C25 | −1.3 (2) |
| C4—C3—C13—O1 | −79.52 (13) | C7—C21—C26—C25 | −178.53 (15) |
| C2—C3—C13—O1 | 167.27 (12) | C5—C6—N1—C1 | 169.39 (14) |
| C4—C3—C13—C14 | 153.16 (12) | C5—C6—N1—C2 | −58.99 (16) |
| C2—C3—C13—C14 | 39.95 (17) | C3—C2—N1—C6 | 54.13 (18) |
| C4—C3—C13—C12 | 39.16 (13) | C3—C2—N1—C1 | −174.49 (15) |
| C2—C3—C13—C12 | −74.05 (14) | C16—C12—N2—C11 | 35.17 (18) |
| N2—C12—C13—O1 | −8.82 (17) | C5—C12—N2—C11 | 162.16 (13) |
| C16—C12—C13—O1 | −134.36 (13) | C13—C12—N2—C11 | −84.36 (16) |
| C5—C12—C13—O1 | 101.15 (14) | C16—C12—N2—C8 | −86.64 (13) |
| N2—C12—C13—C14 | 112.01 (12) | C5—C12—N2—C8 | 40.35 (13) |
| C16—C12—C13—C14 | −13.54 (14) | C13—C12—N2—C8 | 153.84 (12) |
| C5—C12—C13—C14 | −138.02 (11) | C10—C11—N2—C12 | −163.50 (14) |
| N2—C12—C13—C3 | −125.10 (12) | C10—C11—N2—C8 | −40.99 (16) |
| C16—C12—C13—C3 | 109.35 (12) | C9—C8—N2—C12 | −176.64 (12) |
| C5—C12—C13—C3 | −15.13 (13) | C7—C8—N2—C12 | −46.94 (13) |
| O1—C13—C14—C15 | 131.29 (14) | C9—C8—N2—C11 | 50.03 (15) |
| C3—C13—C14—C15 | −103.02 (15) | C7—C8—N2—C11 | 179.73 (11) |

*Hydrogen-bond geometry (Å, º)*

| D—H···A | D—H | H···A | D···A | D—H···A |
|---|---|---|---|---|
| O1—H1···N2 | 0.82 | 2.13 | 2.6497 (16) | 121 |
| C19—H19···O1[i] | 0.93 | 2.76 | 3.659 (2) | 163 |

Symmetry code: (i) −x, y−1/2, −z−1/2.