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# 2,4-Bis(2-methylphenyl)-3-azabicyclo-[3.3.1]nonan-9-one *O*-methyloxime

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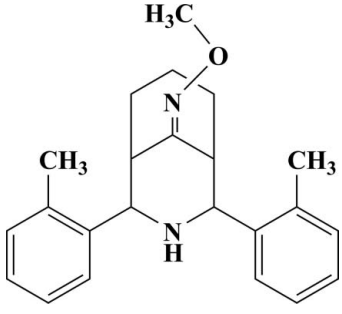
Key indicators: single-crystal X-ray study; $T$ = 298 K; mean $\sigma$(C–C) = 0.003 Å; disorder in main residue; $R$ factor = 0.060; $wR$ factor = 0.169; data-to-parameter ratio = 15.3.

The molecule of the title compound, $C_{23}H_{28}N_2O$, exists in a twin-chair conformation, with equatorial orientation of the *ortho*-tolyl groups on both sides of the secondary amino group. The title oxime compound and its ketone precursor 2,4-bis(2-methylphenyl)-3-azabicyclo[3.3.1]nonan-9-one exhibit similar stereochemistries, with the orientation of the *o*-tolyl rings almost identical in both compounds. In the title compound, the tolyl rings are at an angle of 23.77 (3)° with respect to one another; the angle in the precursor is 29.4 (1)° [Vijayalakshmi, Parthasarathi, Venkatraj & Jeyaraman (2000), *Acta Cryst.* C**56**, 1240–1241]. The cyclohexane ring and the oxime ether are disordered over two alternative orientations, with a refined site-occupancy ratio of 0.813 (2):0.186 (4). The crystal structure of the title compound is stabilized by intermolecular N—H$\cdots\pi$ interactions.

## Related literature

For the synthesis and biological activities of oxime derivatives of 3-azabicyclo[3.3.1]nonan-9-ones, see: Parthiban *et al.* (2009*a*,*b*, 2010*a*,*b*); Jeyaraman & Avila (1981). For related structures with similar conformations, see: Vijayalakshmi *et al.* (2000); Parthiban *et al.* (2009*c*,*d*). For ring-puckering parameters, see: Cremer & Pople (1975); Nardelli (1983).




## Experimental

### Crystal data

$C_{23}H_{28}N_2O$
$M_r$ = 348.47
Monoclinic, $P2_1/n$
$a$ = 6.9700 (9) Å
$b$ = 15.3476 (16) Å
$c$ = 18.354 (2) Å
$\beta$ = 94.622 (4)°
$V$ = 1957.0 (4) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.07 mm$^{-1}$
$T$ = 298 K
0.32 × 0.27 × 0.15 mm

### Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
$T_{min}$ = 0.977, $T_{max}$ = 0.989
12742 measured reflections
4416 independent reflections
2070 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.043

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.169
$S$ = 1.01
4416 reflections
288 parameters
38 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.14 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.19 e Å$^{-3}$

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2* and *SAINT* (Bruker, 2004); data reduction: *SAINT* and *XPREP* (Bruker, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZL2305).

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# supporting information

*Acta Cryst.* (2010). E**66**, o2978 [https://doi.org/10.1107/S1600536810043436]

## 2,4-Bis(2-methylphenyl)-3-azabicyclo[3.3.1]nonan-9-one *O*-methyloxime

**P. Parthiban, V. Ramkumar and Yeon Tae Jeong**

### S1. Comment

Nitrogen containing heterocyclic oximes and oxime ethers are very important molecules in the field of medicinal chemistry due to their broad spectrum of biological activities *viz.* antifungal, antibacterial, antimycobacterial, analgesic, antagonistic, anticancer, antiinflammatory, local anesthetic and hypotensive activity (Parthiban *et al.*, 2009*a*,*b*, 2010*a*,*b*; Jeyaraman & Avila, 1981). Since the stereochemistry of bio-active molecules is a major criterion for their biological response, it is of immense help to establish the stereochemistry of the newly synthesized molecules. Accordingly, we have synthesized the title oxime ether to examine its conformation and orientation of the substituents.

In the crystal structure, the oxime unit is partially flipped thus inducing disorder for the oxime (N2/O1/C22) and also for the piperidine ring N1/C1/C2/C8/C6/C7 and the cylcohexane ring C2–C6/C8 over two orientations. The site occupancy ratio refined to 0.813 (2) to 0.186 (4). The tolyl rings do not participate in the disorder (Fig. 3).

An analysis of the six-membered piperidine ring gave the following: According to Nardelli (Nardelli, 1983), the smallest displacement asymmetry parameters $q_2$ and $q_3$ are 0.052 (4) and -0.614 (4) Å, respectively. According to Cremer and Pople (Cremer & Pople, 1975), the ring puckering parameters such as total puckering amplitude $Q_T$ and phase angle $\theta$ are 0.616 (4) Å and 175.5 (4)°. Thus, all parameters strongly support a near ideal chair conformation for the piperidine ring N1/C1/C2/C8/C6/C7. Similarly, the analysis of cyclohexane ring C2–C6/C8 indicates that it also adopts a chair conformation. It is, however, deviating more from the ideal chair with puckering parameters $Q_T$ and $\theta$ of 0.553 (7) Å and 169.2 (9)°, and $q_2$ and $q_3$ of 0.108 (9) and -0.543 (8) Å, respectively.

The torsion angles C8—C6—C7—C15 and C8—C2—C1—C9 of the *ortho*-tolyl rings are -177.2 (3) and 179.2 (3)° and they are orientated at an angle of 23.77 (3)° with respect to one another, whereas in its ketone precursor, 2,4-bis(2-methylphenyl)-3-azabicyclo[3.3.1]nonan-9-one, they are oriented at an angle of 29.4 (1)° (Vijayalakshmi *et al.*, 2000). The crystal structure of the title compound is stabilized by intermolecular N—H···$\pi$ interactions with N1—H1···*Cg*1 = 2.633 Å, (*Cg*: C15–C20; symmetry operator = 1 - *x*, 2 - *y*, -*z*.)

Thus, the detailed crystallographic study of asymmetry parameters, ring puckering parameters and torsion angles calculated for the title compound proves that the bicyclic moiety exists in a twin-chair conformation with equatorial orientation of the *ortho*-tolyl rings on both sides of the secondary amino group.

### S2. Experimental

The title compound was synthesized by adding 0.501 g *O*-methylhydroxylamine hydrochloride (0.006 mol) and 2.04 g sodium acetate trihydrate (0.015 mol) in a hot ethanolic solution of 1.597 g 2,4-bis(2-methylphenyl)-3-azabicyclo-[3.3.1]nonan-9-one (0.005 mol) (Parthiban *et al.*, 2010*b*). The content was refluxed at 345–350 K till completion of the reaction; the progress and completion of the reaction was monitored by TLC. After the consumption of starting material, the content of the flask was concentrated and water was added. Then, the precipitated oxime ether was separated by filtration, washed with an excess of water, and dried in vacuum. X-ray diffraction quality crystals of 2,4-bis(2-methyl-

phenyl)-3-azabicyclo[3.3.1]nonan-9-one *O*-methyloxime were obtained by slow evaporation from ethanol.

### S3. Refinement

The cyclohexane ring C2–C6/C8 and the oxime ether N2/O1/C22 are disordered over two orientations with a refined site occupancy ratio of 0.813 (2) to 0.186 (4). The two moieties were restrained to have similar geometries. The atoms N2b, O1b and C22b of the minor moiety were restrained to have similar anisotropic displacement parameters. The ADPs of all other disordered atoms in the minor moiety were constrained to be identical to those of their counterparts in the major moiety.

The nitrogen H atom was located in a difference Fourier map and refined isotropically. Other H atoms were fixed geometrically and allowed to ride on the parent C atoms with aromatic C—H = 0.93 Å, methylene C—H = 0.97 Å, methine C—H = 0.98 Å and methyl C—H = 0.96 Å. The displacement parameters were set for phenyl, methylene and aliphatic H atoms at $U_{iso}$(H) = 1.2$U_{eq}$(C) and for methyl H atoms at $U_{iso}$(H) = 1.5$U_{eq}$(C).

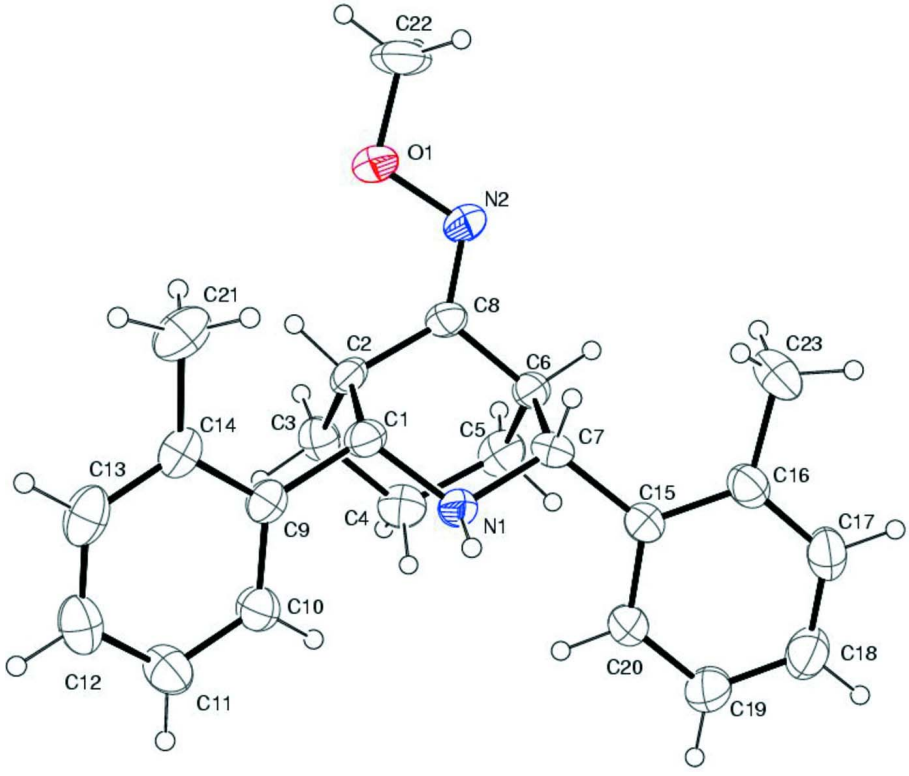


**Figure 1**

Anistropic displacement representation of the molecule with atoms represented with 30% probability ellipsoids. The minor moiety is omitted for clarity.

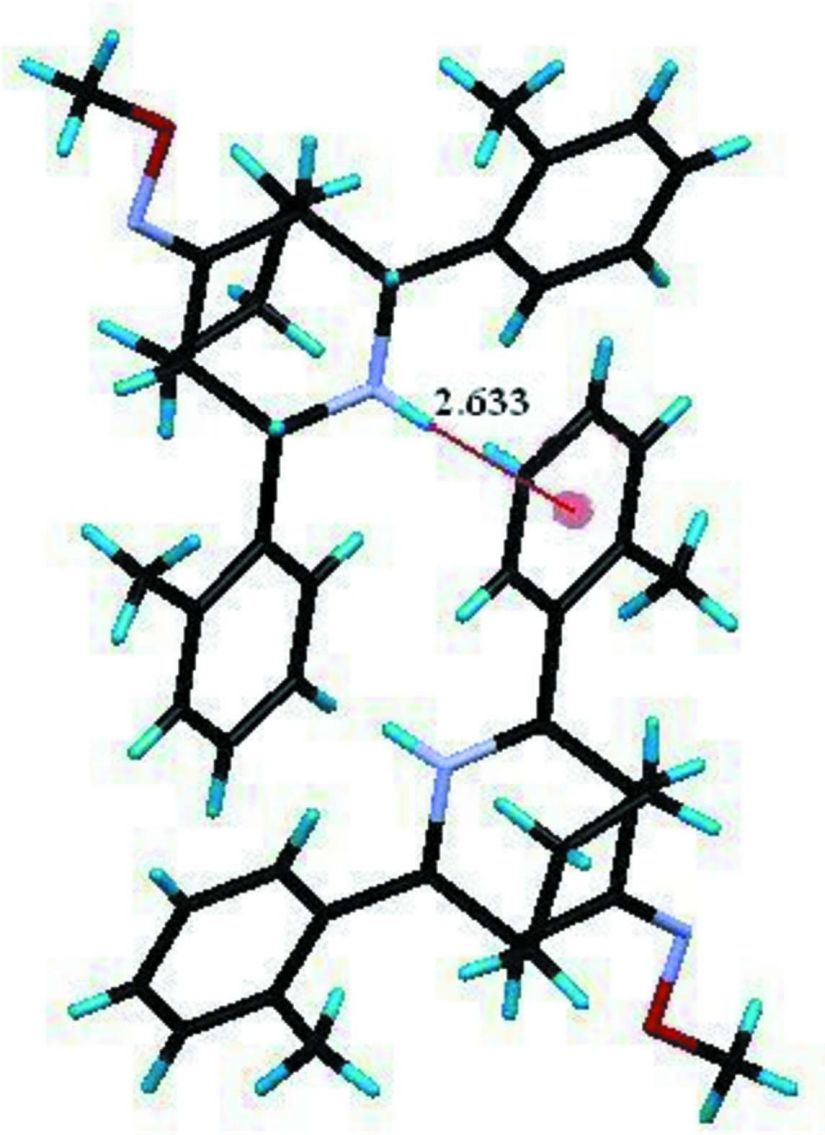


**Figure 2**

Packing diagram showing the N—H···$\pi$ interaction. N1—H1···*Cg*1 = 2.633Å [*Cg*: C15–C20] and symmetry operator = 1 - *x*, 2 - *y*, -*z*.

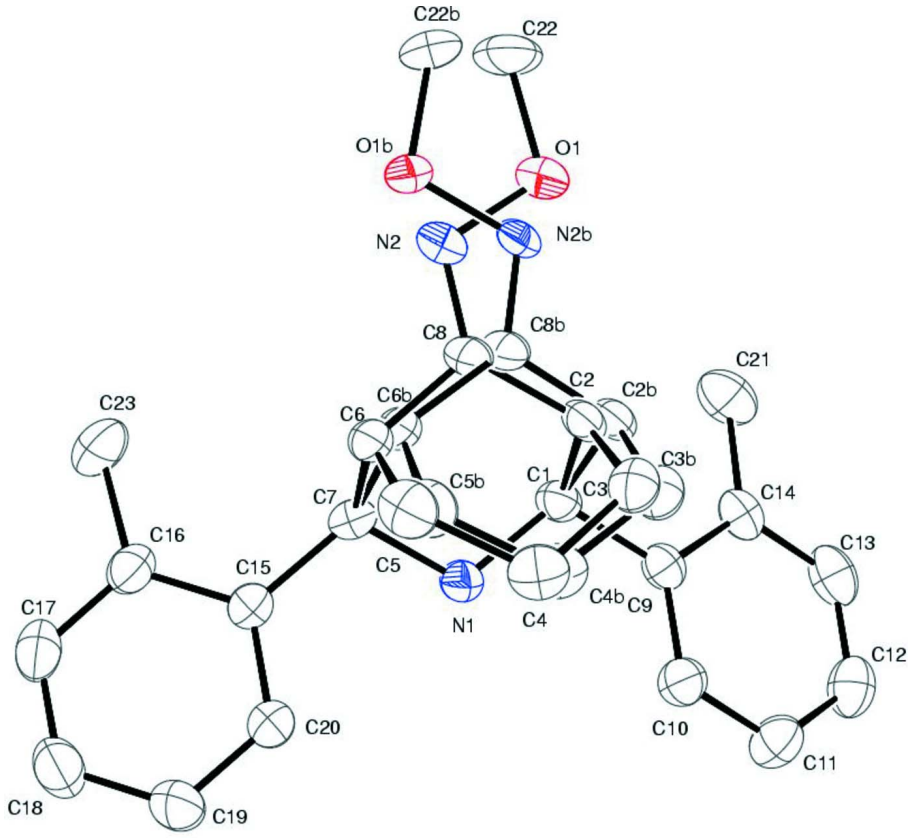


**Figure 3**

*ORTEP* (H atoms are removed for clarity) showing the disorder in two orientations.

### 2,4-Bis(2-methylphenyl)-3-azabicyclo[3.3.1]nonan-9-one *O*-methyloxime

*Crystal data*

$C_{23}H_{28}N_2O$
$M_r = 348.47$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 6.9700\ (9)$ Å
$b = 15.3476\ (16)$ Å
$c = 18.354\ (2)$ Å
$\beta = 94.622\ (4)°$
$V = 1957.0\ (4)$ Å$^3$
$Z = 4$
$F(000) = 752$
$D_x = 1.183$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2129 reflections
$\theta = 2.2–20.4°$
$\mu = 0.07$ mm$^{-1}$
$T = 298$ K
Block, colourless
0.32 × 0.27 × 0.15 mm

*Data collection*

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
$T_{min} = 0.977$, $T_{max} = 0.989$
12742 measured reflections
4416 independent reflections
2070 reflections with $I > 2\sigma(I)$
$R_{int} = 0.043$
$\theta_{max} = 28.3°$, $\theta_{min} = 1.7°$
$h = -9\rightarrow9$
$k = -20\rightarrow13$
$l = -24\rightarrow20$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.060$
$wR(F^2) = 0.169$
$S = 1.01$
4416 reflections
288 parameters
38 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
$w = 1/[\sigma^2(F_o^2) + (0.0708P)^2 + 0.1996P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.14$ e Å$^{-3}$
$\Delta\rho_{min} = -0.19$ 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* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| N1 | 0.6085 (3) | 0.86925 (11) | 0.08349 (9) | 0.0470 (5) | |
| C1 | 0.5497 (3) | 0.81766 (13) | 0.14551 (11) | 0.0461 (6) | |
| H1 | 0.4087 | 0.8169 | 0.1428 | 0.055* | |
| C7 | 0.5361 (3) | 0.83350 (13) | 0.01217 (12) | 0.0497 (6) | |
| H7 | 0.3953 | 0.8317 | 0.0100 | 0.060* | |
| C9 | 0.6212 (3) | 0.85822 (13) | 0.21810 (12) | 0.0462 (6) | |
| C10 | 0.7833 (3) | 0.91132 (15) | 0.22295 (13) | 0.0563 (6) | |
| H10 | 0.8457 | 0.9223 | 0.1810 | 0.068* | |
| C11 | 0.8540 (4) | 0.94796 (17) | 0.28793 (14) | 0.0663 (7) | |
| H11 | 0.9614 | 0.9840 | 0.2895 | 0.080* | |
| C12 | 0.7663 (4) | 0.93131 (18) | 0.34982 (15) | 0.0704 (8) | |
| H12 | 0.8140 | 0.9552 | 0.3942 | 0.084* | |
| C13 | 0.6056 (4) | 0.87857 (17) | 0.34608 (14) | 0.0670 (8) | |
| H13 | 0.5467 | 0.8670 | 0.3887 | 0.080* | |
| C14 | 0.5287 (3) | 0.84220 (15) | 0.28101 (13) | 0.0533 (6) | |
| C15 | 0.5949 (3) | 0.88982 (14) | −0.05005 (12) | 0.0485 (6) | |
| C16 | 0.4936 (3) | 0.88717 (14) | −0.11942 (13) | 0.0554 (6) | |
| C17 | 0.5643 (4) | 0.93407 (17) | −0.17588 (13) | 0.0654 (7) | |
| H17 | 0.4987 | 0.9317 | −0.2220 | 0.078* | |
| C18 | 0.7258 (4) | 0.98333 (18) | −0.16638 (15) | 0.0737 (8) | |
| H18 | 0.7717 | 1.0131 | −0.2056 | 0.088* | |
| C19 | 0.8199 (4) | 0.9886 (2) | −0.09852 (15) | 0.0850 (9) | |
| H19 | 0.9277 | 1.0241 | −0.0907 | 0.102* | |
| C20 | 0.7563 (4) | 0.94157 (17) | −0.04161 (14) | 0.0716 (8) | |

| | | | | | |
|---|---|---|---|---|---|
| H20 | 0.8242 | 0.9448 | 0.0040 | 0.086* | |
| C21 | 0.3492 (4) | 0.78778 (18) | 0.28148 (15) | 0.0799 (9) | |
| H21A | 0.2570 | 0.8069 | 0.2431 | 0.120* | |
| H21B | 0.2956 | 0.7940 | 0.3278 | 0.120* | |
| H21C | 0.3804 | 0.7277 | 0.2738 | 0.120* | |
| C23 | 0.3094 (4) | 0.8366 (2) | −0.13412 (16) | 0.0958 (11) | |
| H23A | 0.3383 | 0.7757 | −0.1376 | 0.144* | |
| H23B | 0.2439 | 0.8561 | −0.1792 | 0.144* | |
| H23C | 0.2285 | 0.8459 | −0.0949 | 0.144* | |
| C2 | 0.6198 (9) | 0.7228 (3) | 0.1364 (2) | 0.0452 (10) | 0.814 (5) |
| H2 | 0.5669 | 0.6871 | 0.1743 | 0.054* | 0.814 (5) |
| C3 | 0.8388 (9) | 0.7096 (8) | 0.1418 (3) | 0.0547 (14) | 0.814 (5) |
| H3A | 0.8937 | 0.7353 | 0.1871 | 0.066* | 0.814 (5) |
| H3B | 0.8661 | 0.6476 | 0.1439 | 0.066* | 0.814 (5) |
| C4 | 0.9363 (12) | 0.7491 (12) | 0.0785 (4) | 0.0608 (14) | 0.814 (5) |
| H4A | 1.0673 | 0.7275 | 0.0795 | 0.073* | 0.814 (5) |
| H4B | 0.9421 | 0.8119 | 0.0844 | 0.073* | 0.814 (5) |
| C5 | 0.8307 (10) | 0.7274 (7) | 0.0047 (3) | 0.0628 (12) | 0.814 (5) |
| H5A | 0.8582 | 0.6675 | −0.0075 | 0.075* | 0.814 (5) |
| H5B | 0.8804 | 0.7643 | −0.0323 | 0.075* | 0.814 (5) |
| C6 | 0.6115 (9) | 0.7394 (2) | 0.0025 (2) | 0.0491 (10) | 0.814 (5) |
| H6 | 0.5532 | 0.7158 | −0.0438 | 0.059* | 0.814 (5) |
| C8 | 0.5378 (8) | 0.6901 (3) | 0.0635 (2) | 0.0475 (11) | 0.814 (5) |
| C2B | 0.621 (4) | 0.7216 (11) | 0.1560 (11) | 0.0452 (10) | 0.186 (5) |
| H2B | 0.5691 | 0.6932 | 0.1980 | 0.054* | 0.186 (5) |
| C3B | 0.843 (4) | 0.715 (4) | 0.1583 (18) | 0.0547 (14) | 0.186 (5) |
| H3C | 0.8985 | 0.7459 | 0.2011 | 0.066* | 0.186 (5) |
| H3D | 0.8799 | 0.6543 | 0.1636 | 0.066* | 0.186 (5) |
| C4B | 0.928 (6) | 0.751 (6) | 0.091 (2) | 0.0608 (14) | 0.186 (5) |
| H4C | 1.0571 | 0.7279 | 0.0894 | 0.073* | 0.186 (5) |
| H4D | 0.9406 | 0.8141 | 0.0970 | 0.073* | 0.186 (5) |
| C5B | 0.814 (5) | 0.733 (4) | 0.0191 (17) | 0.0628 (12) | 0.186 (5) |
| H5C | 0.8508 | 0.6755 | 0.0023 | 0.075* | 0.186 (5) |
| H5D | 0.8504 | 0.7749 | −0.0167 | 0.075* | 0.186 (5) |
| C6B | 0.594 (5) | 0.7349 (11) | 0.0214 (12) | 0.0491 (10) | 0.186 (5) |
| H6B | 0.5385 | 0.7044 | −0.0223 | 0.059* | 0.186 (5) |
| C8B | 0.546 (4) | 0.6829 (17) | 0.0853 (12) | 0.0475 (11) | 0.186 (5) |
| N2 | 0.4164 (5) | 0.6292 (2) | 0.0473 (2) | 0.0555 (9) | 0.814 (5) |
| O1 | 0.3550 (4) | 0.59007 (17) | 0.11187 (14) | 0.0658 (9) | 0.814 (5) |
| C22 | 0.2246 (7) | 0.5220 (3) | 0.0898 (3) | 0.0928 (17) | 0.814 (5) |
| H22A | 0.1227 | 0.5449 | 0.0570 | 0.139* | 0.814 (5) |
| H22B | 0.1715 | 0.4978 | 0.1320 | 0.139* | 0.814 (5) |
| H22C | 0.2916 | 0.4773 | 0.0654 | 0.139* | 0.814 (5) |
| N2B | 0.427 (2) | 0.6218 (8) | 0.0948 (8) | 0.048 (4) | 0.186 (5) |
| O1B | 0.3413 (16) | 0.5918 (7) | 0.0264 (5) | 0.060 (3) | 0.186 (5) |
| C22B | 0.220 (3) | 0.5208 (13) | 0.0398 (10) | 0.087 (7) | 0.186 (5) |
| H22D | 0.2958 | 0.4735 | 0.0608 | 0.130* | 0.186 (5) |
| H22E | 0.1535 | 0.5021 | −0.0054 | 0.130* | 0.186 (5) |

| | | | | | |
|---|---|---|---|---|---|
| H22F | 0.1277 | 0.5384 | 0.0731 | 0.130* | 0.186 (5) |
| H1N | 0.563 (4) | 0.927 (2) | 0.0881 (15) | 0.104* | |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N1 | 0.0589 (12) | 0.0416 (10) | 0.0405 (11) | −0.0091 (9) | 0.0041 (9) | −0.0006 (9) |
| C1 | 0.0432 (13) | 0.0455 (12) | 0.0500 (14) | −0.0055 (10) | 0.0066 (10) | 0.0049 (11) |
| C7 | 0.0505 (14) | 0.0450 (13) | 0.0526 (15) | −0.0115 (10) | −0.0024 (11) | −0.0044 (11) |
| C9 | 0.0466 (14) | 0.0456 (13) | 0.0471 (14) | 0.0057 (10) | 0.0077 (11) | 0.0050 (10) |
| C10 | 0.0569 (15) | 0.0651 (15) | 0.0479 (15) | −0.0076 (12) | 0.0104 (12) | −0.0052 (12) |
| C11 | 0.0605 (17) | 0.0771 (18) | 0.0612 (18) | −0.0052 (14) | 0.0041 (14) | −0.0142 (14) |
| C12 | 0.082 (2) | 0.0784 (19) | 0.0500 (17) | 0.0130 (16) | 0.0007 (15) | −0.0095 (14) |
| C13 | 0.080 (2) | 0.0710 (17) | 0.0530 (17) | 0.0211 (15) | 0.0244 (14) | 0.0091 (14) |
| C14 | 0.0569 (15) | 0.0541 (14) | 0.0503 (15) | 0.0102 (12) | 0.0142 (12) | 0.0102 (12) |
| C15 | 0.0528 (14) | 0.0475 (13) | 0.0445 (14) | −0.0041 (11) | 0.0006 (11) | −0.0035 (11) |
| C16 | 0.0612 (16) | 0.0500 (14) | 0.0529 (15) | 0.0001 (12) | −0.0074 (12) | −0.0072 (12) |
| C17 | 0.083 (2) | 0.0656 (17) | 0.0454 (15) | 0.0104 (15) | −0.0069 (14) | −0.0010 (13) |
| C18 | 0.077 (2) | 0.0842 (19) | 0.0602 (18) | −0.0015 (16) | 0.0084 (16) | 0.0196 (15) |
| C19 | 0.071 (2) | 0.105 (2) | 0.077 (2) | −0.0352 (17) | −0.0080 (16) | 0.0304 (18) |
| C20 | 0.0695 (19) | 0.0869 (19) | 0.0552 (16) | −0.0322 (15) | −0.0137 (14) | 0.0161 (14) |
| C21 | 0.074 (2) | 0.086 (2) | 0.084 (2) | −0.0038 (16) | 0.0332 (16) | 0.0165 (16) |
| C23 | 0.095 (2) | 0.102 (2) | 0.083 (2) | −0.0286 (19) | −0.0386 (18) | 0.0025 (17) |
| C2 | 0.0569 (16) | 0.0393 (12) | 0.040 (3) | −0.0069 (11) | 0.011 (2) | 0.0081 (16) |
| C3 | 0.0604 (17) | 0.051 (2) | 0.052 (4) | 0.0061 (13) | 0.002 (2) | −0.005 (3) |
| C4 | 0.0500 (17) | 0.0626 (19) | 0.071 (4) | 0.0018 (15) | 0.012 (2) | −0.005 (4) |
| C5 | 0.075 (2) | 0.057 (2) | 0.060 (3) | 0.0040 (18) | 0.025 (2) | −0.008 (3) |
| C6 | 0.067 (2) | 0.0449 (14) | 0.035 (3) | −0.0150 (13) | 0.007 (2) | −0.0068 (14) |
| C8 | 0.0547 (16) | 0.0316 (15) | 0.055 (3) | −0.0055 (13) | 0.001 (2) | 0.004 (2) |
| C2B | 0.0569 (16) | 0.0393 (12) | 0.040 (3) | −0.0069 (11) | 0.011 (2) | 0.0081 (16) |
| C3B | 0.0604 (17) | 0.051 (2) | 0.052 (4) | 0.0061 (13) | 0.002 (2) | −0.005 (3) |
| C4B | 0.0500 (17) | 0.0626 (19) | 0.071 (4) | 0.0018 (15) | 0.012 (2) | −0.005 (4) |
| C5B | 0.075 (2) | 0.057 (2) | 0.060 (3) | 0.0040 (18) | 0.025 (2) | −0.008 (3) |
| C6B | 0.067 (2) | 0.0449 (14) | 0.035 (3) | −0.0150 (13) | 0.007 (2) | −0.0068 (14) |
| C8B | 0.0547 (16) | 0.0316 (15) | 0.055 (3) | −0.0055 (13) | 0.001 (2) | 0.004 (2) |
| N2 | 0.069 (2) | 0.0400 (19) | 0.057 (2) | −0.0111 (15) | 0.003 (2) | 0.010 (2) |
| O1 | 0.074 (2) | 0.0531 (17) | 0.0686 (18) | −0.0232 (13) | −0.0030 (13) | 0.0137 (12) |
| C22 | 0.082 (3) | 0.066 (2) | 0.126 (4) | −0.041 (2) | −0.020 (3) | 0.017 (3) |
| N2B | 0.073 (10) | 0.029 (8) | 0.042 (8) | −0.011 (6) | 0.004 (8) | 0.008 (7) |
| O1B | 0.085 (8) | 0.045 (6) | 0.047 (6) | −0.028 (5) | −0.003 (5) | −0.002 (5) |
| C22B | 0.096 (13) | 0.070 (10) | 0.090 (14) | −0.050 (9) | −0.018 (13) | 0.000 (12) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| N1—C7 | 1.470 (3) | C2—C3 | 1.536 (5) |
| N1—C1 | 1.472 (3) | C2—H2 | 0.9800 |
| N1—H1N | 0.94 (3) | C3—C4 | 1.519 (5) |
| C1—C9 | 1.518 (3) | C3—H3A | 0.9700 |

| | | | |
|---|---|---|---|
| C1—C2 | 1.549 (4) | C3—H3B | 0.9700 |
| C1—C2B | 1.563 (15) | C4—C5 | 1.525 (5) |
| C1—H1 | 0.9800 | C4—H4A | 0.9700 |
| C7—C15 | 1.515 (3) | C4—H4B | 0.9700 |
| C7—C6 | 1.552 (4) | C5—C6 | 1.536 (5) |
| C7—C6B | 1.570 (15) | C5—H5A | 0.9700 |
| C7—H7 | 0.9800 | C5—H5B | 0.9700 |
| C9—C14 | 1.389 (3) | C6—C8 | 1.477 (4) |
| C9—C10 | 1.390 (3) | C6—H6 | 0.9800 |
| C10—C11 | 1.374 (3) | C8—N2 | 1.280 (5) |
| C10—H10 | 0.9300 | C2B—C8B | 1.483 (16) |
| C11—C12 | 1.357 (4) | C2B—C3B | 1.548 (17) |
| C11—H11 | 0.9300 | C2B—H2B | 0.9800 |
| C12—C13 | 1.380 (4) | C3B—C4B | 1.513 (17) |
| C12—H12 | 0.9300 | C3B—H3C | 0.9700 |
| C13—C14 | 1.386 (3) | C3B—H3D | 0.9700 |
| C13—H13 | 0.9300 | C4B—C5B | 1.520 (17) |
| C14—C21 | 1.505 (3) | C4B—H4C | 0.9700 |
| C15—C20 | 1.375 (3) | C4B—H4D | 0.9700 |
| C15—C16 | 1.406 (3) | C5B—C6B | 1.543 (17) |
| C16—C17 | 1.385 (3) | C5B—H5C | 0.9700 |
| C16—C23 | 1.506 (3) | C5B—H5D | 0.9700 |
| C17—C18 | 1.355 (4) | C6B—C8B | 1.478 (16) |
| C17—H17 | 0.9300 | C6B—H6B | 0.9800 |
| C18—C19 | 1.362 (3) | C8B—N2B | 1.275 (16) |
| C18—H18 | 0.9300 | N2—O1 | 1.424 (4) |
| C19—C20 | 1.372 (3) | O1—C22 | 1.422 (4) |
| C19—H19 | 0.9300 | C22—H22A | 0.9600 |
| C20—H20 | 0.9300 | C22—H22B | 0.9600 |
| C21—H21A | 0.9600 | C22—H22C | 0.9600 |
| C21—H21B | 0.9600 | N2B—O1B | 1.422 (13) |
| C21—H21C | 0.9600 | O1B—C22B | 1.414 (14) |
| C23—H23A | 0.9600 | C22B—H22D | 0.9600 |
| C23—H23B | 0.9600 | C22B—H22E | 0.9600 |
| C23—H23C | 0.9600 | C22B—H22F | 0.9600 |
| C2—C8 | 1.499 (4) | | |

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|---|---|---|---|
| C7—N1—C1 | 113.01 (16) | C3—C2—H2 | 108.0 |
| C7—N1—H1N | 109.5 (17) | C1—C2—H2 | 108.0 |
| C1—N1—H1N | 108.5 (18) | C4—C3—C2 | 113.6 (5) |
| N1—C1—C9 | 111.47 (17) | C4—C3—H3A | 108.8 |
| N1—C1—C2 | 108.2 (2) | C2—C3—H3A | 108.8 |
| C9—C1—C2 | 113.3 (2) | C4—C3—H3B | 108.8 |
| N1—C1—C2B | 119.8 (10) | C2—C3—H3B | 108.8 |
| C9—C1—C2B | 101.5 (8) | H3A—C3—H3B | 107.7 |
| N1—C1—H1 | 107.9 | C3—C4—C5 | 112.3 (5) |
| C9—C1—H1 | 107.9 | C3—C4—H4A | 109.2 |
| C2—C1—H1 | 107.9 | C5—C4—H4A | 109.2 |

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| C2B—C1—H1 | 107.7 | C3—C4—H4B | 109.2 |
| N1—C7—C15 | 111.35 (17) | C5—C4—H4B | 109.2 |
| N1—C7—C6 | 110.8 (2) | H4A—C4—H4B | 107.9 |
| C15—C7—C6 | 109.3 (2) | C4—C5—C6 | 113.9 (5) |
| N1—C7—C6B | 101.4 (9) | C4—C5—H5A | 108.8 |
| C15—C7—C6B | 123.3 (9) | C6—C5—H5A | 108.8 |
| N1—C7—H7 | 108.5 | C4—C5—H5B | 108.8 |
| C15—C7—H7 | 108.5 | C6—C5—H5B | 108.8 |
| C6—C7—H7 | 108.5 | H5A—C5—H5B | 107.7 |
| C6B—C7—H7 | 102.9 | C8—C6—C5 | 108.9 (4) |
| C14—C9—C10 | 118.7 (2) | C8—C6—C7 | 104.3 (3) |
| C14—C9—C1 | 121.0 (2) | C5—C6—C7 | 117.0 (5) |
| C10—C9—C1 | 120.3 (2) | C8—C6—H6 | 108.8 |
| C11—C10—C9 | 121.9 (2) | C5—C6—H6 | 108.8 |
| C11—C10—H10 | 119.1 | C7—C6—H6 | 108.8 |
| C9—C10—H10 | 119.1 | N2—C8—C6 | 117.6 (4) |
| C12—C11—C10 | 119.7 (3) | N2—C8—C2 | 130.4 (4) |
| C12—C11—H11 | 120.1 | C6—C8—C2 | 112.0 (3) |
| C10—C11—H11 | 120.1 | C8B—C2B—C3B | 106.1 (18) |
| C11—C12—C13 | 119.2 (3) | C8B—C2B—C1 | 100.6 (16) |
| C11—C12—H12 | 120.4 | C3B—C2B—C1 | 112 (3) |
| C13—C12—H12 | 120.4 | C8B—C2B—H2B | 112.5 |
| C12—C13—C14 | 122.4 (2) | C3B—C2B—H2B | 112.5 |
| C12—C13—H13 | 118.8 | C1—C2B—H2B | 112.5 |
| C14—C13—H13 | 118.8 | C4B—C3B—C2B | 114 (2) |
| C13—C14—C9 | 118.1 (2) | C4B—C3B—H3C | 108.7 |
| C13—C14—C21 | 118.8 (2) | C2B—C3B—H3C | 108.7 |
| C9—C14—C21 | 123.0 (2) | C4B—C3B—H3D | 108.7 |
| C20—C15—C16 | 117.7 (2) | C2B—C3B—H3D | 108.7 |
| C20—C15—C7 | 120.9 (2) | H3C—C3B—H3D | 107.6 |
| C16—C15—C7 | 121.3 (2) | C3B—C4B—C5B | 115 (2) |
| C17—C16—C15 | 118.7 (2) | C3B—C4B—H4C | 108.5 |
| C17—C16—C23 | 118.9 (2) | C5B—C4B—H4C | 108.5 |
| C15—C16—C23 | 122.4 (2) | C3B—C4B—H4D | 108.5 |
| C18—C17—C16 | 122.4 (2) | C5B—C4B—H4D | 108.5 |
| C18—C17—H17 | 118.8 | H4C—C4B—H4D | 107.5 |
| C16—C17—H17 | 118.8 | C4B—C5B—C6B | 115 (2) |
| C17—C18—C19 | 119.0 (3) | C4B—C5B—H5C | 108.5 |
| C17—C18—H18 | 120.5 | C6B—C5B—H5C | 108.5 |
| C19—C18—H18 | 120.5 | C4B—C5B—H5D | 108.5 |
| C18—C19—C20 | 120.2 (3) | C6B—C5B—H5D | 108.5 |
| C18—C19—H19 | 119.9 | H5C—C5B—H5D | 107.5 |
| C20—C19—H19 | 119.9 | C8B—C6B—C5B | 107.3 (19) |
| C19—C20—C15 | 122.0 (2) | C8B—C6B—C7 | 122.3 (19) |
| C19—C20—H20 | 119.0 | C5B—C6B—C7 | 105 (3) |
| C15—C20—H20 | 119.0 | C8B—C6B—H6B | 107.0 |
| C14—C21—H21A | 109.5 | C5B—C6B—H6B | 107.0 |
| C14—C21—H21B | 109.5 | C7—C6B—H6B | 107.0 |

| | | | |
|---|---|---|---|
| H21A—C21—H21B | 109.5 | N2B—C8B—C6B | 134 (2) |
| C14—C21—H21C | 109.5 | N2B—C8B—C2B | 111.2 (19) |
| H21A—C21—H21C | 109.5 | C6B—C8B—C2B | 113.1 (16) |
| H21B—C21—H21C | 109.5 | C8—N2—O1 | 110.7 (4) |
| C16—C23—H23A | 109.5 | C22—O1—N2 | 107.5 (3) |
| C16—C23—H23B | 109.5 | C8B—N2B—O1B | 110.5 (14) |
| H23A—C23—H23B | 109.5 | C22B—O1B—N2B | 108.2 (11) |
| C16—C23—H23C | 109.5 | O1B—C22B—H22D | 109.5 |
| H23A—C23—H23C | 109.5 | O1B—C22B—H22E | 109.5 |
| H23B—C23—H23C | 109.5 | H22D—C22B—H22E | 109.5 |
| C8—C2—C3 | 108.6 (4) | O1B—C22B—H22F | 109.5 |
| C8—C2—C1 | 108.1 (3) | H22D—C22B—H22F | 109.5 |
| C3—C2—C1 | 116.0 (6) | H22E—C22B—H22F | 109.5 |
| C8—C2—H2 | 108.0 | | |

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|---|---|---|---|
| C7—N1—C1—C9 | −178.06 (17) | C17—C18—C19—C20 | 2.8 (5) |
| C7—N1—C1—C2 | 56.7 (3) | C18—C19—C20—C15 | −1.6 (5) |
| C1—N1—C7—C15 | 178.36 (18) | C16—C15—C20—C19 | −0.9 (4) |
| C1—N1—C7—C6 | −59.8 (3) | C7—C15—C20—C19 | 175.5 (3) |
| N1—C1—C9—C14 | 155.62 (19) | N1—C1—C2—C8 | −56.6 (4) |
| C2—C1—C9—C14 | −82.1 (3) | C9—C1—C2—C8 | 179.3 (3) |
| N1—C1—C9—C10 | −25.4 (3) | N1—C1—C2—C3 | 65.6 (4) |
| C2—C1—C9—C10 | 97.0 (3) | C9—C1—C2—C3 | −58.5 (4) |
| C14—C9—C10—C11 | 0.1 (3) | C8—C2—C3—C4 | 53.6 (8) |
| C1—C9—C10—C11 | −178.9 (2) | C1—C2—C3—C4 | −68.4 (8) |
| C9—C10—C11—C12 | 1.1 (4) | C2—C3—C4—C5 | −46.2 (12) |
| C10—C11—C12—C13 | −0.9 (4) | C3—C4—C5—C6 | 45.7 (13) |
| C11—C12—C13—C14 | −0.5 (4) | C4—C5—C6—C8 | −53.0 (9) |
| C12—C13—C14—C9 | 1.7 (4) | C4—C5—C6—C7 | 64.9 (9) |
| C12—C13—C14—C21 | −178.0 (2) | N1—C7—C6—C8 | 59.8 (4) |
| C10—C9—C14—C13 | −1.4 (3) | C15—C7—C6—C8 | −177.2 (3) |
| C1—C9—C14—C13 | 177.60 (19) | N1—C7—C6—C5 | −60.6 (4) |
| C10—C9—C14—C21 | 178.3 (2) | C15—C7—C6—C5 | 62.4 (4) |
| C1—C9—C14—C21 | −2.7 (3) | C5—C6—C8—N2 | −119.0 (6) |
| N1—C7—C15—C20 | 25.6 (3) | C7—C6—C8—N2 | 115.4 (6) |
| C6—C7—C15—C20 | −97.1 (3) | C5—C6—C8—C2 | 62.0 (6) |
| N1—C7—C15—C16 | −158.2 (2) | C7—C6—C8—C2 | −63.6 (5) |
| C6—C7—C15—C16 | 79.1 (3) | C3—C2—C8—N2 | 118.8 (8) |
| C20—C15—C16—C17 | 2.1 (3) | C1—C2—C8—N2 | −114.6 (6) |
| C7—C15—C16—C17 | −174.2 (2) | C3—C2—C8—C6 | −62.4 (6) |
| C20—C15—C16—C23 | −176.7 (3) | C1—C2—C8—C6 | 64.2 (6) |
| C7—C15—C16—C23 | 7.0 (4) | C6—C8—N2—O1 | −178.2 (4) |
| C15—C16—C17—C18 | −1.0 (4) | C2—C8—N2—O1 | 0.6 (8) |
| C23—C16—C17—C18 | 177.9 (3) | C8—N2—O1—C22 | −178.6 (5) |
| C16—C17—C18—C19 | −1.5 (4) | | |