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# 2,4-Bis(2-ethoxyphenyl)-7-methyl-3-azabicyclo[3.3.1]nonan-9-one

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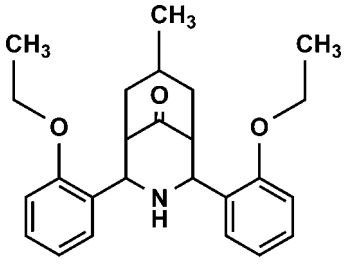
Key indicators: single-crystal X-ray study; $T$ = 298 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.042; $wR$ factor = 0.117; data-to-parameter ratio = 14.4.

The crystal structure of the title compound, $C_{25}H_{31}NO_3$, exists in a twin-chair conformation with an equatorial orientation of the *ortho*-ethoxyphenyl groups. According to Cremer and Pople [Cremer & Pople (1975), *J. Am. Chem. Soc.* **97**, 1354–1358], both the piperidone and cyclohexanone rings are significantly puckered with total puckering amplitutdes $Q_T$ of 0.5889 (18) and 0.554 (2) Å, respectively. The *ortho*-ethoxyphenyl groups are located on either side of the secondary amino group and make a dihedral angle of 12.41 (4)° with respect to each other. The methyl group on the cyclohexanone part occupies an exocyclic equatorial disposition. The crystal packing is stabilized by weak van der Waals interactions.

## Related literature

For the synthesis and biological activity of 3-azabicyclo-[3.3.1]nonan-9-ones, see: Jeyaraman & Avila (1981); Barker *et al.* (2005); Parthiban *et al.* (2009*a*, 2010*b*,*c*, 2011). For related structures, see: Parthiban *et al.* (2009*b*,*c*, 2010*a*,*c*); Cox *et al.* (1985); Smith-Verdier *et al.* (1983); Padegimas & Kovacic (1972). For ring puckering parameters, see: Cremer & Pople (1975); Nardelli (1983).




## Experimental

### Crystal data

$C_{25}H_{31}NO_3$
$M_r$ = 393.51
Monoclinic, $P2_1/n$
$a$ = 10.3147 (6) Å
$b$ = 11.8817 (6) Å
$c$ = 18.7809 (10) Å
$\beta$ = 100.866 (2)°
$V$ = 2260.4 (2) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.08 mm$^{-1}$
$T$ = 298 K
0.35 × 0.28 × 0.15 mm

### Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
$T_{min}$ = 0.974, $T_{max}$ = 0.989
12446 measured reflections
3876 independent reflections
2415 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.025

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.117
$S$ = 1.03
3876 reflections
269 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.12 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.13 e Å$^{-3}$

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2* and *SAINT-Plus* (Bruker, 2004); data reduction: *SAINT-Plus* 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: LW2064).

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# supplementary materials

*Acta Cryst.* (2011). E**67**, o1475-o1476 [ **doi:10.1107/S1600536811018472** ]

# 2,4-Bis(2-ethoxyphenyl)-7-methyl-3-azabicyclo[3.3.1]nonan-9-one

**P. Parthiban, V. Ramkumar, D. H. Park and Y. T. Jeong**

## Comment

The 3-azabicycle nucleus is an important class of pharmacophore due to its broad spectrum of biological activities such as antibacterial, antimycobacterial, antifungal, anticancer, antitussive, antiinflammatory, sedative, antipyretic and calcium antagonistic activity (Jeyaraman & Avila, 1981; Barker *et al.*, 2005; Parthiban *et al.*, 2009*a*, 2010*b*, 2010*c*, 2011). Its biological significane prompted the medicinal chemists to synthesize some structural analogs. Since the stereochemistry plays an important role in biological actions, it is of immense help to establish the stereochemistry of the synthesized bio-potent molecules. Since several stereomers are possible for the synthesized title compound along with different conformations such as chair-chair (Parthiban *et al.*, 2009*b*, 2009*c*, 2010*a*; Cox *et al.*, 1985), chair-boat (Parthiban *et al.*, 2010*c*; Smith-Verdier *et al.*, 1983) and boat-boat (Padegimas & Kovacic, 1972), the title compound was undertaken for the present single-crystal XRD study to establish the stereochemistry.

The analysis of torsion angles, asymmetry parameters and puckering parameters calculated for the title compound shows that the piperidine ring slightly deviates the ideal chair conformation. According to Cremer & Pople, the total puckering amplitude, $Q_T$ is 0.5889 (18) Å and the phase angle θ is 7.19 (18)° (Cremer & Pople, 1975) for the piperidine ring. Also according to Nardelli, the smallest displacement asymmetry parameters $q_2$ and $q_3$ are 0.0741 (18) and 0.5843 (18)°, respectively (Nardelli, 1983).

The cyclohexanone ring deviates more than the piperidone ring from the ideal chair conformation. According to Cremer and Pople the $Q_T$ = 0.554 (2) and θ = 12.2 (2)° (Cremer & Pople, 1975) and by Nardelli, $q_2$ = 0.118 (2) and $q_3$ = 0.541 (2)° (Nardelli, 1983).

The torsion angles of C8—C6—C7—C15 and C8—C2—C1—C9 are -179.07 (14) and 176.83 (14)°, respectively.

The above detailed analysis of the title compound $C_{25}H_{31}NO_3$, clearly shows that the compound exists in a twin-chair conformation with an equatorial orientation of the *ortho*-ethoxyphenyl units on both sides of the secondary amino group. The *ortho*-ethoxyphenyl groups are orientated at a dihedral angle of 12.41 (4)° with respect to each other. The methyl group attached to the cyclohexanone part occupies an exocyclic equatorial disposition. The crystal packing is stabilized by weak van der Waals interactions.

## Experimental

The 7-methyl-2,4-*bis*(2-ethoxyphenyl)-3-azabicyclo[3.3.1]nonan-9-one was synthesized by a modified and an optimized Mannich condensation in one-pot, using 2-ethoxybenzaldehyde (0.1 mol, 15.02 g/13.94 ml), 4-methylcyclohexanone (0.05 mol, 5.61 g/6.14 ml) and ammonium acetate (0.075 mol, 5.78 g) in a 50 ml of absolute ethanol. The mixture was gently warmed on a hot plate at 303–308 K (30–35° C) with moderate stirring till the complete consumption of the starting materials, which was monitored by TLC. At the end, the crude azabicyclic ketone was separated by filtration and gently washed with

1:5 cold ethanol-ether mixture. X-ray diffraction quality crystals of the title compound were obtained by slow evaporation from ethanol.

### Refinement

The nitrogen H atom was located in a difference Fourier map and refined isotropically. Other hydrogen atoms were fixed geometrically and allowed to ride on the parent carbon atoms with aromatic C—H = 0.93 Å, aliphatic C—H = 0.98Å and methylene C—H = 0.97 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)..

### Figures

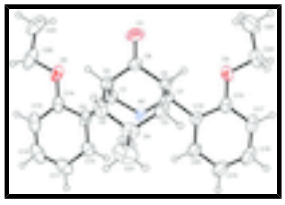


Fig. 1. Anistropic displacement representation of the molecule with atoms represented with 30% probability ellipsoids.

### 2,4-Bis(2-ethoxyphenyl)-7-methyl-3-azabicyclo[3.3.1]nonan-9-one

*Crystal data*

| | |
|---|---|
| $C_{25}H_{31}NO_3$ | $F(000)$ = 848 |
| $M_r$ = 393.51 | $D_x$ = 1.156 Mg m$^{-3}$ |
| Monoclinic, $P2_1/n$ | Mo $K\alpha$ radiation, λ = 0.71073 Å |
| Hall symbol: -P 2yn | Cell parameters from 3407 reflections |
| $a$ = 10.3147 (6) Å | θ = 2.6–21.8° |
| $b$ = 11.8817 (6) Å | μ = 0.08 mm$^{-1}$ |
| $c$ = 18.7809 (10) Å | $T$ = 298 K |
| β = 100.866 (2)° | Block, colourless |
| $V$ = 2260.4 (2) Å$^3$ | 0.35 × 0.28 × 0.15 mm |
| $Z$ = 4 | |

*Data collection*

| | |
|---|---|
| Bruker APEXII CCD area-detector diffractometer | 3876 independent reflections |
| Radiation source: fine-focus sealed tube | 2415 reflections with $I > 2\sigma(I)$ |
| graphite | $R_{int}$ = 0.025 |
| φ and ω scans | $\theta_{max}$ = 25.6°, $\theta_{min}$ = 2.0° |
| Absorption correction: multi-scan (*SADABS*; Bruker, 2004) | $h$ = −10→11 |
| $T_{min}$ = 0.974, $T_{max}$ = 0.989 | $k$ = −14→14 |
| 12446 measured reflections | $l$ = −22→16 |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full | Secondary atom site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)] = 0.042$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.117$ | H atoms treated by a mixture of independent and constrained refinement |
| $S = 1.03$ | $w = 1/[\sigma^2(F_o^2) + (0.047P)^2 + 0.3732P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| 3876 reflections | $(\Delta/\sigma)_{max} < 0.001$ |
| 269 parameters | $\Delta\rho_{max} = 0.12$ e Å$^{-3}$ |
| 0 restraints | $\Delta\rho_{min} = -0.13$ 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 | 1.07118 (17) | 0.68920 (14) | 0.12439 (9) | 0.0475 (4) |
| H1 | 1.1292 | 0.6243 | 0.1379 | 0.057* |
| C2 | 0.99532 (18) | 0.71301 (15) | 0.18699 (9) | 0.0553 (5) |
| H2 | 1.0598 | 0.7203 | 0.2323 | 0.066* |
| C3 | 0.9088 (2) | 0.81806 (16) | 0.17738 (11) | 0.0676 (6) |
| H3A | 0.8763 | 0.8312 | 0.2219 | 0.081* |
| H3B | 0.9629 | 0.8821 | 0.1698 | 0.081* |
| C4 | 0.7921 (2) | 0.81182 (16) | 0.11504 (11) | 0.0659 (6) |
| H4 | 0.8258 | 0.8178 | 0.0698 | 0.079* |
| C5 | 0.72060 (18) | 0.70069 (16) | 0.11430 (10) | 0.0585 (5) |
| H5A | 0.6605 | 0.6933 | 0.0681 | 0.070* |
| H5B | 0.6678 | 0.7023 | 0.1519 | 0.070* |

| | | | | |
|---|---|---|---|---|
| C6 | 0.80887 (17) | 0.59660 (14) | 0.12573 (8) | 0.0489 (4) |
| H6 | 0.7547 | 0.5311 | 0.1324 | 0.059* |
| C7 | 0.88565 (16) | 0.57076 (14) | 0.06386 (8) | 0.0454 (4) |
| H7 | 0.9362 | 0.5012 | 0.0759 | 0.054* |
| C8 | 0.90968 (17) | 0.61391 (15) | 0.19325 (9) | 0.0522 (5) |
| C9 | 1.15495 (18) | 0.78883 (15) | 0.11170 (10) | 0.0522 (5) |
| C10 | 1.1188 (2) | 0.86086 (16) | 0.05364 (11) | 0.0643 (5) |
| H10 | 1.0419 | 0.8464 | 0.0202 | 0.077* |
| C11 | 1.1939 (3) | 0.95391 (18) | 0.04389 (15) | 0.0868 (7) |
| H11 | 1.1680 | 1.0011 | 0.0042 | 0.104* |
| C12 | 1.3066 (3) | 0.9759 (2) | 0.09300 (18) | 0.0975 (8) |
| H12 | 1.3576 | 1.0384 | 0.0866 | 0.117* |
| C13 | 1.3453 (2) | 0.9065 (2) | 0.15186 (15) | 0.0861 (7) |
| H13 | 1.4219 | 0.9224 | 0.1852 | 0.103* |
| C14 | 1.27035 (19) | 0.81263 (17) | 0.16149 (11) | 0.0629 (5) |
| C15 | 0.79067 (17) | 0.55378 (15) | −0.00749 (9) | 0.0495 (5) |
| C16 | 0.71335 (18) | 0.45680 (16) | −0.01864 (10) | 0.0569 (5) |
| C17 | 0.6211 (2) | 0.4422 (2) | −0.08214 (12) | 0.0741 (6) |
| H17 | 0.5700 | 0.3773 | −0.0892 | 0.089* |
| C18 | 0.6060 (2) | 0.5244 (3) | −0.13440 (12) | 0.0885 (8) |
| H18 | 0.5439 | 0.5150 | −0.1768 | 0.106* |
| C19 | 0.6809 (2) | 0.6193 (2) | −0.12479 (11) | 0.0871 (7) |
| H19 | 0.6707 | 0.6743 | −0.1607 | 0.105* |
| C20 | 0.7721 (2) | 0.63362 (18) | −0.06142 (10) | 0.0671 (6) |
| H20 | 0.8223 | 0.6991 | −0.0551 | 0.080* |
| C21 | 0.6966 (3) | 0.9105 (2) | 0.11769 (15) | 0.1095 (9) |
| H21A | 0.6663 | 0.9092 | 0.1630 | 0.164* |
| H21B | 0.6225 | 0.9037 | 0.0785 | 0.164* |
| H21C | 0.7414 | 0.9803 | 0.1133 | 0.164* |
| C22 | 1.4219 (2) | 0.7531 (3) | 0.26893 (13) | 0.1002 (9) |
| H22A | 1.4220 | 0.8246 | 0.2938 | 0.120* |
| H22B | 1.4965 | 0.7517 | 0.2442 | 0.120* |
| C23 | 1.4315 (3) | 0.6584 (3) | 0.32186 (15) | 0.1209 (11) |
| H23A | 1.3550 | 0.6583 | 0.3441 | 0.181* |
| H23B | 1.5093 | 0.6676 | 0.3585 | 0.181* |
| H23C | 1.4365 | 0.5883 | 0.2970 | 0.181* |
| C24 | 0.6380 (2) | 0.29441 (18) | 0.03834 (13) | 0.0819 (7) |
| H24A | 0.6386 | 0.2398 | 0.0000 | 0.098* |
| H24B | 0.5510 | 0.3284 | 0.0315 | 0.098* |
| C25 | 0.6690 (3) | 0.2388 (2) | 0.10965 (16) | 0.1147 (10) |
| H25A | 0.7545 | 0.2041 | 0.1155 | 0.172* |
| H25B | 0.6037 | 0.1823 | 0.1127 | 0.172* |
| H25C | 0.6691 | 0.2936 | 0.1472 | 0.172* |
| H1N | 1.0230 (17) | 0.6437 (14) | 0.0263 (9) | 0.054 (6)* |
| N1 | 0.97762 (14) | 0.66179 (12) | 0.05778 (8) | 0.0461 (4) |
| O1 | 0.92001 (15) | 0.55398 (12) | 0.24612 (7) | 0.0837 (5) |
| O2 | 1.30161 (13) | 0.73934 (13) | 0.21804 (7) | 0.0743 (4) |
| O3 | 0.73609 (13) | 0.37931 (11) | 0.03597 (7) | 0.0719 (4) |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0384 (11) | 0.0552 (10) | 0.0466 (10) | −0.0026 (8) | 0.0018 (8) | −0.0035 (8) |
| C2 | 0.0484 (12) | 0.0740 (12) | 0.0406 (9) | −0.0126 (10) | 0.0011 (8) | −0.0080 (9) |
| C3 | 0.0705 (15) | 0.0652 (13) | 0.0748 (13) | −0.0188 (11) | 0.0337 (12) | −0.0210 (10) |
| C4 | 0.0688 (14) | 0.0615 (12) | 0.0749 (13) | 0.0125 (11) | 0.0328 (12) | 0.0059 (10) |
| C5 | 0.0434 (12) | 0.0802 (13) | 0.0527 (11) | 0.0004 (10) | 0.0108 (9) | −0.0027 (9) |
| C6 | 0.0473 (11) | 0.0536 (10) | 0.0453 (10) | −0.0099 (9) | 0.0079 (8) | 0.0020 (8) |
| C7 | 0.0407 (11) | 0.0469 (9) | 0.0463 (9) | −0.0020 (8) | 0.0023 (8) | −0.0020 (7) |
| C8 | 0.0509 (12) | 0.0644 (11) | 0.0411 (10) | 0.0019 (9) | 0.0079 (8) | 0.0055 (9) |
| C9 | 0.0403 (11) | 0.0596 (11) | 0.0575 (11) | −0.0062 (9) | 0.0114 (9) | −0.0097 (9) |
| C10 | 0.0566 (13) | 0.0667 (12) | 0.0722 (13) | −0.0074 (11) | 0.0190 (10) | 0.0011 (10) |
| C11 | 0.0885 (19) | 0.0707 (15) | 0.1100 (19) | −0.0106 (14) | 0.0411 (16) | 0.0105 (13) |
| C12 | 0.085 (2) | 0.0778 (17) | 0.141 (2) | −0.0335 (15) | 0.0503 (18) | −0.0152 (17) |
| C13 | 0.0608 (15) | 0.0964 (18) | 0.1040 (19) | −0.0290 (14) | 0.0232 (13) | −0.0319 (15) |
| C14 | 0.0456 (13) | 0.0740 (13) | 0.0710 (13) | −0.0138 (11) | 0.0159 (11) | −0.0201 (11) |
| C15 | 0.0416 (11) | 0.0620 (11) | 0.0441 (10) | −0.0024 (9) | 0.0057 (8) | −0.0078 (9) |
| C16 | 0.0497 (12) | 0.0677 (12) | 0.0534 (11) | −0.0044 (10) | 0.0100 (9) | −0.0124 (10) |
| C17 | 0.0576 (14) | 0.0973 (16) | 0.0650 (13) | −0.0179 (12) | 0.0051 (11) | −0.0303 (13) |
| C18 | 0.0680 (17) | 0.140 (2) | 0.0504 (13) | −0.0093 (16) | −0.0059 (11) | −0.0146 (15) |
| C19 | 0.0799 (17) | 0.123 (2) | 0.0516 (12) | −0.0099 (16) | −0.0059 (12) | 0.0122 (13) |
| C20 | 0.0629 (14) | 0.0857 (14) | 0.0490 (11) | −0.0103 (11) | 0.0014 (10) | 0.0052 (10) |
| C21 | 0.119 (2) | 0.0863 (17) | 0.140 (2) | 0.0412 (16) | 0.0676 (19) | 0.0127 (16) |
| C22 | 0.0462 (15) | 0.170 (3) | 0.0793 (16) | −0.0167 (16) | −0.0019 (13) | −0.0290 (18) |
| C23 | 0.088 (2) | 0.179 (3) | 0.0807 (18) | 0.021 (2) | −0.0231 (15) | −0.0037 (19) |
| C24 | 0.0777 (16) | 0.0658 (13) | 0.1047 (18) | −0.0212 (12) | 0.0236 (14) | −0.0183 (13) |
| C25 | 0.122 (2) | 0.0769 (17) | 0.147 (3) | −0.0075 (16) | 0.029 (2) | 0.0255 (17) |
| N1 | 0.0390 (9) | 0.0585 (9) | 0.0413 (8) | −0.0041 (7) | 0.0088 (7) | −0.0067 (7) |
| O1 | 0.0861 (11) | 0.1035 (11) | 0.0580 (8) | −0.0004 (9) | 0.0049 (7) | 0.0331 (8) |
| O2 | 0.0478 (9) | 0.0991 (11) | 0.0686 (9) | −0.0109 (8) | −0.0083 (7) | −0.0111 (8) |
| O3 | 0.0709 (10) | 0.0583 (8) | 0.0812 (9) | −0.0193 (7) | 0.0010 (8) | −0.0064 (7) |

*Geometric parameters (Å, °)*

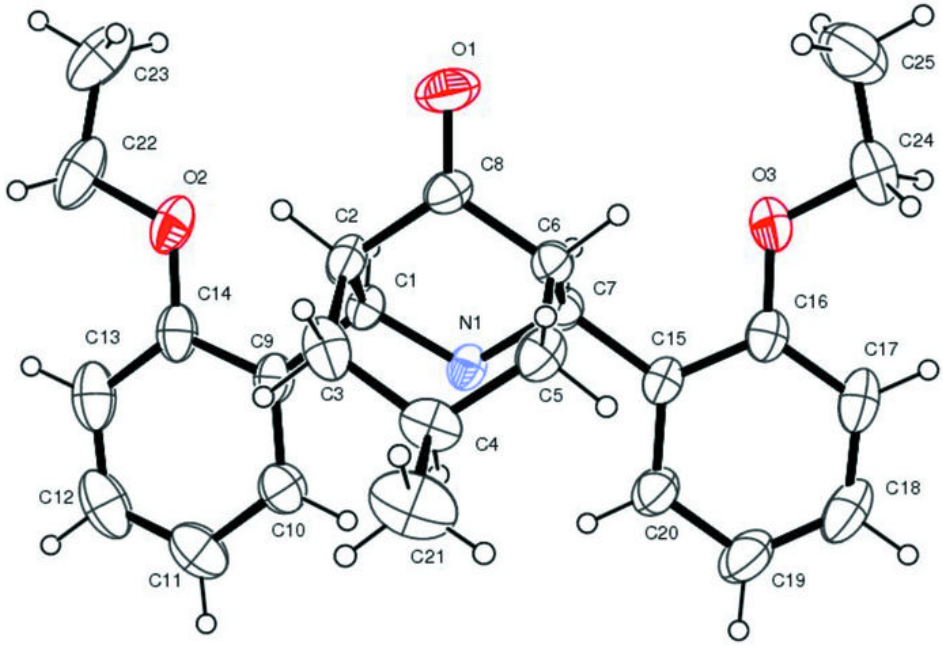
| | | | |
|---|---|---|---|
| C1—N1 | 1.465 (2) | C13—H13 | 0.9300 |
| C1—C9 | 1.511 (2) | C14—O2 | 1.364 (2) |
| C1—C2 | 1.556 (2) | C15—C20 | 1.375 (2) |
| C1—H1 | 0.9800 | C15—C16 | 1.395 (2) |
| C2—C8 | 1.490 (2) | C16—O3 | 1.365 (2) |
| C2—C3 | 1.525 (3) | C16—C17 | 1.389 (3) |
| C2—H2 | 0.9800 | C17—C18 | 1.372 (3) |
| C3—C4 | 1.515 (3) | C17—H17 | 0.9300 |
| C3—H3A | 0.9700 | C18—C19 | 1.360 (3) |
| C3—H3B | 0.9700 | C18—H18 | 0.9300 |
| C4—C5 | 1.511 (3) | C19—C20 | 1.381 (3) |
| C4—C21 | 1.539 (3) | C19—H19 | 0.9300 |
| C4—H4 | 0.9800 | C20—H20 | 0.9300 |

| | | | |
|---|---|---|---|
| C5—C6 | 1.527 (2) | C21—H21A | 0.9600 |
| C5—H5A | 0.9700 | C21—H21B | 0.9600 |
| C5—H5B | 0.9700 | C21—H21C | 0.9600 |
| C6—C8 | 1.495 (2) | C22—O2 | 1.426 (2) |
| C6—C7 | 1.555 (2) | C22—C23 | 1.493 (4) |
| C6—H6 | 0.9800 | C22—H22A | 0.9700 |
| C7—N1 | 1.457 (2) | C22—H22B | 0.9700 |
| C7—C15 | 1.517 (2) | C23—H23A | 0.9600 |
| C7—H7 | 0.9800 | C23—H23B | 0.9600 |
| C8—O1 | 1.2101 (19) | C23—H23C | 0.9600 |
| C9—C10 | 1.381 (3) | C24—O3 | 1.436 (2) |
| C9—C14 | 1.397 (3) | C24—C25 | 1.474 (3) |
| C10—C11 | 1.382 (3) | C24—H24A | 0.9700 |
| C10—H10 | 0.9300 | C24—H24B | 0.9700 |
| C11—C12 | 1.366 (3) | C25—H25A | 0.9600 |
| C11—H11 | 0.9300 | C25—H25B | 0.9600 |
| C12—C13 | 1.377 (3) | C25—H25C | 0.9600 |
| C12—H12 | 0.9300 | N1—H1N | 0.848 (18) |
| C13—C14 | 1.388 (3) | | |
| N1—C1—C9 | 110.10 (14) | C12—C13—H13 | 119.9 |
| N1—C1—C2 | 109.96 (14) | C14—C13—H13 | 119.9 |
| C9—C1—C2 | 111.15 (14) | O2—C14—C13 | 123.9 (2) |
| N1—C1—H1 | 108.5 | O2—C14—C9 | 116.01 (17) |
| C9—C1—H1 | 108.5 | C13—C14—C9 | 120.1 (2) |
| C2—C1—H1 | 108.5 | C20—C15—C16 | 117.59 (16) |
| C8—C2—C3 | 108.30 (15) | C20—C15—C7 | 122.44 (16) |
| C8—C2—C1 | 107.82 (14) | C16—C15—C7 | 119.90 (15) |
| C3—C2—C1 | 115.19 (15) | O3—C16—C17 | 123.62 (18) |
| C8—C2—H2 | 108.5 | O3—C16—C15 | 115.60 (15) |
| C3—C2—H2 | 108.5 | C17—C16—C15 | 120.78 (19) |
| C1—C2—H2 | 108.5 | C18—C17—C16 | 119.5 (2) |
| C4—C3—C2 | 114.42 (15) | C18—C17—H17 | 120.2 |
| C4—C3—H3A | 108.7 | C16—C17—H17 | 120.2 |
| C2—C3—H3A | 108.7 | C19—C18—C17 | 120.7 (2) |
| C4—C3—H3B | 108.7 | C19—C18—H18 | 119.7 |
| C2—C3—H3B | 108.7 | C17—C18—H18 | 119.7 |
| H3A—C3—H3B | 107.6 | C18—C19—C20 | 119.6 (2) |
| C5—C4—C3 | 111.43 (15) | C18—C19—H19 | 120.2 |
| C5—C4—C21 | 110.60 (18) | C20—C19—H19 | 120.2 |
| C3—C4—C21 | 110.98 (18) | C15—C20—C19 | 121.9 (2) |
| C5—C4—H4 | 107.9 | C15—C20—H20 | 119.1 |
| C3—C4—H4 | 107.9 | C19—C20—H20 | 119.1 |
| C21—C4—H4 | 107.9 | C4—C21—H21A | 109.5 |
| C4—C5—C6 | 115.42 (15) | C4—C21—H21B | 109.5 |
| C4—C5—H5A | 108.4 | H21A—C21—H21B | 109.5 |
| C6—C5—H5A | 108.4 | C4—C21—H21C | 109.5 |
| C4—C5—H5B | 108.4 | H21A—C21—H21C | 109.5 |
| C6—C5—H5B | 108.4 | H21B—C21—H21C | 109.5 |
| H5A—C5—H5B | 107.5 | O2—C22—C23 | 107.5 (2) |

| | | | |
|---|---|---|---|
| C8—C6—C5 | 107.99 (14) | O2—C22—H22A | 110.2 |
| C8—C6—C7 | 106.87 (14) | C23—C22—H22A | 110.2 |
| C5—C6—C7 | 115.41 (14) | O2—C22—H22B | 110.2 |
| C8—C6—H6 | 108.8 | C23—C22—H22B | 110.2 |
| C5—C6—H6 | 108.8 | H22A—C22—H22B | 108.5 |
| C7—C6—H6 | 108.8 | C22—C23—H23A | 109.5 |
| N1—C7—C15 | 110.55 (13) | C22—C23—H23B | 109.5 |
| N1—C7—C6 | 110.04 (13) | H23A—C23—H23B | 109.5 |
| C15—C7—C6 | 110.56 (13) | C22—C23—H23C | 109.5 |
| N1—C7—H7 | 108.5 | H23A—C23—H23C | 109.5 |
| C15—C7—H7 | 108.5 | H23B—C23—H23C | 109.5 |
| C6—C7—H7 | 108.5 | O3—C24—C25 | 108.05 (19) |
| O1—C8—C2 | 124.61 (16) | O3—C24—H24A | 110.1 |
| O1—C8—C6 | 123.69 (17) | C25—C24—H24A | 110.1 |
| C2—C8—C6 | 111.70 (14) | O3—C24—H24B | 110.1 |
| C10—C9—C14 | 118.18 (18) | C25—C24—H24B | 110.1 |
| C10—C9—C1 | 122.21 (16) | H24A—C24—H24B | 108.4 |
| C14—C9—C1 | 119.57 (17) | C24—C25—H25A | 109.5 |
| C9—C10—C11 | 121.7 (2) | C24—C25—H25B | 109.5 |
| C9—C10—H10 | 119.1 | H25A—C25—H25B | 109.5 |
| C11—C10—H10 | 119.1 | C24—C25—H25C | 109.5 |
| C12—C11—C10 | 119.4 (2) | H25A—C25—H25C | 109.5 |
| C12—C11—H11 | 120.3 | H25B—C25—H25C | 109.5 |
| C10—C11—H11 | 120.3 | C7—N1—C1 | 115.58 (13) |
| C11—C12—C13 | 120.6 (2) | C7—N1—H1N | 108.7 (12) |
| C11—C12—H12 | 119.7 | C1—N1—H1N | 106.8 (12) |
| C13—C12—H12 | 119.7 | C14—O2—C22 | 119.78 (18) |
| C12—C13—C14 | 120.1 (2) | C16—O3—C24 | 118.36 (16) |
| N1—C1—C2—C8 | 54.66 (18) | C11—C12—C13—C14 | 0.5 (4) |
| C9—C1—C2—C8 | 176.83 (14) | C12—C13—C14—O2 | 179.8 (2) |
| N1—C1—C2—C3 | −66.38 (19) | C12—C13—C14—C9 | −0.4 (3) |
| C9—C1—C2—C3 | 55.80 (19) | C10—C9—C14—O2 | 179.74 (17) |
| C8—C2—C3—C4 | −54.3 (2) | C1—C9—C14—O2 | 2.3 (2) |
| C1—C2—C3—C4 | 66.4 (2) | C10—C9—C14—C13 | −0.1 (3) |
| C2—C3—C4—C5 | 45.8 (2) | C1—C9—C14—C13 | −177.49 (17) |
| C2—C3—C4—C21 | 169.49 (17) | N1—C7—C15—C20 | −17.0 (2) |
| C3—C4—C5—C6 | −45.3 (2) | C6—C7—C15—C20 | 105.11 (19) |
| C21—C4—C5—C6 | −169.20 (17) | N1—C7—C15—C16 | 166.35 (16) |
| C4—C5—C6—C8 | 52.7 (2) | C6—C7—C15—C16 | −71.5 (2) |
| C4—C5—C6—C7 | −66.8 (2) | C20—C15—C16—O3 | 179.34 (17) |
| C8—C6—C7—N1 | −56.66 (17) | C7—C15—C16—O3 | −3.9 (2) |
| C5—C6—C7—N1 | 63.43 (17) | C20—C15—C16—C17 | 0.0 (3) |
| C8—C6—C7—C15 | −179.07 (14) | C7—C15—C16—C17 | 176.80 (16) |
| C5—C6—C7—C15 | −58.98 (18) | O3—C16—C17—C18 | −179.4 (2) |
| C3—C2—C8—O1 | −117.4 (2) | C15—C16—C17—C18 | −0.1 (3) |
| C1—C2—C8—O1 | 117.36 (19) | C16—C17—C18—C19 | 0.4 (4) |
| C3—C2—C8—C6 | 62.90 (18) | C17—C18—C19—C20 | −0.7 (4) |
| C1—C2—C8—C6 | −62.34 (18) | C16—C15—C20—C19 | −0.2 (3) |
| C5—C6—C8—O1 | 118.6 (2) | C7—C15—C20—C19 | −176.98 (18) |

| | | | |
|---|---|---|---|
| C7—C6—C8—O1 | −116.67 (19) | C18—C19—C20—C15 | 0.6 (3) |
| C5—C6—C8—C2 | −61.71 (19) | C15—C7—N1—C1 | 177.62 (14) |
| C7—C6—C8—C2 | 63.03 (18) | C6—C7—N1—C1 | 55.21 (18) |
| N1—C1—C9—C10 | 18.0 (2) | C9—C1—N1—C7 | −176.73 (14) |
| C2—C1—C9—C10 | −104.10 (19) | C2—C1—N1—C7 | −53.93 (19) |
| N1—C1—C9—C14 | −164.69 (16) | C13—C14—O2—C22 | −4.1 (3) |
| C2—C1—C9—C14 | 73.2 (2) | C9—C14—O2—C22 | 176.08 (18) |
| C14—C9—C10—C11 | 0.4 (3) | C23—C22—O2—C14 | −177.42 (19) |
| C1—C9—C10—C11 | 177.78 (18) | C17—C16—O3—C24 | −18.1 (3) |
| C9—C10—C11—C12 | −0.3 (3) | C15—C16—O3—C24 | 162.60 (17) |
| C10—C11—C12—C13 | −0.1 (4) | C25—C24—O3—C16 | −167.01 (19) |

**Fig. 1**


O1
C23
C25
C22
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C8
O3
C2
C6
C1
C7
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N1
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C13
C9
C15
C17
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C4
C12
C18
C10
C20
C21
C11
C19