

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

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

 P. Parthiban,^a V. Ramkumar,^b Dong Ho Park^c and Yeon Tae Jeong^{a*}

^aDepartment of Image Science and Engineering, Pukyong National University, Busan 608 737, Republic of Korea, ^bDepartment of Chemistry, Indian Institute of Technology Madras, Chennai, Tamilnadu, India, and ^cDepartment of Biomedical Chemistry, Inje University, Gimhae, Gyeongnam 621 749, Republic of Korea
Correspondence e-mail: ykjeong@pknu.ac.kr

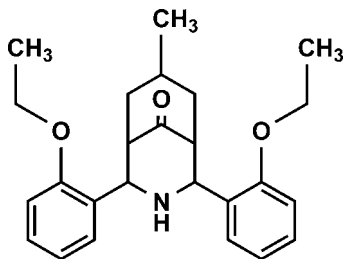
Received 12 May 2011; accepted 16 May 2011

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

The crystal structure of the title compound, $\text{C}_{25}\text{H}_{31}\text{NO}_3$, 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 amplitudes 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.* (2009a, 2010b,c, 2011). For related structures, see: Parthiban *et al.* (2009b,c, 2010a,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

$\text{C}_{25}\text{H}_{31}\text{NO}_3$	$V = 2260.4$ (2) Å ³
$M_r = 393.51$	$Z = 4$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 10.3147$ (6) Å	$\mu = 0.08$ mm ⁻¹
$b = 11.8817$ (6) Å	$T = 298$ K
$c = 18.7809$ (10) Å	$0.35 \times 0.28 \times 0.15$ mm
$\beta = 100.866$ (2)°	

Data collection

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

Refinement

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

This research was supported by the Corporate-affiliated Research Institute for Academic-Industrial-Institutional Cooperation Improvement (Business No. S7080008110). The authors acknowledge the Department of Chemistry, IIT Madras, for the X-ray data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LW2064).

References

- Barker, D., Lin, D. H. S., Carland, J. E., Chu, C. P. Y., Chebib, M., Brimble, M. A., Savage, G. P. & McLeod, M. D. (2005). *Bioorg. Med. Chem.* **13**, 4565–4575.
- Bruker (2004). APEX2, XPREP, SAINT-Plus and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.
- Cox, P. J., McCabe, P. H., Milne, N. J. & Sim, G. A. (1985). *J. Chem. Soc. Chem. Commun.* pp. 626–628.
- Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Jeyaraman, R. & Avila, S. (1981). *Chem. Rev.* **81**, 149–174.
- Nardelli, M. (1983). *Acta Cryst.* **C39**, 1141–1142.
- Padegimas, S. J. & Kovacic, P. (1972). *J. Org. Chem.* **37**, 2672–2676.
- Parthiban, P., Aridoss, G., Rathika, P., Ramkumar, V. & Kabilan, S. (2009a). *Bioorg. Med. Chem. Lett.* **19**, 6981–6985.
- Parthiban, P., Ramkumar, V., Amirthaganesan, S. & Jeong, Y. T. (2009b). *Acta Cryst.* **E65**, o1356.
- Parthiban, P., Ramkumar, V. & Jeong, Y. T. (2010a). *Acta Cryst.* **E66**, o48–o49.
- Parthiban, P., Ramkumar, V., Kim, M. S., Son, S. M. & Jeong, Y. T. (2009c). *Acta Cryst.* **E65**, o1383.
- Parthiban, P., Rathika, P., Park, K. S. & Jeong, Y. T. (2010b). *Monatsh. Chem.* **141**, 79–93.
- Parthiban, P., Rathika, P., Ramkumar, V., Son, S. M. & Jeong, Y. T. (2010c). *Bioorg. Med. Chem. Lett.* **20**, 1642–1647.

Parthiban, P., Subalakshmi, V., Balasubramanian, K., Islam, Md. N., Choi, J. S. & Jeong, Y. T. (2011). *Bioorg. Med. Chem. Lett.* **21**, 2287–2296.
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

Smith-Verdier, P., Florencio, F. & García-Blanco, S. (1983). *Acta Cryst.* **C39**, 101–103.

supporting information

Acta Cryst. (2011). E67, 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, Dong Ho Park and Yeon Tae Jeong****S1. Comment**

The 3-azabicyclo 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.*, 2009a, 2010b, 2010c, 2011). Its biological significance 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 stereoisomers are possible for the synthesized title compound along with different conformations such as chair-chair (Parthiban *et al.*, 2009b, 2009c, 2010a; Cox *et al.*, 1985), chair-boat (Parthiban *et al.*, 2010c; 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 $\theta = 12.2$ (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.

S2. 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.

S3. 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 = 0.98 Å and methylene C—H = 0.97 Å. The displacement parameters were set for phenyl, methylene and aliphatic H atoms at $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ and for methyl H atoms at $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$.

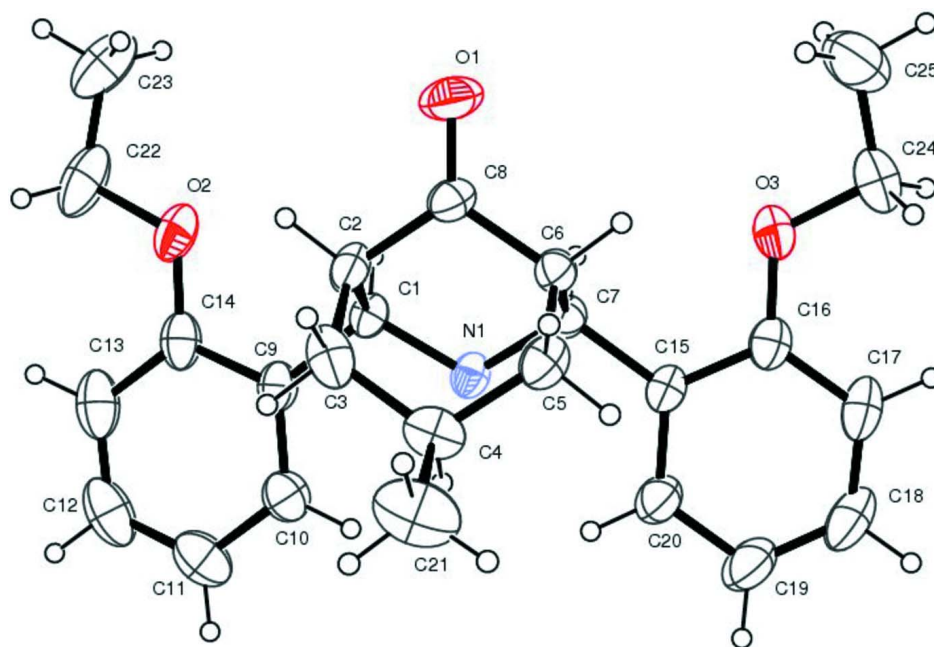


Figure 1

Anisotropic 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

$\text{C}_{25}\text{H}_{31}\text{NO}_3$

$M_r = 393.51$

Monoclinic, $P2_1/n$

Hall symbol: $-P\ 2_1n$

$a = 10.3147\ (6)\ \text{\AA}$

$b = 11.8817\ (6)\ \text{\AA}$

$c = 18.7809\ (10)\ \text{\AA}$

$\beta = 100.866\ (2)^\circ$

$V = 2260.4\ (2)\ \text{\AA}^3$

$Z = 4$

$F(000) = 848$

$D_x = 1.156\ \text{Mg m}^{-3}$

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

Cell parameters from 3407 reflections

$\theta = 2.6\text{--}21.8^\circ$

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

$T = 298\ \text{K}$

Block, colourless

$0.35 \times 0.28 \times 0.15\ \text{mm}$

Data collection

Bruker APEXII CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan
(*SADABS*; Bruker, 2004)

$T_{\text{min}} = 0.974$, $T_{\text{max}} = 0.989$

12446 measured reflections

3876 independent reflections

2415 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.025$

$\theta_{\text{max}} = 25.6^\circ$, $\theta_{\text{min}} = 2.0^\circ$

$h = -10 \rightarrow 11$

$k = -14 \rightarrow 14$

$l = -22 \rightarrow 16$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.117$
 $S = 1.03$
 3876 reflections
 269 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
 $w = 1/[\sigma^2(F_o^2) + (0.047P)^2 + 0.3732P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.12 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.13 \text{ e } \text{\AA}^{-3}$

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
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 (Å²)

	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)
