

2-[(4-*tert*-Butylanilino)(phenyl)methyl]-cyclohexanone

Hoong-Kun Fun,^{a*} Suchada Chantrapromma,^{b‡} Sankappa Rai,^c Prakash Shetty^d and Arun M. Isloor^e

^aX-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, ^bCrystal Materials Research Unit, Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand, ^cSyngene International Ltd, Biocon Park, plot No. 2 & 3, Bommasandra 4th Phase, Jigani Link Road, Bangalore 560 100, India, ^dDepartment of Printing, Manipal Institute of Technology, Manipal 576 104, India, and ^eDepartment of Chemistry, National Institute of Technology - Karnataka, Surathkal, Mangalore 575 025, India
Correspondence e-mail: hkfun@usm.my

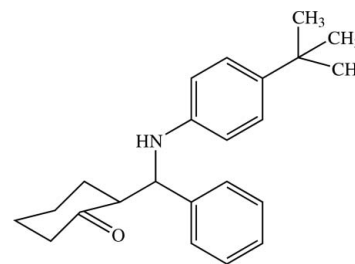
Received 9 February 2009; accepted 11 February 2009

Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.047; wR factor = 0.124; data-to-parameter ratio = 19.1.

In the molecule of the title compound, $\text{C}_{23}\text{H}_{29}\text{NO}$, the cyclohexanone ring has been distorted from the standard chair conformation by the ketone group such that part of the ring is almost flat. The remaining [(4-*tert*-butylanilino)(phenyl)methyl] portion of the molecule is in an equatorial position on the cyclohexanone ring. The dihedral angle between the two benzene rings is $81.52(8)^\circ$. In the crystal packing, molecules are linked by $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds into infinite one-dimensional chains along the a axis and these chains are stacked down the c axis. The crystal structure is further stabilized by weak $\text{C}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\pi$ interactions.

Related literature

For values of bond lengths, see: Allen *et al.* (1987). For details of hydrogen-bond motifs, see: Bernstein *et al.* (1995). For information on the Mannich reaction, see: Kobayashi & Ishitani (1999); Bohme & Haake (1976). For background to the bioactivity and applications of beta-amino carbonyl compounds, see, for example: Arend *et al.* (1988); Isloor, Sunil *et al.* (2009); Isloor, Kalluraya *et al.* (2009); Jadhav *et al.* (2008); Kalluraya *et al.* (2001). For puckering parameters, see: Cremer & Pople, (1975). For the stability of the temperature controller, see Cosier & Glazer (1986).



Experimental

Crystal data

$\text{C}_{23}\text{H}_{29}\text{NO}$
 $M_r = 335.47$
 Triclinic, $P\bar{1}$
 $a = 6.5315(2)$ Å
 $b = 12.3946(3)$ Å
 $c = 12.8853(3)$ Å
 $\alpha = 62.973(1)^\circ$
 $\beta = 86.347(2)^\circ$
 $\gamma = 85.103(2)^\circ$
 $V = 925.46(4)$ Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.07$ mm⁻¹
 $T = 100$ K
 $0.52 \times 0.41 \times 0.11$ mm

Data collection

Bruker SMART APEXII CCD
 area-detector diffractometer
 Absorption correction: multi-scan
 (SADABS; Bruker, 2005)
 $T_{\min} = 0.953$, $T_{\max} = 0.992$
 17722 measured reflections
 4449 independent reflections
 3584 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.027$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.047$
 $wR(F^2) = 0.124$
 $S = 1.07$
 4449 reflections
 233 parameters
 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.39$ e Å⁻³
 $\Delta\rho_{\min} = -0.23$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1N1}\cdots\text{O1}^{\text{i}}$	0.89 (2)	2.35 (2)	3.2050 (16)	161.6 (19)
$\text{C9}-\text{H9A}\cdots\text{O1}$	0.93	2.59	3.1146 (19)	116
$\text{C2}-\text{H2A}\cdots\text{Cg1}^{\text{ii}}$	0.97	2.60	3.4992 (19)	155
$\text{C23}-\text{H23C}\cdots\text{Cg1}^{\text{iii}}$	0.96	2.99	3.747 (2)	137

Symmetry codes: (i) $x - 1, y, z$; (ii) $-x + 2, -y + 1, -z + 1$; (iii) $-x + 1, -y + 1, -z + 2$. Cg1 is the centroid of the C14-C19 ring.

Data collection: APEX2 (Bruker, 2005); cell refinement: APEX2; data reduction: SAINT (Bruker, 2005); program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL and PLATON (Spek, 2009).

AMI is grateful to the Director, NITK, Surathkal, India, for providing research facilities. SR thanks Dr Gautam Das, Syngene International Ltd, Bangalore, India, for allocation of research resources. The authors also thank the Universiti Sains Malaysia for the Research University Golden Goose grant No. 1001/PFIZIK/811012.

[‡] Additional correspondence author, e-mail: suchada.c@psu.ac.th.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2573).

References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
- Arend, M., Westermanb, B. & Risch, N. (1988). *Angew. Chem.* **37**, 1044–1070.
- Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
- Bohme, H. & Haake, M. (1976). *Advances in Organic Chemistry*, edited by E. C. Taylor, p. 107. New York: John Wiley and Sons.
- Bruker (2005). *APEX2, SAINT and SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.
- Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
- Isloor, A. M., Kalluraya, B. & Shetty, P. (2009). *Eur. J. Med. Chem.* Submitted.
- Isloor, A. M., Sunil, D., Shetty, P. & Satyamoorthy, K. (2009). *Eur. J. Med. Chem.* Submitted.
- Jadhav, V. J., Kulkarni, M. V., Rasal, V. P., Biradar, S. S. & Vinay, M. D. (2008). *Eur. J. Med. Chem.* **43**, 1721–1729.
- Kalluraya, B., Isloor, A. M., Chimbalkar, R. & Shenoy, S. (2001). *Indian J. Heterocycl. Chem.* pp. 239–240.
- Kobayashi, S. & Ishitani, H. (1999). *Chem. Rev.* **99**, 1069–1094.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.

supporting information

Acta Cryst. (2009). E65, o539–o540 [doi:10.1107/S1600536809004954]

2-[(4-*tert*-Butylanilino)(phenyl)methyl]cyclohexanone

Hoong-Kun Fun, Suchada Chantrapromma, Sankappa Rai, Prakash Shetty and Arun M. Isloor

S1. Comment

Mannich reactions are among the most important carbon-carbon bond forming reactions in organic synthesis (Kobayashi & Ishitani, 1999). They provide beta-amino carbonyl compounds, which are important synthetic intermediates for various pharmaceuticals and natural products (Arend *et al.*, 1988). They exhibit wide variety of pharmaceutical properties such as anti cancer (Isloor, Sunil *et al.*, 2009), analgesic (Isloor, Kalluraya *et al.*, 2009), anti-inflammatory (Jadhav *et al.*, 2008), antimicrobial (Kalluraya *et al.*, 2001) activities. The increasing popularity of the Mannich reaction has been fueled by the ubiquitous nature of nitrogen containing compounds in drugs and natural products (Bohme & Haake, 1976). Prompted by the biological activity of these derivatives, we have synthesized the title compound (I) and report its structure here, Fig 1. The cyclohexanone ring has been distorted from the standard chair conformation by the ketone group such that the C2 C1 O1 C6 part of the ring is almost flat with puckering parameter $Q = 0.5181(16)\text{\AA}$, and $\theta = 159.23(18)^\circ$ and $\varphi = 9.3(5)^\circ$ (Cremer & Pople, 1975). The [4-(*tert*-butyl)anilino](phenyl)methyl substituent group is equatorially attached to the ring at atom C6 with torsion angles $C5-C6-C7-C8 = -57.80(16)^\circ$ and $C5-C6-C7-N1 = 69.13(15)^\circ$. The two benzene rings are nearly perpendicular to each other with a dihedral angle of $81.52(8)^\circ$ between them. The bond distances have normal values (Allen *et al.*, 1987).

A weak intramolecular $C9-H9A\cdots O1$ interaction generates an $S(7)$ ring motif (Bernstein *et al.*, 1995) (Table 1) and effects the solid state conformation of the molecule. In the crystal structure $N-H\cdots O$ hydrogen bonds (Table 1, Fig. 2) link the molecules into infinite one-dimensional chains along the a axis and these chains are stacked down the c axis. The crystal is further stabilized by weak $C-H\cdots O$ and $C-H\cdots\pi$ interactions (Table 1); $Cg1$ is the centroid of the C14–C19 ring (Table 1).

S2. Experimental

The title compound was obtained by vigorously stirring a solution of cyclohexanone (0.5 g, 5.0 mmol), benzaldehyde (0.53 g, 5.0 mmol) and 4-*tert*-butylaniline (0.75 g, 5.0 mmol) in dry acetonitrile (5 ml). Trifluoro acetic acid (0.57 g, 5 mmol) was then added. The reaction mixture was stirred at room temperature for 2 h. After standing for 1 hr, the solvent was removed and the crude product was purified by column chromatography using ethyl acetate and petroleum ether (1:1 v:v) as eluants. The product was further recrystallized using 10 ml of hot ethanol. The yield was 1 g (58%), M_p 439–441 K.

S3. Refinement

The amine H atom was located in a difference map and refined isotropically. The remaining H atoms were placed in calculated positions with $d(C-H) = 0.93\text{\AA}$, $U_{iso} = 1.2U_{eq}(C)$ for aromatic, 0.98\AA , $U_{iso} = 1.2U_{eq}(C)$ for CH, 0.97\AA , $U_{iso} = 1.2U_{eq}(C)$ for CH_2 and 0.96\AA , $U_{iso} = 1.5U_{eq}(C)$ for CH_3 atoms. A rotating group model was used for the methyl groups. The highest residual electron density peak is located at 0.72\AA from C8 and the deepest hole is located at 1.03\AA

from C16.

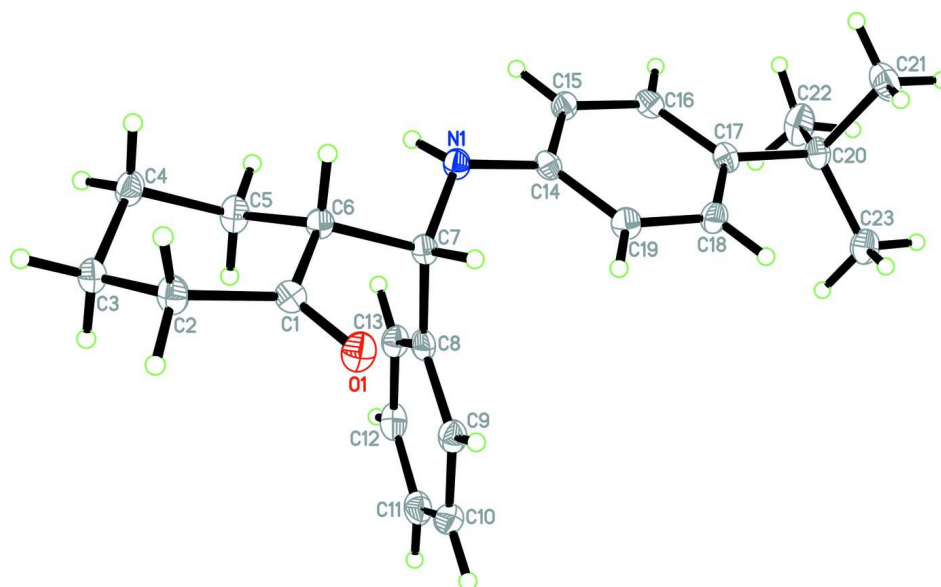


Figure 1

The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

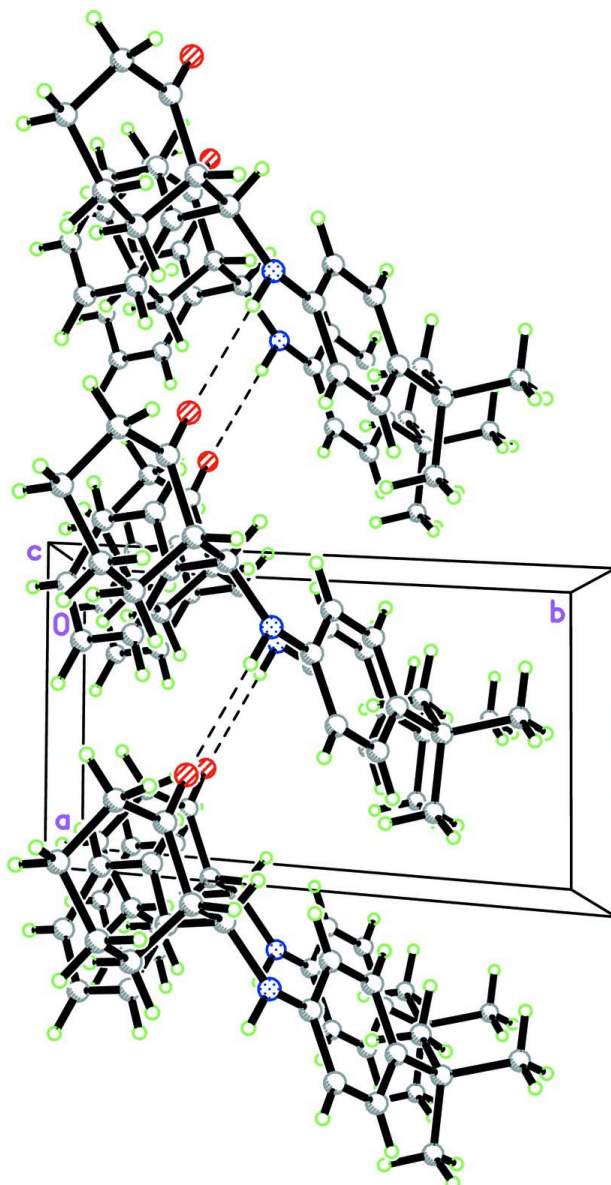


Figure 2

The packing diagram of (I), viewed along the *c* axis, showing molecular chains along the *a* axis. Hydrogen bonds are shown as dashed lines.

2-[(4-*tert*-Butylanilino)(phenyl)methyl]cyclohexanone

Crystal data

$C_{23}H_{29}NO$

$M_r = 335.47$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 6.5315 (2) \text{ \AA}$

$b = 12.3946 (3) \text{ \AA}$

$c = 12.8853 (3) \text{ \AA}$

$\alpha = 62.973 (1)^\circ$

$\beta = 86.347 (2)^\circ$

$\gamma = 85.103 (2)^\circ$

$V = 925.46 (4) \text{ \AA}^3$

$Z = 2$

$F(000) = 364$

$D_x = 1.204 \text{ Mg m}^{-3}$

Melting point = 439–441 K

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

Cell parameters from 4449 reflections

$\theta = 1.8\text{--}28.0^\circ$

$\mu = 0.07 \text{ mm}^{-1}$
 $T = 100 \text{ K}$

Plate, colorless
 $0.52 \times 0.41 \times 0.11 \text{ mm}$

Data collection

Bruker SMART APEXII CCD area-detector
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 Detector resolution: 8.33 pixels mm^{-1}
 ω scans
 Absorption correction: multi-scan
 (SADABS; Bruker, 2005)
 $T_{\min} = 0.953$, $T_{\max} = 0.992$

17722 measured reflections
 4449 independent reflections
 3584 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.027$
 $\theta_{\max} = 28.0^\circ$, $\theta_{\min} = 1.8^\circ$
 $h = -8 \rightarrow 8$
 $k = -16 \rightarrow 16$
 $l = -16 \rightarrow 16$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.047$
 $wR(F^2) = 0.124$
 $S = 1.07$
 4449 reflections
 233 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
 $w = 1/[\sigma^2(F_o^2) + (0.0465P)^2 + 0.607P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.39 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.23 \text{ e } \text{\AA}^{-3}$

Special details

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

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

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

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	1.32798 (15)	0.71893 (10)	0.43723 (9)	0.0200 (2)
N1	0.75282 (19)	0.58289 (11)	0.55557 (10)	0.0157 (3)
C1	1.1976 (2)	0.75037 (12)	0.36390 (12)	0.0147 (3)
C2	1.2523 (2)	0.82454 (13)	0.23594 (12)	0.0183 (3)
H2A	1.2974	0.7691	0.2037	0.022*
H2B	1.3680	0.8714	0.2299	0.022*
C3	1.0809 (2)	0.91152 (13)	0.16072 (12)	0.0176 (3)
H3A	1.0622	0.9816	0.1759	0.021*
H3B	1.1192	0.9396	0.0790	0.021*
C4	0.8804 (2)	0.84862 (14)	0.18722 (12)	0.0189 (3)
H4A	0.8968	0.7809	0.1684	0.023*

H4B	0.7727	0.9051	0.1396	0.023*
C5	0.8193 (2)	0.80271 (14)	0.31607 (12)	0.0194 (3)
H5A	0.6875	0.7670	0.3308	0.023*
H5B	0.8042	0.8707	0.3346	0.023*
C6	0.9799 (2)	0.70785 (12)	0.39536 (12)	0.0146 (3)
H6A	0.9781	0.6384	0.3785	0.018*
C7	0.9298 (2)	0.65755 (12)	0.52741 (12)	0.0144 (3)
H7A	1.0474	0.6032	0.5682	0.017*
C8	0.9016 (2)	0.75555 (12)	0.56920 (12)	0.0153 (3)
C9	1.0660 (2)	0.78409 (13)	0.61390 (12)	0.0183 (3)
H9A	1.1930	0.7419	0.6200	0.022*
C10	1.0424 (3)	0.87519 (14)	0.64956 (13)	0.0220 (3)
H10A	1.1535	0.8941	0.6785	0.026*
C11	0.8531 (3)	0.93747 (14)	0.64179 (13)	0.0233 (3)
H11A	0.8377	0.9994	0.6641	0.028*
C12	0.6871 (2)	0.90756 (14)	0.60086 (13)	0.0216 (3)
H12A	0.5592	0.9480	0.5977	0.026*
C13	0.7104 (2)	0.81741 (13)	0.56449 (12)	0.0184 (3)
H13A	0.5980	0.7981	0.5368	0.022*
C14	0.6904 (2)	0.51586 (12)	0.67217 (12)	0.0147 (3)
C15	0.4914 (2)	0.47478 (13)	0.69928 (12)	0.0159 (3)
H15A	0.4004	0.4953	0.6396	0.019*
C16	0.4279 (2)	0.40402 (12)	0.81375 (12)	0.0165 (3)
H16A	0.2945	0.3788	0.8288	0.020*
C17	0.5577 (2)	0.36937 (12)	0.90732 (12)	0.0159 (3)
C18	0.7558 (2)	0.41071 (13)	0.87917 (12)	0.0173 (3)
H18A	0.8472	0.3892	0.9389	0.021*
C19	0.8215 (2)	0.48285 (13)	0.76503 (12)	0.0172 (3)
H19A	0.9541	0.5094	0.7503	0.021*
C20	0.4898 (2)	0.28876 (13)	1.03410 (12)	0.0178 (3)
C21	0.5531 (3)	0.15560 (14)	1.06508 (14)	0.0280 (4)
H21A	0.4873	0.1327	1.0142	0.042*
H21B	0.5121	0.1048	1.1444	0.042*
H21C	0.6996	0.1461	1.0560	0.042*
C22	0.2558 (2)	0.30252 (16)	1.05089 (14)	0.0277 (4)
H22A	0.1889	0.2703	1.0078	0.042*
H22B	0.2133	0.3869	1.0233	0.042*
H22C	0.2191	0.2590	1.1321	0.042*
C23	0.5907 (3)	0.32236 (15)	1.11896 (13)	0.0235 (3)
H23A	0.7367	0.3055	1.1169	0.035*
H23B	0.5370	0.2754	1.1965	0.035*
H23C	0.5616	0.4072	1.0966	0.035*
H1N1	0.646 (3)	0.6162 (18)	0.5096 (17)	0.030 (5)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0126 (5)	0.0257 (6)	0.0188 (5)	-0.0004 (4)	-0.0006 (4)	-0.0076 (4)

N1	0.0128 (6)	0.0171 (6)	0.0142 (6)	-0.0028 (5)	-0.0003 (5)	-0.0043 (5)
C1	0.0119 (6)	0.0148 (6)	0.0168 (7)	0.0009 (5)	0.0008 (5)	-0.0072 (5)
C2	0.0135 (7)	0.0216 (7)	0.0163 (7)	-0.0015 (5)	0.0028 (5)	-0.0059 (6)
C3	0.0182 (7)	0.0177 (7)	0.0140 (7)	-0.0012 (5)	0.0004 (5)	-0.0048 (6)
C4	0.0169 (7)	0.0221 (7)	0.0150 (7)	-0.0011 (6)	-0.0028 (5)	-0.0058 (6)
C5	0.0121 (7)	0.0243 (8)	0.0161 (7)	-0.0004 (5)	0.0000 (5)	-0.0044 (6)
C6	0.0121 (6)	0.0158 (6)	0.0147 (7)	-0.0012 (5)	-0.0003 (5)	-0.0057 (5)
C7	0.0110 (6)	0.0150 (6)	0.0140 (6)	-0.0008 (5)	-0.0001 (5)	-0.0039 (5)
C8	0.0159 (7)	0.0151 (6)	0.0117 (6)	-0.0024 (5)	0.0017 (5)	-0.0032 (5)
C9	0.0155 (7)	0.0205 (7)	0.0153 (7)	-0.0028 (5)	0.0007 (5)	-0.0048 (6)
C10	0.0262 (8)	0.0234 (8)	0.0158 (7)	-0.0079 (6)	-0.0007 (6)	-0.0074 (6)
C11	0.0362 (9)	0.0175 (7)	0.0152 (7)	-0.0017 (6)	0.0016 (6)	-0.0067 (6)
C12	0.0236 (8)	0.0206 (7)	0.0161 (7)	0.0040 (6)	0.0006 (6)	-0.0054 (6)
C13	0.0162 (7)	0.0207 (7)	0.0151 (7)	-0.0008 (5)	0.0001 (5)	-0.0056 (6)
C14	0.0142 (7)	0.0125 (6)	0.0156 (7)	0.0002 (5)	0.0012 (5)	-0.0051 (5)
C15	0.0137 (7)	0.0160 (7)	0.0163 (7)	-0.0005 (5)	-0.0025 (5)	-0.0058 (5)
C16	0.0126 (6)	0.0156 (7)	0.0199 (7)	-0.0018 (5)	0.0014 (5)	-0.0068 (6)
C17	0.0160 (7)	0.0138 (6)	0.0157 (7)	0.0006 (5)	0.0021 (5)	-0.0054 (5)
C18	0.0148 (7)	0.0189 (7)	0.0162 (7)	0.0010 (5)	-0.0027 (5)	-0.0063 (6)
C19	0.0116 (6)	0.0193 (7)	0.0187 (7)	-0.0016 (5)	0.0006 (5)	-0.0070 (6)
C20	0.0168 (7)	0.0176 (7)	0.0150 (7)	-0.0005 (5)	0.0018 (5)	-0.0042 (6)
C21	0.0387 (10)	0.0179 (7)	0.0216 (8)	-0.0027 (7)	0.0058 (7)	-0.0046 (6)
C22	0.0184 (8)	0.0376 (9)	0.0187 (8)	-0.0029 (7)	0.0047 (6)	-0.0059 (7)
C23	0.0262 (8)	0.0249 (8)	0.0168 (7)	-0.0020 (6)	0.0009 (6)	-0.0072 (6)

Geometric parameters (Å, °)

O1—C1	1.2172 (17)	C11—C12	1.382 (2)
N1—C14	1.3996 (18)	C11—H11A	0.9300
N1—C7	1.4657 (17)	C12—C13	1.389 (2)
N1—H1N1	0.89 (2)	C12—H12A	0.9300
C1—C2	1.5149 (19)	C13—H13A	0.9300
C1—C6	1.5244 (19)	C14—C19	1.399 (2)
C2—C3	1.529 (2)	C14—C15	1.4019 (19)
C2—H2A	0.9700	C15—C16	1.388 (2)
C2—H2B	0.9700	C15—H15A	0.9300
C3—C4	1.521 (2)	C16—C17	1.398 (2)
C3—H3A	0.9700	C16—H16A	0.9300
C3—H3B	0.9700	C17—C18	1.397 (2)
C4—C5	1.5281 (19)	C17—C20	1.5376 (19)
C4—H4A	0.9700	C18—C19	1.392 (2)
C4—H4B	0.9700	C18—H18A	0.9300
C5—C6	1.537 (2)	C19—H19A	0.9300
C5—H5A	0.9700	C20—C23	1.535 (2)
C5—H5B	0.9700	C20—C22	1.535 (2)
C6—C7	1.5462 (18)	C20—C21	1.536 (2)
C6—H6A	0.9800	C21—H21A	0.9600
C7—C8	1.5307 (19)	C21—H21B	0.9600

C7—H7A	0.9800	C21—H21C	0.9600
C8—C9	1.392 (2)	C22—H22A	0.9600
C8—C13	1.398 (2)	C22—H22B	0.9600
C9—C10	1.394 (2)	C22—H22C	0.9600
C9—H9A	0.9300	C23—H23A	0.9600
C10—C11	1.384 (2)	C23—H23B	0.9600
C10—H10A	0.9300	C23—H23C	0.9600
C14—N1—C7	119.50 (12)	C12—C11—C10	119.94 (14)
C14—N1—H1N1	111.8 (13)	C12—C11—H11A	120.0
C7—N1—H1N1	115.8 (13)	C10—C11—H11A	120.0
O1—C1—C2	120.57 (12)	C11—C12—C13	120.31 (14)
O1—C1—C6	121.72 (12)	C11—C12—H12A	119.8
C2—C1—C6	117.49 (12)	C13—C12—H12A	119.8
C1—C2—C3	116.04 (12)	C12—C13—C8	120.49 (14)
C1—C2—H2A	108.3	C12—C13—H13A	119.8
C3—C2—H2A	108.3	C8—C13—H13A	119.8
C1—C2—H2B	108.3	C19—C14—N1	122.84 (13)
C3—C2—H2B	108.3	C19—C14—C15	117.33 (12)
H2A—C2—H2B	107.4	N1—C14—C15	119.78 (13)
C4—C3—C2	110.53 (12)	C16—C15—C14	121.08 (13)
C4—C3—H3A	109.5	C16—C15—H15A	119.5
C2—C3—H3A	109.5	C14—C15—H15A	119.5
C4—C3—H3B	109.5	C15—C16—C17	122.24 (13)
C2—C3—H3B	109.5	C15—C16—H16A	118.9
H3A—C3—H3B	108.1	C17—C16—H16A	118.9
C3—C4—C5	110.11 (12)	C18—C17—C16	116.14 (13)
C3—C4—H4A	109.6	C18—C17—C20	121.38 (13)
C5—C4—H4A	109.6	C16—C17—C20	122.47 (13)
C3—C4—H4B	109.6	C19—C18—C17	122.45 (13)
C5—C4—H4B	109.6	C19—C18—H18A	118.8
H4A—C4—H4B	108.2	C17—C18—H18A	118.8
C4—C5—C6	111.74 (12)	C18—C19—C14	120.75 (13)
C4—C5—H5A	109.3	C18—C19—H19A	119.6
C6—C5—H5A	109.3	C14—C19—H19A	119.6
C4—C5—H5B	109.3	C23—C20—C22	107.70 (13)
C6—C5—H5B	109.3	C23—C20—C21	108.66 (13)
H5A—C5—H5B	107.9	C22—C20—C21	109.07 (13)
C1—C6—C5	112.30 (11)	C23—C20—C17	111.26 (12)
C1—C6—C7	112.15 (11)	C22—C20—C17	111.07 (12)
C5—C6—C7	114.88 (11)	C21—C20—C17	109.02 (12)
C1—C6—H6A	105.5	C20—C21—H21A	109.5
C5—C6—H6A	105.5	C20—C21—H21B	109.5
C7—C6—H6A	105.5	H21A—C21—H21B	109.5
N1—C7—C8	113.40 (11)	C20—C21—H21C	109.5
N1—C7—C6	108.22 (11)	H21A—C21—H21C	109.5
C8—C7—C6	113.68 (11)	H21B—C21—H21C	109.5
N1—C7—H7A	107.1	C20—C22—H22A	109.5

C8—C7—H7A	107.1	C20—C22—H22B	109.5
C6—C7—H7A	107.1	H22A—C22—H22B	109.5
C9—C8—C13	118.57 (13)	C20—C22—H22C	109.5
C9—C8—C7	120.52 (13)	H22A—C22—H22C	109.5
C13—C8—C7	120.91 (13)	H22B—C22—H22C	109.5
C8—C9—C10	120.75 (14)	C20—C23—H23A	109.5
C8—C9—H9A	119.6	C20—C23—H23B	109.5
C10—C9—H9A	119.6	H23A—C23—H23B	109.5
C11—C10—C9	119.90 (14)	C20—C23—H23C	109.5
C11—C10—H10A	120.1	H23A—C23—H23C	109.5
C9—C10—H10A	120.1	H23B—C23—H23C	109.5
O1—C1—C2—C3	-149.78 (14)	C9—C10—C11—C12	1.3 (2)
C6—C1—C2—C3	35.51 (18)	C10—C11—C12—C13	-1.7 (2)
C1—C2—C3—C4	-45.58 (17)	C11—C12—C13—C8	0.2 (2)
C2—C3—C4—C5	58.62 (16)	C9—C8—C13—C12	1.7 (2)
C3—C4—C5—C6	-62.20 (16)	C7—C8—C13—C12	-178.96 (13)
O1—C1—C6—C5	148.61 (14)	C7—N1—C14—C19	-22.0 (2)
C2—C1—C6—C5	-36.74 (17)	C7—N1—C14—C15	160.68 (12)
O1—C1—C6—C7	17.48 (18)	C19—C14—C15—C16	-0.1 (2)
C2—C1—C6—C7	-167.88 (12)	N1—C14—C15—C16	177.38 (13)
C4—C5—C6—C1	49.83 (16)	C14—C15—C16—C17	-0.5 (2)
C4—C5—C6—C7	179.57 (12)	C15—C16—C17—C18	0.3 (2)
C14—N1—C7—C8	-59.87 (16)	C15—C16—C17—C20	-178.69 (13)
C14—N1—C7—C6	173.04 (12)	C16—C17—C18—C19	0.4 (2)
C1—C6—C7—N1	-161.06 (11)	C20—C17—C18—C19	179.42 (13)
C5—C6—C7—N1	69.13 (15)	C17—C18—C19—C14	-1.0 (2)
C1—C6—C7—C8	72.02 (15)	N1—C14—C19—C18	-176.59 (13)
C5—C6—C7—C8	-57.80 (16)	C15—C14—C19—C18	0.8 (2)
N1—C7—C8—C9	141.87 (13)	C18—C17—C20—C23	34.25 (18)
C6—C7—C8—C9	-93.95 (15)	C16—C17—C20—C23	-146.77 (14)
N1—C7—C8—C13	-37.48 (17)	C18—C17—C20—C22	154.21 (14)
C6—C7—C8—C13	86.69 (15)	C16—C17—C20—C22	-26.80 (19)
C13—C8—C9—C10	-2.1 (2)	C18—C17—C20—C21	-85.57 (17)
C7—C8—C9—C10	178.51 (12)	C16—C17—C20—C21	93.41 (17)
C8—C9—C10—C11	0.7 (2)		

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1M1 \cdots O1 ⁱ	0.89 (2)	2.35 (2)	3.2050 (16)	161.6 (19)
C9—H9A \cdots O1	0.93	2.59	3.1146 (19)	116
C2—H2A \cdots Cg1 ⁱⁱ	0.97	2.60	3.4992 (19)	155
C23—H23C \cdots Cg1 ⁱⁱⁱ	0.96	2.99	3.747 (2)	137

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