Table 4. *Torsion angles* (°) *with e.s.d.'s in parentheses*

Signs are given according to the convention of Klyne & Prelog (1960).

N(1)–C(1)–C(2)–N(2)	6·4 (12)	N(21)–N(2)–C(2)–C(1)	−135·2 (8)
N(11)–N(1)–C(1)–C(2)	49·8 (11)	C(21)–N(21)–N(2)–C(2)	178·2 (7)
C(11)–N(11)–N(1)–C(1)	−175·0 (7)	C(22)–C(21)–N(21)–N(2)	67·3 (11)
C(12)–C(11)–N(11)–N(1)	−54·5 (12)	N(22)–C(22)–C(21)–N(21)	3·9 (15)
N(12)–C(12)–C(11)–N(11)	−0·7 (14)	O(21)–N(22)–C(22)–C(21)	3·8 (18)
O(11)–N(12)–C(12)–C(11)	−34·1 (13)		

N(1) deviate significantly, probably for the relief of intramolecular short contacts. The dihedral angles of the paired planes (I)–(II), (I)–(III) and (II)–(III) are 1·8, 68·1 and 66·4°, respectively. Some torsion angles in the molecule are listed in Table 4.

The conformations around the two azoxy groups are both *trans*. The nitro group bound to ring (II) is considerably twisted out of the six-membered-ring plane, while that bound to ring (III) is not. Rotations about the C(1)–N(1) and C(2)–N(2) bonds are fixed so as to minimize the unusual contacts between the two azoxy groups and also to stagger the nitro groups over the azoxy group moieties.

As shown in Fig. 2, the crystal structure consists of columns of parallel-stacked six-membered rings. The columns are parallel to the crystallographic *a* axis and the stacking distance is 3·8 Å (*a*/2), which is larger than that reported for the overlap interactions of an aromatic ring system. As a whole, the molecules are well packed and are nearly parallel to the (10$\bar{1}$) plane without short intermolecular contacts other than weak van der Waals interactions.

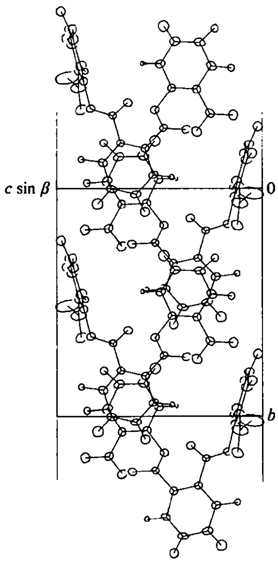


Fig. 2. The *a*-axis projection of the packing of the molecules.

References

GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* A**27**, 368–376.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.

KLYNE, W. & PRELOG, V. (1960). *Experientia*, **16**, 521–523.

LEUENBERGER, C., HOESCH, L. & DRIEDING, A. S. (1980). *Chem. Commun.* pp. 1197–1198.

OAE, S., TSUJIMOTO, N. & NAKANISHI, A. (1973). *Bull. Chem. Soc. Jpn*, **46**, 535–539.

TAMURA, Y., TSUJIMOTO, N., IKEDA, M. & TOMITA, K. (1971). Third Int. Congr. of Heterocycl. Chem., Sendai, Japan, Abstr. B-27-3, pp. 252–255.

UNICS (1979). *The Universal Crystallographic Computing System – Osaka.* Library of Programs, Computing Center, Osaka Univ.

Acta Cryst. (1983). C**39**, 101–103

Structure of 3-Benzyl-7-methyl-3,7-diazabicyclo[3.3.1]nonan-9-one, $C_{15}H_{20}N_2O$

BY P. SMITH-VERDIER, F. FLORENCIO AND S. GARCÍA-BLANCO

Departamento de Rayos X, Instituto 'Rocasolano', CSIC, Serrano 119, *Madrid*-6, *Spain*

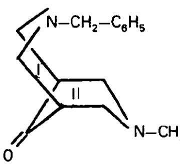
(*Received* 14 *June* 1982; *accepted* 15 *September* 1982)

Abstract. $M_r = 244{\cdot}4$ (5), monoclinic, space group $P2_1/n$, $a = 9{\cdot}779$ (2), $b = 16{\cdot}208$ (3), $c = 8{\cdot}596$ (2) Å, $\beta = 90{\cdot}69$ (1)°, $V = 1362$ Å^3, $Z = 4$, $d_m = 1{\cdot}20$, $d_x = 1{\cdot}191$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 0{\cdot}5580$ mm, $T = 294$ K. Final $R = 0{\cdot}050$ for 2177 observed reflections. The bicyclic system adopts a

0108-2701/83/010101-03$01.50

chair–boat conformation. The $C_6H_5CH_2$ and CH_3 groups are in equatorial positions.

Introduction. A study is being carried out in the Departamento de Química Orgánica de la Facultad de Farmacia de la Universidad Complutense, Madrid, Spain to obtain bicyclic-derivative compounds, which could have pharmacological activity. As part of this study, the structure of the title compound has been determined.



Experimental. Crystals supplied by Drs Galvez and Martínez, crystal used: 0·3 × 0·2 × 0·35 mm, unit-cell parameters determined by least-squares fit of angular settings for 15 high-angle reflections, Philips 1100 automated diffractometer, monochromated Cu $K\alpha$, systematic absences consistent with space group $P2_1/n$; intensities collected on same diffractometer, ω–2θ scanning mode, $\theta = 75°$, no absorption correction, 2293 independent, 2177 observed with $I > 2\sigma(I)$, fluctuations in intensities of two standard reflections less than 3·8%, $R_{\rm int}$ 0·010; scattering factors taken from *International Tables for X-ray Crystallography* (1974); structure solved by *MULTAN* 80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), subsequent refinement carried out by least-squares methods using unit weights with XRAY 70 system of programs (Stewart, Kundell & Baldwin, 1970), function minimized $\sum w|F_o - F_c|^2$, anisotropic temperature factors refined for non-H atoms, all H atoms located in a difference electron-density map and their positional and isotropic thermal parameters also refined, $R_w = 0{\cdot}057$; no significantly large positive or negative electron-density peaks in a final difference Fourier synthesis, largest positive region 0·16 e Å^{-3}, largest negative region 0·17 e Å^{-3}, $F(000) = 528$.

Discussion. The final atomic parameters are contained in Table 1.* Fig. 1 indicates the atomic-numbering scheme. A projection of the crystal structure along the *c* axis is presented in Fig. 2.

Bond lengths and angles are given in Table 2. The molecule consists of two piperidone rings joined by a common ketone bridge with a benzyl ring attached to N(3) and a methyl group attached to N(7). The mean of

Table 1. *Atomic coordinates* (×10⁵, ×10³ *for* H) *and isotropic thermal parameters* (×10⁴, ×10³ *for* H)

For non-H atoms, $U_{\rm eq} = \frac{1}{3}\sum_i\sum_j U_{ij}\, a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j \cos(a_i, a_j)$. For H atoms, $T = \exp[-8\pi^2U(\sin\theta/\lambda)^2]$.

	x	y	z	$U_{\rm eq}/U$(Å^2)
C(1)	33437 (21)	29017 (14)	−30294 (24)	499 (7)
C(2)	40193 (21)	21422 (15)	−22428 (26)	547 (8)
N(3)	30312 (16)	17110 (11)	−12704 (20)	478 (6)
C(4)	18611 (21)	14262 (13)	−21894 (26)	512 (7)
C(5)	11326 (20)	21591 (13)	−29599 (24)	475 (7)
C(6)	6381 (21)	27964 (14)	−17626 (26)	527 (7)
N(7)	12954 (18)	35937 (11)	−20367 (21)	530 (6)
C(8)	27824 (23)	35212 (14)	−18448 (27)	557 (8)
C(9)	21733 (21)	25722 (13)	−39852 (25)	481 (7)
C(10)	37076 (23)	10287 (15)	−4342 (27)	574 (8)
C(11)	27961 (21)	6278 (14)	7593 (24)	500 (7)
C(12)	26341 (25)	−2168 (15)	7913 (29)	616 (8)
C(13)	18496 (29)	−5928 (15)	19369 (34)	722 (10)
C(14)	12218 (27)	−1194 (16)	30444 (30)	674 (9)
C(15)	13646 (25)	7277 (15)	30114 (27)	602 (8)
C(16)	21511 (23)	11002 (14)	18733 (26)	547 (8)
C(17)	7551 (28)	42269 (16)	−9867 (31)	715 (10)
O(1)	21175 (16)	25766 (10)	−54004 (17)	597 (6)
H(11)	402 (2)	316 (1)	−372 (2)	22 (5)
H(21)	486 (2)	230 (1)	−146 (3)	42 (7)
H(22)	440 (2)	172 (1)	−308 (2)	23 (5)
H(41)	122 (2)	116 (1)	−155 (3)	32 (6)
H(42)	213 (2)	106 (1)	−306 (3)	35 (6)
H(51)	36 (2)	193 (1)	−366 (2)	24 (5)
H(61)	85 (2)	259 (1)	−73 (2)	30 (6)
H(62)	−36 (2)	287 (1)	−187 (3)	42 (7)
H(81)	319 (2)	405 (1)	−202 (3)	32 (6)
H(82)	305 (2)	335 (1)	−66 (2)	27 (5)
H(101)	460 (3)	130 (2)	11 (3)	55 (8)
H(102)	403 (2)	58 (1)	−122 (3)	40 (7)
H(121)	311 (2)	−56 (1)	−3 (3)	37 (6)
H(131)	169 (3)	−126 (2)	186 (3)	66 (9)
H(141)	70 (3)	−36 (2)	380 (3)	55 (10)
H(151)	84 (2)	114 (1)	380 (3)	49 (7)
H(161)	230 (2)	169 (1)	192 (3)	43 (7)
H(171)	105 (2)	410 (2)	26 (3)	53 (7)
H(172)	−22 (3)	433 (2)	−121 (3)	67 (9)
H(173)	123 (3)	481 (2)	−121 (3)	64 (9)

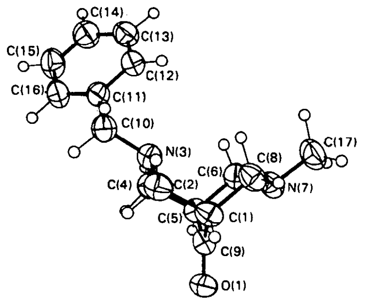


Fig. 1. Perspective view of the molecule showing atomic labelling.

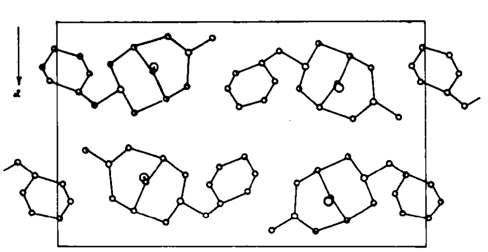


Fig. 2. Projection of the crystal structure along **c**.

---

* Lists of structure factors, anisotropic thermal parameters and conformatonal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38132 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

the two C–C in the ketone bridge is 1·505 Å; these bonds should be shorter since each involves the trigonal bridgehead C atom.

The bicyclic system shows a chair–boat conformation as can be deduced from the torsion angles (Table 3) and the deposited conformational parameters (Cano, Foces-Foces & García-Blanco, 1977). Both rings are very near an ideal conformation and they have dominant mirror symmetry [C_s plane through N(3), N(7), C(9), O(1), C(17)].

Table 2. *Bond lengths* (Å) *and valence angles* (°) *with e.s.d.'s in parentheses*

C(1)–C(2)	1·549 (3)	N(7)–C(8)	1·396 (4)
C(1)–C(8)	1·536 (3)	N(7)–C(17)	1·469 (3)
C(1)–C(9)	1·500 (3)	C(9)–O(1)	1·217 (3)
C(2)–N(3)	1·463 (3)	C(10)–C(11)	1·514 (4)
N(3)–C(4)	1·458 (3)	C(11)–C(12)	1·378 (3)
N(3)–C(10)	1·472 (3)	C(11)–C(16)	1·384 (3)
C(4)–C(5)	1·532 (3)	C(12)–C(13)	1·396 (4)
C(5)–C(6)	1·540 (3)	C(13)–C(14)	1·373 (4)
C(5)–C(9)	1·511 (3)	C(14)–C(15)	1·380 (4)
C(6)–N(7)	1·464 (3)	C(15)–C(16)	1·390 (3)

C(8)–C(1)–C(9)	108·6 (2)	C(8)–N(7)–C(17)	110·5 (2)
C(2)–C(1)–C(9)	106·0 (2)	C(1)–C(8)–N(7)	109·9 (2)
C(2)–C(1)–C(8)	112·6 (2)	C(1)–C(9)–C(5)	110·7 (2)
C(1)–C(2)–N(3)	110·4 (2)	C(5)–C(9)–O(1)	124·3 (2)
C(2)–N(3)–C(10)	110·0 (2)	C(1)–C(9)–O(1)	124·8 (2)
C(2)–N(3)–C(4)	111·2 (2)	N(3)–C(10)–C(11)	112·9 (2)
C(4)–N(3)–C(10)	111·8 (2)	C(10)–C(11)–C(16)	120·6 (2)
N(3)–C(4)–C(5)	110·3 (2)	C(10)–C(11)–C(12)	120·6 (2)
C(4)–C(5)–C(9)	106·4 (2)	C(12)–C(11)–C(16)	118·8 (2)
C(4)–C(5)–C(6)	112·3 (2)	C(11–C(12)–C(13)	120·8 (2)
C(6)–C(5)–C(9)	108·2 (2)	C(12)–C(13)–C(14)	120·0 (3)
C(5)–C(6)–N(7)	110·0 (2)	C(13)–C(14)–C(15)	119·7 (2)
C(6)–N(7)–C(17)	110·9 (2)	C(14)–C(15)–C(16)	120·2 (2)
C(6)–N(7)–C(8)	110·4 (2)	C(11)–C(16)–C(15)	120·5 (2)

Table 3. *Torsion angles* (°)

C(8)–C(1)–C(9)–C(5)	−58·9 (2)	C(2)–N(3)–C(4)–C(5)	−60·1 (2)
C(1)–C(9)–C(5)–C(6)	58·0 (2)	N(3)–C(4)–C(5)–C(9)	59·8 (2)
C(9)–C(5)—–N(7)	1·9 (2)	C(4)–C(5)–C(9)–C(1)	−62·9 (2)
C(5)–C(6)–N(7)–C(8)	−61·8 (2)	C(5)–C(9)–C(1)–C(2)	62·4 (2)
C(6)–N(7)–C(8)–C(1)	61·0 (2)	C(4)–N(3)–C(10)–C(11)	−63·8 (2)
N(7)–C(8)–C(1)–C(9)	−0·4 (6)	C(2)–N(3)–C(10)–C(11)	172·2 (2)
C(9)–C(1)–C(2)–N(3)	−59·5 (2)	N(3)–C(10)–C(11)–C(12)	129·8 (2)
C(1)–C(2)–N(3)–C(4)	59·9 (2)	N(3)–C(10)–C(11)–C(16)	−52·7 (3)

None of the intermolecular distances is less than a normal van der Waals contact. The closest approach is 3·350 Å and is that of the O of one molecule to C(16) of a molecule related to the first by symmetry of type $x,y,z + 1$.

We thank the Centro de Proceso de Datos del Ministerio de Educación y Ciencia (Madrid) for the use of the 1108 Univac computer.

References

Cano, F. H., Foces-Foces, C. & García-Blanco, S. (1977). *Tetrahedron*, **33**, 797–802.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L. Germain, G., Declercq, J. P. & Woolfson, M. M. (1980). *MULTAN* 80. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Stewart, J. M., Kundell, F. A. & Baldwin, J. C. (1970). The XRAY 70 system. Computer Science Center, Univ. of Maryland, College Park, Maryland.

Acta Cryst. (1983). C**39**, 103–105

Structure of 1,3,9,9,10,10-Hexamethyl-2,4,6,8-tetraoxatricyclo$[3.3.1.1^{3,7}]$decane, $C_{12}H_{20}O_4$

By Setsuo Kashino and Masao Haisa

Department of Chemistry, Faculty of Science, Okayama University, Tsushima, Okayama 700, *Japan*

and Sadao Tsuboi, Tsuyoshi Ono and Akira Takeda

Department of Synthetic Chemistry, School of Engineering, Okayama University, Tsushima, Okayama 700, *Japan*

(*Received* 9 *June* 1982; *accepted* 22 *July* 1982)

Abstract. $M_r = 228·29$, orthorhombic, $Pbna$, $a = 11·043$ (1), $b = 15·847$ (1), $c = 6·764$ (1) Å, $V = 1183·6$ (2) Å^3, $Z = 4$, $D_m = 1·24$, $D_x = 1·281$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 0·74$ mm^{-1}, $F(000) = 496$; the final $R = 0·052$ for 853 non-zero reflexions. The molecule retains twofold symmetry in the crystals. The tetraoxaadamantane skeleton reveals no significant distortion. The average dimensions are: O–C 1·426 (2), C–C 1·536 (3) Å; O–C–O 110·2 (2), C–O–C 111·5 (2)°.

0108-2701/83/010103-03$01.50