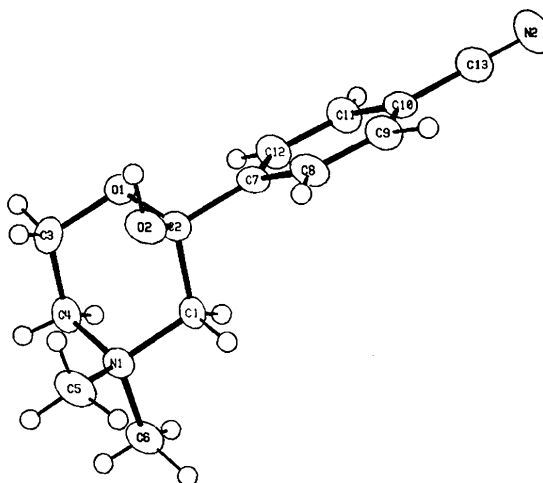


Table 3. Bond lengths (Å), bond angles (°) and torsion angles (°)

O1—C2	1.415 (4)	C2—C7	1.522 (5)
O1—C3	1.447 (4)	C3—C4	1.493 (5)
O2—C2	1.400 (4)	C7—C8	1.393 (5)
N1—C1	1.513 (4)	C7—C12	1.389 (4)
N1—C4	1.507 (4)	C8—C9	1.386 (5)
N1—C5	1.515 (4)	C9—C10	1.394 (5)
N1—C6	1.504 (5)	C10—C11	1.394 (5)
N2—C13	1.136 (5)	C10—C13	1.439 (5)
C1—C2	1.521 (5)	C11—C12	1.378 (5)
C2—O1—C3	111.8 (3)	O1—C3—C4	110.8 (3)
C1—N1—C4	107.9 (3)	N1—C4—C3	110.5 (3)
C1—N1—C5	112.7 (3)	C2—C7—C8	120.2 (3)
C1—N1—C6	108.0 (3)	C2—C7—C12	120.2 (3)
C4—N1—C5	111.5 (3)	C8—C7—C12	119.4 (3)
C4—N1—C6	109.5 (3)	C7—C8—C9	120.3 (3)
C5—N1—C6	107.2 (3)	C8—C9—C10	119.6 (3)
N1—C1—C2	114.2 (3)	C9—C10—C11	120.3 (3)
O1—C2—O2	111.3 (3)	C9—C10—C13	120.0 (3)
O1—C2—C1	111.8 (3)	C11—C10—C13	119.7 (3)
O1—C2—C7	105.8 (3)	C10—C11—C12	119.5 (3)
O2—C2—C1	107.4 (3)	C7—C12—C11	120.9 (3)
O2—C2—C7	113.5 (3)	N2—C13—C10	179.3 (4)
C1—C2—C7	107.0 (3)		
C3—O1—C2—O2	-65.9 (4)	O2—C2—C7—C8	26.6 (6)
C3—O1—C2—C1	54.2 (5)	O2—C2—C7—C12	-158.4 (4)
C3—O1—C2—C7	170.3 (3)	C1—C2—C7—C8	-91.6 (5)
C2—O1—C3—C4	-61.0 (5)	C1—C2—C7—C12	83.4 (5)
C4—N1—C1—C2	49.7 (5)	O1—C3—C4—N1	61.6 (5)
C5—N1—C1—C2	-73.9 (5)	C2—C7—C8—C9	175.2 (4)
C6—N1—C1—C2	167.9 (4)	C12—C7—C8—C9	0.2 (7)
C1—N1—C4—C3	-54.7 (5)	C2—C7—C12—C11	-176.1 (4)
C5—N1—C4—C3	69.6 (5)	C8—C7—C12—C11	-1.0 (7)
C6—N1—C4—C3	-172.0 (4)	C7—C8—C9—C10	0.8 (7)
N1—C1—C2—O1	-50.3 (5)	C8—C9—C10—C11	-0.9 (7)
N1—C1—C2—O2	72.1 (4)	C8—C9—C10—C13	178.2 (4)
N1—C1—C2—C7	-165.7 (4)	C9—C10—C11—C12	0.1 (7)
O1—C2—C7—C8	149.0 (4)	C13—C10—C11—C12	-179.0 (4)
O1—C2—C7—C12	-36.0 (5)	C10—C11—C12—C7	0.8 (7)

Fig. 1. $[C_{13}H_{17}N_2O_2]^+$, 40% ellipsoids (Johnson, 1965).

pharmacological activity of related compounds: Anderson, Corey, Force, Jensen, Matz & Rivard (1966).

References

- ANDERSON, E. L., COREY, J. E. JR, FORCE, E. E., JENSEN, E. M., MATZ, R. S. & RIVARD, D. E. (1966). *J. Med. Chem.* **9**, 211–213.
- FRENZ, B. A. (1978). In *Computing in Crystallography*, edited by H. SCHENK, R. OLTJOF-HAEZKAMP, H. VAN KONINGSVELD & G. C. BASSI, pp. 64–71. Delft Univ. Press.
- GARCIA, J. G. (1986). MS Thesis. Louisiana State Univ., Baton Rouge, Louisiana, USA.
- GARCIA-GUJARDO, J. G., FRONCZEK, F. R. & GANDOUR, R. D. (1986). *Acta Cryst.* **C42**, 1535–1537.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- JOHNSON, C. K. (1965). *ORTEP: A Fortran Thermal-Ellipsoid Plot Program for Crystal Structure Illustrations*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

Related literature. Synthesis: Garcia (1986); structure of 4,4-dimethyl-2-oxomorpholinium bromide: Garcia-Guajardo, Fronczek & Gandour (1986); synthesis and

Acta Cryst. (1988). **C44**, 1690–1692

Structure of a Hexahydrobenzo[*a*]quinolizine Derivative

BY JOCHEN ANTEL AND GEORGE M. SHEDRICK

Institut für Anorganische Chemie der Universität, Tammannstrasse 4, D-3400 Göttingen, Federal Republic of Germany

AND GUNTER BRILL AND LUTZ-F. TIETZE

Institut für Organische Chemie der Universität, Tammannstrasse 2, D-3400 Göttingen, Federal Republic of Germany

(Received 30 March 1988; accepted 11 May 1988)

Abstract. Racemic (2*R*,3*R*,11*bS*)-1,2,3,4,6,7-Hexahydro-9,10-dimethoxy-3-methoxycarbonyl-11*bH*-benzo[*a*]quinolizine-2-carbonitrile, $C_{18}H_{22}N_2O_4$, $M_r =$

330.38, triclinic, $P\bar{1}$, $a = 8.459$ (2), $b = 9.671$ (2), $c = 11.017$ (2) Å, $\alpha = 106.64$ (3), $\beta = 99.86$ (3), $\gamma = 98.90$ (2)°, $V = 830.72$ Å³, $Z = 2$, $D_x = 1.32$ Mg m⁻³,

0108-2701/88/091690-03\$03.00

© 1988 International Union of Crystallography

$\lambda(\text{Mo } K\alpha) = 0.71069 \text{ \AA}$, $\mu = 0.09 \text{ mm}^{-1}$, $F(000) = 352$, $T = 298 \text{ K}$, $R = 0.064$ for 1586 observed reflections. The structure was investigated to determine the relative configuration, which could not be established unambiguously by NMR. The piperidine ring consisting of C(1), C(2), C(3), C(4), N(5), and C(11b) adopts a chair conformation, whereas the second piperidine moiety shows a twisted half-chair conformation.

Experimental. Crystal size $0.3 \times 0.3 \times 0.4 \text{ mm}$. Stoe-Siemens four-circle diffractometer, monochromated Mo $K\alpha$ radiation, profile-fitting mode involving variable scan width and speed (Clegg, 1981). 3471 reflections measured, $2\theta_{\text{max}} 45^\circ$, $\pm h \pm k \pm l$, three check reflections with no significant intensity change. 2152 unique reflections ($R_{\text{int}} = 0.031$), of which 1586 with $F > 3\sigma(F)$ were used for all calculations (SHELXS86, Sheldrick, 1985; SHELX76, Sheldrick, 1976). Cell constants refined from $\pm 2\theta$ values of 29 reflections in the range $20\text{--}25^\circ$. Absorption and extinction corrections were not necessary. Structure solution by direct methods. Refinement on F to $R = 0.064$, $wR = 0.063$; all non-H atoms anisotropic, H atoms were included using a riding model [$\text{C-H } 0.96 \text{ \AA}$, $U(\text{H}) = 1.2U_{\text{eq}}(\text{C})$], 226 parameters, $S = 1.45$, weighting scheme $w^{-1} = \sigma^2(F) + 0.0006F^2$ which led to a featureless analysis of variance in terms of $\sin\theta$ and F_o , max. $\Delta/\sigma = 0.21$, max. and min. height in final $\Delta\rho$ map 0.29 and -0.29 e \AA^{-3} respectively. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). The main reason for the relatively high R index was probably the poor quality of the crystal.

Atomic parameters are given in Table 1, selected bond distances and angles in Table 2.* Fig. 1 shows a thermal ellipsoid plot with the atom numbering.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51022 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

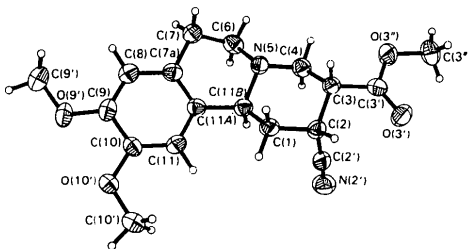


Fig. 1. View of the title compound showing atom-numbering scheme.

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$)

Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
C(1)	2259 (4)	3901 (4)	3982 (4)	40 (2)
C(2)	3099 (4)	5533 (4)	4489 (4)	37 (2)
C(2')	4198 (5)	5858 (4)	3644 (4)	42 (2)
N(2')	5054 (5)	6050 (4)	2999 (4)	61 (2)
C(3)	1811 (4)	6483 (4)	4566 (4)	36 (2)
C(3')	2634 (5)	8100 (4)	5109 (4)	40 (2)
O(3')	4049 (4)	8558 (3)	5682 (3)	61 (1)
O(3'')	1626 (3)	8980 (3)	4915 (3)	50 (1)
C(3'')	2298 (6)	10555 (4)	5483 (5)	60 (2)
C(4)	524 (4)	6007 (4)	3289 (4)	39 (2)
N(5)	-238 (3)	4439 (3)	2947 (3)	34 (1)
C(6)	-1622 (5)	3981 (4)	1806 (4)	44 (2)
C(7)	-2586 (4)	2467 (4)	1671 (4)	44 (2)
C(7A)	-1470 (5)	1385 (4)	1687 (4)	37 (2)
C(8)	-2129 (5)	-146 (4)	1182 (4)	42 (2)
C(9)	-1135 (5)	-1156 (4)	1122 (4)	40 (2)
O(9')	-1657 (3)	-2660 (3)	633 (3)	54 (1)
C(9')	-3355 (5)	-3236 (5)	151 (5)	65 (2)
C(10)	564 (5)	-636 (4)	1567 (4)	37 (2)
O(10')	1475 (3)	-1709 (3)	1434 (3)	47 (1)
C(10')	3202 (5)	-1218 (5)	1930 (5)	53 (2)
C(11)	1204 (5)	855 (4)	2083 (4)	37 (2)
C(11A)	203 (5)	1889 (4)	2155 (4)	35 (2)
C(11B)	992 (4)	3528 (4)	2708 (4)	35 (2)

Table 2. Bond lengths (\AA) and bond angles ($^\circ$)

C(1)–C(2)	1.523 (5)	C(1)–C(11B)	1.525 (5)
C(2)–C(2')	1.484 (7)	C(2)–C(3)	1.528 (6)
C(2')–N(2')	1.129 (7)	C(3)–C(3')	1.507 (5)
C(3)–C(4)	1.525 (5)	C(3')–O(3')	1.202 (5)
C(3')–O(3'')	1.327 (6)	O(3'')–C(3'')	1.449 (4)
C(4)–N(5)	1.465 (5)	N(5)–C(6)	1.471 (4)
N(5)–C(11B)	1.474 (5)	C(6)–C(7)	1.515 (6)
C(7)–C(7A)	1.516 (6)	C(7A)–C(8)	1.407 (5)
C(7A)–C(11A)	1.378 (5)	C(8)–C(9)	1.379 (6)
C(9)–O(9')	1.368 (5)	C(9)–C(10)	1.397 (5)
O(9')–C(9')	1.406 (5)	C(10)–O(10')	1.375 (5)
C(10)–C(11)	1.371 (5)	O(10')–C(11)	1.421 (5)
C(11)–C(11A)	1.402 (6)	C(11A)–C(11B)	1.520 (5)
C(2)–C(1)–C(11B)	111.4 (3)	C(1)–C(2)–C(2')	109.9 (3)
C(1)–C(2)–C(3)	110.1 (3)	C(2')–C(2)–C(3)	111.5 (4)
C(2)–C(2')–N(2')	177.0 (5)	C(2)–C(3)–C(3')	110.4 (3)
C(2)–C(3)–C(4)	111.5 (3)	C(3')–C(3)–C(4)	116.2 (4)
C(3)–C(3')–O(3')	123.8 (4)	C(3)–C(3')–O(3'')	113.2 (3)
O(3')–C(3')–O(3'')	123.0 (4)	C(3')–O(3'')–C(3'')	116.5 (3)
C(3)–C(4)–N(5)	109.4 (3)	C(4)–N(5)–C(6)	111.0 (3)
C(4)–N(5)–C(11B)	110.3 (3)	C(6)–N(5)–C(11B)	110.6 (3)
N(5)–C(6)–C(7)	108.8 (4)	C(6)–C(7)–C(7A)	111.2 (3)
C(7)–C(7A)–C(8)	120.2 (3)	C(7)–C(7A)–C(11A)	120.4 (3)
C(3)–C(7A)–C(11A)	119.4 (4)	C(7A)–C(8)–C(9)	121.5 (3)
C(8)–C(9)–O(9')	125.7 (3)	C(8)–C(9)–C(10)	118.8 (3)
O(9')–C(9)–C(10)	115.5 (4)	C(9)–O(9')–C(9')	117.6 (3)
C(9)–C(10)–O(10')	115.3 (3)	C(9)–C(10)–C(11)	119.8 (4)
O(10')–C(10)–C(11)	124.9 (3)	C(10)–O(10')–C(11)	116.7 (3)
C(10)–C(11)–C(11A)	121.8 (3)	C(7A)–C(11A)–C(11)	118.7 (3)
C(7A)–C(11A)–C(11B)	122.0 (4)	C(11)–C(11A)–C(11B)	119.3 (3)
C(1)–C(11B)–N(5)	108.1 (3)	C(1)–C(11B)–C(11A)	112.0 (3)
N(5)–C(11B)–C(11A)	111.8 (3)		

Related literature. For the preparation of the compound see Brill (1986). For the preparation of some related compounds see Tietze & Brill (1987). For a structure of a hexahydrobenzo[*a*]quinolizine derivative see Carpy, H'Naïfi & Leger (1984).

We thank the Deutsche Forschungsgemeinschaft and the Fonds der Chemischen Industrie for financial support.

References

- BRILL, G. (1986). Thesis. Univ. of Göttingen, Federal Republic of Germany.
- CARPY, A., H'NAÏFI, A. & LEGER, J.-M. (1984). *Acta Cryst.* C40, 1965-1967.
- CLEGG, W. (1981). *Acta Cryst.* A37, 22-28.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- SHELDRIK, G. M. (1976). *SHELX76*. Crystal structure refinement program. Univ. of Cambridge. Modified by G. M. SHELDRIK.
- SHELDRIK, G. M. (1985). *SHELXS86*. In *Crystallographic Computing 3*, edited by G. M. SHELDRIK, C. KRÜGER & R. GODDARD, pp. 175-189. Oxford Univ. Press.
- TIETZE, L.-F. & BRILL, G. (1987). *Justus Liebig's Ann. Chem.* pp. 311-319.

Book Review

Works intended for notice in this column should be sent direct to the Book-Review Editor (R. O. Gould, University of Edinburgh, West Mains Road, Edinburgh EH9 3JJ, Scotland). As far as practicable books will be reviewed in a country different from that of publication.

Acta Cryst. (1988). C44, 1692

Stereochemistry of organometallic and inorganic compounds. Edited by IVAN BERNAL. Pp. x + 246. Amsterdam: Elsevier, 1987. Price Dfl 195.00, US\$ 95.00.

The publication of Volume 2 of this series reflects the enormous current interest in the coordination chemistry of macrocyclic ligands, and fills a gap in the review literature that has developed since the publication of G. A. Melson's *Coordination Chemistry of Macrocyclic Compounds* in 1979. The volume consists of three chapters. The first, 'Stereochemistry of Metallic Macrocyclics' by Jan C. A. Boeyens and Susan M. Dobson (102 pp., 208 references), gives an excellent account of the conformational characteristics of transition-metal-coordinated macrocycles. The article reviews the chemistry of both large and small ring N-, S- and P-donor systems, and relates the observed stereochemistries at the metal centre (obtained usually by X-ray crystallography) with conformational properties and preferences of the coordinated ligand. Chapter 2 by H.-J. Buschmann on 'Thermodynamic and Stereochemical Aspects of the Macrocyclic and Cryptate Effects' (84 pp., 149 references) is equally successful, and concentrates on O-donor polyether crown and cryptate chemistry. A good historical perspective is retained throughout, and the many thermodynamic parameters quoted are restricted to compact tables, thus

making the article very approachable and informative. Perhaps the least successful chapter in this volume is the last one by Karen E. Matthes and David Parker on 'Stereochemical Aspects of Macrocyclic Complexes of Transition Metal Ions' (36 pp., 100 references). This article deals with selected aspects of the chemistry of second- and third-row transition-metal ions and also includes sections on copper, nickel, manganese and chromium. Reviewing a vast topic such as macrocyclic chemistry, by its very nature, necessitates selectivity. However, considering its title, this article is short and the examples too selective. It might have been more successful if this section had been incorporated into Chapter 1 to give two large chapters on transition- and main-group-element chemistry respectively. This notwithstanding, Chapter 3 concentrates on unusual aspects of heavy-metal macrocyclic chemistry and, therefore, gives an important overview of new developments in this area.

In general, Volume 2 is an excellent addition to this series. Most importantly, each chapter concentrates on work in the recent literature (up to mid-1986) and the timeliness of this well produced volume is therefore retained.

MARTIN SCHRÖDER

*Department of Chemistry
University of Edinburgh
West Mains Road
Edinburgh EH9 3JJ
Scotland*