

Table 1. Final fractional coordinates ($\times 10^4$) and mean isotropic temperature factors ($\times 10^3$) with e.s.d.'s in parentheses

For non-H atoms $U_{\text{iso}} = \frac{1}{3}(U_{11} + U_{22} + U_{33})$.

	x	y	z	U_{iso} (\AA^2)
O(1)	7503 (2)	2911 (8)	1884 (2)	115 (2)
N(1)	8369 (2)	1463 (6)	3433 (2)	82 (2)
C(2)	6836 (3)	3309 (9)	2454 (2)	82 (2)
C(21)	5679 (2)	3279 (7)	1818 (2)	73 (2)
C(22)	4969 (3)	4923 (8)	1920 (3)	88 (3)
C(23)	3900 (3)	4850 (9)	1365 (3)	88 (3)
C(24)	3494 (3)	3142 (8)	678 (2)	76 (2)
C(25)	4184 (3)	1513 (9)	535 (3)	83 (2)
C(26)	5256 (3)	1601 (8)	1108 (3)	81 (3)
O(2)	2404 (2)	3174 (6)	187 (2)	99 (2)
C(27)	1941 (5)	1288 (14)	-430 (5)	111 (4)
C(3)	7226 (3)	1495 (7)	3266 (2)	77 (2)
C(31)	6979 (2)	2003 (6)	4179 (2)	64 (2)
C(32)	6316 (3)	551 (7)	4451 (2)	71 (2)
C(33)	6070 (3)	999 (7)	5273 (2)	72 (2)
C(34)	6478 (2)	2946 (7)	5827 (2)	66 (2)
C(35)	7145 (3)	4415 (8)	5572 (3)	79 (2)
C(36)	7393 (3)	3919 (7)	4750 (3)	81 (2)
O(3)	6179 (2)	3233 (5)	6627 (2)	88 (2)
C(37)	6522 (5)	5292 (10)	7188 (4)	115 (4)
C(4)	8946 (4)	-489 (10)	4008 (4)	106 (3)
C(1)	8381 (3)	1496 (9)	2431 (3)	92 (3)
C(11)	9408 (3)	2470 (7)	2378 (3)	84 (2)
C(12)	9985 (3)	4107 (9)	3019 (4)	106 (4)
C(13)	10859 (4)	5122 (12)	2908 (5)	134 (4)
C(14)	11181 (4)	4451 (15)	2168 (6)	145 (5)
C(15)	10628 (5)	2853 (15)	1531 (5)	144 (5)
C(16)	9727 (4)	1808 (10)	1626 (4)	117 (3)
H(2)	6994 (22)	4846 (61)	2738 (20)	63 (8)
H(22)	5238 (26)	6086 (74)	2358 (24)	87 (12)
H(23)	3463 (32)	6090 (94)	1360 (30)	127 (16)
H(25)	3952 (26)	151 (80)	64 (25)	109 (12)
H(26)	5671 (24)	628 (63)	1072 (22)	63 (11)
H(27)	1102 (32)	1765 (83)	-756 (27)	111 (14)
H(28)	2164 (45)	-7 (120)	6 (40)	148 (25)
H(29)	2264 (29)	1073 (91)	-962 (29)	127 (16)
H(3)	6878 (18)	62 (51)	2957 (16)	45 (7)
H(32)	6057 (23)	-842 (65)	4087 (20)	74 (10)
H(33)	4352 (24)	4990 (69)	4520 (22)	92 (10)
H(35)	7389 (27)	5731 (83)	5867 (24)	93 (12)
H(36)	7861 (25)	4955 (64)	4568 (22)	84 (10)
H(37)	6181 (30)	5217 (88)	7695 (29)	115 (14)
H(38)	6287 (35)	6676 (89)	6701 (33)	132 (18)
H(39)	7410 (33)	5152 (88)	7463 (28)	128 (15)
H(41)	8859 (29)	-464 (85)	4634 (28)	112 (13)
H(42)	9743 (35)	-356 (77)	4107 (27)	117 (13)
H(43)	8664 (43)	-2423 (152)	3653 (40)	183 (23)
H(1)	8286 (28)	-108 (83)	2163 (26)	101 (13)
H(12)	9725 (35)	4721 (107)	3515 (34)	143 (17)
H(13)	11341 (39)	5922 (103)	3389 (32)	134 (18)
H(14)	11703 (35)	5338 (91)	2034 (27)	130 (16)
H(15)	10736 (34)	2666 (106)	947 (35)	138 (18)
H(16)	9339 (42)	700 (121)	1114 (36)	166 (23)

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

O(1)—C(1)	1.416 (5)	C(31)—C(32)	1.378 (5)
C(1)—N(1)	1.469 (5)	C(32)—C(33)	1.378 (6)
N(1)—C(3)	1.457 (4)	C(33)—C(34)	1.375 (5)
C(3)—C(2)	1.524 (6)	C(34)—C(35)	1.376 (6)
C(2)—O(1)	1.440 (5)	C(35)—C(36)	1.384 (6)
N(1)—C(4)	1.448 (6)	C(36)—C(31)	1.374 (5)
C(2)—C(21)	1.489 (4)	O(3)—C(34)	1.374 (4)
C(21)—C(22)	1.386 (6)	O(3)—C(37)	1.421 (6)
C(22)—C(23)	1.366 (6)	C(1)—C(11)	1.511 (6)
C(23)—C(24)	1.370 (6)	C(11)—C(12)	1.357 (6)
C(24)—C(25)	1.384 (6)	C(12)—C(13)	1.367 (8)
C(25)—C(26)	1.375 (5)	C(13)—C(14)	1.355 (12)
C(26)—C(21)	1.380 (5)	C(14)—C(15)	1.328 (10)
O(2)—C(24)	1.372 (4)	C(15)—C(16)	1.397 (9)
O(2)—C(27)	1.404 (8)	C(16)—C(11)	1.367 (7)
C(3)—C(31)	1.513 (5)		
C(1)—O(1)—C(2)	109.0 (3)	C(32)—C(31)—C(36)	118.2 (3)
O(1)—C(2)—C(3)	103.4 (3)	C(31)—C(32)—C(33)	121.2 (3)
C(2)—C(3)—N(1)	100.5 (3)	C(32)—C(33)—C(34)	119.8 (4)
C(3)—N(1)—C(1)	102.7 (2)	C(33)—C(34)—C(35)	120.0 (3)
N(1)—C(1)—O(1)	105.4 (3)	C(33)—C(34)—O(3)	115.0 (3)
O(1)—C(2)—C(21)	111.0 (3)	O(3)—C(34)—C(35)	124.9 (3)
C(3)—C(2)—C(21)	117.6 (4)	C(34)—O(3)—C(37)	117.3 (4)
C(2)—C(21)—C(22)	121.5 (3)	C(34)—C(35)—C(36)	119.3 (4)
C(2)—C(21)—C(26)	121.8 (4)	C(35)—C(36)—C(31)	121.5 (4)
C(22)—C(21)—C(26)	116.6 (3)	O(1)—C(1)—C(11)	109.9 (4)
C(21)—C(22)—C(23)	121.7 (4)	N(1)—C(1)—C(11)	113.1 (3)
C(22)—C(23)—C(24)	120.6 (4)	C(1)—C(11)—C(12)	121.5 (4)
C(23)—C(24)—C(25)	119.3 (3)	C(1)—C(11)—C(16)	119.4 (4)
C(23)—C(24)—O(2)	116.0 (4)	C(12)—C(11)—C(16)	118.9 (4)
C(24)—O(2)—C(27)	117.7 (4)	C(11)—C(12)—C(13)	120.8 (6)
O(2)—C(24)—C(25)	124.7 (4)	C(12)—C(13)—C(14)	120.2 (6)
C(24)—C(25)—C(26)	119.2 (4)	C(13)—C(14)—C(15)	119.9 (7)
C(25)—C(26)—C(21)	122.5 (4)	C(14)—C(15)—C(16)	120.7 (7)
C(2)—C(3)—C(31)	115.5 (3)	C(15)—C(16)—C(11)	119.3 (5)
N(1)—C(3)—C(31)	113.8 (2)	C(1)—N(1)—C(4)	113.1 (4)
C(3)—C(31)—C(32)	120.3 (3)	C(3)—N(1)—C(4)	114.5 (4)
C(3)—C(31)—C(36)	121.5 (3)		

Discussion. The final coordinates with e.s.d.'s are listed in Table 1* with atoms numbered arbitrarily as shown in Fig. 1. Bond lengths and angles are given in Table 2.

The *p*-methoxyphenyl substituents bound to atoms C(2) and C(3) are in the *trans* configuration, whereas the phenyl group bound to C(1) is in the *cis* configuration with respect to the *p*-methoxyphenyl of C(3) and in the *trans* configuration with respect to the methyl group bound to N(1). The angles between the best planes fitted by least squares through the phenyl rings are: $\angle I, II = 50.4 (2)$, $\angle I, III = 17.0 (2)$, $\angle II, III = 65.3 (2)^\circ$, where I, II and III represent the phenyl rings connected to atoms C(1), C(2) and C(3), respectively. The oxazolidine ring is in an almost ideal half-chair conformation, with atoms N and C(3) displaced by 0.32 (2) and 0.36 (2) \AA on opposite sides of the plane through C(1), O and C(2).

The authors wish to thank Dr M. Hursthouse for data collection.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38472 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- BARTNIK, R. & MLOSTOŃ, G. (1983). In preparation.
 BARTNIK, R. & MLOSTOŃ, G. (1981). *Topics in Chemistry of Heterocyclic Compounds*, edited by J. KOVAČ, p. 118. Bratislava, Czechoslovakia.

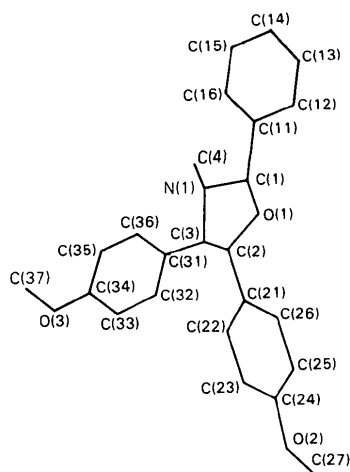


Fig. 1. The atom-numbering scheme.

MAIN, P., WOOLFSON, M. M., LESSINGER, L., GERMAIN, G. & DECLERCQ, J. P. (1976). *MULTAN76. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

SKARŻYŃSKI, T. (1982a). *Acta Cryst.* B38, 3110–3111.
SKARŻYŃSKI, T. (1982b). *Acta Cryst.* B38, 3112–3113.
SKARŻYŃSKI, T., DEREWENDA, Z., BRZozowski, A. M. & MLOSTOŃ, G. (1982). *Acta Cryst.* B38, 3113–3115.

Acta Cryst. (1983). C39, 1053–1055

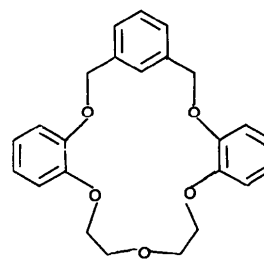
1,4,7,14,22-Pentaoxa[7]orthocyclo[2]metacyclo[2]orthocyclophane, C₂₄H₂₄O₅

BY GABRIELA WEBER* AND PETER G. JONES

Anorganisch-Chemisches Institut der Universität, Tammannstrasse 4, D-3400 Göttingen, Federal Republic of Germany

(Received 16 February 1983; accepted 28 March 1983)

Abstract. $M_r = 392.46$, orthorhombic, $Cmc2_1$, $a = 23.444$ (6), $b = 10.852$ (3), $c = 7.983$ (2) Å, $V = 2031.0$ Å³, $Z = 4$, $D_x = 1.283$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.083$ mm⁻¹, $F(000) = 832$, $T = 291$ K, $R = 0.034$ for 1601 reflections. With a crystallographic mirror generating the second half of the molecule, the macrocycle adopts a 'dentist's chair' conformation in which the five hetero-atoms are co-planar to within ± 0.008 Å.



(1)

Introduction. The pyridino analogue (2) [in which the 'inner' C(10) is replaced by N] of the title ligand (1) forms a variety of adducts (including clathrates) with small neutral molecules (Vögtle, Müller & Weber, 1980), but the title ligand itself does not. In general, crown ethers based on xylene compounds reveal a relatively low coordination ability (e.g. Cram & Cram, 1978; cf. van Zon, de Jong, Reinhoudt, Torny & Onwezen, 1981; de Boer, Reinhoudt, Harkema, van Hummel & de Jong, 1982), and few structures of such complexes with H-bond donors are known (e.g. Goldberg, 1975; Reinhoudt, den Hertog & de Jong, 1981). Hence it seems that the pyridine base plays an important rôle in secondary interactions, particularly in the uptake of alcohols (Weber & Vögtle, 1980; Weber, Vögtle, Josel, Newkome & Puckett, 1983), though a methanol complex of a bicyclic polyether containing only O as hetero-atoms has recently been reported (Bandy, Hughes & Truter, 1982).

An X-ray analysis of the present compound (1) might, therefore, provide structural information as to the differing coordination behaviours of (1) and (2).

Experimental. Sample kindly provided by Dr E. Weber (no relation), University of Bonn, D-5300 Federal Republic of Germany, crystal $ca 0.6 \times 0.6 \times 0.4$ mm, grown from methanol/ethyl acetate; Stoe four-circle diffractometer, cell dimensions from 2θ angles for 36 reflections ($20 < 2\theta < 25^\circ$); 4308 profile-fitted (Clegg, 1981) data up to $2\theta = 50^\circ$ (excluding systematic absences), $-27 \leq h \leq 27$, $-2 \leq k \leq 12$, $-9 \leq l \leq 9$, 1826 unique ($R_{\text{int}} = 0.019$), 1601 intensities $> 2\sigma(I)$; structure solved by multisolution direct methods and refined anisotropically by blocked-cascade least squares on F , H atoms included in idealized positions (C–H = 0.96 Å) as 'riding atoms' [except H(10) refined freely], $U(\text{H}_i) = 1.2 U_{\text{eq}}(\text{C}_i)$; empirical extinction correction coefficient $x = 5.6$ (2) $\times 10^{-6}$, where $F' = F/(1.0 + xF^2/\sin 2\theta)^{0.25}$; 138 parameters, $wR = 0.031$, $w^{-1} = \sigma^2(F) + 0.00015F^2$, $S = 1.42$, slope of normal probability plot = 1.29, max. shift/e.s.d. = 0.04, mean 0.007, largest peak/hole ≤ 0.22 e Å⁻³; scattering factors from *International Tables for X-ray Crystallography* (1974), program for structure solution, refinement, etc. (SHELXTL) written by Professor G. M. Sheldrick, for diffractometer control by Dr W. Clegg, University of Göttingen.

* Author for correspondence.