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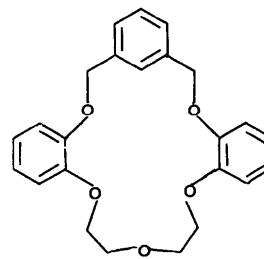
1,4,7,14,22-Pentaoxa[7]orthocyclo[2]metacyclo[2]orthocyclophane, C₂₄H₂₄O₅

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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.

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Table 1. Atom coordinates ($\times 10^5$, H $\times 10^4$) and isotropic temperature factors ($\text{\AA}^2 \times 10^4$)

	x	y	z	U_{eq}^*
H(10)	5000	1116 (22)	429 (33)	622
O(1)	50000	45359 (12)	44580†	535 (5)
C(2)	45035 (7)	52590 (15)	42740 (25)	588 (6)
C(3)	39861 (7)	44812 (16)	45230 (26)	589 (6)
O(4)	39356 (4)	36370 (11)	31584 (18)	627 (4)
C(5)	35095 (6)	27754 (15)	32225 (26)	528 (5)
C(6)	35072 (6)	19029 (15)	19334 (25)	528 (6)
O(7)	39326 (4)	20327 (11)	7731 (18)	629 (4)
C(8)	39361 (7)	12033 (15)	-6064 (28)	611 (6)
C(9)	44847 (7)	13755 (13)	-15453 (24)	514 (5)
C(10)	50000	12531 (19)	-7156 (34)	517 (8)
C(11)	44924 (8)	16190 (16)	-32478 (26)	634 (6)
C(12)	50000	17372 (22)	-40876 (35)	711 (11)
C(13)	30943 (7)	10050 (16)	18881 (28)	642 (6)
C(14)	26800 (8)	9601 (19)	31353 (32)	752 (7)
C(15)	26782 (8)	18090 (20)	43898 (33)	776 (8)
C(16)	30939 (7)	27225 (18)	44415 (29)	659 (6)

* $U_{eq} = \frac{1}{3}$ (trace of the orthogonalized U_{ij} matrix) (except for H).

† Fixed to define the origin on the polar axis.

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

Primed atoms are related by $1-x, y, z$.			
C(2)—O(1)	1.412 (3)	C(2)—C(3)	1.491 (3)
C(3)—O(4)	1.428 (3)	C(5)—O(4)	1.369 (3)
C(5)—C(6)	1.398 (4)	C(6)—O(7)	1.368 (3)
C(8)—O(7)	1.422 (3)	C(8)—C(9)	1.500 (4)
C(10)—C(9)	1.384 (3)		
C(11)—C(12)	1.372 (3)	C(11)—C(9)	1.385 (4)
C(13)—C(6)	1.374 (3)	C(13)—C(14)	1.392 (4)
C(14)—C(15)	1.361 (4)	C(15)—C(16)	1.391 (4)
C(16)—C(5)	1.378 (4)	C(10)—H(10)	0.93 (3)
C(2')—O(1)—C(2)	111.1 (3)	O(1)—C(2)—C(3)	110.0 (2)
C(2)—C(3)—O(4)	109.2 (2)	C(3)—O(4)—C(5)	118.0 (2)
O(4)—C(5)—C(6)	115.9 (3)	O(4)—C(5)—C(16)	124.8 (3)
C(6)—C(5)—C(16)	119.3 (3)	C(5)—C(6)—O(7)	115.2 (2)
C(5)—C(6)—C(13)	120.1 (3)	O(7)—C(6)—C(13)	124.7 (3)
C(6)—O(7)—C(8)	117.6 (2)	O(7)—C(8)—C(9)	108.2 (2)
C(8)—C(9)—C(10)	119.8 (3)	C(8)—C(9)—C(11)	121.7 (3)
C(10)—C(9)—C(11)	118.5 (3)	C(9)—C(10)—C(9')	121.6 (3)
C(12)—C(11)—C(9)	120.6 (3)	C(11)—C(12)—C(11')	120.3 (4)
C(6)—C(13)—C(14)	119.8 (3)	C(13)—C(14)—C(15)	120.3 (3)
C(14)—C(15)—C(16)	120.1 (3)	C(5)—C(16)—C(15)	120.3 (3)

Discussion. Final atom parameters are given in Table 1.* Bond lengths and angles (Table 2) exhibit no anomalies except that some $C_{sp^2}-C_{sp^2}$ distances [mean 1.38 (1) \AA] appear somewhat shortened, probably due to anisotropic motion of atoms involved. Widened angles $O(4)-C(5)-C(16) = 124.8 (3)$ and $O(7)-C(6)-C(13) = 124.7 (3)^\circ$ [compensated by $O(4)-C(5)-C(6) = 115.9 (3)$ and $C(5)-C(6)-O(7) = 115.2 (2)^\circ$, respectively] are a common feature of 'benzo-crowns' (e.g. Bush & Truter, 1972; Hanson, Hughes & Truter, 1976; Hanson, 1978).

The unique sequence $ag^- a asa ag^+ a$ [starting with $C(2')-O(1)-C(2)-C(3)$] of nearly ideal endocyclic

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, and torsion angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38484 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

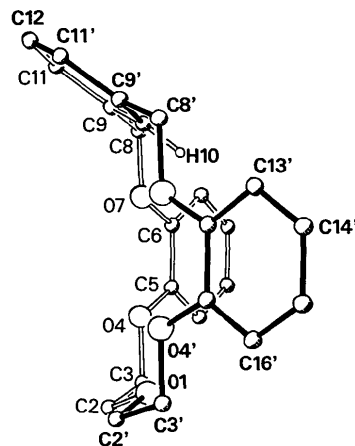


Fig. 1. A perspective view of the macrocycle; radii are arbitrary. Primed and unprimed atoms are related by a mirror running through O(1), C(10), H(10), C(12) [and H(12)]. The 'dentist's chair' conformation is easily recognized on rotating the figure through 90° clockwise.

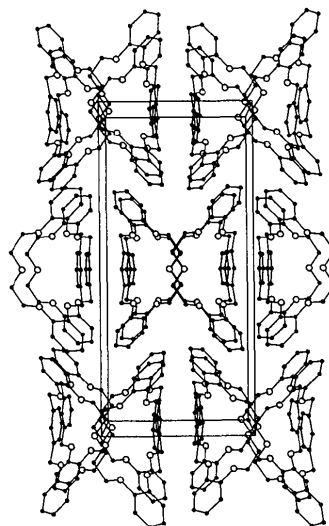


Fig. 2. The packing of molecules viewed along c .

torsion angles* characterizes the strain-free conformation of the macrocycle (Fig. 1) in which the five O atoms and C(10) are co-planar to within $\pm 0.09 \text{\AA}$ [cf. 'co-planarity' of six O atoms in uncomplexed '18-crown-6' (Maverick, Seiler, Schweizer & Dunitz, 1980) $\pm 0.36 \text{\AA}$]. Neglecting C(10), hetero-atoms are co-planar to within as little as $\pm 0.008 (2) \text{\AA}$, C(10) then deviating by $0.185 (2)$ and H(10) by $-0.54 (2) \text{\AA}$. The latter plane subtends dihedral angles of $53.7 (7)^\circ$ with aromatic moiety $A = C(9), C(10), C(9'), C(11), C(12)$ and $C(11')$ and of $35.8 (7)^\circ$ with $B = C(5), C(6)$ and $C(13)$ to $C(16)$ [$A/B = 61.2 (5)$, $B/B' = 71.6 (5)^\circ$].

* See deposit footnote.

There are no unusually close intermolecular contacts; the packing is illustrated in Fig. 2.

A more detailed discussion will be given when structural data of the pyridine analogue (2) are available.

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Structure of Tefludazine, 2-{4-[3-(4-Fluorophenyl)-6-(trifluoromethyl)-1-indanyl]piperazinyl}ethanol, C₂₂H₂₄F₄N₂O

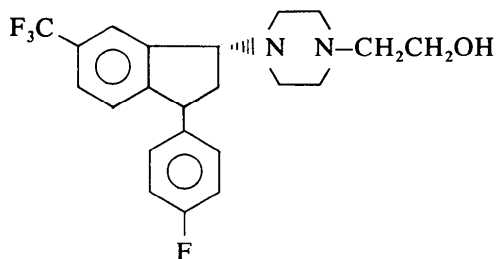
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Abstract. $M_r = 408.44$, monoclinic, $P2_1/c$, $a = 13.20$ (1), $b = 13.378$ (6), $c = 12.55$ (1) Å, $\beta = 112.60$ (8)°, $V = 2046$ (3) Å³, $Z = 4$, $D_m = 1.328$ Mg m⁻³, $D_x = 1.326$ (2) Mg m⁻³, Mo K α , $\lambda = 0.71069$ Å, $\mu = 0.101$ mm⁻¹, $F(000) = 856$, room temperature, $R = 0.055$ for 2191 diffractometer-collected reflections. The two substituents of the indan ring system are situated *trans* to each other.

Introduction. The potent and long-acting neuroleptic compound tefludazine was developed by systematic variation of structural components (Bøgesø, 1983). The structure determination was undertaken in order to confirm the relative configuration proposed on the basis of the NMR spectra.



Experimental. Crystals grown by Dr K. P. Bøgesø, H. Lundbeck & Co A/S, from a mixture of diisopropyl ether and hexane, unit-cell parameters from least-

squares refinement of the θ angles ($|\theta| \leq 15^\circ$) for 24 reflections, Nonius CAD-3 diffractometer, D_m by flotation, irregularly shaped crystal, of approximate size 0.30 × 0.34 × 0.36 mm, mounted in silicon grease in a glass capillary, ω -scan technique, 3974 reflections measured in one quadrant with $(\sin \theta)/\lambda \leq 0.599$ Å⁻¹, $-13 \leq h \leq 15$, $-15 \leq k \leq 0$, $-14 \leq l \leq 0$, 2192 with $I_{net} \geq 3.0\sigma(I)$, where σ is the standard deviation from counting statistics; intensity of the three standard reflections did not vary significantly, no absorption correction; structure solved with *MULTAN77* (Main, Lessinger, Woolfson, Germain & Declercq, 1977), trial structure refined with *XRAY72* (Stewart, Kruger, Ammon, Dickinson & Hall, 1972); final cycles of refinement, based on $|F|$, included one scale factor and positional and anisotropic thermal parameters for all non-H atoms; H atoms located in difference electron density map included in refinement as a fixed contribution in calculated positions (C–H = 1.0 Å), with a common thermal parameter ($B = 4.0$ Å²); position of the hydroxyl hydrogen atom taken directly from the difference electron density map; unit weights [weight analysis showed them to be satisfactory] were used, final $R = R_w = 0.055$, $S = 1.53$, ratios of maximum and average shift to error in the last refinement cycle were 0.32 and 0.12, respectively, minimum and maximum heights in final difference Fourier synthesis were -0.2 and 0.3 e Å⁻³, respectively, X-ray scattering factors for H from Stewart Davidson & Simpson