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N,*N*′,*N*″,*N*‴-Bis(*m*-xylyl)bis(4,13-diaza-1,7,10-trioxacyclopentadecane): a Multidentate Macrocyclic Complexing Agent Containing Two 15-Crown-5 Rings

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Abstract. $C_{36}H_{56}N_4O_6$, $M_r = 640{\cdot}84$, orthorhombic, *Pbca*, $a = 22{\cdot}460$ (9), $b = 17{\cdot}520$ (8), $c = 9{\cdot}04$ (1) Å, $V = 3557{\cdot}2$ Å^3, $Z = 4$, $D_x = 1{\cdot}20$ g cm^{-3}, Mo $K\alpha$ radiation, $\lambda = 0{\cdot}71069$ Å, $\mu = 0{\cdot}76$ cm^{-1}, room temperature, $R = 0.073$ for 970 reflections with $F > 6\sigma(F)$. The molecule has symmetry $\bar{1}$ (*i*). In the crystal the central cavity of the whole molecule, bounded by two 15-membered rings, is nearly closed and could not accommodate a cation; a pair of C atoms come within 2·3 Å of the inversion centre. All the O atoms point towards the centres of their 15-membered rings. Substantial conformational change must occur when this multidentate chelating agent forms a complex with a cation.

Experimental. The title compound was prepared in 30% yield from α,α-dibromo-*m*-xylene and 1,10-diaza-4,7,13-trioxacyclopentadecane, in a solution of K_2CO_3 and MeCN, and recrystallized from dichloromethane and heptane. Crystals were provided by Professor S. Mageswaren and Professor I. O. Sutherland, Liverpool University.

Crystal 0·28 × 0·28 × 0·12 mm, preliminary oscillation and Weissenberg photographs gave unit cell and space group. Stoe Stadi-2 diffractometer, graphite monochromator, *a* and *b* parameters refined from 12 reflections with $14 \le 2\theta \le 24°$, Layers $l =$ 0–8, with h 0–24, k 0–18, $2\theta_{max} = 50°$, recorded using ω scans, one standard reflection per layer, no significant variations in intensity. 2791 measured reflections, 2335 independent reflections, R_{int} 0·02. Lorentz and polarization corrections. Structure solved by direct methods (*SHELX*86, Sheldrick, 1986) and difference Fourier maps. Least-squares refinement, on F, using 970 reflections with $F > 6\sigma(F)$, since data was rather weak. Most H atoms located in difference Fourier maps; all H atoms, placed at calculated positions, included in F with $U = 0{\cdot}05$ Å^2, but not refined. Anisotropic vibration

Table 1. *Fractional atomic coordinates with e.s.d.'s in parentheses*

$U_{eq} = (U_{11} + U_{22} + U_{33})/3$.

	x	*y*	*z*	U_{eq} (Å^2)
N(1)	0·1328 (3)	0·1506 (4)	0·1782 (6)	0·0471 (46)
N(2)	−0·1172 (3)	0·0286 (3)	0·2508 (7)	0·0408 (44)
O(1)	0·0155 (3)	0·2276 (3)	0·1560 (7)	0·0597 (41)
O(2)	−0·1060 (2)	0·1998 (3)	0·1466 (6)	0·0581 (41)
O(3)	0·0446 (3)	0·0435 (3)	0·3086 (6)	0·0435 (39)
C(1)	0·1026 (3)	0·1999 (4)	0·2855 (8)	0·0480 (58)
C(2)	0·0658 (4)	0·2615 (5)	0·2194 (10)	0·0706 (71)
C(3)	−0·0254 (4)	0·2783 (5)	0·0952 (11)	0·0517 (69)
C(4)	−0·0758 (4)	0·2347 (5)	0·0284 (9)	0·0578 (67)
C(5)	−0·1457 (4)	0·1415 (5)	0·0992 (10)	0·0715 (75)
C(6)	−0·1611 (3)	0·0884 (4)	0·2247 (9)	0·0499 (58)
C(7)	−0·0609 (3)	0·0564 (4)	0·3148 (8)	0·0508 (60)
C(8)	−0·0094 (4)	0·0135 (5)	0·2541 (10)	0·0663 (75)
C(9)	0·0942 (4)	0·0199 (4)	0·2228 (10)	0·0543 (67)
C(10)	0·1446 (3)	0·0742 (4)	0·2392 (9)	0·0457 (61)
C(11)	0·1866 (3)	0·1866 (4)	0·1197 (8)	0·0325 (50)
C(12)	0·2052 (2)	0·1537 (2)	−0·0262 (5)	0·0389 (21)
C(13)	0·2619 (2)	0·1716 (2)	−0·0788 (5)	0·0463 (22)
C(14)	0·2807 (2)	0·1440 (2)	−0·2158 (5)	0·0535 (24)
C(15)	0·2427 (2)	0·0985 (2)	−0·3003 (5)	0·0508 (23)
C(16)	0·1860 (2)	0·0805 (2)	−0·2477 (5)	0·0413 (20)
C(17)	0·1673 (2)	0·1081 (2)	−0·1107 (5)	0·0402 (22)
C(18)	−0·1411 (3)	−0·0315 (4)	0·3399 (8)	0·0778 (65)

Table 2. *Selected molecular geometry* (*in* Å *and* °)

Mean bond lengths

C—C	(not aromatic)	1·490 (6)
C—N		1·459 (12)
C—O		1·407 (17)

Torsion angles

N(1)—C(1)—C(2)—O(1)	71·4 (8)
N(2)—C(7)—C(8)—O(3)	176·3 (6)
O(1)—C(3)—C(4)—O(2)	67·1 (8)
O(2)—C(5)—C(6)—N(2)	−82·9 (8)
O(3)—C(9)—C(10)—N(1)	−65·7 (8)
C(1)—C(2)—O(1)—C(3)	176·9 (7)
C(3)—C(4)—O(2)—C(5)	−165·2 (7)
C(4)—C(3)—O(1)—C(2)	178·6 (7)
C(5)—C(6)—N(2)—C(7)	70·8 (8)
C(6)—C(5)—O(2)—C(4)	160·7 (7)
C(7)—C(8)—O(3)—C(9)	−162·9 (6)
C(8)—C(7)—N(2)—C(6)	−143·6 (7)
C(9)—C(10)—N(1)—C(1)	85·8 (8)
C(10)—C(9)—O(3)—C(8)	156·6 (7)
C(10)—N(1)—C(1)—C(2)	−153·9 (7)

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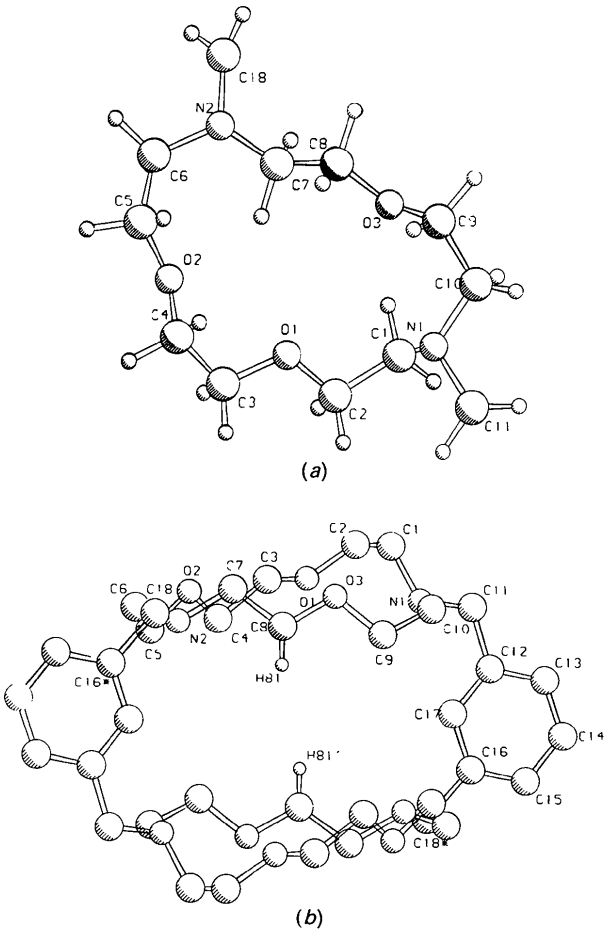

Fig. 1. The numbering scheme for the title compound. (*a*) View of one constituent 15-membered ring; C(18) and C(11) are joined to (centrosymmetrically related) benzene rings. (*b*) View of the whole molecule (hydrogens omitted for clarity) showing that in the crystal conformation there is no large central cavity. [Both views drawn by *PLUTO* (Motherwell, 1976).]

parameters refined for all C, N and O atoms; benzene rings constrained as regular hexagons with C—C = 1·395 Å. $R = 0{\cdot}073$, $wR = 0{\cdot}065$, 174 parameters, $S = 1{\cdot}261$, weight $= 1/[\sigma^2(F) + 0{\cdot}00065\,(F)^2]$, maximum shift/e.s.d. $\leq 0{\cdot}4$, maximum and minimum electron density in final difference map 0·37, $-0{\cdot}41$ e Å^{-3}. The relatively high value of the final R factor and the need to omit many of the weaker intensities in the refinement reflects the overall weakness of the diffraction data obtainable. Refinement with *SHELX* (Sheldrick, 1976) using atomic scattering factors therein. Table 1 gives atom positions* and Table 2 selected information on bond lengths and angles. Views of the molecule are shown in Fig. 1.

Related literature. Like other crown ethers the compound is an efficient and selective multidentate chelating agent, *e.g.* $H_3N^+—CH_2—CH_2—N^+H_3$ are accommodated (Sutherland, 1985).

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* 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 54273 (6 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 4′-Demethyl-9-(3″-thymidyl)-epipodophyllotoxin

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Abstract. {5*R*-[5α,5aβ,8aα,9β(*R**)]}-5,5a,8a,9-Tetrahydro-5-(4-hydroxy-3,5-dimethoxyphenyl)-9-{[1″-(2″-deoxy-β-D-ribofuranosyl)-5‴-methyluracil]oxy}-furo[3′,4′:6,7]naphtho[2,3-*d*]-1,3-dioxol-6(5a*H*)-one, $C_{31}H_{32}N_2O_{12}$, $M_r = 624{\cdot}60$, monoclinic, $P2_1$, $a = 6{\cdot}724$ (1), $b = 16{\cdot}251$ (2), $c = 13{\cdot}7598$ (8) Å, $\beta = 93{\cdot}292$ (9)°, $V = 1501{\cdot}0$ (3) Å^3, $Z = 2$, $D_x = 1{\cdot}382$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 8{\cdot}63$ cm^{-1}, $F(000) = 656$, $T = 296$ K, final $R = 0{\cdot}057$ for 1679 unique reflections.

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