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Structure of the *cis–transoid–cis* Isomer of 2,3,11,12-Tetra-anisyl-18-crown-6: *rel*-(2*R*,3*S*,11*S*,12*R*)-2,3,11,12-Tetrakis(4-methoxyphenyl)-1,4,7,10,13,16-hexaoxacyclooctadecane

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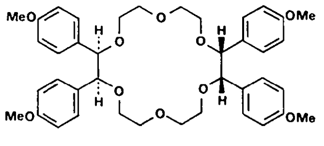
Abstract. $C_{40}H_{48}O_{10}$, $M_r = 689$, monoclinic, $P2_1/a$, $a = 8{\cdot}238\,(2)$, $b = 20{\cdot}616\,(7)$, $c = 10{\cdot}883\,(2)$ Å, $\beta = 99{\cdot}99\,(2)°$, $V = 1820$ Å^3, $Z = 2$, $D_x = 1{\cdot}26$ Mg m^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1{\cdot}54178$ Å, $\mu = 0{\cdot}70$ mm^{-1}, $F(000) = 736$, room temperature, $R = 0{\cdot}040$ for 1636 observed reflections with $|F_o| > 3\sigma(|F_o|)$. The 18-membered ring of the title compound (2) adopts a conformation with two *anti* and four *gauche* OCH_2CH_2O units which is reminiscent of the conformation of 18-crown-6: all four anisyl groups are axial: the vicinal groups have an *anti* relationship to each other.

Introduction. The synthesis of the *cis–cisoid–cis* isomer (1) (Pears, Shahriari-Zavareh, Stoddart, Crosby, Allwood, Slawin & Williams, 1988; paper I)* from a reaction of *meso*-bis(4-methoxyphenyl)ethane-1,2-diol with diethyleneglycol bistosylate in dimethylformamide in the presence of sodium hydride as base was accompanied (Pears, 1985) by the formation of the configurational diastereoisomer (2) with the *cis–transoid–cis* configuration. Here, we describe the crystal structure analysis of (2) and compare our findings with those already published (Weber, Sheldrick, Burgemeister, Dietl, Mannschreck & Merz, 1984; Blasius, Rausch, Andreetti & Rebizant, 1984) for *cis–transoid–cis*-2,3,11,12-tetraphenyl-18-crown-6 (7),* prior to investigating (Pears, Shahriari-Zavareh, Stoddart, Crosby, Allwood & Williams, 1988) the ability of (2) to form a crystalline adduct with ammonia–borane.



(2)

Experimental. Single crystals of (2), suitable for X-ray crystallography, were grown (Pears, 1985) at room

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[*] In order to avoid repetition in the presentation of configurational formulae, the reader is referred to Fig. 1 in paper I.

[*] See previous footnote.

temperature from dichloromethane layered with *n*-pentane and had m.p. 467–469 K. Crystal size 0·45 × 0·28 × 0·13 mm. Refined unit-cell parameters obtained by centring 15 reflections. Nicolet *R*3*m* diffractometer. 1869 independent reflections ($\theta \leq 50°$) measured, Cu $K\alpha$ radiation (graphite monochromator), ω scan. 1636 [$|F_o| > 3\sigma(|F_o|)$] considered observed, index range h −8/8, k 0/20, l 0/10; two check reflections measured every 50 reflections, net count constant, Lorentz and polarization corrections, no absorption correction. Structure solved by direct methods; non-hydrogen atoms refined anisotropically; positions of H atoms calculated (C—H = 0·96 Å); H atoms assigned isotropic thermal parameters, $U(\text{H}) = 1{\cdot}2\ U_{\text{eq}}(\text{C})$, allowed to ride on parent C atoms. The methoxy CH_3 groups refined as rigid bodies. An empirical extinction correction was applied ($g = 0{\cdot}00400$). Refinement using F magnitudes by block-cascade full-matrix least squares; $R = 0{\cdot}040$; $wR = 0{\cdot}046$ [$w^{-1} = \sigma^2(F) + 0{\cdot}0011F^2$]. $(\Delta/\sigma)_{\text{max}} = 0{\cdot}20$; residual electron density in difference map within −0·14 and 0·15 e Å^{-3}; atomic scattering factors and dispersion corrections from *International Tables for X-ray Crystallography* (1974). Computations carried out on an Eclipse S140 computer using the *SHELXTL* program system (Sheldrick, 1983).

Discussion. Table 1* lists the fractional atomic coordinates of the non-hydrogen atoms, Table 2 the bond lengths and angles, and selected torsion angles.

The structure of (2) is illustrated in Fig. 1 which includes the atomic numbering scheme. There is a crystallographic centre of symmetry at the centre of the macrocyclic ring. Fig. 2 presents a view of (2) approximately at right angles to that shown in Fig. 1; it emphasizes the fact that the four anisyl residues lie in approximately parallel planes to each other. The two pairs of vicinal anisyl groups are axial and antiperiplanar [171·0 (2)°] to each other with those at C(8) and C(8′) being directed over the opposite faces of the macrocyclic ring which adopts a conformation (with two *anti* and four *gauche* bismethylenedioxy units) closely matching that of 18-crown-6 (Dunitz & Seiler, 1974) and the *cis–transoid–cis* isomer (7) of 2,3,11,12-tetraphenyl-18-crown-6 (Weber *et al.*, 1984; Blasius *et al.*, 1984). In the case of all four anisyl groups, the methoxy functions are aligned approximately coplanar (Makriyannis & Fesik, 1982; Kruse & Cha, 1982; Mersh, Saunders & Matlin, 1983) with their associated rings.

Table 1. *Atom coordinates* (×10^4) *and equivalent isotropic temperature factors* (Å^2 × 10^3)

	x	y	z	U_{eq}*
O(1)	8307 (2)	−1713 (1)	9158 (2)	53 (1)
C(2)	6596 (3)	−1566 (1)	8915 (3)	58 (1)
C(3)	6164 (3)	−952 (1)	8178 (2)	58 (1)
O(4)	6479 (2)	−401 (1)	8955 (2)	50 (1)
C(5)	5979 (3)	176 (1)	8290 (2)	57 (1)
C(6)	6560 (3)	751 (1)	9095 (3)	53 (1)
O(7)	8286 (2)	813 (1)	9195 (1)	47 (1)
C(8)	8991 (3)	1306 (1)	10036 (2)	45 (1)
C(9)	10801 (3)	1323 (1)	9863 (2)	46 (1)
C(10)	8815 (3)	1159 (1)	11366 (2)	43 (1)
C(11)	8075 (3)	1594 (1)	12057 (2)	46 (1)
C(12)	7915 (3)	1463 (1)	13290 (2)	50 (1)
C(13)	8495 (3)	885 (1)	13822 (2)	47 (1)
C(14)	9231 (3)	445 (1)	13142 (2)	53 (1)
C(15)	9392 (3)	581 (1)	11930 (2)	51 (1)
C(16)	11041 (3)	1570 (1)	8602 (2)	45 (1)
C(17)	10567 (3)	2191 (1)	8185 (2)	53 (1)
C(18)	10807 (3)	2403 (1)	7033 (2)	58 (1)
C(19)	11527 (3)	2005 (1)	6258 (2)	52 (1)
C(20)	12029 (3)	1393 (1)	6655 (3)	57 (1)
C(21)	11788 (3)	1185 (1)	7823 (2)	53 (1)
O(22)	8382 (2)	705 (1)	15015 (2)	68 (1)
C(23)	7642 (4)	1145 (2)	15750 (3)	73 (1)
O(24)	11658 (3)	2258 (1)	5122 (2)	73 (1)
C(25)	12326 (4)	1848 (2)	4271 (3)	76 (1)

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

Table 2. *Bond lengths* (Å), *angles* (°) *and selected torsion angles* (°)

O(1)–C(2)	1·421 (3)	O(1)–C(9′)	1·432 (3)
C(2)–C(3)	1·506 (4)	C(3)–O(4)	1·414 (3)
O(4)–C(5)	1·415 (3)	C(5)–C(6)	1·503 (4)
C(6)–O(7)	1·412 (3)	O(7)–C(8)	1·422 (3)
C(8)–C(9)	1·535 (3)	C(8)–C(10)	1·511 (3)
C(9)–C(16)	1·508 (3)	C(9)–O(1′)	1·432 (3)
C(10)–C(11)	1·378 (3)	C(10)–C(15)	1·386 (3)
C(11)–C(12)	1·398 (3)	C(12)–C(13)	1·373 (3)
C(13)–C(14)	1·377 (4)	C(13)–O(22)	1·369 (3)
C(14)–C(15)	1·378 (4)	C(16)–C(17)	1·391 (3)
C(16)–C(21)	1·381 (4)	C(17)–C(18)	1·375 (4)
C(18)–C(19)	1·382 (4)	C(19)–C(20)	1·374 (4)
C(19)–O(24)	1·364 (3)	C(20)–C(21)	1·388 (4)
O(22)–C(23)	1·416 (4)	O(24)–C(25)	1·433 (4)

C(2)–O(1)–C(9′)	112·5 (2)	O(1)–C(2)–C(3)	114·5 (2)
C(2)–C(3)–O(4)	110·8 (2)	C(3)–O(4)–C(5)	111·3 (2)
O(4)–C(5)–C(6)	109·2 (2)	C(5)–C(6)–O(7)	109·3 (2)
C(6)–O(7)–C(8)	114·1 (2)	O(7)–C(8)–C(9)	103·6 (2)
O(7)–C(8)–C(10)	112·2 (2)	C(9)–C(8)–C(10)	112·3 (2)
C(8)–C(9)–C(16)	113·8 (2)	C(8)–C(9)–O(1′)	107·8 (2)
C(16)–C(9)–O(1′)	110·9 (2)	C(8)–C(10)–C(11)	121·2 (2)
C(8)–C(10)–C(15)	120·9 (2)	C(11)–C(10)–C(15)	117·9 (2)
C(10)–C(11)–C(12)	121·5 (2)	C(11)–C(12)–C(13)	119·3 (2)
C(12)–C(13)–C(14)	119·9 (2)	C(12)–C(13)–O(22)	124·0 (2)
C(14)–C(13)–O(22)	116·1 (2)	C(13)–C(14)–C(15)	120·4 (2)
C(10)–C(15)–C(14)	121·0 (2)	C(9)–C(16)–C(17)	122·2 (2)
C(9)–C(16)–C(21)	120·5 (2)	C(17)–C(16)–C(21)	117·3 (2)
C(16)–C(17)–C(18)	120·9 (2)	C(17)–C(18)–C(19)	120·7 (2)
C(18)–C(19)–C(20)	119·5 (2)	C(18)–C(19)–O(24)	115·8 (2)
C(20)–C(19)–O(24)	124·7 (2)	C(19)–C(20)–C(21)	119·2 (3)
C(16)–C(21)–C(20)	122·3 (2)	C(13)–O(22)–C(23)	117·9 (2)
C(19)–O(24)–C(25)	117·4 (2)		

C(9′)–O(1)–C(2)–C(3)	76·9 (3)	O(7)–C(8)–C(9)–O(1′)	−168·8 (2)
O(1)–C(2)–C(3)–O(4)	−79·2 (3)	C(8)–C(9)–O(1′)–C(2′)	156·7 (3)
C(2)–C(3)–O(4)–C(5)	−176·3 (2)	O(7)–C(8)–C(9)–C(16)	67·8 (2)
C(3)–O(4)–C(5)–C(6)	−171·8 (2)	C(10)–C(8)–C(9)–C(16)	−171·0 (2)
O(4)–C(5)–C(6)–O(7)	72·3 (3)	C(10)–C(8)–C(9)–O(1′)	−47·6 (2)
C(5)–C(6)–O(7)–C(8)	−174·2 (2)	C(9)–C(8)–C(10)–C(15)	−60·9 (3)
C(6)–O(7)–C(8)–C(9)	−174·0 (2)	C(8)–C(9)–C(16)–C(17)	60·8 (3)

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* Lists of structure-factor amplitudes, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44785 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

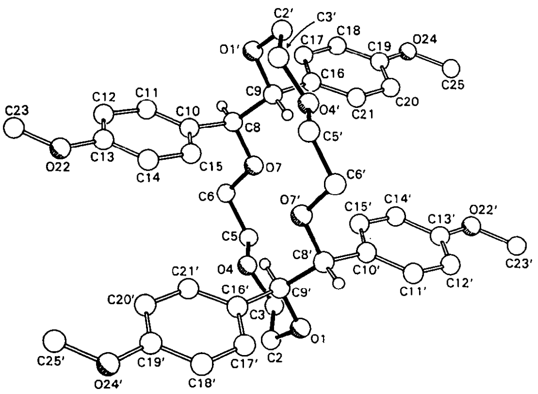

Fig. 1. Ball-and-stick representation of the structure of (2) showing the atomic numbering scheme. The 18-crown-6 ring is highlighted with solid C—C and C—O bonds.

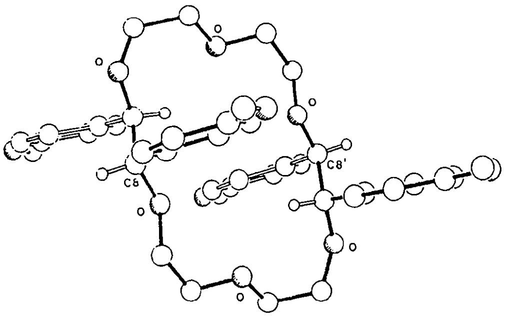

Fig. 2. A ball-and-stick representation of the structure of (2) looking down on the 18-membered ring. The macrocyclic ring, together with the two nearest anisyl groups, is highlighted with solid C—C and C—O bonds.

There are no contacts of less than normal van der Waals distances between symmetry-related molecules.

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A 1:2 Adduct Between the *cis–transoid–cis* Isomer of 2,3,11,12-Tetra-anisyl-18-crown-6 and Ammonia–Borane

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(*Received* 7 *September* 1987; *accepted* 17 *February* 1988)

Abstract. *rel*-(2*R*,3*S*,11*S*,12*R*)-2,3,11,12-Tetrakis(4-methoxyphenyl)-1,4,7,10,13,16-hexaoxacyclooctadecane–ammonia–borane (1/2/2), $C_{40}H_{48}O_{10}.B_2H_6N_2H_6$, $M_r = 751$, monoclinic, $P2_1/c$, $a = 8{\cdot}224\,(2)$, $b = 8{\cdot}848\,(3)$, $c = 28{\cdot}47\,(1)$ Å, $\beta = 90{\cdot}56\,(3)°$, $V = 2072$ Å^3, $Z = 2$, $D_x = 1{\cdot}21$ Mg m^{-3}, $\lambda(\text{Cu}\ K\alpha) = 1{\cdot}54178$ Å, $\mu = 0{\cdot}65$ mm^{-1}, $F(000) = 808$, room temperature, $R = 0{\cdot}051$ for 1730 unique observed

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