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

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Abstract. *rel*-(2*R*,3*S*,11*R*,12*S*)-2,3,11,12-Tetrakis(4-methoxyphenyl)-1,4,7,10,13,16-hexaoxacyclooctadecane–ammonia–borane (1/1/1), $C_{40}H_{48}O_{10}.BH_3$-NH_3, $M_r = 720$, monoclinic, $P2_1/n$, $a = 11{\cdot}078$ (4), $b = 18{\cdot}010$ (2), $c = 20{\cdot}639$ (9) Å, $\beta = 101{\cdot}07$ (3)°, $V = 4041$ Å^3, $Z = 4$, $D_x = 1{\cdot}19$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 0{\cdot}65$ mm^{-1}, $F(000) = 1544$, room temperature, $R = 0{\cdot}045$ for 3503 unique observed reflections with $|F_o| > 3\sigma(|F_o|)$. With BH_3NH_3, the title receptor (1) forms an extremely strong 1:1 adduct which does not dissociate during chromatography on silica gel: the substrate is hydrogen bonded through its NH_3 group to the nearer oxygen atoms on the unsubstituted face of (1), the macrocycle of which adopts an all-*gauche* conformation: the unsubstituted face carries two equatorial and two protective axial anisyl substituents.

Introduction. The ammonia–borane complex (BH_3-NH_3) is an excellent substrate (Colquhoun, Stoddart & Williams, 1986) for forming crystalline adducts with 18-crown-6 (Colquhoun, Jones, Maud, Stoddart & Williams, 1984) and its derivatives (Allwood, Shahriari-Zavareh, Stoddart & Williams, 1984; Alston, Stoddart, Wolstenholme, Allwood & Williams, 1985; Shahriari-Zavareh, Stoddart, Williams, Allwood & Williams, 1985). In some cases, adducts of a face-to-face type have been isolated with a 1:1 stoichiometry and, in other cases, two molecules of the BH_3NH_3 complex are bound simultaneously to opposite faces of the macrocyclic polyether ring, affording a 2:1 adduct. In view of the fact that the *cis–cisoid–cis* isomer (1)* of 2,3,11,12-tetra-anisyl-18-crown-6 exhibits (Pears, 1985) very much slower chromatographic mobility on silica gel than does the *cis–transoid–cis* isomer (2), we decided to investigate the ability of (1) to form an adduct with BH_3NH_3. We were to witness a remarkable result. Not only does (1) form a crystalline 1:1 adduct with BH_3NH_3, but this adduct does not dissociate and remains intact during chromatography on silica gel using ethyl acetate–dichloromethane as eluant. Indeed, the [(1).BH_3NH_3] adduct is eluted in advance of the free macrocycle (1). These observations indicate that the *cis–cisoid–cis* isomer (1) of 2,3,11,12-tetra-anisyl-18-crown-6 forms an extremely strong adduct with BH_3NH_3. Here, we report on the solid-state structure of [(1).BH_3NH_3] and discuss the result in the context of the quite amazing chromatographic properties described above.

Experimental. Ammonia–borane (22 mg) was added to (1) (50 mg) dissolved in dichloromethane (5 ml). The solvent was removed under vacuum to afford a white solid. This was dissolved in dichloromethane which was layered with *n*-pentane. After 2 days, crystals of [(1).BH_3NH_3] (found: C 66·8; H 7·7; N 1·8%. C_{40}-$H_{54}O_{10}NB$ requires C 66·8; H 7·7; N 1·7%), which had m.p. 428–431 K, were isolated and were found to be suitable for X-ray structural investigation (Pears, 1985). Crystal size 0·83 × 0·48 × 0·33 mm. Refined unit-cell parameters obtained by centring 16 reflections. Nicolet *R*3*m* diffractometer. 4123 independent reflections ($\theta \leq 50°$) measured, Cu $K\alpha$ radiation (graphite monochromator), ω scan. 3503 [$|F_o| > 3\sigma(|F_o|)$] considered observed, index range h −10/10, k 0/17, l 0/20; two check reflections measured every 50 reflections, net count constant; data brought to uniform scale, Lorentz and polarization corrections, no absorption correction. Structure solved by direct methods; non-hydrogen atoms refined anisotropically; positions of NH_3 and BH_3 hydrogen atoms clearly resolved in ΔF

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* In order to avoid repetition in the presentation of configurational formulae, the reader is referred to formula (1) and Fig. 1 of Pears, Shahriari-Zavareh, Stoddart, Crosby, Allwood, Slawin & Williams (1988).

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map, both these groups and the methoxy CH_3 groups refined as rigid bodies (B—H = 1·08, N—H = 0·96 Å); remaining H-atom positions calculated (C—H = 0·96 Å), assigned isotropic thermal parameters, $U(\mathrm{H}) = 1{\cdot}2\,U_{\mathrm{eq}}(X)$ (X = C, N, B) allowed to ride on parent X atoms. An empirical extinction correction was applied [$g = 0{\cdot}0029\,(3)$]. Refinement using F magnitudes by block-cascade full-matrix least squares; $R = 0{\cdot}045$; $wR = 0{\cdot}052$ [$w^{-1} = \sigma^2(F) + 0{\cdot}0007F^2$]. $(\Delta/\sigma)_{\max} = 0{\cdot}09$; residual electron density in difference map within −0·14 and +0·20 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 [(1).BH_3NH_3] is illustrated in Fig. 1 which includes the atomic numbering scheme. Fig. 2 shows space-filling representations of the 1:1 adduct viewed from opposite sides of the macrocyclic ring.

Whereas in the free macrocycle (1) there was an absence of local symmetry in the 18-membered ring, in [(1).BH_3NH_3] the geometry of the macrocycle adopts (Fig. 1) the classical all-*gauche* conformation with local pseudo-D_{3d} symmetry (Wipff, Weiner & Kollman, 1982; Wipff, Kollman & Lehn, 1983). One pair of *cisoid* anisyl groups at C(9) and C(17) are axial and the other pair at C(8) and C(18) are equatorial. Both pairs of *cis*-anisyl groups have a *gauche* relationship. The BH_3NH_3 is bound (Fig. 2*a*) in a perching mode (Trueblood, Knobler, Lawrence & Stevens, 1982) to the unsubstituted face of the macrocycle, the two axial anisyl groups being twisted so as to obscure approach of a second substrate to the opposite tetra-substituted face.

The NH_3 group in BH_3NH_3 is hydrogen bonded to O(4), O(10) and O(16) with N···O distances of 2·90 (1), 2·95 (1) and 2·97 (1) Å, respectively. The corresponding N—H···O angles at H(*a*), H(*c*) and H(*b*) respectively are 179, 175 and 177°. The angles between the COC planes and HO vectors are 7, 12 and 16° for H(*a*), H(*c*) and H(*b*) respectively. The N···O distances to the O atoms on the opposite face, O(1), O(7) and O(13), are 3·19 (1), 3·31 (1) and 3·10 (1) Å respectively. The N atom is displaced 1·23 (1) Å from the best mean plane of the six O atoms in the macrocycle and the angle between the B—N axis and this plane is 85·2 (1)°. The NH_3 and BH_3 groups in the substrate are staggered with respect to each other, the smallest H—N—B—H torsion angle being 55°.

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

	x	y	z	U_{eq}*
O(1)	12137 (1)	7929 (1)	−248 (1)	73 (1)
C(2)	13071 (2)	7746 (2)	307 (1)	87 (1)
C(3)	12982 (2)	6947 (2)	469 (1)	87 (1)
O(4)	11896 (2)	6830 (1)	712 (1)	86 (1)
C(5)	11692 (3)	6093 (2)	874 (2)	94 (1)
C(6)	10655 (3)	6069 (2)	1238 (1)	95 (1)
O(7)	9539 (2)	6254 (1)	805 (1)	75 (1)
C(8)	8516 (2)	6278 (1)	1130 (1)	69 (1)
C(9)	7412 (2)	6616 (1)	660 (1)	69 (1)
O(10)	7697 (1)	7386 (1)	604 (1)	72 (1)
C(11)	6658 (2)	7822 (1)	318 (1)	78 (1)
C(12)	7060 (2)	8601 (1)	239 (1)	73 (1)
O(13)	7815 (1)	8626 (1)	−237 (1)	63 (1)
C(14)	8253 (2)	9350 (1)	−324 (1)	66 (1)
C(15)	9093 (2)	9324 (1)	−807 (1)	70 (1)
O(16)	10204 (1)	8949 (1)	−511 (1)	68 (1)
C(17)	11064 (2)	8882 (1)	−939 (1)	65 (1)
C(18)	12288 (2)	8646 (1)	−506 (1)	70 (1)
C(19)	8217 (2)	5528 (1)	1389 (1)	65 (1)
C(20)	8230 (3)	4881 (1)	1024 (1)	81 (1)
C(21)	7928 (3)	4208 (2)	1261 (1)	81 (1)
C(22)	7585 (2)	4158 (2)	1865 (1)	70 (1)
C(23)	7590 (3)	4788 (1)	2238 (1)	76 (1)
C(24)	7912 (2)	5460 (1)	1999 (1)	73 (1)
C(25)	7059 (2)	6250 (1)	−4 (1)	68 (1)
C(26)	7771 (3)	6302 (2)	−480 (1)	85 (1)
C(27)	7413 (3)	5962 (2)	−1098 (1)	93 (1)
C(28)	6341 (3)	5566 (2)	−1231 (1)	94 (1)
C(29)	5616 (3)	5523 (2)	−774 (2)	104 (1)
C(30)	5967 (3)	5860 (2)	−169 (1)	87 (1)
C(31)	10631 (2)	8375 (1)	−1517 (1)	60 (1)
C(32)	10702 (3)	8597 (2)	−2145 (1)	81 (1)
C(33)	10382 (3)	8131 (2)	−2677 (1)	98 (1)
C(34)	9981 (3)	7432 (2)	−2593 (1)	84 (1)
C(35)	9856 (2)	7201 (2)	−1975 (2)	83 (1)
C(36)	10187 (2)	7675 (2)	−1439 (1)	72 (1)
C(37)	13338 (2)	8666 (1)	−884 (1)	65 (1)
C(38)	13954 (2)	9319 (1)	−957 (1)	76 (1)
C(39)	14907 (3)	9334 (2)	−1301 (1)	80 (1)
C(40)	15289 (2)	8702 (2)	−1565 (1)	71 (1)
C(41)	14693 (2)	8051 (2)	−1494 (1)	78 (1)
C(42)	13723 (2)	8041 (2)	−1162 (1)	79 (1)
O(43)	7295 (2)	3465 (1)	2053 (1)	92 (1)
C(44)	6990 (4)	3379 (2)	2675 (2)	119 (2)
O(45)	5922 (2)	5191 (1)	−1819 (1)	129 (1)
C(46)	6575 (4)	5261 (2)	−2323 (2)	150 (2)
O(47)	9724 (2)	7003 (1)	−3149 (1)	124 (1)
C(48)	9318 (4)	6276 (2)	−3073 (2)	156 (2)
O(49)	16261 (2)	8771 (1)	−1886 (1)	90 (1)
C(50)	16854 (3)	8109 (2)	−2026 (2)	105 (2)
N(1)	10163 (2)	8039 (1)	689 (1)	66 (1)
B(1)	10541 (3)	8557 (2)	1317 (2)	84 (1)

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

It is interesting to note that in [(1).BH_3NH_3] all three N—H···O hydrogen-bond distances are less than 3 Å. This is in contrast to the distances observed in all other 1:1 adducts with 18-crown-6 (Colquhoun *et al.*, 1984) and substituted 18-crown-6 derivatives (Allwood *et al.*, 1984; Alston *et al.*, 1985; Shahriari-Zavareh *et al.*, 1985) where at least two of the N···O distances are greater than 3 Å. This almost certainly reflects a stronger binding in [(1).BH_3NH_3] than in any of the previously characterized 1:1 adducts and is consistent with its unusual chromatographic behaviour. In this context, it is interesting to note that the *cis–cisoid–cis* isomer (6) of 2,3,11,12-tetraphenyl-18-crown-6 forms (Merz, Eichner & Tomahogh, 1981) a notably stronger

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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 44784 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

O(1)–C(2)	1·427 (3)	O(1)–C(18)	1·415 (3)
C(2)–C(3)	1·482 (5)	C(3)–O(4)	1·406 (4)
O(4)–C(5)	1·394 (3)	C(5)–C(6)	1·488 (5)
C(6)–O(7)	1·418 (3)	O(7)–C(8)	1·424 (3)
C(8)–C(9)	1·533 (3)	C(8)–C(19)	1·509 (4)
C(9)–O(10)	1·429 (3)	C(9)–C(25)	1·502 (4)
O(10)–C(11)	1·422 (3)	C(11)–C(12)	1·488 (4)
C(12)–O(13)	1·409 (3)	O(13)–C(14)	1·412 (3)
C(14)–C(15)	1·490 (4)	C(15)–O(16)	1·432 (3)
O(16)–C(17)	1·424 (3)	C(17)–C(18)	1·533 (3)
C(17)–C(31)	1·504 (3)	C(18)–C(37)	1·521 (4)
C(19)–C(20)	1·387 (4)	C(19)–C(24)	1·370 (4)
C(20)–C(21)	1·369 (4)	C(21)–C(22)	1·374 (4)
C(22)–C(23)	1·368 (4)	C(22)–O(43)	1·361 (3)
C(23)–C(24)	1·377 (4)	C(25)–C(26)	1·377 (4)
C(25)–C(30)	1·382 (4)	C(26)–C(27)	1·401 (4)
C(27)–C(28)	1·366 (4)	C(28)–C(29)	1·354 (5)
C(28)–O(45)	1·388 (3)	C(29)–C(30)	1·375 (4)
C(31)–C(32)	1·371 (4)	C(31)–C(36)	1·372 (4)
C(32)–C(33)	1·372 (4)	C(33)–C(34)	1·355 (4)
C(34)–C(35)	1·372 (4)	C(34)–O(47)	1·366 (3)
C(35)–C(36)	1·389 (4)	C(37)–C(38)	1·379 (4)
C(37)–C(42)	1·367 (4)	C(38)–C(39)	1·381 (4)
C(39)–C(40)	1·364 (4)	C(40)–C(41)	1·364 (4)
C(40)–O(49)	1·374 (3)	C(41)–C(42)	1·381 (4)
O(43)–C(44)	1·399 (5)	O(45)–C(46)	1·382 (5)
O(47)–C(48)	1·399 (4)	O(49)–C(50)	1·415 (4)
N(1)–B(1)	1·584 (4)		

C(2)–O(1)–C(18)	113·1 (2)	O(1)–C(2)–C(3)	109·6 (2)
C(2)–C(3)–O(4)	108·9 (2)	C(3)–O(4)–C(5)	114·6 (2)
O(4)–C(5)–C(6)	108·9 (2)	C(5)–C(6)–O(7)	109·9 (2)
C(6)–O(7)–C(8)	112·8 (2)	O(7)–C(8)–C(9)	108·9 (2)
O(7)–C(8)–C(19)	112·8 (2)	C(9)–C(8)–C(19)	112·0 (2)
C(8)–C(9)–O(10)	105·9 (2)	C(8)–C(9)–C(25)	116·1 (2)
O(10)–C(9)–C(25)	111·9 (2)	C(9)–O(10)–C(11)	113·1 (2)
O(10)–C(11)–C(12)	109·1 (2)	C(11)–C(12)–O(13)	109·6 (2)
C(12)–O(13)–C(14)	112·4 (2)	O(13)–C(14)–C(15)	109·3 (2)
C(14)–C(15)–O(16)	109·0 (2)	C(15)–O(16)–C(17)	113·4 (2)
O(16)–C(17)–C(18)	106·5 (2)	O(16)–C(17)–C(31)	113·2 (2)
C(18)–C(17)–C(31)	113·9 (2)	O(1)–C(18)–C(17)	108·4 (2)
O(1)–C(18)–C(37)	111·9 (2)	C(17)–C(18)–C(37)	111·9 (2)
C(8)–C(19)–C(20)	122·0 (2)	C(8)–C(19)–C(24)	120·9 (2)
C(20)–C(19)–C(24)	117·0 (2)	C(19)–C(20)–C(21)	121·2 (3)
C(20)–C(21)–C(22)	120·7 (3)	C(21)–C(22)–C(23)	118·9 (3)
C(21)–C(22)–O(43)	116·1 (2)	C(23)–C(22)–O(43)	125·0 (3)
C(22)–C(23)–C(24)	119·8 (3)	C(19)–C(24)–C(23)	122·3 (2)
C(9)–C(25)–C(26)	122·5 (2)	C(9)–C(25)–C(30)	120·5 (3)
C(26)–C(25)–C(30)	116·9 (2)	C(25)–C(26)–C(27)	121·3 (3)
C(26)–C(27)–C(28)	119·5 (3)	C(27)–C(28)–C(29)	120·0 (3)
C(27)–C(28)–O(45)	124·0 (3)	C(29)–C(28)–O(45)	116·0 (3)
C(28)–C(29)–C(30)	120·4 (3)	C(25)–C(30)–C(29)	121·9 (3)
C(17)–C(31)–C(32)	120·4 (2)	C(17)–C(31)–C(36)	121·9 (2)
C(32)–C(31)–C(36)	117·7 (2)	C(31)–C(32)–C(33)	121·7 (3)
C(32)–C(33)–C(34)	120·2 (3)	C(33)–C(34)–C(35)	119·6 (3)
C(33)–C(34)–O(47)	115·9 (3)	C(35)–C(34)–O(47)	124·5 (3)
C(34)–C(35)–C(36)	119·7 (3)	C(31)–C(36)–C(35)	120·9 (3)
C(18)–C(37)–C(38)	121·3 (2)	C(18)–C(37)–C(42)	121·7 (2)
C(38)–C(37)–C(42)	116·9 (3)	C(37)–C(38)–C(39)	121·1 (2)
C(38)–C(39)–C(40)	121·1 (3)	C(39)–C(40)–C(41)	118·5 (3)
C(39)–C(40)–O(49)	116·7 (2)	C(41)–C(40)–O(49)	124·8 (2)
C(40)–C(41)–C(42)	120·2 (3)	C(37)–C(42)–C(41)	122·2 (3)
C(22)–O(43)–C(44)	118·3 (2)	C(28)–O(45)–C(46)	118·6 (3)
C(34)–O(47)–C(48)	117·0 (3)	C(40)–O(49)–C(50)	117·4 (2)

C(18) O(1) C(2)–C(3)	−169·6 (2)	C(14)–C(15)–O(16)–C(17)	178·9 (2)
O(1)–C(2)–C(3)–O(4)	−68·6 (3)	C(15)–O(16)–C(17)–C(18)	166·9 (3)
C(2)–C(3)–O(4)–C(5)	179·0 (2)	O(16)–C(17)–C(18)–O(1)	64·8 (2)
C(3)–O(4)–C(5)–C(6)	169·5 (2)	C(2)–O(1)–C(18)–C(17)	−162·2 (2)
O(4)–C(5)–C(6)–O(7)	68·8 (3)	O(7)–C(8)–C(9)–C(25)	54·3 (3)
C(5)–C(6)–O(7)–C(8)	−177·0 (2)	C(19)–C(8)–C(9)–O(10)	164·1 (2)
C(6)–O(7)–C(8)–C(9)	169·9 (2)	C(19)–C(8)–C(9)–C(25)	−71·0 (3)
O(7)–C(8)–C(9)–O(10)	−70·4 (2)	O(16)–C(17)–C(18)–C(37)	−171·4 (2)
C(8)–C(9)–O(10)–C(11)	−165·5 (2)	C(31)–C(17)–C(18)–O(1)	−60·8 (3)
C(9)–O(10)–C(11)–C(12)	−176·3 (2)	C(31)–C(17)–C(18)–C(37)	63·1 (3)
O(10)–C(11)–C(12)–O(13)	67·2 (3)	C(9)–C(8)–C(19)–C(20)	81·2 (3)
C(11)–C(12)–O(13)–C(14)	−178·6 (2)	C(8)–C(9)–C(25)–C(26)	−70·3 (3)
C(12)–O(13)–C(14)–C(15)	177·1 (2)	C(18)–C(17)–C(31)–C(36)	71·9 (3)
O(13)–C(14)–C(15)–O(16)	−69·9 (2)	C(17)–C(18)–C(37)–C(38)	83·3 (3)

complex with ammonium picrate in chloroform than do the other configurational diasteroisomers [(7)–(9)].

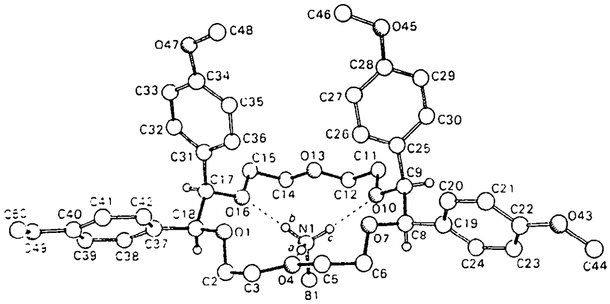

Fig. 1. Ball-and-stick representation of the structure of [(1).BH_3NH_3] showing the atomic numbering scheme. The 18-crown-6 ring is highlighted with solid C–C and C–O bonds.

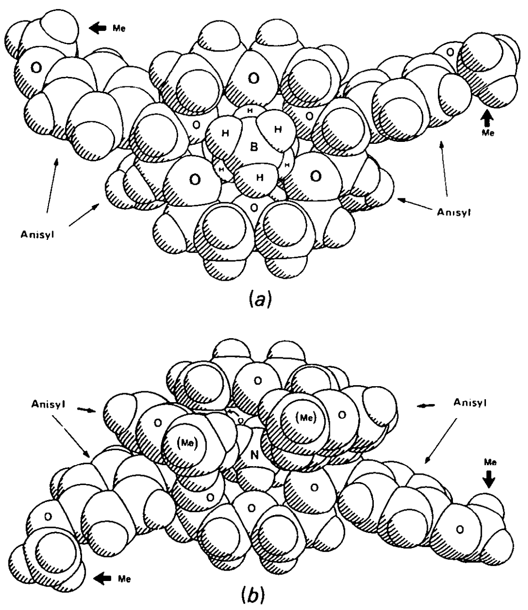

Fig. 2. Space-filling representation of [(1).BH_3NH_3] viewed from (*a*) the sterically accessible unsubstituted face of the macrocycle showing the docking of BH_3NH_3 from in front and (*b*) the sterically hindered tetra-substituted face of the macrocycle with two axial (and two equatorial) anisyl groups, and showing the docking of BH_3NH_3 from behind.

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

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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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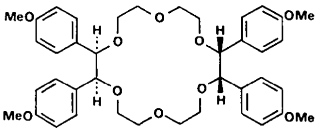
Abstract. $C_{40}H_{48}O_{10}$, $M_r = 689$, monoclinic, $P2_1/a$, $a = 8{\cdot}238\ (2)$, $b = 20{\cdot}616\ (7)$, $c = 10{\cdot}883\ (2)$ Å, $\beta = 99{\cdot}99\ (2)°$, $V = 1820$ Å^3, $Z = 2$, $D_x = 1{\cdot}26$ Mg m^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}54178$ Å, $\mu = 0{\cdot}70$ mm^{-1}, $F(000) = 736$, 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.

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