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

BY DAVID A. PEARS, HOOSHANG SHAHRIARI-ZAVAREH AND J. FRASER STODDART

Department of Chemistry, The University, Sheffield S3 7HF, England

JOHN CROSBY

Organics Division, Imperial Chemical Industries PLC, Blackley, Manchester M9 3DA, England

AND BILLY L. ALLWOOD, ALEXANDRA M. Z. SLAWIN AND DAVID J. WILLIAMS

Department of Chemistry, Imperial College, London SW7 2AY, England

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Abstract. $C_{40}H_{48}O_{10}$, $M_r = 689$, triclinic, $P\bar{1}$, $a = 10{\cdot}960\,(2)$, $b = 13{\cdot}194\,(2)$, $c = 13{\cdot}785\,(4)$ Å, $\alpha = 85{\cdot}76\,(2)$, $\beta = 81{\cdot}87\,(2)$, $\gamma = 73{\cdot}14\,(1)^\circ$, $V = 1889$ Å^3, $Z = 2$, $D_x = 1{\cdot}21$ Mg m^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1{\cdot}54178$ Å, $\mu = 0{\cdot}67$ mm^{-1}, $F(000) = 736$, room temperature, $R = 0{\cdot}073$ for 3294 observed reflections with $|F_o| > 3\sigma(|F_o|)$. There are two *anti* and four *gauche* OCH_2-CH_2O units present in the conformation adopted by the 18-membered ring of the title compound (1): two of the anisyl groups are axial and two are equatorial: the vicinal groups have a *gauche* relationship to each other.

Introduction. There has been considerable interest of late in the solid-state structures of 2,3,11,12-tetra-aryl-18-crown-6 derivatives, such as (1)–(5) (Fig. 1), where the four anisyl substituents have the *cis–cisoid–cis, cis–transoid–cis, trans–cisoid–trans, trans–transoid–trans* and *cis–trans* configurations respectively. They provide (Weber, Sheldrick, Burgemeister, Dietl, Mannschreck & Merz, 1984) an ideal set of configurational diastereoisomers on which to investigate (Merz, Eichner & Tomahogh, 1981) the dependence of metal (*e.g.* Na^+ and K^+) and inorganic (*e.g.* H_3O^+ and NH_4^+) ion complexation strengths upon relative configurations. Unlike the analogous series of dicyclohexano-18-crown-6 derivatives (Burden, Coxon, Stoddart & Wheatley, 1977), where the fused six-membered rings introduce (Coxon, Laidler, Pettman & Stoddart, 1978) additional rigid configurational constraints into the macrocyclic polyether ring, the 2,3,11,12-tetraphenyl-18-crown-6 derivatives (6)–(10) shown in Fig. 1 are only subject to configurational influences and associated nonbonded interactions between the four substituents. Furthermore, since the *trans–transoid–trans* (4) and *cis–trans* (5) isomers are chiral, the potential exists (Stoddart, 1987*a,b*) to employ them either as chiral reagents (Allwood, Shahriari-Zavareh, Stoddart & Williams, 1984) or as chiral catalysts (Colquhoun, Stoddart & Williams, 1986). In exhibiting their molecular recognition properties, chiral crown ethers can also discriminate between enantiomeric substrates. Indeed, the (*RRRR*)-enantiomer of the *trans–transoid–trans* isomer (4) has been shown (Dietl, Merz & Tomahogh, 1982) to extract (*R*)-phenylglycine methyl ester hydroperchlorate preferentially into deuteriochloroform from an aqueous solution of its

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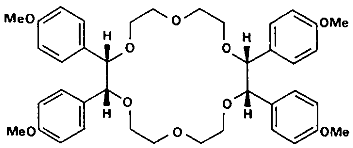
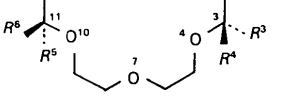
racemic modification. In the parent 2,3,11,12-tetraphenyl series (Fig. 1) of 18-crown-6 derivatives (6)–(10), the crystal structures of the *cis–transoid–cis* (7) and *trans–cisoid–trans* (8) isomers have been reported (Weber *et al.,* 1984; Blasius, Rausch, Andreetti & Rebizant, 1984), along with those of the 1:1 complexes of the *cis–transoid–cis* (7) and *trans–cisoid–trans* (8) isomers with sodium iodide (Weber *et al.,* 1984), the 1:2 complex of the *trans–cisoid–trans* isomer (8) with ammonia–borane (Allwood *et al.,* 1984), and the 1:1 adduct of the (*RRRR*)-enantiomer of the *trans–transoid–trans* isomer (9) with ammonia–borane (Allwood *et al.,* 1984). Ammonia–borane is an ideal substrate for binding to the 18-crown-6 constitution (Colquhoun, Jones, Maud, Stoddart & Williams, 1984; Alston, Stoddart, Wolstenholme, Allwood & Williams, 1985). It has the merit that it is neutral and so removes the problems raised by the counterions that necessarily accompany cationic substrates. It is also a reducing agent and so its complexes are potentially useful (Allwood *et al.,* 1984; Shahriari-Zavareh, Stoddart, Williams, Allwood & Williams, 1985; Colquhoun, Stoddart & Williams, 1986; Stoddart, 1984, 1987*a,b*) as reagents in chemical reactions.

This is the first of a series of eight short papers which describe the X-ray structural analyses of compounds (1)–(4) and their complexes with ammonia–borane. Here, we report and discuss the solid-state structure of *cis–cisoid–cis*-2,3,11,12-tetra-anisyl-18-crown-6 (1) in the knowledge that Weber *et al.* (1984) have reported that single crystals of the *cis–cisoid–cis* isomer (6) of 2,3,11,12-tetraphenyl-18-crown-6 suitable for X-ray structural determination could not be grown.



(1)

Experimental. Reaction of *meso*-bis(4-methoxyphenyl)-ethane-1,2-diol with diethyleneglycol bistosylate in dimethylformamide in the presence of sodium hydride as base gave (Pears, 1985) a mixture of products which contained both the *cis–cisoid–cis* (1) and *cis–transoid–cis* (2) isomers of 2,3,11,12-tetra-anisyl-18-crown-6. When the crude product was subjected to column chromatography on silica gel using dichloromethane–diethyl ether as eluant, the first fraction was found to correspond to the *cis–cisoid–cis* isomer (1). Single crystals of (1), suitable for X-ray crystallography, were grown at room temperature over 2 days in dichloromethane layered with *n*-pentane and had m.p. 431–433 K. Crystal size 0·50 × 0·43 × 0·13 mm. Refined unit-cell parameters obtained by centring 18 reflections. Nicolet *R*3*m* diffractometer. 3878 independent reflections ($\theta \leq 50°$) measured, Cu $K\alpha$ radiation (graphite monochromator), ω-scan. 3294 [$|F_o| > 3\sigma(|F_o|)$] considered observed, index range h −10/10, k −12/13, l 0/13; 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 a combination of direct methods and ΔE map recycling; non-hydrogen atoms refined anisotropically; positions of H atoms calculated (C—H = 0·96 Å), assigned isotropic thermal parameters $U(\mathrm{H}) = 1{\cdot}2\ U_{\mathrm{eq}}(\mathrm{C})$, allowed to ride on parent C atoms. All the OMe groups have high anisotropic thermal parameters indicating possible disorder. Two alternative sites for C(46) were refined with occupancies of 0·4 and 0·6. The disorder in the O(47) methoxy group was resolved into two discrete alternative orientations each of 0·5 occupancy. Each orientation was refined subject to a C—O distance constraint of 1·45 Å. Slight disorder in the polyether chain was resolved at O(4) into two discrete orientations of 0·8 and 0·2. An empirical extinction correction was applied [$g = 0{\cdot}0074\,(16)$]. Refinement using F magnitudes by block-cascade full-matrix least squares; $R = 0{\cdot}073$; $wR = 0{\cdot}088$ [$w^{-1} = \sigma^2(F) + 0{\cdot}0017F^2$]. $(\Delta/\sigma)_{\max} = 0{\cdot}35$; residual electron density in difference map within −0·21 and +0·40 e Å^{-3}; atomic scattering factors and dispersion corrections from *International Tables for X-ray Crystallography* (1974). Computation carried out on an Eclipse S140 computer using the *SHELXTL* program system (Sheldrick, 1983).



Compound number	Relative configuration	R^1	R^2	R^3	R^4	R^5	R^6	R^7	R^8
(1)	*cis–cisoid–cis*	*p*-MeOC$_6$H$_4$	H	*p*-MeOC$_6$H$_4$	H	*p*-MeOC$_6$H$_4$	H	*p*-MeOC$_6$H$_4$	H
(2)	*cis–transoid–cis*	*p*-MeOC$_6$H$_4$	H	*p*-MeOC$_6$H$_4$	H	H	*p*-MeOC$_6$H$_4$	H	*p*-MeOC$_6$H$_4$
(3)	*trans–cisoid–trans*	*p*-MeOC$_6$H$_4$	H	H	*p*-MeOC$_6$H$_4$	H	*p*-MeOC$_6$H$_4$	*p*-MeOC$_6$H$_4$	H
(4)	*trans–transoid–trans*	*p*-MeOC$_6$H$_4$	H	H	*p*-MeOC$_6$H$_4$	*p*-MeOC$_6$H$_4$	H	H	*p*-MeOC$_6$H$_4$
(5)	*cis–trans*	*p*-MeOC$_6$H$_4$	H	*p*-MeOC$_6$H$_4$	H	*p*-MeOC$_6$H$_4$	H	H	*p*-MeOC$_6$H$_4$
(6)	*cis–cisoid–cis*	C$_6$H$_5$	H	C$_6$H$_5$	H	C$_6$H$_5$	H	C$_6$H$_5$	H
(7)	*cis–transoid–cis*	C$_6$H$_5$	H	C$_6$H$_5$	H	H	C$_6$H$_5$	H	C$_6$H$_5$
(8)	*trans–cisoid–trans*	C$_6$H$_5$	H	H	C$_6$H$_5$	H	C$_6$H$_5$	C$_6$H$_5$	H
(9)	*trans–transoid–trans*	C$_6$H$_5$	H	H	C$_6$H$_5$	C$_6$H$_5$	H	H	C$_6$H$_5$
(10)	*cis–trans*	C$_6$H$_5$	H	C$_6$H$_5$	H	C$_6$H$_5$	H	H	C$_6$H$_5$

Fig. 1. The sets of five configurational diastereoisomers for the 2,3,11,12-tetra-anisyl- (1)–(5) and 2,3,11,12-tetraphenyl- (6)–(10) 18-crown-6 derivatives. In compounds (4), (5), (9) and (10), the absolute configurations at the chiral centres are all (*R*).

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

	x	y	z	U_{eq}*
O(1)	6152 (2)	1444 (2)	−3624 (2)	82 (1)
C(2)	7120 (4)	484 (3)	−3575 (3)	88 (2)
C(3)	7397 (3)	234 (2)	−2567 (3)	77 (2)
O(4*a*)	7957 (3)	−867 (2)	−2441 (2)	80 (1)
O(4*b*)	8634 (6)	−446 (9)	−2399 (8)	111 (5)
C(5)	8612 (4)	−1198 (3)	−1601 (3)	99 (2)
C(6)	7774 (4)	−1009 (3)	−668 (3)	94 (2)
O(7)	7591 (3)	37 (2)	−386 (2)	83 (1)
C(8)	6608 (4)	374 (3)	392 (3)	79 (2)
C(9)	6061 (4)	1568 (3)	269 (3)	78 (2)
O(10)	5471 (3)	1720 (2)	−610 (2)	94 (1)
C(11)	4637 (4)	2729 (3)	−756 (3)	92 (2)
C(12)	4166 (5)	2779 (4)	−1698 (3)	117 (2)
O(13)	3316 (3)	3735 (2)	−1886 (2)	120 (1)
C(14)	2733 (5)	3806 (5)	−2725 (3)	164 (3)
C(15)	3278 (4)	4069 (5)	−3600 (3)	132 (3)
O(16)	4542 (2)	3467 (2)	−3943 (2)	90 (1)
C(17)	4632 (4)	2625 (3)	−4546 (3)	79 (2)
C(18)	6006 (3)	1873 (3)	−4591 (3)	72 (2)
C(19)	7021 (4)	45 (3)	1398 (3)	73 (2)
C(20)	6133 (4)	14 (3)	2203 (3)	83 (2)
C(21)	6472 (4)	−273 (3)	3116 (3)	85 (2)
C(22)	7744 (4)	−560 (3)	3249 (3)	81 (2)
C(23)	8658 (4)	−521 (3)	2466 (3)	87 (2)
C(24)	8311 (4)	−220 (3)	1552 (3)	85 (2)
C(25)	7056 (4)	2160 (3)	243 (3)	76 (2)
C(26)	7102 (4)	2715 (3)	1048 (3)	80 (2)
C(27)	8036 (4)	3216 (3)	1061 (3)	88 (2)
C(28)	8949 (4)	3177 (3)	265 (4)	90 (2)
C(29)	8904 (5)	2663 (3)	−556 (4)	100 (2)
C(30)	7972 (5)	2169 (3)	−556 (3)	99 (2)
C(31)	7031 (3)	2377 (3)	−4992 (3)	66 (1)
C(32)	7357 (4)	3086 (3)	−4459 (3)	88 (2)
C(33)	8263 (5)	3560 (4)	−4825 (4)	107 (3)
C(34)	8892 (4)	3341 (4)	−5773 (4)	107 (2)
C(35)	8601 (4)	2644 (4)	−6304 (4)	105 (2)
C(36)	7680 (4)	2171 (3)	−5922 (3)	80 (2)
C(37)	4328 (3)	3007 (3)	−5585 (3)	72 (2)
C(38)	4505 (4)	3943 (3)	−6002 (3)	77 (2)
C(39)	4259 (4)	4269 (3)	−6942 (3)	77 (2)
C(40)	3793 (3)	3658 (3)	−7476 (3)	74 (2)
C(41)	3628 (4)	2722 (3)	−7090 (3)	91 (2)
C(42)	3857 (4)	2409 (3)	−6131 (3)	86 (2)
O(43)	7980 (3)	−843 (2)	4200 (2)	114 (1)
C(44)	9252 (5)	−1165 (6)	4384 (4)	176 (4)
O(45)	9898 (3)	3651 (3)	304 (3)	127 (2)
C(46*a*)	10673 (9)	3743 (7)	−496 (8)	152 (5)
C(46*b*)	10123 (16)	4234 (11)	1052 (10)	140 (8)
O(47*a*)	9669 (6)	4068 (5)	−5911 (6)	121 (4)
C(48*a*)	10497 (12)	3905 (11)	−6840 (8)	157 (7)
O(47*b*)	9926 (6)	3605 (5)	−6409 (4)	142 (4)
C(48*b*)	10407 (14)	4321 (10)	−5911 (13)	151 (8)
O(49)	3506 (3)	3915 (2)	−8423 (2)	103 (1)
C(50)	3663 (6)	4887 (4)	−8852 (3)	126 (3)

* 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)	1·403 (4)	O(1)–C(18)	1·440 (4)
C(2)–C(3)	1·469 (5)	C(3)–O(4*a*)	1·419 (4)
C(3)–O(4*b*)	1·437 (8)	O(4*a*)–C(5)	1·435 (5)
O(4*b*)–C(5)	1·455 (11)	C(5)–C(6)	1·455 (5)
C(6)–O(7)	1·399 (5)	O(7)–C(8)	1·394 (4)
C(8)–C(9)	1·527 (5)	C(8)–C(19)	1·527 (6)
C(9)–O(10)	1·432 (5)	C(9)–C(25)	1·510 (6)
O(10)–C(11)	1·405 (4)	C(11)–C(12)	1·456 (7)
C(12)–O(13)	1·374 (5)	O(13)–C(14)	1·386 (6)
C(14)–C(15)	1·348 (7)	C(15)–O(16)	1·407 (5)
O(16)–C(17)	1·386 (5)	C(17)–C(18)	1·539 (5)
C(17)–C(37)	1·549 (5)	C(18)–C(31)	1·495 (6)
C(19)–C(20)	1·377 (5)	C(19)–C(24)	1·399 (6)
C(20)–C(21)	1·371 (6)	C(21)–C(22)	1·372 (6)
C(22)–C(23)	1·374 (5)	C(22)–O(43)	1·388 (5)
C(23)–C(24)	1·378 (6)	C(25)–C(26)	1·369 (6)
C(25)–C(30)	1·384 (6)	C(26)–C(27)	1·372 (7)
C(27)–C(28)	1·368 (6)	C(28)–C(29)	1·356 (7)
C(28)–O(45)	1·369 (6)	C(29)–C(30)	1·362 (8)
C(31)–C(32)	1·366 (6)	C(31)–C(36)	1·371 (5)
C(32)–C(33)	1·351 (8)	C(33)–C(34)	1·388 (8)
C(34)–C(35)	1·330 (8)	C(34)–O(47*a*)	1·448 (9)
C(34)–O(47*b*)	1·449 (8)	C(35)–C(36)	1·370 (7)
C(37)–C(38)	1·394 (5)	C(37)–C(42)	1·359 (6)
C(38)–C(39)	1·389 (5)	C(39)–C(40)	1·357 (6)
C(40)–C(41)	1·375 (6)	C(40)–O(49)	1·392 (5)
C(41)–C(42)	1·405 (6)	O(43)–C(44)	1·392 (7)
O(45)–C(46*a*)	1·318 (11)	O(45)–C(46*b*)	1·400 (16)
O(47*a*)–C(48*a*)	1·445 (13)	O(47*b*)–C(48*b*)	1·444 (18)
O(49)–C(50)	1·437 (6)		

C(2)–O(1)–C(18)	115·8 (2)	O(1)–C(2)–C(3)	111·7 (3)
C(2)–C(3)–O(4*a*)	111·6 (3)	C(2)–C(3)–O(4*b*)	119·7 (5)
C(3)–O(4*a*)–C(5)	117·7 (3)	C(3)–O(4*b*)–C(5)	115·2 (6)
O(4*a*)–C(5)–C(6)	114·3 (4)	O(4*b*)–C(5)–C(6)	126·7 (4)
C(5)–C(6)–O(7)	108·1 (4)	C(6)–O(7)–C(8)	113·1 (3)
O(7)–C(8)–C(9)	106·1 (3)	O(7)–C(8)–C(19)	113·9 (3)
C(9)–C(8)–C(19)	114·1 (3)	C(8)–C(9)–O(10)	106·5 (3)
C(8)–C(9)–C(25)	113·4 (3)	O(10)–C(9)–C(25)	112·0 (3)
C(9)–O(10)–C(11)	116·9 (3)	O(10)–C(11)–C(12)	110·6 (3)
C(11)–C(12)–O(13)	114·2 (4)	C(12)–O(13)–C(14)	117·3 (4)
O(13)–C(14)–C(15)	122·0 (5)	C(14)–C(15)–O(16)	117·6 (4)
C(15)–O(16)–C(17)	114·8 (4)	O(16)–C(17)–C(18)	107·6 (3)
O(16)–C(17)–C(37)	111·5 (3)	C(18)–C(17)–C(37)	110·7 (3)
O(1)–C(18)–C(17)	107·3 (3)	O(1)–C(18)–C(31)	113·0 (3)
C(17)–C(18)–C(31)	113·8 (3)	C(8)–C(19)–C(20)	121·3 (4)
C(8)–C(19)–C(24)	122·0 (3)	C(20)–C(19)–C(24)	116·8 (4)
C(19)–C(20)–C(21)	122·7 (4)	C(20)–C(21)–C(22)	119·8 (4)
C(21)–C(22)–C(23)	119·2 (4)	C(21)–C(22)–O(43)	115·0 (3)
C(23)–C(22)–O(43)	125·8 (4)	C(22)–C(23)–C(24)	120·8 (4)
C(19)–C(24)–C(23)	120·7 (4)	C(9)–C(25)–C(26)	120·1 (3)
C(9)–C(25)–C(30)	123·9 (4)	C(26)–C(25)–C(30)	116·0 (4)
C(25)–C(26)–C(27)	121·2 (4)	C(26)–C(27)–C(28)	120·9 (4)
C(27)–C(28)–C(29)	119·4 (5)	C(27)–C(28)–O(45)	120·0 (4)
C(29)–C(28)–O(45)	120·6 (4)	C(28)–C(29)–C(30)	118·9 (4)
C(25)–C(30)–C(29)	123·5 (4)	C(18)–C(31)–C(32)	121·9 (3)
C(18)–C(31)–C(36)	122·1 (4)	C(32)–C(31)–C(36)	116·0 (4)
C(31)–C(32)–C(33)	122·2 (4)	C(32)–C(33)–C(34)	120·3 (5)
C(33)–C(34)–C(35)	118·8 (5)	C(33)–C(34)–O(47*a*)	103·1 (5)
C(35)–C(34)–O(47*a*)	137·9 (6)	C(33)–C(34)–O(47*b*)	138·2 (6)
C(35)–C(34)–O(47*b*)	102·9 (5)	C(34)–C(35)–C(36)	120·2 (4)
C(31)–C(36)–C(35)	122·6 (4)	C(17)–C(37)–C(38)	123·6 (4)
C(17)–C(37)–C(42)	119·0 (4)	C(38)–C(37)–C(42)	117·3 (4)
C(37)–C(38)–C(39)	123·4 (4)	C(38)–C(39)–C(40)	118·3 (4)
C(39)–C(40)–C(41)	119·6 (4)	C(39)–C(40)–O(49)	123·0 (4)
C(41)–C(40)–O(49)	117·4 (4)	C(40)–C(41)–C(42)	121·6 (4)
C(37)–C(42)–C(41)	119·6 (4)	C(22)–O(43)–C(44)	118·1 (4)
C(28)–O(45)–C(46*a*)	120·2 (6)	C(28)–O(45)–C(46*b*)	130·7 (7)
C(34)–O(47*a*)–C(48*a*)	112·0 (8)	C(34)–O(47*b*)–C(48*b*)	109·9 (8)
C(40)–O(49)–C(50)	119·1 (4)		

C(18)–O(1)–C(2)–C(3)	166·9 (3)	C(11)–C(12)–O(13)–C(14)	−173·9 (4)
C(2)–O(1)–C(18)–C(17)	157·7 (4)	C(12)–O(13)–C(14)–C(15)	−87·7 (7)
O(1)–C(2)–C(3)–O(4*a*)	156·8 (3)	O(13)–C(14)–C(15)–C(16)	54·3 (8)
C(1)–C(2)–C(3)–O(4*b*)	−155·5 (6)	C(14)–C(15)–O(16)–C(17)	93·2 (6)
C(2)–C(3)–O(4*a*)–C(5)	162·5 (4)	C(15)–O(16)–C(17)–C(18)	−163·1 (3)
C(2)–C(3)–O(4*b*)–C(5)	−140·0 (6)	O(16)–C(17)–C(18)–O(1)	65·6 (4)
C(3)–O(4*a*)–C(5)–C(6)	67·1 (5)	O(7)–C(8)–C(9)–O(25)	−57·5 (4)
C(3)–O(4*b*)–C(5)–C(6)	−38·1 (12)	C(19)–C(8)–C(9)–O(10)	−167·6 (3)
O(4*a*)–C(5)–C(6)–O(7)	−84·1 (5)	C(19)–C(8)–C(9)–C(25)	68·8 (4)
O(4*b*)–C(5)–C(6)–O(7)	−35·9 (8)	O(16)–C(17)–C(18)–C(31)	−60·1 (4)
C(5)–C(6)–O(7)–C(8)	167·9 (3)	C(37)–C(17)–C(18)–O(1)	−172·3 (3)
C(6)–O(7)–C(8)–C(9)	−151·1 (3)	C(37)–C(17)–C(18)–C(31)	61·9 (4)
O(7)–C(8)–C(9)–O(10)	66·1 (4)	C(9)–C(8)–C(19)–C(20)	78·2 (5)
C(8)–C(9)–O(10)–C(11)	165·1 (3)	C(8)–C(9)–C(25)–C(30)	75·4 (5)
C(9)–O(10)–C(11)–C(12)	177·1 (4)	C(18)–C(17)–C(37)–C(42)	87·0 (4)
O(10)–C(11)–C(12)–O(13)	178·5 (4)	C(17)–C(18)–C(31)–C(32)	73·8 (4)

Discussion. Table 1* lists the fractional atomic coordinates of the non-hydrogen atoms, Table 2 the bond lengths and angles and selected C—C and C—O torsion angles associated with the polyether ring and substituent anisyl groups.

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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 44783 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The structure of (1) is illustrated in Fig. 2 which includes the atomic numbering scheme. Fig. 3 shows a space-filling representation of the molecule looking into the opposite face of the macrocycle from that viewed in Fig. 2.

Although there is a surprising absence of symmetry in the structure, the maximum deviation [by O(16) in fact] of the oxygen atoms from the mean plane of the macrocycle is only 0·58 (2) Å. The polyether ring adopts a conformation containing two *anti* bismethylenedioxy units [O(1) to O(4) and O(10) to O(13)], a feature which also characterizes (Dunitz & Seiler, 1974) the crystal structure of 18-crown-6. Nevertheless, the remainder of the macrocycle departs significantly from the conformation of the parent compound. This conformational difference is probably a result of the location of the four anisyl substituents at C(8), C(9), C(17) and C(18), two of which assume equatorial orientations [C(8) and C(17)] and two of which assume axial orientations [C(9) and C(18)]. These axial anisyl groups can be seen (Figs. 2 and 3) to dominate one of the faces of the macrocycle while the methylene groups at C(3) and C(12) are directed inwards thus helping to fill the potential cavity. Both pairs of vicinal anisyl groups are oriented *gauche* with respect to each other. 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.

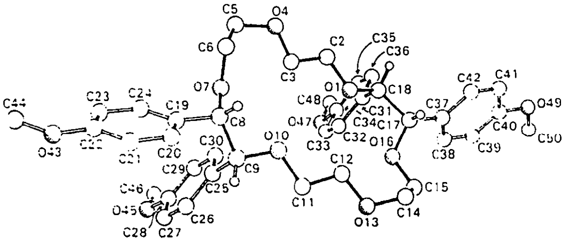

Fig. 2. Ball-and-stick representation of the structure of (1) showing the atomic numbering scheme. The 18-crown-6 ring is highlighted with solid C—C and C—O bonds.

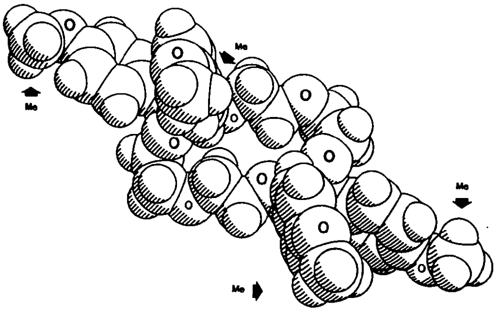

Fig. 3. A space-filling representation of (1) viewed from the opposite face of the macrocycle to that chosen for the ball-and-stick representation in Fig. 2.

Despite the potential for intermolecular aromatic–aromatic interactions (Burley & Petsko, 1985, 1986; Moody, Owusu, Slawin, Spencer, Stoddart, Thomas & Williams, 1987), the molecules are loosely packed with no intermolecular contacts of less than van der Waals distances.

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