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

The structure of (2) is illustrated in Fig. 1 which gives a side-on view of the molecule and includes the numbering scheme: Fig. 2 gives a plan view down the crystallographic twofold axis of a space-filling representation of (2).

The macrobicyclic diazapolyether disulfonamide (2) is symmetric with a crystallographic twofold axis passing through the C(20)—C(20') bond of the central polyether chain and between the faces of the trisubstituted aromatic rings. The symmetry of this structure is in marked contrast to the pronounced molecular asymmetry displayed by the all-oxygen analogue (1). Also, the characteristic V-shaped conformation adopted by (1), in both its free and second-sphere coordinating forms (Alston, Slawin, Stoddart & Williams, 1984) is no longer present in (2). The molecular cavity in (2) is totally self-filling (Fig. 2) with the two trisubstituted aromatic rings lying almost parallel to each other (4° between their mean planes) and with an inter-ring separation of between 3·45 and 3·54 Å. There are only two significant departures (see Table 2) from conventional *gauche* geometries about the C—C bonds in the polyether chains; these are about the C(3)—C(4) and C(3')—C(4') bonds [−178·3 (2)°]. There is also a small pyramidal distortion at N(5) and

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42759 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

N(5'), each lying 0·23 (1) Å from the plane of their adjacent carbon and sulfur atoms with a consequent opening out of the bond angles (Table 2) around the nitrogen atom. The bond angles (Table 2) around the sulfur atom reveal a slightly distorted tetrahedral arrangement of bonds. The length [1·632 (2) Å] of the N—S bond indicates that it has partial double-bond character as a result of π bonding between the lone pair of electrons on the nitrogen atom and the empty $3d$ orbitals on the sulfur atom. The conformational orientation of the tosyl groups with respect to the —CH₂—N—CH₂— fragments on the two associated chains is similar to that observed (Smith-Verdier, Garcia-Blanco & Florencio, 1976) in the X-ray structure of the methanesulfonamide of piperidine-4-spiro-2'-(1,3-dioxolane).

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Structure of a Chiral Bisanisylene Crown Ether, 1,4:1',4':3,6:3',6'-Tetraanhydro-2,2':5,5'-bis-O-[2-methoxy-1,3-phenylenebis(methylene)]di-D-mannitol

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Abstract. $C_{30}H_{36}O_{10}$, $M_r = 556·6$, monoclinic, $C2$, $a = 13·255$ (3), $b = 15·479$ (3), $c = 14·187$ (3) Å, $\beta = 106·36$ (2)°, $V = 2793$ Å³, $Z = 4$, $D_x = 1·32$ g cm⁻³, $\lambda(Cu\ K\alpha) = 1·54178$ Å, $\mu = 7·9$ cm⁻¹, $F(000) = 1184$, room temperature, $R = 0·064$ for 1880 unique observed reflections. In this chiral bisanisylene macrocycle DD-(3), the dianhydromannitolo units adopt a 'back-to-back' geometry with respect to each other. This is in
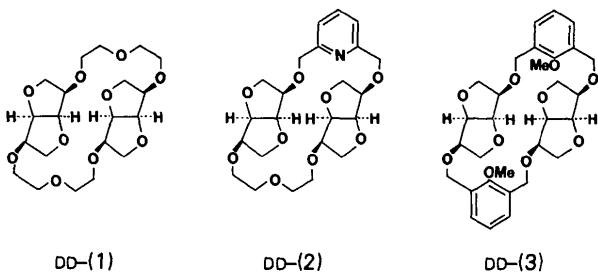
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$C_{30}H_{36}O_{10}$

striking contrast to the 'face-to-back' geometry displayed by 1,4:1',4':3,6:3',6'-tetraanhydro-2,2':5,5'-bis-(O-oxydiethylene)di-D-mannitol [DD-(1)] and 1,4:-1',4':3,6:3',6'-tetraanhydro-5,5'-O-oxydiethylene-2,2'-O-[pyridine-2,6-diylbis(methylene)]di-D-mannitol [DD-(2)]. The molecule DD-(3) has approximate non-crystallographic C_2 symmetry about an axis passing through the centre of, and normal to, the mean plane of the macrocyclic ring. In the solid state, the molecule has diastereotopic faces with the two aryl methoxy groups both oriented away from the more open of the two ring faces.

Introduction. In order to confer greater rigidity upon receptor molecules such as DD-(1) (Metcalfe, Stoddart, Jones, Crawshaw, Quick & Williams, 1981; Metcalfe, Stoddart, Jones, Crawshaw, Gavuzzo & Williams, 1981) and DD-(2) (Crawshaw, Stoddart & Williams, 1986) containing 1,4:3,6-dianhydro-D-mannitol, it was decided to appeal to 2-substituted-1,3-xylylene units as constitutional building blocks and rigid spacer groups. Since the first report (Vögtle & Weber, 1974) of the use of these units in the synthesis of macrocyclic hosts, they have found wide application (McKervey & Mulholland, 1977; Newcomb, Moore & Cram, 1977; van der Leij, Oosternik, Hall & Reinhoudt, 1981; McKervey & O'Connor, 1982). In this paper, we report on the results of an X-ray crystallographic investigation of the macrocyclic compound DD-(3) incorporating two dianhydromannitolo rings and two m-anisylene units.

DD-(1)   DD-(2)   DD-(3)

Experimental. Single crystals of DD-(3) (found: C 64·8, H 6·5%; $C_{30}H_{36}O_{10}$ requires C 64·7, H 6·5%), suitable for X-ray crystallography, were grown (Crawshaw, 1982) at room temperature from ethyl acetate–light petroleum (b.p. 333–353 K) and had m.p. 508 K and $[\alpha]_D^{20°C} = +112\cdot6°$ [CHCl$_3$, 0·82 g dm^{-3}]. Crystal size 0·30 × 0·30 × 0·10 mm. Refined unit-cell parameters by centring 22 reflections. Nicolet $R3m$ diffractometer. 1964 independent reflections ($\theta \le 58°$) measured, Cu $K\alpha$ radiation (graphite monochromator), ω scan. 1880 [$|F_o| > 3\sigma(|F_o|)$] considered observed, index range h −13/13, k 0/16, l 0/15; two check reflections measured every 50 reflections, net count constant; data brought to uniform scale, Lorentz and polarization corrections, no absorption correction. Structure solved

Table 1. *Atom coordinates* $(\times 10^4)$ *and equivalent isotropic temperature factors* $(\text{Å}^2 \times 10^3)$

	x	y	z	U_{eq}*
C(1)	7613 (4)	7856 (3)	6067 (4)	62 (2)
O(2)	8682 (3)	7596 (2)	6218 (3)	70 (1)
C(3)	9424 (4)	8186 (4)	6780 (4)	63 (2)
C(4)	10493 (5)	7952 (10)	6705 (6)	140 (5)
O(5)	11200 (3)	8385 (5)	7524 (4)	119 (3)
C(6)	10707 (4)	8515 (4)	8257 (4)	68 (2)
C(7)	9583 (4)	8219 (4)	7835 (4)	71 (2)
O(8)	9547 (5)	7398 (5)	8250 (5)	155 (3)
C(9)	10555 (8)	7150 (6)	8765 (9)	183 (5)
C(10)	11154 (6)	7949 (5)	9107 (6)	98 (3)
O(11)	12239 (4)	7794 (3)	9395 (4)	122 (2)
C(12)	12843 (7)	8420 (5)	10048 (6)	116 (3)
C(13)	13100 (5)	9161 (4)	9521 (5)	81 (2)
C(14)	12609 (4)	9957 (3)	9370 (4)	60 (2)
C(15)	12836 (5)	10615 (4)	8852 (4)	72 (2)
C(16)	12259 (6)	11463 (4)	8644 (5)	83 (2)
O(17)	11673 (4)	11607 (3)	7694 (3)	97 (2)
C(18)	10838 (4)	11019 (4)	7323 (4)	70 (2)
C(19)	10329 (5)	11172 (8)	6252 (5)	107 (3)
O(20)	9314 (3)	10828 (5)	6054 (3)	118 (3)
C(21)	8995 (5)	10804 (4)	6907 (5)	69 (2)
C(22)	9909 (4)	11082 (4)	7726 (4)	67 (2)
O(23)	9711 (4)	11939 (4)	7979 (4)	119 (2)
C(24)	8796 (7)	12250 (5)	7279 (6)	121 (3)
C(25)	8139 (5)	11434 (4)	6922 (4)	68 (2)
O(26)	7390 (3)	11617	6012 (3)	74 (1)
C(27)	6543 (5)	11021 (4)	5697 (5)	74 (2)
C(28)	6845 (4)	10182 (4)	5294 (4)	59 (2)
C(29)	7001 (4)	9425 (3)	5827 (4)	55 (2)
C(30)	7366 (4)	8680 (3)	5485 (4)	54 (2)
C(31)	7543 (4)	8721 (4)	4569 (4)	62 (2)
C(32)	7380 (5)	9470 (4)	4026 (4)	71 (2)
C(33)	7039 (4)	10189 (4)	4394 (4)	67 (2)
O(34)	6847 (3)	9407 (3)	6754 (3)	78 (2)
C(35)	5864 (7)	9075 (7)	6791 (7)	123 (4)
C(36)	13911 (5)	9046 (6)	9077 (6)	109 (3)
C(37)	14195 (6)	9711 (8)	8525 (6)	115 (4)
C(38)	13634 (6)	10492 (6)	8418 (5)	103 (3)
O(39)	11765 (3)	10061 (3)	9789 (3)	85 (2)
C(40)	12018 (8)	10446 (7)	10714 (6)	116 (4)

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

by direct methods; non-H atoms refined aniso-tropically; positions of H atoms calculated (C—H = 0·96 Å), assigned isotropic thermal parameters, $U(\text{H}) = 1\cdot2\ U_{eq}(\text{C})$, allowed to ride on parent C atoms. The two methyl groups were refined as rigid bodies. Refinement using F magnitudes by block-cascade full-matrix least squares; $R = 0\cdot064$; $wR = 0\cdot075$ [$w^{-1} = \sigma^2(F) + 0\cdot002F^2$]. $(\Delta/\sigma)_{max} = 0\cdot15$; residual electron density in difference map within $-0\cdot22$ and $+0\cdot51$ 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 *SHELXTL* program system (Sheldrick, 1983).

Discussion. Table 1* lists the fractional atomic coordinates of the non-H atoms, Table 2 the bond lengths and

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters, and a figure of a skeletal representation of DD-(3) have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42739 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

angles and selected C—C and C—O torsional angles associated with the dianhydromannitolo rings.

The structure of DD-(3) is illustrated in Fig. 1 which includes the atomic-numbering scheme and torsional angles for a selected macrocyclic periphery. Fig. 2 shows a space-filling representation of the molecule corresponding to the skeletal view illustrated in Fig. 1.

The molecule has approximate non-crystallographic C_2 symmetry about an axis passing through the centre of, and normal to, the mean plane of the macrocyclic ring. In striking contrast with DD-(1) and DD-(2), which both possess (Metcalfe, Stoddart, Jones, Crawshaw, Quick & Williams, 1981; Crawshaw et al., 1986) 'face-to-back' relative geometries for the pairs of dianhydromannitolo rings, in DD-(3) these residues are oriented 'back-to-back' (Fig. 1). Despite this gross conformational feature, which results in a contraction of the inner region of the macrocycle, the spacing effect of the two anisylene units still creates (Fig. 2) a slight opening of the central portion of the receptor cavity. There is an obvious shearing of the two anisylene units with respect to each other which allows the two dianhydromannitolo residues to approach each other more closely, thus helping to fill the cavity. From the point of view of creating a spacious receptor site the potential benefits of the rigid anisylene units, together

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

C(1)—O(2)	1·431 (6)	C(1)—C(30)	1·503 (7)
O(2)—C(3)	1·412 (6)	C(3)—C(4)	1·495 (10)
C(3)—C(7)	1·452 (8)	C(4)—O(5)	1·436 (11)
O(5)—C(6)	1·390 (9)	C(6)—C(7)	1·512 (7)
C(6)—C(10)	1·474 (9)	C(7)—O(8)	1·407 (10)
O(8)—C(9)	1·384 (12)	C(9)—C(10)	1·476 (13)
C(10)—O(11)	1·400 (9)	O(11)—C(12)	1·422 (9)
C(12)—C(13)	1·460 (11)	C(13)—C(14)	1·382 (8)
C(13)—C(36)	1·402 (12)	C(14)—C(15)	1·338 (9)
C(14)—O(39)	1·417 (8)	C(15)—C(16)	1·506 (9)
C(15)—C(38)	1·378 (11)	C(16)—O(17)	1·371 (7)
O(17)—C(18)	1·414 (8)	C(18)—C(19)	1·497 (8)
C(18)—C(22)	1·500 (9)	C(19)—O(20)	1·401 (9)
O(20)—C(21)	1·391 (9)	C(21)—C(22)	1·486 (7)
C(21)—C(25)	1·500 (9)	C(22)—O(23)	1·418 (9)
O(23)—C(24)	1·418 (9)	C(24)—C(25)	1·537 (10)
C(25)—O(26)	1·418 (6)	O(26)—C(27)	1·424 (7)
C(27)—C(28)	1·516 (8)	C(28)—C(29)	1·379 (7)
C(28)—C(33)	1·372 (8)	C(29)—C(30)	1·391 (7)
C(29)—O(34)	1·387 (7)	C(30)—C(31)	1·384 (8)
C(31)—C(32)	1·375 (8)	C(32)—C(33)	1·360 (9)
O(34)—C(35)	1·416 (11)	C(36)—C(37)	1·408 (14)
C(37)—C(38)	1·404 (14)	O(39)—C(40)	1·393 (9)

O(2)—C(1)—C(30)	112·0 (5)	C(1)—O(2)—C(3)	113·9 (4)
O(2)—C(3)—C(4)	109·0 (6)	O(2)—C(3)—C(7)	118·7 (5)
C(4)—C(3)—C(7)	102·5 (5)	C(3)—C(4)—O(5)	104·2 (8)
C(4)—O(5)—C(6)	109·6 (5)	O(5)—C(6)—C(7)	106·2 (5)
O(5)—C(6)—C(10)	111·1 (6)	C(7)—C(6)—C(10)	105·5 (5)
C(3)—C(7)—C(6)	105·0 (5)	C(3)—C(7)—O(8)	112·8 (6)
C(6)—C(7)—O(8)	104·7 (5)	C(7)—O(8)—C(9)	109·3 (7)
O(8)—C(9)—C(10)	106·9 (8)	C(6)—C(10)—C(9)	100·0 (6)
C(6)—C(10)—O(11)	119·0 (7)	C(9)—C(10)—O(11)	111·5 (7)
C(10)—O(11)—C(12)	114·7 (6)	O(11)—C(12)—C(13)	111·8 (7)
C(12)—C(13)—C(14)	127·8 (7)	C(12)—C(13)—C(36)	116·7 (7)
C(14)—C(13)—C(36)	115·4 (7)	C(13)—C(14)—C(15)	126·6 (6)
C(13)—C(14)—O(39)	115·3 (5)	C(15)—C(14)—O(39)	118·1 (5)
C(14)—C(15)—C(16)	126·2 (6)	C(14)—C(15)—C(38)	117·3 (6)
C(16)—C(15)—C(38)	116·4 (6)	C(15)—C(16)—O(17)	116·7 (5)
C(16)—O(17)—C(18)	115·4 (5)	O(17)—C(18)—C(19)	111·0 (6)
O(17)—C(18)—C(22)	117·1 (5)	C(19)—C(18)—C(22)	101·1 (5)
C(18)—C(19)—O(20)	106·0 (6)	C(19)—O(20)—C(21)	110·2 (5)
O(20)—C(21)—C(22)	106·9 (5)	O(20)—C(21)—C(25)	114·3 (5)
C(22)—C(21)—C(25)	104·7 (5)	C(18)—C(22)—C(21)	105·1 (5)
C(18)—C(22)—O(23)	112·8 (5)	C(21)—C(22)—O(23)	107·5 (5)
C(22)—O(23)—C(24)	108·8 (5)	O(23)—C(24)—C(25)	104·2 (6)
C(21)—C(25)—C(24)	100·3 (5)	C(21)—C(25)—O(26)	117·1 (5)
C(24)—C(25)—O(26)	108·8 (5)	C(25)—O(26)—C(27)	116·4 (4)
O(26)—C(27)—C(28)	113·8 (5)	C(27)—C(28)—C(29)	122·4 (5)
C(27)—C(28)—C(33)	118·8 (5)	C(29)—C(28)—C(33)	118·6 (5)
C(28)—C(29)—C(30)	121·8 (5)	C(28)—C(29)—O(34)	119·9 (5)
C(30)—C(29)—O(34)	118·3 (5)	C(1)—C(30)—C(29)	123·7 (5)
C(1)—C(30)—C(31)	119·1 (5)	C(29)—C(30)—C(31)	117·2 (5)
C(30)—C(31)—C(32)	121·6 (5)	C(31)—C(32)—C(33)	119·4 (6)
C(28)—C(33)—C(32)	121·4 (6)	C(29)—O(34)—C(35)	115·2 (5)
C(13)—C(36)—C(37)	121·1 (8)	C(36)—C(37)—C(38)	118·2 (8)
C(15)—C(38)—C(37)	121·4 (8)	C(14)—O(39)—C(40)	115·8 (6)

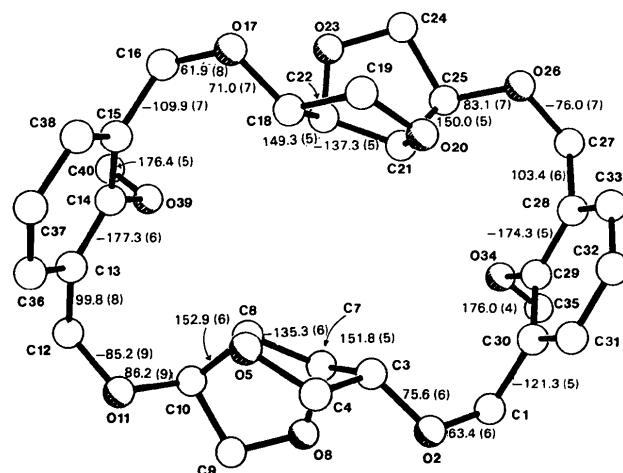|			
C(1)—O(2)—C(3)—C(4)	−167·7 (6)	C(16)—O(17)—C(18)—C(19)	−173·6 (6)
C(2)—C(3)—C(4)—O(5)	−161·7 (7)	O(17)—C(18)—C(19)—O(20)	−157·2 (7)
C(3)—C(4)—O(5)—C(6)	25·4 (10)	C(18)—C(19)—O(20)—C(21)	24·2 (10)
C(4)—O(5)—C(6)—C(7)	−5·4 (9)	C(19)—O(20)—C(21)—C(22)	−5·2 (8)
O(5)—C(6)—C(7)—O(8)	101·8 (6)	O(20)—C(21)—C(22)—O(23)	104·7 (6)
C(6)—C(7)—O(8)—C(9)	−7·4 (9)	C(21)—C(22)—O(23)—C(24)	−7·8 (8)
C(7)—O(8)—C(9)—C(10)	28·5 (11)	C(22)—O(23)—C(24)—C(25)	28·8 (8)
O(8)—C(9)—C(10)—O(11)	−163·7 (8)	O(23)—C(24)—C(25)—O(26)	−161·0 (6)
C(9)—C(10)—O(11)—C(12)	−158·2 (8)	C(24)—C(25)—O(26)—C(27)	−164·2 (6)

Fig. 1. Ball-and-stick representation of the structure of DD-(3) showing the atomic-numbering scheme and the torsional angles (°) around the macrocyclic periphery.
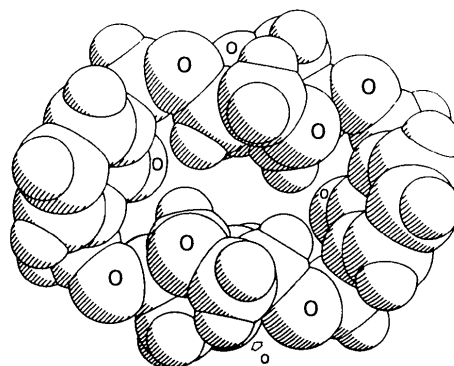

Fig. 2. A space-filling representation of DD-(3) corresponding to the skeletal view in Fig. 1.

with a 'face-to-face' arrangement of the dianhydro-mannitolo residues, can be readily appreciated. Interestingly, the four C—O bonds [e.g. C(1)—O(2) etc.] that link the rigid portions of DD-(3) all adopt (Fig. 1) gauche geometries in contrast to the anti* arrangements normally associated with polyether molecules (Goldberg, 1980, 1984). The two methoxy methyl groups adopt conformations about the C(aryl)—O bonds corresponding to minimum conjugation of these groups with the aromatic rings. The angles between the mean planes of C(14)—O(39)—C(40) and C(29)—O(34)—C(35) and the mean planes of their respective aryl rings are 88 (1) and 83 (1)°, respectively. Surprisingly, the two methyl groups are directed away from the central cavity region of the macrocycle rather than helping to fill it. The molecule has diastereotopic faces with the two aryl methoxy groups both lying on the more hindered face. The two aryl groups are tilted slightly with respect to each other with a cleft angle of 34 (1)° between the mean planes of the aromatic rings. Significantly, the shapes of the two dianhydromannitolo residues match (Table 2) each other very closely (equivalent torsional angles within 5°) in DD-(3), whereas this is not the case in DD-(2), which displays (Crawshaw et al., 1986) internal differences between equivalent torsional angles of just greater than 50°.

There are four intermolecular contacts of less than 3·4 Å. The shortest of these is 3·22 (1) Å between C(38) and O(8) with an associated H···O distance of 2·45 Å.

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* In the context of this and the following paper 'gauche' and 'anti' are synonymous with synclinal and antiperiplanar, respectively.

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Structure of a Chiral Macrobicyclic Crown Ether, 1,4:1',4':3,6:3',6'-Tetraanhydro-2,2':5,5'-O-[2,2'-(3,6-dioxaoctane-1,8-diyldioxy)bis(1,3-phenylene)tetrakis(methylene)]di-D-mannitol Dichloromethane Solvate

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Abstract. $C_{34}H_{42}O_{12}.CH_2Cl_2$, $M_r = 727·63$, orthorhombic, $P2_12_12_1$, $a = 12·337$ (2), $b = 15·538$ (2), $c = 18·191$ (4) Å, $V = 3487$ Å^3, $Z = 4$, $D_x = 1·39$ g cm^{-3}, $\lambda(Cu\ K\alpha) = 1·54178$ Å, $\mu = 22·1$ cm^{-1}, $F(000) = 1536$, room temperature, $R = 0·049$ for 2414 unique observed reflections. The chiral macrocycle