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

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Abstract. *rel*-(2*R*,3*R*,11*S*,12*S*)-2,3,11,12-Tetrakis(4-methoxyphenyl)-1,4,7,10,13,16-hexaoxacyclooctadecane–ammonia–borane (1/2/2), $C_{40}H_{48}O_{10}.B_2H_6N_2H_6$, $M_r = 751$, triclinic, $P\bar{1}$, $a = 8{\cdot}742\,(2)$, $b = 13{\cdot}727\,(4)$, $c = 18{\cdot}616\,(5)$ Å, $\alpha = 92{\cdot}01\,(3)$, $\beta = 97{\cdot}76\,(2)$, $\gamma = 102{\cdot}98\,(2)°$, $V = 2151$ Å^3, $Z = 2$, $D_x = 1{\cdot}16$ Mg m^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1{\cdot}54178$ Å, $\mu = 0{\cdot}63$ mm^{-1}, $F(000) = 808$, room temperature, $R = 0{\cdot}051$ for 3914 unique observed reflections with $|F_o| > 3\sigma(|F_o|)$. In two crystallographically independent centrosymmetric 1:2 adducts, two molecules of BH_3NH_3 are hydrogen bonded through their NH_3 groups, each by two-point binding to two of the nearer oxygen atoms on the homotopic faces of the title receptor (3) wherein the macrocycle adopts an all-*gauche* conformation: all four anisyl substituents are equatorial.

Introduction. It is known that *trans–cisoid–trans*-2,3,11,12-tetraphenyl-18-crown-6 (8)* forms a 1:1 complex with sodium iodide (Weber, Sheldrick, Burgemeister, Dietl, Mannschreck & Merz, 1984) and a 1:2 adduct $[(8).(BH_3NH_3)_2]$ with ammonia–borane (Allwood, Shahriari-Zavareh, Stoddart & Williams, 1984). We therefore expected the *trans–cisoid–trans* isomer (3)† of 2,3,11,12-tetra-anisyl-18-crown-6 (paper V) to crystallize with two molar equivalents of ammonia–borane and so afford $[(3).(BH_3NH_3)_2]$. This paper reports that this expectation was fulfilled and establishes that the solid-state structure of $[(3).(BH_3NH_3)_2]$ is very similar to that of $[(8).(BH_3NH_3)_2]$.

Experimental. Addition of an excess of ammonia–borane to a solution of (3) in dichloromethane afforded (Pears, 1985) a solid residue on evaporation of the solvent. Crystallization of this product from dichloromethane layered with *n*-pentane provided single crystals of $[(3).(BH_3NH_3)_2]$ which were found to be suitable for X-ray structural investigation. Crystal size: $0{\cdot}50 \times 0{\cdot}40 \times 0{\cdot}10$ mm. Refined unit-cell parameters obtained from setting angles of 18 reflections. Nicolet *R*3*m* diffractometer. 4437 independent reflections ($\theta \leq 50°$) measured, Cu $K\alpha$ radiation (graphite monochromator), ω scan. 3914 $[|F_o| > 3\sigma(|F_o|)]$ considered observed, index range h −8/8, k −13/13, l 0/18; two check reflections measured every 50 reflections, net count constant; Lorentz and polarization corrections, no absorption correction. Structure solved by direct methods. Successive ΔF maps revealed disorder in the C(16) anisyl group of one of the independent crown moieties. This portion of the molcule was refined as two overlapping orientations, each of half-weight occupancy; non-hydrogen atoms refined anisotropically; BH_3, NH_3 and CH_3 groups refined as idealized rigid bodies (B—H 1·08, N—H 0·96 Å). Positions of other H atoms calculated (C—H 0·96 Å); H atoms assigned isotropic thermal parameters, $U(\mathrm{H}) = 1{\cdot}2\ U_{\mathrm{eq}}(X)$ (X = C, N, B), and allowed to ride on parent X atoms. An empirical extinction correction was applied ($g = 0{\cdot}00508$). Refinement using F magnitudes by block-cascade full-matrix least squares; $R = 0{\cdot}051$; $wR = 0{\cdot}061$ $[w^{-1} = \sigma^2(F) + 0{\cdot}00060F^2]$. $(\Delta/\sigma)_{\mathrm{max}} = 0{\cdot}286$; residual electron density in difference map within $-0{\cdot}16$ and $0{\cdot}35$ e Å^{-3}; atomic scattering factors and dispersion corrections from *International Tables for X-ray*

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

† See previous footnote.

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Crystallography (1974). Computations carried out on an Eclipse S140 computer using the *SHELXTL* program system (Sheldrick, 1983).

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

	x	y	z	U_{eq}†
O(1)	6471 (2)	3268 (2)	9998 (1)	90 (1)
C(2)	8118 (4)	3617 (3)	10290 (2)	96 (1)
C(3)	8288 (4)	4265 (3)	10962 (2)	104 (2)
O(4)	7894 (2)	5181 (2)	10778 (1)	92 (1)
C(5)	8294 (4)	5906 (3)	11366 (2)	113 (2)
C(6)	7825 (4)	6834 (3)	11127 (2)	114 (2)
O(7)	6131 (2)	6635 (2)	10963 (1)	91 (1)
C(8)	5529 (3)	7520 (2)	10986 (2)	78 (1)
C(9)	6248 (3)	2782 (2)	9292 (1)	72 (1)
C(10)	5835 (3)	8017 (2)	11740 (1)	63 (1)
C(11)	6476 (4)	9009 (2)	11891 (1)	77 (1)
C(12)	6706 (4)	9470 (2)	12587 (1)	74 (1)
C(13)	6281 (3)	8898 (2)	13139 (1)	66 (1)
C(14)	5651 (3)	7888 (2)	13006 (1)	74 (1)
C(15)	5434 (3)	7457 (2)	12316 (2)	71 (1)
C(16)	6945 (3)	1874 (2)	9288 (1)	72 (1)
C(17)	7999 (20)	1809 (9)	8802 (8)	68 (4)
C(18)	8402 (8)	899 (5)	8668 (3)	82 (3)
C(19)	8328 (14)	95 (6)	9110 (5)	79 (4)
C(20)	7438 (15)	178 (6)	9667 (5)	77 (5)
C(21)	6877 (6)	1049 (3)	9717 (3)	66 (2)
O(24)	9032 (4)	−713 (2)	9037 (2)	74 (2)
C(25)	8655 (8)	−1548 (4)	9517 (3)	88 (3)
C(16*)	6945 (3)	1874 (2)	9288 (1)	72 (1)
C(17*)	7999 (21)	1809 (10)	8802 (7)	68 (4)
C(18*)	8556 (6)	842 (3)	8832 (3)	80 (3)
C(19*)	7943 (10)	247 (4)	9364 (4)	56 (5)
C(20*)	7017 (15)	425 (7)	9885 (5)	95 (4)
C(21*)	6401 (6)	1273 (4)	9834 (2)	60 (2)
O(24*)	8415 (4)	−649 (3)	9454 (2)	96 (2)
C(25*)	9337 (8)	−966 (5)	8913 (4)	125 (4)
O(22)	6471 (3)	9252 (2)	13851 (1)	97 (1)
C(23)	6897 (5)	10295 (2)	14011 (2)	102 (2)
O(26)	4480 (2)	2911 (1)	4380 (1)	62 (1)
C(27)	4949 (4)	2323 (2)	4952 (1)	74 (1)
C(28)	6503 (3)	2872 (2)	5372 (2)	76 (1)
O(29)	6271 (2)	3705 (1)	5775 (1)	61 (1)
C(30)	7568 (3)	4135 (2)	6324 (1)	71 (1)
C(31)	7249 (4)	5032 (2)	6686 (1)	72 (1)
O(32)	7191 (2)	5769 (1)	6169 (1)	63 (1)
C(33)	2433 (3)	3232 (2)	3506 (1)	57 (1)
C(34)	2819 (3)	2566 (2)	4102 (1)	56 (1)
C(35)	694 (3)	2926 (2)	3179 (1)	59 (1)
C(36)	218 (4)	2463 (2)	2502 (1)	80 (1)
C(37)	−1404 (4)	2196 (3)	2203 (2)	90 (1)
C(38)	−2480 (3)	2418 (2)	2603 (2)	78 (1)
C(39)	−2031 (4)	2876 (2)	3282 (2)	85 (1)
C(40)	−466 (3)	3127 (2)	3570 (2)	76 (1)
C(41)	2394 (3)	1472 (2)	3843 (1)	56 (1)
C(42)	1240 (3)	788 (2)	4113 (1)	67 (1)
C(43)	832 (3)	−213 (2)	3868 (1)	72 (1)
C(44)	1578 (3)	−548 (2)	3345 (2)	68 (1)
C(45)	2767 (3)	128 (2)	3076 (2)	74 (1)
C(46)	3152 (3)	1116 (2)	3320 (1)	69 (1)
O(47)	−4109 (3)	2193 (2)	2362 (1)	119 (1)
C(48)	−4685 (5)	1764 (3)	1664 (2)	132 (2)
O(49)	1264 (2)	−1510 (1)	3053 (1)	91 (1)
C(50)	−35 (4)	−2201 (2)	3249 (2)	105 (2)
N(1)	3530 (3)	4732 (2)	5489 (1)	71 (1)
B(1)	2043 (6)	4273 (4)	5867 (3)	126 (2)
N(2)	4012 (3)	4526 (2)	10649 (1)	94 (1)
B(2)	2959 (7)	4216 (4)	11283 (3)	134 (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)	1·435 (3)	O(1)–C(9)	1·422 (3)
C(2)–C(3)	1·480 (5)	C(3)–O(4)	1·417 (4)
O(4)–C(5)	1·406 (4)	C(5)–C(6)	1·487 (6)
C(6)–O(7)	1·432 (4)	O(7)–C(8)	1·430 (4)
C(8)–C(10)	1·502 (4)	C(8)–C(9′)	1·528 (4)
C(9)–C(16)	1·505 (4)	C(9)–C(16*)	1·505 (4)

Table 2 (*cont.*)

C(9)–C(8′)	1·528 (4)	C(10)–C(11)	1·355 (4)
C(10)–C(15)	1·379 (4)	C(11)–C(12)	1·391 (4)
C(12)–C(13)	1·356 (4)	C(13)–C(14)	1·372 (4)
C(13)–O(22)	1·372 (3)	C(14)–C(15)	1·367 (4)
C(16)–C(17)	1·391 (18)	C(16)–C(21)	1·404 (5)
C(17)–C(18)	1·395 (16)	C(18)–C(19)	1·394 (11)
C(19)–C(20)	1·394 (16)	C(19)–O(24)	1·395 (11)
C(20)–C(21)	1·395 (12)	O(24)–C(25)	1·485 (6)
C(16*)–C(17*)	1·390 (16)	C(16*)–C(21*)	1·397 (5)
C(17*)–C(18*)	1·394 (18)	C(18*)–C(19*)	1·393 (9)
C(19*)–C(20*)	1·394 (15)	C(19*)–O(24*)	1·390 (8)
C(20*)–C(21*)	1·389 (13)	O(24*)–C(25*)	1·483 (8)
O(22)–C(23)	1·406 (4)	O(26)–C(17)	1·429 (3)
O(26)–C(34)	1·439 (3)	C(27)–C(28)	1·495 (4)
C(28)–O(29)	1·414 (3)	O(29)–C(30)	1·421 (3)
C(30)–C(31)	1·482 (4)	C(31)–O(32)	1·424 (3)
O(32)–C(33′)	1·427 (3)	C(33)–C(34)	1·514 (3)
C(33)–C(35)	1·517 (3)	C(33)–O(32′)	1·427 (3)
C(34)–C(41)	1·509 (3)	C(35)–C(36)	1·359 (3)
C(35)–C(40)	1·395 (4)	C(36)–C(37)	1·412 (4)
C(37)–C(38)	1·355 (5)	C(38)–C(39)	1·359 (4)
C(38)–O(47)	1·394 (3)	C(39)–C(40)	1·362 (4)
C(41)–C(42)	1·378 (3)	C(41)–C(46)	1·383 (4)
C(42)–C(43)	1·383 (4)	C(43)–C(44)	1·364 (4)
C(44)–C(45)	1·391 (4)	C(44)–O(49)	1·363 (3)
C(45)–C(46)	1·366 (4)	O(47)–C(48)	1·388 (4)
O(49)–C(50)	1·406 (4)	N(1)–B(1)	1·579 (6)
N(2)–B(2)	1·605 (6)		

C(2)–O(1)–C(9)	112·2 (2)	O(1)–C(2)–C(3)	109·9 (3)
C(2)–C(3)–O(4)	109·2 (3)	C(3)–O(4)–C(5)	112·6 (2)
O(4)–C(5)–C(6)	108·9 (3)	C(5)–C(6)–O(7)	109·1 (3)
C(6)–O(7)–C(8)	113·2 (2)	O(7)–C(8)–C(10)	111·7 (2)
O(7)–C(8)–C(9′)	107·6 (2)	C(10)–C(8)–C(9′)	110·9 (2)
O(1)–C(9)–C(16)	112·2 (2)	O(1)–C(9)–C(16*)	112·2 (2)
O(1)–C(9)–C(8′)	108·5 (2)	C(16)–C(9)–C(8′)	110·0 (2)
C(16*)–C(9)–C(8′)	110·0 (2)	C(8)–C(10)–C(11)	123·3 (2)
C(8)–C(10)–C(15)	119·7 (2)	C(11)–C(10)–C(15)	117·1 (2)
C(10)–C(11)–C(12)	122·9 (3)	C(11)–C(12)–C(13)	118·5 (2)
C(12)–C(13)–C(14)	120·1 (2)	C(12)–C(13)–O(22)	124·4 (2)
C(14)–C(13)–O(22)	115·5 (2)	C(13)–C(14)–C(15)	120·0 (3)
C(10)–C(15)–C(14)	121·4 (2)	C(9)–C(16)–C(17)	118·1 (6)
C(9)–C(16)–C(21)	134·4 (3)	C(17)–C(16)–C(21)	107·3 (6)
C(16)–C(17)–C(18)	119·7 (10)	C(17)–C(18)–C(19)	128·4 (9)
C(18)–C(19)–C(20)	111·7 (9)	C(18)–C(19)–O(24)	126·9 (8)
C(20)–C(19)–O(24)	121·5 (7)	C(19)–C(20)–C(21)	116·6 (8)
C(16)–C(21)–C(20)	133·1 (6)	C(19)–O(24)–C(25)	118·0 (5)
C(9)–C(16*)–C(17*)	124·2 (6)	C(9)–C(16*)–C(21*)	108·7 (3)
C(17*)–C(16*)–C(21*)	126·1 (6)	C(16*)–C(17*)–C(18*)	120·6 (9)
C(17*)–C(18*)–C(19*)	108·0 (8)	C(18*)–C(19*)–C(20*)	131·1 (7)
C(18*)–C(19*)–O(24*)	117·1 (7)	C(20*)–C(19*)–O(24*)	111·6 (7)
C(19*)–C(20*)–C(21*)	117·3 (8)	C(16*)–C(21*)–C(20*)	112·9 (6)
C(19*)–O(24*)–C(25*)	117·7 (5)	C(13)–O(22)–C(23)	118·3 (2)
C(27)–O(26)–C(34)	111·2 (2)	O(26)–C(27)–C(28)	109·5 (2)
C(27)–C(28)–O(29)	109·1 (2)	C(28)–O(29)–C(30)	113·6 (2)
O(29)–C(30)–C(31)	109·4 (2)	C(30)–C(31)–O(32)	108·7 (2)
C(31)–O(32)–C(33′)	113·2 (2)	C(34)–C(33)–C(35)	111·3 (2)
C(34)–C(33)–O(32′)	106·6 (2)	C(35)–C(33)–O(32′)	110·4 (2)
O(26)–C(34)–C(33)	107·4 (2)	O(26)–C(34)–C(41)	112·1 (2)
C(33)–C(34)–C(41)	112·4 (2)	C(33)–C(35)–C(36)	121·7 (3)
C(33)–C(35)–C(40)	120·2 (2)	C(36)–C(35)–C(40)	118·1 (2)
C(35)–C(36)–C(37)	121·0 (3)	C(36)–C(37)–C(38)	118·5 (3)
C(37)–C(38)–C(39)	121·5 (3)	C(37)–C(38)–O(47)	123·5 (3)
C(39)–C(38)–O(47)	115·0 (3)	C(38)–C(39)–C(40)	119·7 (3)
C(35)–C(40)–C(39)	121·2 (3)	C(34)–C(41)–C(42)	121·8 (2)
C(34)–C(41)–C(46)	121·0 (2)	C(42)–C(41)–C(46)	117·2 (2)
C(41)–C(42)–C(43)	121·8 (3)	C(42)–C(43)–C(44)	120·1 (2)
C(43)–C(44)–C(45)	118·8 (2)	C(43)–C(44)–O(49)	125·5 (2)
C(45)–C(44)–O(49)	115·7 (3)	C(44)–C(45)–C(46)	120·5 (3)
C(41)–C(46)–C(45)	121·6 (2)	C(38)–O(47)–C(48)	119·5 (3)
C(44)–O(49)–C(50)	118·0 (3)		

C(9)–O(1)–C(2)–C(3)	170·0 (3)	C(34)–O(26)–C(27)–C(28)	−162·9 (2)
C(2)–O(1)–C(9)–C(8′)	−177·5 (2)	O(26)–C(27)–C(28)–O(29)	70·3 (3)
O(1)–C(2)–C(3)–O(4)	−70·5 (3)	C(27)–C(28)–C(29)–C(30)	165·7 (2)
C(2)–C(3)–O(4)–C(5)	−169·2 (3)	C(28)–O(29)–C(30)–C(31)	176·9 (2)
C(3)–O(4)–C(5)–C(6)	−179·3 (3)	O(29)–C(30)–C(31)–O(32)	−65·0 (3)
O(4)–C(5)–C(6)–O(7)	66·0 (4)	C(30)–C(31)–O(32)–C(33′)	−154·8 (2)
C(5)–C(6)–O(7)–C(8)	158·6 (3)	C(31)–O(32)–C(33′)–C(34′)	−170·4 (2)
C(6)–O(7)–C(8)–C(9)	172·8 (2)	O(32′)–C(33)–C(34)–O(26)	−59·5 (2)
O(7)–C(8)–C(9′)–O(1′)	−56·3 (2)	C(27)–O(26)–C(34)–C(33)	179·7 (2)
O(7′)–C(8′)–C(9)–C(16)	179·3 (2)	C(35)–C(33)–C(34)–O(26)	−179·8 (2)
C(10′)–C(8′)–C(9)–C(16)	−58·2 (3)	C(35)–C(33)–C(34)–C(41)	56·5 (3)
O(1)–C(9)–C(8′)–C(10′)	178·8 (2)	O(32′)–C(33)–C(34)–C(41)	176·9 (2)
C(9′)–C(8)–C(10)–C(15)	70·3 (4)	C(34)–C(33)–C(35)–C(40)	72·8 (3)
C(8′)–C(9)–C(16)–C(21)	−73·1 (4)	C(33)–C(34)–C(41)–C(46)	64·4 (3)
C(8′)–C(9)–C(16′)–C(21′)	−70·4 (3)		

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 $[(3).(BH_3NH_3)_2]$ is illustrated in Fig. 1 which includes the atomic numbering scheme for the crystallographically independent adducts *A* and *B*. Fig. 2 shows a space-filling representation of adduct *B* viewed normal to the plane of the macrocyclic ring.

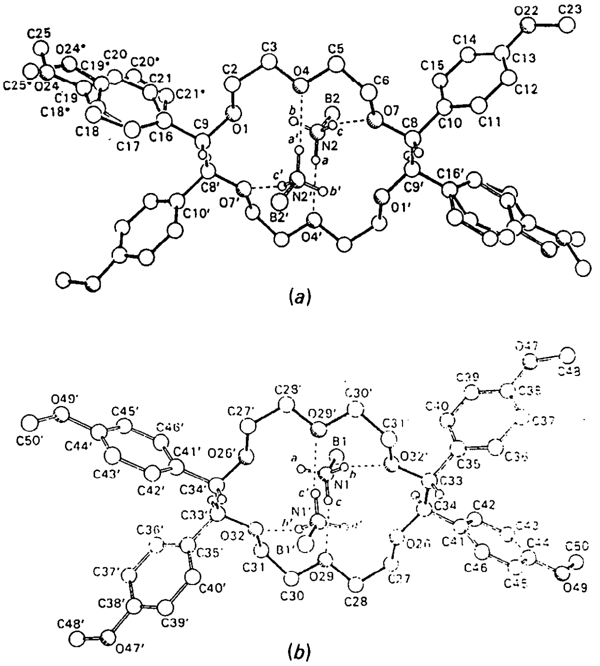

Fig. 1. Ball-and-stick representations of the structure of $[(3).(BH_3NH_3)_2]$ showing the atomic numbering scheme in the crystallographically independent 1:2 adducts, (*a*) adduct *A* and (*b*) adduct *B*. In both cases the B—N bonds and the 18-crown-6 ring are highlighted with solid B—N, C—C and C—O bonds. In (*a*), the two orientations adopted by one of the anisyl groups are illustrated using open and closed bonds.

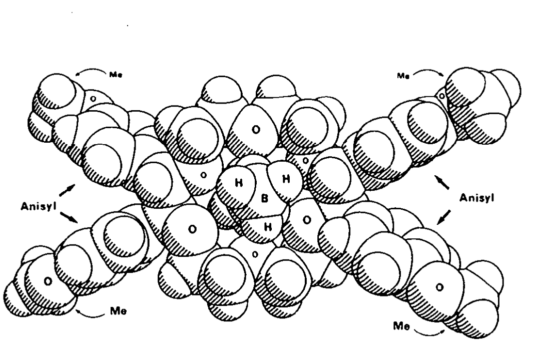

Fig. 2. Space-filling representation of adduct *B* of $[(3).(BH_3NH_3)_2]$ viewed normal to the plane of the macrocyclic ring.

Table 3. *The* N···O *contact distances and relevant hydrogen-bond parameters, where appropriate, for adducts A and B of* $[(3).(BH_3NH_3)_2]$ *listed in order of increasing* N···O *distances*

Contact	*R*(N···O) (Å)	ψ (°)*	θ (°)†
N(2)···O(4′)	3·02 (1)	167‡	4‡
N(2)···O(7)	3·06 (1)	169§	3§
N(2)···O(4)	3·29 (1)	—	—
N(2)···O(7′)	3·33 (1)	—	—
N(2)···O(1)	3·36 (1)	154¶	16¶
N(2)···O(1′)	3·37 (1)	—	—
N(1)···O(29)	3·04 (1)	163‡	0‡
N(1)···O(32′)	3·08 (1)	166¶	6¶
N(1)···O(29′)	3·24 (1)	—	—
N(1)···O(32)	3·26 (1)	—	—
N(1)···O(26′)	3·29 (1)	163§	23§
N(1)···O(26)	3·49 (1)	—	—

* N—H···O angles.
† Angles between COC planes and HO vectors.
‡ H(*c*).
§ H(*a*).
¶ H(*b*).

The structural differences between adducts *A* and *B* are slight (compare Figs. 1*a* and 1*b*) and involve principally alternative relative orientations of methoxy groups all of which are aligned approximately coplanar with their associated aromatic rings. In adduct *A*, one of the symmetry-related pairs of anisyl substituents is disordered (see *Experimental*) resulting in the methoxy groups lying in opposite directions with respect to each other whilst in adduct *B* the methoxy groups are ordered (see Figs. 1*a* and 1*b*). Thus, to all intents and purposes, the gross structural features of adducts *A* and *B* are the same. On adduct formation with two molecules of BH_3NH_3, the 18-crown-6 ring undergoes major conformational changes from a geometry (paper V) very similar to that of 18-crown-6 (Dunitz & Seiler, 1974) to adopt the omnipresent all-*gauche* conformation with local pseudo-D_{3d} symmetry (Wipff, Weiner & Kollman, 1982; Wipff, Kollman & Lehn, 1983). The O atoms in the macrocyclic rings alternate 0·18 (1) and 0·19 (1) Å respectively above and below the mean plane of these six atoms in adducts *A* and *B*. In both cases, all four anisyl substituents assume equatorial orientations in a manner reminiscent of the four phenyl groups in *trans–cisoid–trans*-tetraphenyl-18-crown-6 (Allwood *et al.*, 1984).

In adducts *A* and *B*, the BH_3NH_3 molecules are bound (Figs. 1 and 2) in perching modes (Trueblood, Knobler, Lawrence & Stevens, 1982) to opposite faces of the macrocycle. Details of the noncovalent bonding pattern are given in Table 3. Note that in both adducts one of the hydrogen bonds is significantly longer than the other two, a feature which characterizes the 1:2 adducts of *cis–transoid–cis*-tetraphenyl-18-crown-6 (2) (Pears, Shahriari-Zavareh, Stoddart, Crosby, Allwood & Williams, 1988*a*; paper IV) and *trans–cisoid–trans*-tetraphenyl-18-crown-6 (7) (Allwood *et al.*,

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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 44788 (29 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

1984). The through-the-ring N···N′ distances are 3·30 (1) and 3·32 (1) Å in adducts *A* and *B* respectively: these values compare with 3·21 (1) Å in [(2).$(BH_3NH_3)_2$] and 3·59 (1) Å in [(7).$(BH_3NH_3)_2$]. The NH_3 and BH_3 groups in the substrate are in a somewhat distorted staggered relationship to each other, the smallest H—N—B—H torsion angles being 37 and 44° in adducts *A* and *B* respectively. The N atoms are displaced 1·64 (1) and 1·66 (1) Å from the best mean planes of the six O atoms in the macrocycles and the B—N bonds are inclined 11 and 10° to the normals to these planes. The corresponding parameters in [(7).$(BH_3NH_3)_2$] are (Allwood *et al.*, 1984) 1·78 Å and 24°.

There are no contacts of less than normal van der Waals distances between the 1:2 adducts.

The similarities in the solid-state structures of [(3).$(BH_3NH_3)_2$] and [(7).$(BH_3NH_3)_2$] are reassuring and give us confidence that more often than not 'snapshots' of the solid state will be a fairly good guide to the supramolecular structures that exist in solution.

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Structure of the Dextrorotatory *trans–transoid–trans* Isomer of 2,3,11,12-Tetra-anisyl-18-crown-6: (2*R*,3*R*,11*R*,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, $P2_1$, $a = 8{\cdot}494\,(1)$, $b = 11{\cdot}419\,(1)$, $c = 19{\cdot}351\,(3)$ Å, $\beta = 91{\cdot}43\,(1)°$, $V = 1876$ Å^3, $Z = 2$, $D_x = 1{\cdot}219$ Mg m^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1{\cdot}54178$ Å, $\mu = 0{\cdot}67$ mm^{-1}, $F(000) = 736$, room temperature, $R = 0{\cdot}054$ for 2311 observed reflections with $|F_o| > 3\sigma(|F_o|)$. The 18-membered ring of the title compound (4) adopts a conformation with local non-crystallographic C_2 symmetry in which all six OCH_2CH_2O units have *gauche* geometry: all four anisyl groups are equatorial: the vicinal groups have a *gauche* relationship to each other.

Introduction. Although good-quality single crystals of *trans–transoid–trans*-2,3,11,12-tetraphenyl-18-crown-

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