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}, $\lambda(\text{Cu }K\alpha) = 1{\cdot}54178$ 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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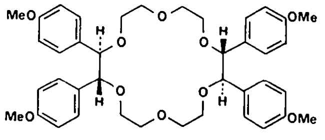
6 (9)[*] with the (*RRRR*)-configuration have been obtained (Allwood, Shahriari-Zavareh, Stoddart & Williams, 1984), their X-ray crystal structure solution has proved intractable to date as a result of (*a*) their crystallization in a non-centrosymmetric space group, (*b*) the presence of more than one independent molecule in the asymmetric unit, and (*c*) additional included solvent molecules. This problem has now been circumvented by investigating the crystal structure of the *trans–transoid–trans* isomer (4) of 2,3,11,12-tetra-anisyl-18-crown-6. This paper describes (*a*) the spontaneous resolution of (4) during crystallization and (*b*) the determination of the solid-state structure of the (*RRRR*)-enantiomer.



(4)

Experimental. Chromatographic separation (Pears, Shahriari-Zavareh, Stoddart, Crosby, Allwood & Williams, 1988*b*; paper V) of the *trans–transoid–trans*-(4) from the *trans–cisoid–trans*-(3) isomer of 2,3,11,12-tetra-anisyl-18-crown-6 was followed by crystallization of the former from 2-methoxyethanol. This procedure not only yielded good single crystals (with m.p. 432–433 K) suitable for X-ray structural investigation, but also effected the spontaneous resolution of the racemic modification obtained by synthesis in a manner that yielded a dextrorotatory sample, the molecules of which can be assigned (Pears, 1985) the (*RRRR*)-configuration. Crystal size: $0.40 \times 0.40 \times 0.40$ mm. Refined unit-cell parameters obtained by centring 18 reflections. Nicolet *R*3*m* diffractometer. 2685 independent reflections ($\theta \leq 58°$) measured, Cu $K\alpha$ radiation (graphite monochromator), ω scan. 2311 [$|F_o| > 3\sigma(|F_o|)$] considered observed, index range h −9/9, k 0/12, l 0/18; two check reflections measured every 50 reflections, net count constant, Lorentz and polarization corrections, no absorption correction. Structure solved, though with some difficulty, by direct methods. Several attempts using automatically selected starting sets of varying sizes failed to produce a meaningful solution. The structure was finally solved using a starting set comprised of three origin-defining terms, a weak link in the convergence map, the four principal contributors to the list of negative quartets and a further 12 automatically selected terms. ΔE map recycling of the fourth best phase solution gave the positions of all non-hydrogen atoms. Non-hydrogen atoms refined anisotropically; positions of H atoms calculated (C—H = 0·96 Å); H atoms assigned isotropic thermal parameters, $U(\text{H}) = 1\cdot2\, U_{\text{eq}}(\text{C})$, allowed to ride on parent C atoms. The methoxy CH_3 groups refined as rigid bodies. An empirical extinction correction was applied ($g = 0\cdot00489$). Refinement using F magnitudes by block-cascade full-matrix least squares; $R = 0\cdot054$, $wR = 0\cdot059$ [$w^{-1} = \sigma^2(F) + 0\cdot0015F^2$]. $(\Delta/\sigma)_{\max} = 0\cdot15$; residual electron density in difference map within −0·19 and 0·24 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 (4) is illustrated in Fig. 1 which includes the atomic numbering scheme. The macrocyclic ring has local non-crystallographic C_2 symmetry about an axis normal to the ring plane. The conformation differs from that (Dunitz & Seiler, 1974) of 18-crown-6 and the previous three configurational diastereoisomers [(1)–(3)] already discussed in papers I, V and by Pears, Shahriari-Zavareh, Stoddart, Crosby, Allwood & Williams (1988*a*; paper III). The sequence of O—C—C—O subunit torsional angles, which reads ag^+g^- ag^-a ag^-a ag^+g^- ag^-a ag^-a starting from O(1), reveals that the macrocycle contains no *anti* bismethylenedioxy units. Both pairs of vicinal anisyl groups are oriented equatorial and approximately *gauche* to each other, though as in *trans–cisoid–trans*-2,3,11,12-tetra-anisyl-18-crown-6 (3) (paper V), the Ar—C—C—Ar torsion angles are again significantly reduced to 42·7 (6) and 48·6 (6)°. Also, in common with the *trans–cisoid–trans* isomer (3), the combination of equatorial orientations of the four anisyl groups, coupled with the reduction in the Ar—C—C—Ar torsion angles, results in both faces of the macrocycle being not obscured.

There are several short contacts between symmetry-related molecules: C(50)···O(16) 3·16 (1), C(47)···C(39) 3·31 (1), O(47)···C(38) 3·38 (1), C(50)···O(7) 3·39 (1), and C(44)···O(13) 3·39 (1) Å. All of these contacts involve methoxy groups, which are aligned approximately coplanar (Makriyannis & Fesik, 1982; Kruse & Cha, 1982; Mersh, Saunders & Matlin, 1983) with their associated aromatic rings.

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

[*] 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 44789 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

	x	y	z	U_{eq}*
O(1)	7143 (4)	4334 (3)	3604 (2)	59 (1)
C(2)	7692 (7)	3422 (6)	4051 (3)	69 (2)
C(3)	8960 (7)	2767 (6)	3700 (3)	77 (2)
O(4)	8419 (5)	2019 (4)	3160 (2)	70 (1)
C(5)	8214 (7)	2604 (5)	2510 (3)	64 (2)
C(6)	7890 (6)	1700 (5)	1967 (3)	58 (2)
O(7)	6339 (4)	1276 (3)	2021 (2)	53 (1)
C(8)	5860 (5)	501 (5)	1486 (2)	49 (2)
C(9)	4070 (6)	505 (5)	1467 (3)	51 (2)
O(10)	3647 (4)	1641 (3)	1244 (2)	60 (1)
C(11)	2038 (6)	1938 (5)	1313 (3)	62 (2)
C(12)	1818 (6)	3191 (5)	1107 (3)	60 (2)
O(13)	2435 (4)	4007 (3)	1596 (2)	60 (1)
C(14)	4072 (6)	4248 (5)	1539 (3)	58 (2)
C(15)	4516 (7)	5221 (5)	2027 (3)	61 (2)
O(16)	4607 (4)	4732 (3)	2706 (2)	56 (1)
C(17)	5059 (6)	5572 (5)	3228 (2)	52 (2)
C(18)	5708 (6)	4860 (5)	3838 (3)	51 (2)
C(19)	6623 (5)	−690 (5)	1567 (3)	51 (2)
C(20)	6796 (6)	−1429 (5)	994 (3)	62 (2)
C(21)	7539 (6)	−2481 (6)	1076 (3)	69 (2)
C(22)	8138 (6)	−2857 (5)	1708 (3)	62 (2)
C(23)	7970 (6)	−2155 (5)	2276 (3)	61 (2)
C(24)	7212 (6)	−1077 (5)	2200 (3)	54 (2)
C(25)	3315 (5)	−447 (5)	1038 (2)	50 (2)
C(26)	3231 (6)	−353 (5)	320 (3)	62 (2)
C(27)	2577 (6)	−1237 (5)	−78 (3)	60 (2)
C(28)	2026 (6)	−2242 (5)	233 (3)	58 (2)
C(29)	2064 (6)	−2349 (6)	943 (3)	64 (2)
C(30)	2716 (6)	−1432 (5)	1337 (3)	58 (2)
C(31)	3681 (5)	6345 (5)	3426 (3)	49 (2)
C(32)	2401 (6)	5861 (5)	3759 (3)	63 (2)
C(33)	1186 (7)	6568 (5)	3967 (3)	73 (2)
C(34)	1225 (6)	7753 (5)	3847 (3)	62 (2)
C(35)	2469 (7)	8238 (5)	3518 (3)	67 (2)
C(36)	3701 (6)	7515 (5)	3320 (3)	58 (2)
C(37)	6009 (5)	5570 (4)	4483 (2)	47 (2)
C(38)	6972 (5)	6556 (5)	4479 (3)	53 (2)
C(39)	7267 (6)	7206 (5)	5069 (3)	56 (2)
C(40)	6577 (6)	6872 (5)	5673 (2)	52 (2)
C(41)	5622 (6)	5929 (5)	5699 (3)	58 (2)
C(42)	5335 (7)	5274 (5)	5102 (3)	58 (2)
O(43)	8884 (5)	−3912 (4)	1714 (2)	77 (2)
C(44)	9731 (8)	−4235 (7)	2331 (4)	84 (3)
O(45)	1404 (5)	−3082 (4)	−207 (2)	73 (1)
C(46)	1036 (7)	−4189 (6)	71 (3)	77 (2)
O(47)	−63 (5)	8380 (4)	4066 (3)	90 (2)
C(48)	−87 (8)	9598 (7)	3972 (5)	104 (3)
O(49)	6960 (4)	7582 (4)	6234 (2)	69 (1)
C(50)	6515 (10)	7181 (6)	6904 (3)	92 (3)

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

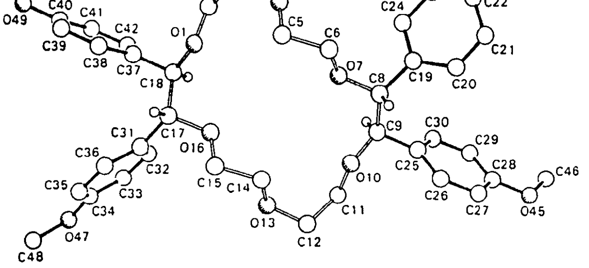

Fig. 1. Ball-and-stick representation of the structure of (4) showing the atomic numbering scheme. The anisyl substituents and the methine hydrogen atoms on the macrocycle are highlighted with solid C—C and C—H bonds.

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

O(1)—C(2)	1·425 (7)	O(1)—C(18)	1·443 (6)
C(2)—C(3)	1·490 (9)	C(3)—O(4)	1·417 (7)
O(4)—C(5)	1·432 (7)	C(5)—C(6)	1·494 (8)
C(6)—O(7)	1·410 (6)	O(7)—C(8)	1·415 (6)
C(8)—C(9)	1·520 (7)	C(8)—C(19)	1·512 (7)
C(9)—O(10)	1·411 (6)	C(9)—C(25)	1·501 (7)
O(10)—C(11)	1·418 (6)	C(11)—C(12)	1·496 (8)
C(12)—O(13)	1·419 (7)	O(13)—C(14)	1·424 (6)
C(14)—C(15)	1·500 (8)	C(15)—O(16)	1·428 (6)
O(16)—C(17)	1·438 (6)	C(17)—C(18)	1·524 (7)
C(17)—C(31)	1·522 (7)	C(18)—C(37)	1·505 (7)
C(19)—C(20)	1·403 (8)	C(19)—C(24)	1·384 (7)
C(20)—C(21)	1·365 (8)	C(21)—C(22)	1·381 (8)
C(22)—C(23)	1·372 (8)	C(22)—O(43)	1·361 (7)
C(23)—C(24)	1·395 (8)	C(25)—C(26)	1·394 (7)
C(25)—C(30)	1·369 (8)	C(26)—C(27)	1·378 (8)
C(27)—C(28)	1·382 (8)	C(28)—C(29)	1·378 (7)
C(28)—O(45)	1·379 (7)	C(29)—C(30)	1·402 (8)
C(31)—C(32)	1·392 (7)	C(31)—C(36)	1·352 (8)
C(32)—C(33)	1·378 (8)	C(33)—C(34)	1·374 (8)
C(34)—C(35)	1·365 (8)	C(34)—O(47)	1·383 (7)
C(35)—C(36)	1·395 (8)	C(37)—C(38)	1·391 (7)
C(37)—C(42)	1·382 (7)	C(38)—C(39)	1·379 (7)
C(39)—C(40)	1·375 (7)	C(40)—C(41)	1·350 (8)
C(40)—O(49)	1·386 (6)	C(41)—C(42)	1·393 (7)
O(43)—C(44)	1·427 (8)	O(45)—C(46)	1·411 (8)
O(47)—C(48)	1·402 (9)	O(49)—C(50)	1·435 (7)

C(2)—O(1)—C(18)	112·3 (4)	O(1)—C(2)—C(3)	108·6 (5)
C(2)—C(3)—O(4)	114·5 (5)	C(3)—O(4)—C(5)	113·3 (4)
O(4)—C(5)—C(6)	108·1 (5)	C(5)—C(6)—O(7)	110·0 (4)
C(6)—O(7)—C(8)	114·4 (4)	O(7)—C(8)—C(9)	106·5 (4)
O(7)—C(8)—C(19)	111·9 (4)	C(9)—C(8)—C(19)	115·5 (4)
C(8)—C(9)—O(10)	104·9 (4)	C(8)—C(9)—C(25)	115·1 (4)
O(10)—C(9)—C(25)	113·3 (4)	C(9)—O(10)—C(11)	115·4 (4)
O(10)—C(11)—C(12)	108·5 (4)	C(11)—C(12)—O(13)	114·1 (4)
C(12)—O(13)—C(14)	114·9 (4)	O(13)—C(14)—C(15)	109·0 (4)
C(14)—C(15)—O(16)	107·3 (4)	C(15)—O(16)—C(17)	113·2 (4)
O(16)—C(17)—C(18)	105·9 (4)	O(16)—C(17)—C(31)	111·7 (4)
C(18)—C(17)—C(31)	112·3 (4)	O(1)—C(18)—C(17)	105·7 (4)
O(1)—C(18)—C(37)	111·0 (4)	C(17)—C(18)—C(37)	113·9 (4)
C(8)—C(19)—C(20)	120·8 (4)	C(8)—C(19)—C(24)	121·5 (5)
C(20)—C(19)—C(24)	117·7 (5)	C(19)—C(20)—C(21)	119·7 (5)
C(20)—C(21)—C(22)	122·4 (6)	C(21)—C(22)—C(23)	119·0 (5)
C(21)—C(22)—O(43)	116·4 (5)	C(23)—C(22)—O(43)	124·6 (5)
C(22)—C(23)—C(24)	119·2 (5)	C(19)—C(24)—C(23)	122·1 (5)
C(9)—C(25)—C(26)	120·4 (5)	C(9)—C(25)—C(30)	121·3 (4)
C(26)—C(25)—C(30)	118·3 (5)	C(25)—C(26)—C(27)	120·7 (5)
C(26)—C(27)—C(28)	120·1 (5)	C(27)—C(28)—C(29)	120·5 (5)
C(27)—C(28)—O(45)	116·0 (4)	C(29)—C(28)—O(45)	123·5 (5)
C(28)—C(29)—C(30)	118·4 (5)	C(25)—C(30)—C(29)	121·9 (5)
C(17)—C(31)—C(32)	120·1 (5)	C(17)—C(31)—C(36)	121·5 (4)
C(32)—C(31)—C(36)	118·4 (5)	C(31)—C(32)—C(33)	120·2 (5)
C(32)—C(33)—C(34)	120·4 (5)	C(33)—C(34)—C(35)	120·1 (5)
C(33)—C(34)—O(47)	115·9 (5)	C(35)—C(34)—O(47)	124·0 (5)
C(34)—C(35)—C(36)	118·9 (5)	C(31)—C(36)—C(35)	122·1 (5)
C(18)—C(37)—C(38)	121·3 (4)	C(18)—C(37)—C(42)	121·4 (5)
C(38)—C(37)—C(42)	117·3 (5)	C(37)—C(38)—C(39)	121·7 (5)
C(38)—C(39)—C(40)	118·8 (5)	C(39)—C(40)—C(41)	121·5 (5)
C(39)—C(40)—O(49)	114·0 (5)	C(41)—C(40)—O(49)	124·5 (4)
C(40)—C(41)—C(42)	119·3 (5)	C(37)—C(42)—C(41)	121·3 (5)
C(22)—O(43)—C(44)	117·5 (5)	C(28)—O(45)—C(46)	118·3 (4)
C(34)—O(47)—C(48)	118·9 (5)	C(40)—O(49)—C(50)	117·3 (5)

C(18)—O(1)—C(2)—C(3)	−168·1 (4)	C(14)—C(15)—O(16)—C(17)	−178·8 (4)
O(1)—C(2)—C(3)—O(4)	74·2 (6)	C(15)—O(16)—C(17)—C(18)	158·6 (4)
C(2)—C(3)—O(4)—C(5)	−85·1 (6)	O(16)—C(17)—C(18)—O(1)	−67·1 (5)
C(3)—O(4)—C(5)—C(6)	−171·3 (4)	C(2)—O(1)—C(18)—C(17)	165·9 (4)
O(4)—C(5)—C(6)—O(7)	−74·6 (5)	O(7)—C(8)—C(9)—C(25)	167·6 (4)
C(5)—C(6)—O(7)—C(8)	−173·9 (4)	C(19)—C(8)—C(9)—O(10)	167·9 (4)
C(6)—O(7)—C(8)—C(9)	159·1 (4)	C(19)—C(8)—C(9)—C(25)	42·7 (6)
O(7)—C(8)—C(9)—O(10)	−67·2 (5)	O(16)—C(17)—C(18)—C(37)	170·8 (4)
C(8)—C(9)—O(10)—C(11)	168·2 (4)	C(31)—C(17)—C(18)—O(1)	170·7 (4)
C(9)—O(10)—C(11)—C(12)	−175·6 (4)	C(31)—C(17)—C(18)—C(37)	48·6 (6)
O(10)—C(11)—C(12)—O(13)	73·1 (6)	O(7)—C(8)—C(19)—C(24)	−21·9 (6)
C(11)—C(12)—O(13)—C(14)	−84·3 (5)	C(8)—C(9)—C(25)—C(26)	77·9 (6)
C(12)—O(13)—C(14)—C(15)	−173·4 (4)	O(16)—C(17)—C(31)—C(32)	−66·4 (6)
O(13)—C(14)—C(15)—O(16)	−76·4 (5)	O(1)—C(18)—C(37)—C(38)	−63·8 (4)

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A 1:1 Adduct Between the *trans–transoid–trans* Isomer of 2,3,11,12-Tetra-anisyl-18-crown-6 with the (*RRRR*)-Configuration and Ammonia–Borane

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Abstract. (2*R*,3*R*,11*R*,12*R*)-2,3,11,12-Tetrakis-4-methoxyphenyl-1,4,7,10,13,16-hexaoxacyclooctadecane–ammonia–borane–methanol (4/4/4/3), $C_{40}H_{48}O_{10}.BH_3NH_3.0{\cdot}75CH_3OH$, $M_r = 744$, triclinic, $P1$, $a = 7{\cdot}995$ (2), $b = 11{\cdot}035$ (2), $c = 13{\cdot}371$ (4) Å, $\alpha = 68{\cdot}34$ (2), $\beta = 74{\cdot}62$ (2), $\gamma = 82{\cdot}84$ (2)°, $V = 1057$ Å^3, $Z = 1$, $D_x = 1{\cdot}17$ Mg m^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1{\cdot}54178$ Å, $\mu = 0{\cdot}65$ mm^{-1}, $F(000) = 399{\cdot}5$, room temperature, $R = 0{\cdot}044$ for 2765 unique observed reflections with $|F_o| > 3\sigma(|F_o|)$. One molecule of BH_3NH_3 is hydrogen bonded through its NH_3 group to the nearer triangle of O atoms on one of the two homotopic faces of the title chiral receptor (4). In adopting an all-*gauche* conformation, the macrocyclic ring carries a pair of vicinal diequatorial anisyl groups and a pair of vicinal diaxial anisyl groups, one *syn* and the other *anti* to the bound BH_3NH_3 substrate molecule: the latter anisyl substituent totally obscures the 'free' face of the macrocycle, thus preventing adduct formation with a second BH_3NH_3 molecule.

Introduction. The fact that enantioselective reductions of aromatic ketones can be performed (Allwood, Shahriari-Zavareh, Stoddart & Williams, 1984) using the ammonia–borane (BH_3NH_3) adducts of the chiral *trans–transoid–trans*-tetraphenyl-18-crown-6 derivatives (9)* gives added significance to a determination of the solid-state structure of these adducts. In this paper, we (*a*) describe the preparation of a 1:1 adduct between the *trans–transoid–trans* isomer (4) of (2*R*,3*R*,11*R*,12*R*)-tetra-anisyl-18-crown-6, (*b*) report on the determination of its crystal structure, and (*c*) compare the structural characteristics of $[(4).BH_3NH_3]$ with those already reported (Allwood *et al.*, 1984) for $[(9).BH_3NH_3]$.

Experimental. Addition of an excess of ammonia–borane to a solution of (+)-(4) in methanol gave a crystalline adduct on removal of the solvent under vacuum. Recrystallization from methanol with *n*-hexane as a precipitant afforded single crystals of

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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*c*; paper VII) and to Fig. 1 of Pears, Shahriari-Zavareh, Stoddart, Crosby, Allwood, Slawin & Williams (1988*a*; paper I).

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