norbornane system to become almost identical in length, C(1)–C(12) = 1·569 (2) Å and C(5)–C(9) = 1·578 (2) Å (see numbering in Fig. 3). The four external valence angles of the norbornane moiety in (3), *e.g.* C(2)–C(1)–C(12), open to an average value of 116·7 (5)°. The equivalence of the C(4)–C(5) bonds in (1), (2) and the reference compounds and with the bond in (3) implies the lengthening is not related to strain transmitted by the four-membered ring.

Molecular mechanics calculations predict that compound (3) is the least strained of the compounds discussed, and the formation of the four-membered ring imparts an additional 26·2 and 35·8 kJ mol⁻¹ of strain energy to (1) and (2). The C(7)-substituted compounds have only 16·7 kJ mol⁻¹ more strain energy than (3).

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The Structure of Tris(dimethyldioxolo)triphenylene

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Abstract. Triphenyleno[2,3-*d*:6,7-*d'*:10,11-*d''*]tri(2,2-dimethyl-1,3-dioxole), $C_{27}H_{24}O_6$, $M_r = 444·48$, monoclinic, $C2/c$, $a = 22·662$ (8), $b = 9·005$ (1), $c = 24·220$ (5) Å, $\beta = 113·95$ (2)°, $V = 4517$ (2) Å³, $Z = 8$, $D_x = 1·307$ g cm⁻³, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0·68$ cm⁻¹, $F(000) = 1872$, $T = 298$ K, final $R = 0·0765$ for 1037 observed unique reflections. Apart from the methyl groups the molecules are planar; the threefold axis perpendicular to the plane of the molecule does not coincide with any of the symmetry operations of the lattice. All intermolecular distances are greater than the sum of van der Waals radii.

Introduction. We report the structure of tris(dimethyldioxolo)triphenylene (TacTPh). The purpose of the investigation has been to gain insight into the packing patterns of peripherally substituted triphenylenes in order to test computer models of the relationship between substitution pattern and molecular organiza-tion in organic radical ion crystals (Jørgensen, Bjørnholm & Bechgaard, 1988). Special attention is given to triphenylenes because of their C_3 symmetry which allows degeneracy of the highest occupied molecular orbital. When packed in regular segregated stacks, such molecules constitute potential candidates as donors in radical ion crystals with intriguing magnetic interactions (Miller, Epstein & Reif, 1988).

Experimental. Ketalization of 2,3,6,7,10,11-hexa-hydroxytriphenylene (Piattelli, Fattorusso, Nicolaus & Magno, 1965) was performed by adding P_2O_5 to a solution of hexahydroxytriphenylene in a 1:1 (v/v) mixture of acetone and acetonitrile followed by reflux for 2 h. Single crystals were obtained by recrystal-lization from butanol.

A crystal of dimensions 0·4 × 0·1 × 0·05 mm was used for data collection on a CAD-4 diffractometer with graphite-monochromatized Mo $K\alpha$ radiation.

Table 1. *Fractional coordinates* (× 10⁴) *and isotropic or equivalent isotropic temperature factors* (Å² × 10³)

$$U_{eq} = \tfrac{1}{3}\sum_i\sum_j U_{ij}a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j.$$

	x	y	z	U/U_eq
C(1)	514 (5)	5353 (11)	1056 (5)	42 (3)
C(2)	95 (6)	5768 (13)	1351 (5)	52 (3)
C(3)	−231 (6)	7117 (13)	1160 (5)	52 (3)
C(4)	−187 (6)	7970 (13)	720 (5)	51 (3)
C(5)	204 (6)	7611 (13)	422 (5)	56 (4)
C(6)	554 (5)	6232 (11)	603 (5)	39 (3)
C(7)	946 (5)	5776 (13)	285 (5)	50 (3)
C(8)	968 (5)	6641 (12)	−207 (5)	50 (3)
C(9)	1361 (5)	6102 (12)	−473 (5)	46 (3)
C(10)	1733 (5)	4844 (12)	−286 (5)	43 (3)
C(11)	1726 (5)	3979 (12)	178 (5)	47 (3)
C(12)	1304 (5)	4414 (13)	468 (5)	47 (3)
C(13)	1256 (5)	3506 (12)	940 (4)	39 (3)
C(14)	1603 (6)	2157 (14)	1096 (5)	58 (4)
C(15)	1571 (6)	1346 (14)	1567 (5)	58 (4)
C(16)	1199 (6)	1835 (13)	1870 (5)	53 (3)
C(17)	846 (6)	3117 (12)	1715 (5)	51 (3)
C(18)	869 (5)	3986 (12)	1227 (5)	41 (3)
C(19)	−795 (7)	9219 (14)	1129 (7)	71 (8)*
C(20)	−439 (6)	10352 (14)	1604 (6)	71 (6)*
C(21)	−1534 (6)	9395 (14)	811 (6)	77 (7)*
C(22)	1944 (7)	5862 (14)	−1045 (6)	63 (7)*
C(23)	2554 (7)	6765 (16)	−879 (6)	83 (8)*
C(24)	1625 (7)	5244 (16)	−1709 (6)	85 (8)*
C(25)	1743 (7)	−273 (15)	2338 (7)	72 (9)*
C(26)	1428 (8)	−1779 (16)	2262 (7)	97 (10)*
C(27)	2350 (7)	−50 (20)	2896 (7)	103 (9)*
O(1)	−657 (4)	7741 (10)	1372 (4)	74 (5)*
O(2)	−557 (4)	9233 (10)	643 (4)	80 (5)*
O(3)	1451 (5)	6730 (10)	−941 (4)	79 (5)*
O(4)	2085 (4)	4604 (9)	−622 (4)	70 (5)*
O(5)	1864 (5)	29 (10)	1810 (4)	85 (5)*
O(6)	1261 (4)	855 (9)	2331 (4)	77 (5)*

* U_{eq}.

Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

C(1)—C(2)	1·45 (2)	C(16)—C(17)	1·37 (2)
C(1)—C(6)	1·39 (2)	C(17)—C(18)	1·44 (2)
C(1)—C(18)	1·44 (2)	C(19)—C(20)	1·50 (2)
C(2)—C(3)	1·40 (2)	C(19)—C(21)	1·54 (2)
C(3)—C(4)	1·35 (2)	C(22)—C(23)	1·51 (2)
C(4)—C(5)	1·39 (2)	C(22)—C(24)	1·57 (2)
C(5)—C(6)	1·44 (2)	C(25)—C(26)	1·50 (2)
C(6)—C(7)	1·45 (2)	C(25)—C(27)	1·50 (2)
C(7)—C(8)	1·44 (2)	O(1)—C(3)	1·38 (2)
C(7)—C(12)	1·44 (2)	O(1)—C(19)	1·44 (2)
C(8)—C(9)	1·38 (2)	O(2)—C(4)	1·38 (2)
C(9)—C(10)	1·37 (2)	O(2)—C(19)	1·48 (2)
C(10)—C(11)	1·37 (2)	O(3)—C(9)	1·36 (2)
C(11)—C(12)	1·45 (2)	O(3)—C(22)	1·47 (2)
C(12)—C(13)	1·45 (2)	O(4)—C(10)	1·37 (2)
C(13)—C(14)	1·41 (2)	O(4)—C(22)	1·48 (2)
C(13)—C(18)	1·39 (2)	O(5)—C(15)	1·36 (2)
C(14)—C(15)	1·40 (2)	O(5)—C(25)	1·45 (2)
C(15)—C(16)	1·40 (2)	O(6)—C(16)	1·39 (2)
		O(6)—C(25)	1·48 (2)

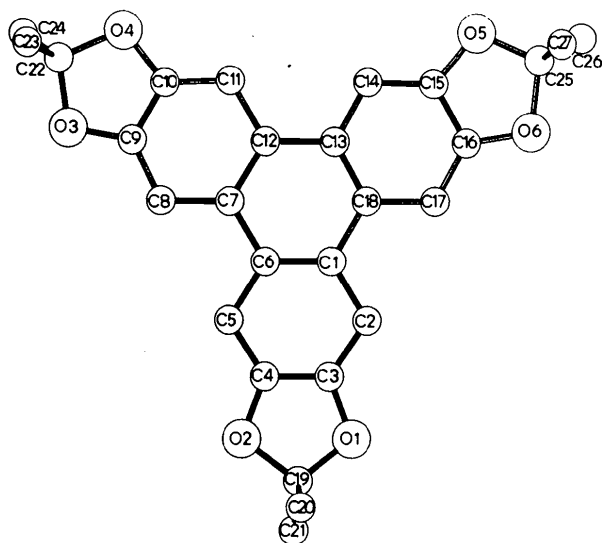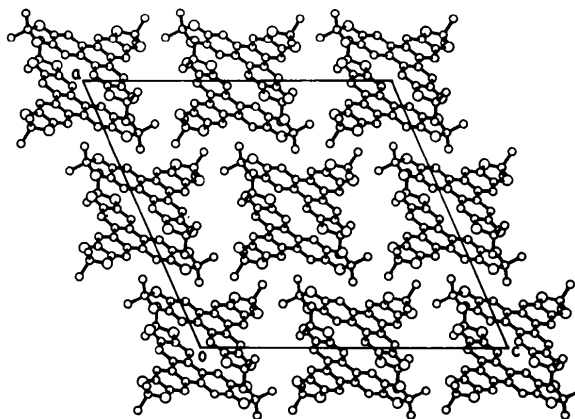|			
C(2)—C(1)—C(6)	121 (1)	C(14)—C(13)—C(18)	122 (1)
C(2)—C(1)—C(18)	119 (1)	C(13)—C(14)—C(15)	119 (1)
C(6)—C(1)—C(18)	120 (1)	C(14)—C(15)—C(16)	118 (1)
C(1)—C(2)—C(3)	115 (1)	C(14)—C(15)—O(5)	132 (2)
C(2)—C(3)—C(4)	124 (1)	C(16)—C(15)—O(5)	110 (1)
C(2)—C(3)—O(1)	126 (1)	C(15)—C(16)—C(17)	124 (1)
C(4)—C(3)—O(1)	110 (1)	C(15)—C(16)—O(6)	109 (1)
C(3)—C(4)—C(5)	123 (1)	C(17)—C(16)—O(6)	127 (1)
C(3)—C(4)—O(2)	110 (1)	C(16)—C(17)—C(18)	118 (1)
C(5)—C(4)—O(2)	127 (1)	C(1)—C(18)—C(13)	121 (1)
C(4)—C(5)—C(6)	116 (1)	C(1)—C(18)—C(17)	120 (1)
C(1)—C(6)—C(5)	121 (1)	C(13)—C(18)—C(17)	119 (1)
C(1)—C(6)—C(7)	121 (1)	C(20)—C(19)—C(21)	117 (1)
C(5)—C(6)—C(7)	118 (1)	C(20)—C(19)—O(1)	111 (1)
C(6)—C(7)—C(8)	122 (1)	C(20)—C(19)—O(2)	110 (1)
C(6)—C(7)—C(12)	118 (1)	C(21)—C(19)—O(1)	108 (1)
C(8)—C(7)—C(12)	121 (1)	C(21)—C(19)—O(2)	106 (1)
C(7)—C(8)—C(9)	116 (1)	O(1)—C(19)—O(2)	104 (1)
C(8)—C(9)—C(10)	124 (1)	C(23)—C(22)—C(24)	117 (2)
C(8)—C(9)—O(3)	126 (1)	C(23)—C(22)—O(3)	110 (1)
C(10)—C(9)—O(3)	110 (1)	C(23)—C(22)—O(4)	108 (1)
C(9)—C(10)—C(11)	122 (1)	C(24)—C(22)—O(3)	107 (1)
C(9)—C(10)—O(4)	111 (1)	C(24)—C(22)—O(4)	109 (1)
C(11)—C(10)—O(4)	127 (1)	O(3)—C(22)—O(4)	104 (1)
C(10)—C(11)—C(12)	118 (1)	C(26)—C(25)—C(27)	118 (1)
C(7)—C(12)—C(11)	119 (1)	C(26)—C(25)—O(5)	107 (1)
C(7)—C(12)—C(13)	121 (1)	C(26)—C(25)—O(6)	110 (1)
C(11)—C(12)—C(13)	121 (1)	C(27)—C(25)—O(5)	107 (1)
C(12)—C(13)—C(14)	119 (1)	C(27)—C(25)—O(6)	109 (1)
C(12)—C(13)—C(18)	119 (1)	O(5)—C(25)—O(6)	105 (1)

Fig. 1. View of the TacTPh molecule with atomic numbering perpendicular to the best plane of the center ring.

Fig. 2. The packing viewed along the *b* axis.

ω–2θ scan, 20 reflections with $10 < \theta < 13\cdot5°$ used for measuring lattice parameters, absorption correction $\mu = 0\cdot68$ cm⁻¹ gave transmission factors 0·98–0·99, maximum $(\sin\theta)/\lambda = 0\cdot5269$ Å⁻¹, $|h| \le 24$, $0 \le k \le 9$, $|l| \le 25$. Three standard reflections showed no intensity reduction during the data collection. 6579 reflections measured gave 2772 unique reflections, R_{int}

$= 0\cdot023$, 1052 reflections with $\sigma(I)/I < 0\cdot33$ were used for refinements. Direct methods and least-squares minimization of $\sum w(\Delta F)^2$ with C(1) to C(18) isotropic
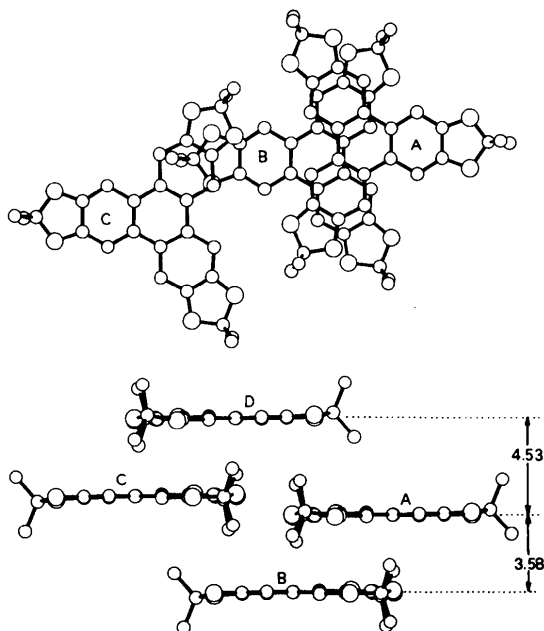
Fig. 3. The overlap. Upper: viewed perpendicular to the best plane of the center ring, lower: perpendicular to the upper view. Distances between the molecules in Å, e.s.d.'s 0·05 Å. Symmetry operations: A (x, y, z), B ($-x, 2-y, -z$), C ($-x, 1-y, -z$), D ($x, 1+y, z$).

and C(19) to C(27) and all O anisotropic, no attempt to locate the H atoms, 208 parameters refined, $R = 0\cdot076$, $wR = 0\cdot097$, $w = [\sigma^2(F_o) + 0\cdot0008F_o^2]^{-1}$, $(\Delta/\sigma)_{max} = 0\cdot014$ in the final cycle, $(\Delta\rho)_{max}/(\Delta\rho)_{min} = 0\cdot33/-0\cdot24$ e Å^{-3}. Computer programs used are $SHELX76$ (Sheldrick, 1976) with atomic scattering factors, and $PLUTO$ (Motherwell, 1978).

Discussion. Final positional and thermal parameters are given in Table 1, bond lengths and angles in Table 2.* The molecule with atomic labels is shown in Fig. 1. The distances from the best plane through C(1)–C(18) to C(19), C(22) and C(25) are 0·20, 0·03 and 0·18 Å, respectively. The three tetrahedra formed by two CH$_3$ groups and two O atoms around C(29), C(22) and C(25) are only slightly distorted. The angles between the normals to the best plane through C(1)–C(18) and the planes through the three benzene rings are 1·70, 1·85 and 1·46°, respectively. The angles between the normal to the molecular plane and **a**, **b** and **c** are 56·8, 59·8 and 67·1°, respectively. The packing is shown in Fig. 2, and the overlap in Fig. 3.

The molecules are not packed in regular segregated stacks. The overlap between two neighbouring molecules A and B in Fig. 3 does not indicate strong interaction.

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Structure of (4S,5R)-6-Bromo-6-deoxyascorbic Acid

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Abstract. C$_6$H$_7$BrO$_5$, $M_r = 239\cdot02$, orthorhombic, $P2_12_12_1$, $a = 12\cdot384$ (4), $b = 8\cdot247$ (5), $c = 8\cdot188$ (4) Å, $V = 836\cdot25$ Å^3, $Z = 4$, $D_x = 1\cdot899$ Mg m^{-3}, λ(Mo $K\alpha) = 0\cdot71073$ Å, $\mu = 4\cdot77$ mm^{-1}, $F(000) = 472$, $R = 0\cdot040$, $wR = 0\cdot041$ for 828 unique reflections having $I \geq 3\sigma(I)$. The γ-lactone ring shows a conformation between twisted $^1T_2$ and envelope ^1E. Intermolecular hydrogen bonds O(3)–

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