contacts. H(8)-*endo* lies only 2·41 (5) Å above C(2) and probably experiences some interaction with the C(2) π system. The C(2)–C(3) π system and the π system of the anhydride may spatially overlap since the C(2)···C(13) and C(3)···O(14) separations are only 2·824 (4) and 2·757 (4) Å. If a homoconjugative interaction exists it does not affect the C(2)–C(3) separation of 1·339 (3) Å which is the same as found in other norbornene and sesquinorbornene systems (Watson, Galloy, Bartlett & Roof, 1981).

A least-squares plane fitted to C(9)C(1)C(5)C(6) *B* shows deviations of 0·11 (1) Å from planarity while planes fitted to C(6)C(7)C(8)C(9) *A*, C(11)C(12)-C(13)O(13)C(14)O(14)O(15) *D*, C(1)C(2)C(3)C(4) *E* and C(1)C(4)C(11)C(12) *F* show maximum deviations of 0·051 (6), 0·043 (6), 0·005 (6), and 0·013 (8) Å. The interplanar angles *A–B*, *B–C*, *A–C*, *D–E* and *E–F* are 115·8 (6), 121·9 (4), 121·9 (3), 119·5 (4) and 110·0 (4)°.

We thank The Robert A. Welch Foundation (P-074) for financial support. This is FASTBIOS Contribution No. 126.

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Tri-[*b,e,h*]-(4,5-dimethoxybenzo)cyclonona-2,5,8-trien-1-one,* $C_{27}H_{28}O_7$

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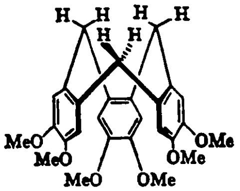
(*Received* 9 *January* 1984; *accepted* 11 *April* 1984)

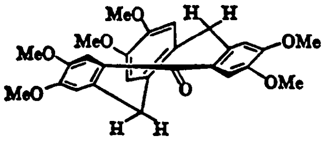
Abstract. $M_r = 464{\cdot}52$, orthorhombic, $P2_12_12_1$, $a = 16{\cdot}735$ (2), $b = 18{\cdot}763$ (3), $c = 7{\cdot}805$ (1) Å, $V = 2450{\cdot}5$ (6) Å^3, $Z = 4$, D_m(flotation, KI + water) = 1·253, $D_x = 1{\cdot}259$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71073$ Å, $\mu = 0{\cdot}7$ cm^{-1}, $F(000) = 984$, $T = 295$ K, final $R = 0{\cdot}054$ for 1244 reflections. The molecule contains a nine-membered ring with a twist conformation, fused to three planar six-membered rings; the whole molecule has roughly C_2 symmetry, with maximum deviation in torsion angles of 16·8° from an exactly symmetrical geometry, and dihedral-angle differences of up to 18·3°. Bond lengths and angles are normal.

Introduction. Cyclotriveratrylene (I) possesses an interesting ring system consisting of a cyclononatriene ring to which three benzo rings are fused; the molecule is believed to have a 'crown' conformation, with the benzo rings forming the three sides of a trigonal pyramid (Lindsey, 1963, 1965; Erdtman, Haglid & Ryhage, 1964; Goldup, Morrison & Smith, 1965; Miller & Gesner, 1965).

For the ketone (II) derived from cyclotriveratrylene, spectroscopic data (UV, IR and NMR) suggest a twist conformation similar to that of cyclohexanone, which is flexible in solution (Anand, Cookson, Halton & Stevens, 1966). Crystals of (II) were made available for X-ray study about twenty years ago, but the problem seemed too difficult to be solved by the techniques of that era; the structure has now been determined by direct methods.



(I)



(II)

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* Alternative IUPAC name: 10,15-dihydro-2,3,7,8,12,13-hexamethoxy-5*H*-tribenzo[*a,d,g*]cyclononen-5-one.

Table 1. *Final positional (fractional* $\times 10^4$*) and isotropic thermal* ($Å^2 \times 10^3$) *parameters with estimated standard deviations in parentheses*

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

	x	y	z	U_{eq}
C(1)	4998 (4)	−1750 (3)	6505 (9)	40
C(2)	4707 (4)	−1838 (3)	8133 (10)	42
C(3)	4106 (4)	−1374 (3)	8765 (9)	41
C(4)	3831 (4)	−837 (3)	7722 (9)	42
C(4a)	4143 (3)	−736 (3)	6061 (9)	40
C(5)	3812 (4)	−119 (3)	5078 (8)	46
C(5a)	4289 (4)	382 (3)	4001 (9)	43
C(6)	3861 (4)	760 (3)	2767 (9)	43
C(7)	4213 (4)	1263 (3)	1779 (10)	49
C(8)	5036 (4)	1416 (3)	1967 (9)	45
C(9)	5462 (4)	1047 (3)	3208 (9)	40
C(9a)	5106 (4)	521 (3)	4229 (8)	39
C(10)	5661 (3)	187 (3)	5529 (8)	39
C(10a)	6240 (4)	−346 (3)	4710 (8)	37
C(11)	7061 (4)	−188 (3)	4804 (8)	41
C(12)	7618 (4)	−633 (3)	4027 (9)	45
C(13)	7354 (4)	−1229 (4)	3113 (10)	48
C(14)	6553 (5)	−1395 (3)	3080 (8)	45
C(14a)	5989 (3)	−948 (3)	3835 (8)	37
C(15)	5109 (4)	−1144 (3)	3706 (8)	42
C(15a)	4726 (3)	−1198 (3)	5451 (9)	39
C(16)	5541 (4)	−2844 (3)	8662 (10)	77
C(17)	3232 (4)	−1078 (3)	11069 (9)	63
C(18)	3062 (5)	1494 (4)	110 (12)	103
C(19)	6160 (4)	2143 (3)	1213 (10)	60
C(20)	8712 (4)	112 (4)	4822 (10)	73
C(21)	7713 (5)	−2136 (4)	1130 (12)	84
O(1)	4948 (3)	−2352 (2)	9276 (6)	58
O(2)	3859 (2)	−1513 (2)	10396 (6)	53
O(3)	3846 (3)	1666 (2)	526 (6)	67
O(4)	5341 (3)	1940 (2)	960 (6)	57
O(5)	8426 (3)	−530 (2)	4059 (6)	62
O(6)	7949 (3)	−1621 (3)	2328 (7)	73
O(7)	3086 (3)	−15 (2)	5184 (6)	74

Experimental. Colourless crystals, 0·15 × 0·09 × 0·1 mm. CAD-4 diffractometer, graphite-monochromatized Mo $K\alpha$ radiation. $\theta \leq 24°$, ω–2θ scan, ω-scan width $(0{\cdot}70 + 0{\cdot}35\tan\theta)°$ extended by 25% on either side of the peak for background measurement, horizontal aperture $(2 + \tan\theta)$mm, vertical aperture 4 mm. Cell parameters by least-squares fit to observed $\sin^2\theta$ values for 25 centred reflections with $12 < \theta < 15°$. Absent reflections $h00$, h odd, $0k0$, k odd, $00l$, l odd ($P2_12_12_1$). Intensity and orientation checks for three standard reflections showed little or no decay throughout data collection. 2206 independent reflections; 1244 with $I \geq 1{\cdot}5\sigma(I)$, where $\sigma^2(I) = S + 2B + [0{\cdot}04(S - B)]^2$, S = total peak count and B = time-averaged background. Lp corrections (no absorption corrections). Structure solved by *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), refined by full-matrix least squares. H positions estimated geometrically with C—H = 1·08 Å and the temperature factor fixed at the isotropic B of the carrier atom. $\sum w(|F_o| - |F_c|)^2$ minimized, $w = 1/\sigma^2(F)$. Electron density in final difference map $\leq 0{\cdot}23$ e Å^{-3}. Final $R = 0{\cdot}054$, $R_w = 0{\cdot}047$ for 1244 reflections, 307 variables, $R = 0{\cdot}119$ for all data. Mean and max. Δ/σ 0·013 and 0·061, respectively. Standard deviation in an observation of unit weight 1·461. Scattering factors from Cromer & Mann (1968) and Stewart, Davidson & Simpson (1965). Local adaptations of standard computer programs *ORFLS* (Busing, Martin & Levy, 1962), *ORFFE* (Busing, Martin & Levy, 1964), *ORTEP*II (Johnson, 1976) and *PLUTO* (Motherwell & Clegg, 1978).

Discussion. Final atomic parameters are in Table 1.* The molecular conformation (Fig. 1) is exactly as previously deduced by Anand *et al.* (1966). The nine-membered ring has a twisted conformation, with three approximately planar four-atom segments (Figs. 1 and 2) linked so that the ring and the whole molecule have roughly C_2 symmetry, with the pseudo-twofold axis along the C=O bond of the planar carbonyl group. Deviations from exact twofold symmetry are seen in the torsion and dihedral angles. In the nine-membered ring the maximum difference in torsion angle is 16·8° (Fig. 2); the two six-membered rings adjacent to the carbonyl group make angles of 41·5 (2) and 23·2 (3)° with the plane of that group, and 88·1 (2) and 79·6 (2)° with the plane of the third six-membered ring. The nine-membered ring conformation is quite similar to that of the related hetero-atom ring in a trithiatribenzo[*a,d,g*]cyclononene (von Deuten, Kopf & Klar, 1979), where the conformation is described as a 'saddle'. The six-membered rings are essentially planar [maximum deviation 0·025 (8) Å]; the methoxy O atoms are slightly displaced from the planes [maximum 0·074 (5) Å] with the methyl groups displaced by amounts as large as 0·366 (9) Å; the methyl groups are all directed away from the adjacent methoxy substituent (Fig. 1).

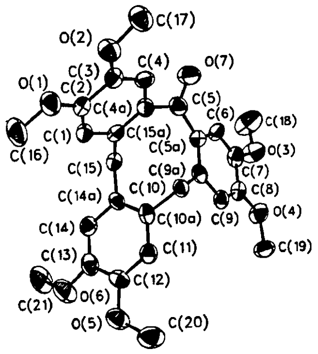

Fig. 1. View of the molecule and the numbering of atoms. The thermal ellipsoids are drawn at the 50% probability level.

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* Lists of structure factors, anisotropic thermal parameters, coordinates of H atoms, torsion angles, least-squares planes and a packing diagram have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39396 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond lengths* (Å) *and angles* (°) *with estimated standard deviations in parentheses* (*see also Fig.* 2)

C(1)–C(2)	1·371 (9)	C(9a)–C(10)	1·511 (8)
C(1)–C(15a)	1·399 (8)	C(10)–C(10a)	1·533 (8)
C(2)–C(3)	1·419 (8)	C(10a)–C(11)	1·407 (7)
C(2)–O(1)	1·375 (7)	C(10a)–C(14a)	1·385 (8)
C(3)–C(4)	1·374 (8)	C(11)–C(12)	1·389 (8)
C(3)–O(2)	1·364 (7)	C(12)–C(13)	1·399 (8)
C(4)–C(4a)	1·410 (9)	C(12)–O(5)	1·367 (6)
C(4a)–C(5)	1·494 (8)	C(13)–C(14)	1·377 (8)
C(4a)–C(15a)	1·390 (8)	C(13)–O(6)	1·381 (8)
C(5)–C(5a)	1·493 (8)	C(14)–C(14a)	1·393 (8)
C(5)–O(7)	1·233 (6)	C(14a)–C(15)	1·522 (8)
C(5a)–C(6)	1·394 (8)	C(15)–C(15a)	1·508 (8)
C(5a)–C(9a)	1·404 (7)	C(16)–O(1)	1·438 (7)
C(6)–C(7)	1·354 (8)	C(17)–O(2)	1·429 (7)
C(7)–C(8)	1·414 (8)	C(18)–O(3)	1·390 (8)
C(7)–O(3)	1·381 (7)	C(19)–O(4)	1·435 (7)
C(8)–C(9)	1·387 (9)	C(20)–O(5)	1·426 (7)
C(8)–O(4)	1·359 (7)	C(21)–O(6)	1·402 (8)
C(9)–C(9a)	1·401 (8)		

C(2)–C(1)–C(15a)	121·2 (6)	C(5a)–C(9a)–C(9)	118·2 (6)
C(1)–C(2)–C(3)	120·0 (6)	C(9)–C(9a)–C(10)	114·5 (5)
C(1)–C(2)–O(1)	125·6 (6)	C(10)–C(10a)–C(11)	117·3 (5)
C(3)–C(2)–O(1)	114·4 (6)	C(11)–C(10a)–C(14a)	119·6 (5)
C(2)–C(3)–C(4)	118·8 (6)	C(10a)–C(11)–C(12)	120·3 (6)
C(2)–C(3)–O(2)	115·0 (6)	C(11)–C(12)–C(13)	119·5 (6)
C(4)–C(3)–O(2)	126·3 (6)	C(11)–C(12)–O(5)	124·8 (6)
C(3)–C(4)–C(4a)	121·3 (6)	C(13)–C(12)–O(5)	115·7 (6)
C(4)–C(4a)–C(5)	116·1 (6)	C(12)–C(13)–C(14)	119·8 (6)
C(4)–C(4a)–C(15a)	119·4 (6)	C(12)–C(13)–O(6)	115·1 (6)
C(4a)–C(5)–O(7)	117·0 (6)	C(14)–C(13)–O(6)	125·0 (6)
C(5a)–C(5)–O(7)	117·6 (6)	C(13)–C(14)–C(14a)	121·0 (6)
C(5)–C(5a)–C(6)	115·8 (5)	C(10a)–C(14a)–C(14)	119·6 (5)
C(6)–C(5a)–C(9a)	119·6 (6)	C(14)–C(14a)–C(15)	118·8 (6)
C(5a)–C(6)–C(7)	121·6 (6)	C(1)–C(15a)–C(4a)	119·3 (6)
C(6)–C(7)–C(8)	120·4 (7)	C(1)–C(15a)–C(15)	116·3 (6)
C(6)–C(7)–O(3)	126·3 (6)	C(2)–O(1)–C(16)	115·9 (5)
C(8)–C(7)–O(3)	113·3 (6)	C(3)–O(2)–C(17)	117·2 (5)
C(7)–C(8)–C(9)	118·1 (7)	C(7)–O(3)–C(18)	117·3 (6)
C(7)–C(8)–O(4)	117·0 (6)	C(8)–O(4)–C(19)	118·1 (6)
C(9)–C(8)–O(4)	124·8 (7)	C(12)–O(5)–C(20)	117·3 (5)
C(8)–C(9)–C(9a)	122·0 (6)	C(13)–O(6)–C(21)	117·3 (6)

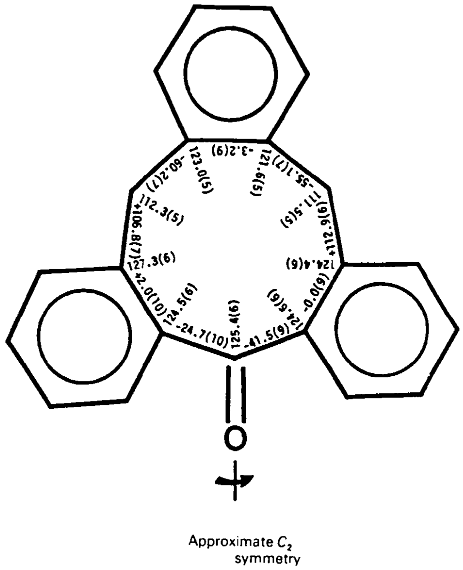

Fig. 2. Bond angles (°) and torsion angles (°) in the nine-membered ring.

Bond lengths and angles in the molecule (Table 2) are generally close to expected values. In the nine-membered ring, the C—CH_2 bonds are in the range 1·508–1·533 (8) Å, mean 1·519 (6) Å,* C—CO are 1·494 (8) and 1·493 (8) Å, and other C—C are 1·390 (8), 1·404 (7), 1·385 (8) Å, mean 1·393 (6) Å. Bond angles in the nine-membered ring are 112·3 (5) and 111·5 (5)°, mean 111·9 (4)° at CH_2, 125·4 (6)° at the carbonyl C atom, and 121·6–127·3 (6)° at the other C(sp^2) atoms. In the six-membered rings, C—C = 1·354–1·419 (8) Å, mean 1·393 (4) Å, and C—C—C = 118·1–122·0 (6)°, mean 120·0 (3)°; C—OMe = 1·359–1·381 (7) Å, mean 1·371 (4) Å, O—Me = 1·390–1·438 (7) Å, mean 1·420 (8) Å, C—O—Me = 115·9–118·1 (6)°, mean 117·2 (3)°.

Intermolecular distances correspond to normal van der Waals interactions; the shortest contact is C⋯O = 3·172 (7) Å.

We thank the Natural Sciences and Engineering Research Council of Canada for financial support, the University of British Columbia Computing Centre for assistance, and Dr I. D. R. Stevens for suggesting the problem and for crystal samples.

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* The errors given for the mean values are calculated from the r.m.s. deviations from the mean.

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