The Structure of the Octahydrophotodimer of Biphenylene

By E. L. Passer and J. G. White

Department of Chemistry, Fordham University, Bronx, New York 10458, *USA*

and N. L. Goldman and A. H. Lerit

Department of Chemistry, Queens College of The City University of New York, Flushing, New York 11367, *USA*

Abstract

The octahydrophotodimer of biphenylene forms monoclinic crystals with $a = 13 \cdot 168$ (2), $b = 8 \cdot 609$ (1), $c = 21 \cdot 324$ (3) Å, $\beta = 134 \cdot 35$ (3)°, space group $P2_1/c$, $Z = 4$. The structure was determined by direct methods and refined by full-matrix least squares to $R = 0 \cdot 062$ for 2771 independent observed counter-diffractometer-measured reflections. The analysis established the molecule to be *syn*-5,12:6,11-dibutanodibenzo[a,e]-cyclooctene, $C_{24}H_{24}$. Considerable strain arises from the linking of the doubly bonded carbon atoms across the cyclooctatetraene ring by tetramethylene groups, causing non-planarity of the ethylenic groups and distortion of bond angles. Additional strain arises from close interhydrogen interactions between the two tetramethylene bridges.

Introduction

The formation of a photodimer from biphenylene (I) has been described by Goldman & Ruden (1968). Structure (II) was assigned to this substance, which was considered to have been formed by *syn* addition of two biphenylene molecules (one photoexcited) across their C(4a)—C(8b) bonds. This assignment was based largely on proton magnetic resonance data and the catalytic reduction of the photodimer to give an octahydrophotodimer, presumed to be (III). The *syn* stereochemistry of the photodimer was later corroborated by a comparison of its infrared and Raman spectra; ten coincidences within 5 cm⁻¹ were observed between 270 and 1620 cm⁻¹, which would be incompatible with a centrosymmetric *anti* dimer (Ziffer & Levin, 1969).

Subsequently, the ^{13}C magnetic resonance spectrum of the photodimer was determined, showing six resonances that were all in the sp^2 region (141·41, 140·60, 134·79, 127·29, 125·20 and 124·16 p.p.m. from tetramethylsilane in chloroform). This finding is inconsistent with (II), but consistent with either (IV) or (V).

(I)

(II)

(III)

(IV)

(V)

(VI)
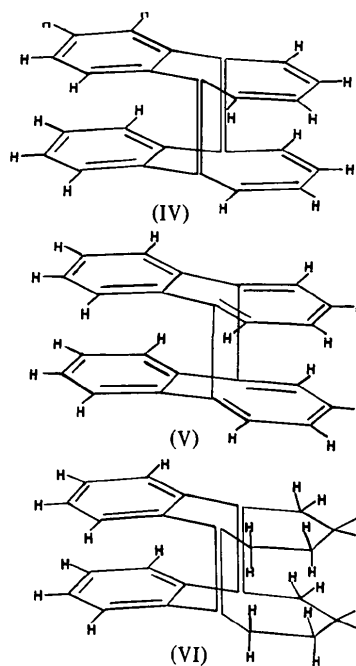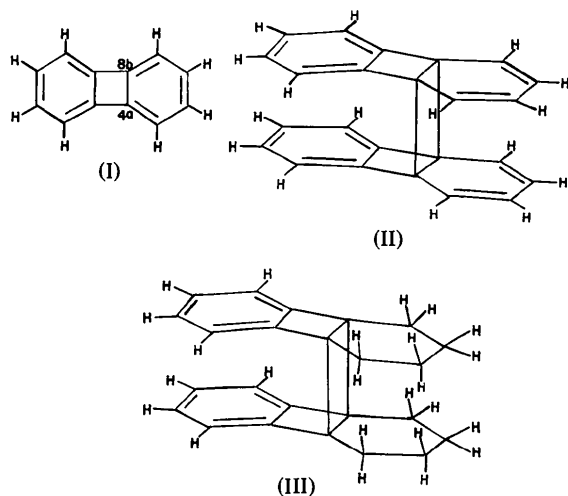
Crystals suitable for an X-ray crystallographic study were not obtained from the photodimer, but were from the octahydrophotodimer. The X-ray results for the latter, as described in the following section, show it to be (VI). In the absence of rearrangement during the hydrogenation, (IV) can be assigned as the structure for the photodimer of biphenylene, probably formed by way of (II); similar conclusions have been reached by Nishida, 1978, and Odaira, 1978 (personal communication from Dr Nishida), on the basis of chemical evidence. Compounds (IV) and (VI), cyclooctadienes with fused benzene rings, are formally cyclooctatetraenes; their systematic names are syn-5,12:6,11-di[1,3]butadienodibenzo[a,e]cyclooctane and syn-5,12:6,11-dibutanodibenzo[a,e]cyclooctene.

Experimental

Crystals, in the form of flat transparent plates, were grown from ethyl acetate solution. For data collection a small piece was cut, measuring $0.25 \times 0.20 \times 0.20$ mm. Unit-cell dimensions and intensity data were measured on an Enraf–Nonius CAD-4 automated diffractometer with Ni-filtered Cu $K\alpha$ radiation, using the ω–2θ scanning mode. The cell parameters were refined by least squares from the diffractometer settings for 15 high-angle reflections.

Crystal data

$C_{24}H_{24}$, $M_r = 312.2$; $a = 13.168$ (2), $b = 8.609$ (1), $c = 21.324$ (3) Å, $\beta = 134.35$ (3)°; $V = 1728.6$ Å^3, $d_m = 1.20$ (flotation), $d_x = 1.20$ Mg m^{-3}, $Z = 4$. Space group $P2_1/c$ ($h0l$, $l = 2n$; $0k0$, $k = 2n$). λ(Cu $K\alpha$) = 1.5418 Å, μ(Cu $K\alpha$) = 0.511 mm^{-1}.

In the range $2\theta < 150°$, 3562 reflections were measured, and of these 3127 were considered observed by the criterion $I/\sigma(I) > 3$. Lorentz and polarization corrections were applied and the structure amplitudes derived. Absorption corrections were not made, and from the crystal dimensions it is calculated that maximum errors due to true absorption are $\sim\pm1\%$ in F_o.

Structure determination and refinement

The structure was solved by direct methods using the program REL (Long, 1965). The set of phases with the highest consistency gave an E map which showed the positions of all the carbon atoms. Refinement was carried out by full-matrix least-squares methods (Busing, Martin & Levy, 1962) using anisotropic temperature factors. A subsequent difference electron-density map showed the positions of all the hydrogen atoms and these were included in the refinement with isotropic temperature factors. At a late stage in the refinement,

Table 1. *Fractional coordinates of the carbon atoms* ($\times 10^4$) *and equivalent isotropic B's*

	x	y	z	B_{eq} (Å^2)
C(1)	6705 (2)	4800 (2)	1276 (1)	4.9 (1)
C(2)	7123 (3)	4758 (4)	832 (1)	6.7 (2)
C(3)	8156 (3)	3783 (4)	1092 (2)	6.7 (1)
C(4)	8805 (2)	2797 (3)	1798 (1)	5.3 (1)
C(5)	9069 (2)	1788 (2)	3025 (1)	4.8 (1)
C(6)	8279 (2)	637 (2)	2953 (1)	3.9 (1)
C(7)	6455 (2)	−889 (2)	1528 (1)	4.4 (1)
C(8)	5068 (2)	−1083 (2)	729 (1)	4.7 (1)
C(9)	4032 (2)	−75 (2)	473 (1)	4.5 (1)
C(10)	4365 (2)	1154 (2)	1007 (1)	4.0 (1)
C(11)	6200 (2)	2675 (2)	2419 (1)	3.6 (1)
C(12)	6961 (2)	3838 (2)	2505 (1)	3.7 (1)
C(13)	7705 (2)	5101 (2)	3184 (1)	4.8 (1)
C(14)	9316 (2)	5146 (2)	3761 (1)	4.7 (1)
C(15)	10146 (2)	3685 (3)	4289 (1)	4.7 (1)
C(16)	10402 (2)	2472 (2)	3886 (1)	4.5 (1)
C(17)	5953 (2)	2383 (3)	3004 (1)	4.8 (1)
C(18)	7077 (3)	1372 (4)	3817 (2)	6.8 (1)
C(19)	7491 (3)	−188 (3)	3697 (1)	6.2 (1)
C(20)	8601 (2)	−127 (3)	3692 (1)	5.7 (1)
C(21)	7325 (2)	3812 (2)	1977 (1)	3.3 (1)
C(22)	8391 (2)	2795 (2)	2243 (1)	3.3 (1)
C(23)	5754 (2)	1374 (2)	1806 (1)	3.2 (1)
C(24)	6815 (2)	343 (2)	2071 (1)	3.4 (1)

Table 2. *Fractional coordinates of the hydrogen atoms* ($\times 10^3$) *and isotropic thermal parameters*

The temperature factors were of the form $\exp(-B \sin^2 \theta/\lambda^2)$.

	x	y	z	B (Å^2)
H(1)	590 (3)	548 (3)	110 (2)	6.3 (5)
H(2)	672 (4)	536 (4)	36 (3)	7.5 (6)
H(3)	856 (4)	385 (4)	85 (2)	8.1 (7)
H(4)	956 (4)	210 (4)	202 (2)	7.1 (6)
H(7)	718 (3)	−155 (3)	170 (2)	4.4 (4)
H(8)	484 (3)	−194 (3)	36 (2)	5.1 (5)
H(9)	307 (3)	−17 (3)	−7 (2)	5.7 (5)
H(10)	361 (3)	186 (3)	83 (2)	4.8 (4)
H(13,1)	721 (3)	620 (3)	285 (2)	6.1 (5)
H(13,2)	753 (3)	492 (3)	356 (2)	6.0 (5)
H(14,1)	950 (3)	541 (3)	340 (2)	6.3 (5)
H(14,2)	966 (3)	601 (3)	414 (2)	5.0 (4)
H(15,1)	1111 (3)	399 (3)	483 (2)	5.8 (5)
H(15,2)	964 (3)	318 (3)	444 (1)	4.6 (4)
H(16,1)	1099 (3)	294 (3)	380 (1)	4.9 (4)
H(16,2)	1096 (3)	168 (3)	431 (2)	5.2 (5)
H(17,1)	592 (3)	333 (4)	321 (2)	6.0 (5)
H(17,2)	503 (3)	192 (3)	263 (2)	6.1 (5)
H(18,1)	799 (4)	215 (4)	424 (2)	7.2 (6)
H(18,2)	670 (4)	123 (4)	410 (2)	8.1 (7)
H(19,1)	784 (3)	−91 (4)	414 (2)	6.4 (6)
H(19,2)	658 (4)	−61 (4)	318 (2)	6.5 (5)
H(20,1)	950 (3)	20 (3)	427 (2)	5.8 (5)
H(20,2)	872 (4)	−138 (5)	362 (3)	10.9 (8)

error analysis showed that the intensities of the reflections with $\sin^2 \theta < 0.2$ were less reliable than the rest and these were omitted together with eight other reflections which appeared to suffer extinction or Renninger effects. Final refinement was carried out

with the remaining 2771 observed reflections. The function minimized was $\sum w(|F_o| - k|F_c|)^2$, where $w = 1/\sigma(F_o)^2$, $\sigma(F_o)$ being derived from the counting statistics. The unobserved reflections were included in the last cycles with a constant weight such that their average $w|\Delta F|^2$ was equal to that for the observed reflections with $F_o < 5\cdot0$.

In the final cycle $R = 0\cdot062$, $R_w = 0\cdot063$ for observed reflections only and $R = 0\cdot071$ for all reflections; $[\sum w|\Delta F|^2/(m - n)]^{1/2} = 1\cdot68$. The atomic scattering factors used were those of Cromer & Waber (1965) for carbon and those of Stewart, Davidson & Simpson (1965) for hydrogen.* Final positional parameters for the carbon atoms and equivalent isotropic B's (Willis & Pryor, 1975) are given in Table 1, and positional parameters and isotropic thermal parameters for the hydrogen atoms in Table 2.

Fig. 2. The cyclododecadiene ring viewed normal to the mean plane of the double bonds. The ring torsion angles (°) (e.s.d.'s 0·2–0·4°), and the short H⋯H interactions (Å) (e.s.d.'s 0·06–0·08 Å) are shown.

Results and discussion

The atomic numbering scheme is shown in Fig. 1. The molecule contains a dibenzocyclooctatetraene system. The two double bonds, C(5)=C(6) and C(11)=C(12), are connected by tetramethylene bridges from C(11) to C(6) and from C(12) to C(5). These bridges, together with both pairs of doubly bonded atoms, form a cyclododecadiene ring. Two additional eight-membered rings also exist. On one side of the molecule, the bridge from C(13)–C(16) and C(5), C(22), C(21) and C(12) form one ring; the other ring is formed by corresponding atoms on the other side. The torsion angles of the cyclododecadiene ring are shown in Fig. 2 which is

* Tables of anisotropic thermal parameters, thermal-analysis data, and structure factors have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35179 (39 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
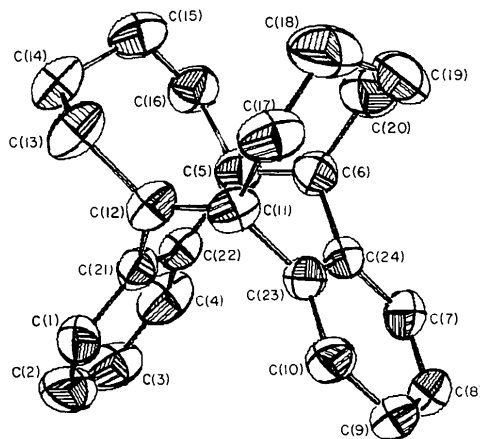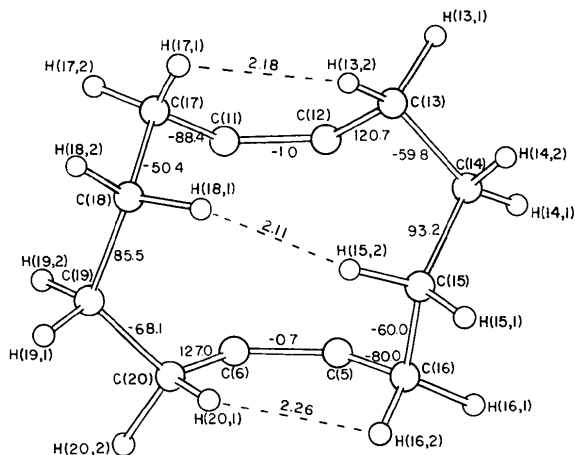
Table 3. *Bond angles involving the carbon atoms* (°)

C(2)–C(1)–C(21)	120·3 (2)	C(1)–C(21)–C(12)	123·0 (2)
C(1)–C(2)–C(3)	120·2 (3)	C(21)–C(12)–C(13)	113·5 (2)
C(2)–C(3)–C(4)	120·6 (3)	C(13)–C(12)–C(11)	126·6 (2)
C(3)–C(4)–C(22)	120·3 (3)	C(12)–C(11)–C(17)	125·8 (2)
C(4)–C(22)–C(21)	119·1 (2)	C(17)–C(11)–C(23)	114·6 (2)
C(22)–C(21)–C(1)	119·5 (2)	C(11)–C(23)–C(10)	123·4 (2)
C(24)–C(7)–C(8)	120·4 (2)	C(7)–C(24)–C(6)	124·5 (2)
C(7)–C(8)–C(9)	120·3 (2)	C(24)–C(6)–C(20)	115·0 (2)
C(8)–C(9)–C(10)	120·2 (2)	C(20)–C(6)–C(5)	125·4 (2)
C(9)–C(10)–C(23)	120·2 (2)	C(6)–C(5)–C(16)	124·3 (2)
C(10)–C(23)–C(24)	119·8 (2)	C(16)–C(5)–C(22)	114·1 (2)
C(23)–C(24)–C(7)	119·1 (2)	C(5)–C(22)–C(4)	123·2 (2)
C(23)–C(11)–C(12)	118·6 (2)	C(5)–C(16)–C(15)	115·0 (2)
C(11)–C(12)–C(21)	119·3 (2)	C(16)–C(15)–C(14)	118·3 (2)
C(12)–C(21)–C(22)	117·5 (2)	C(15)–C(14)–C(13)	116·5 (2)
C(21)–C(22)–C(5)	117·7 (2)	C(14)–C(13)–C(12)	113·3 (2)
C(22)–C(5)–C(6)	118·7 (2)	C(11)–C(17)–C(18)	115·5 (2)
C(5)–C(6)–C(24)	118·3 (2)	C(17)–C(18)–C(19)	118·8 (3)
C(6)–C(24)–C(23)	116·4 (2)	C(18)–C(19)–C(20)	116·1 (3)
C(24)–C(23)–C(11)	116·8 (2)	C(19)–C(20)–C(6)	118·7 (2)

a projection onto the mean plane of the double bonds. It is clear that a pseudo-twofold axis exists perpendicular to this plane. The torsion angles of the cyclooctatetraene ring, which is in the tub form, are given in Table 5.

A thermal-motion analysis was carried out from the anisotropic thermal parameters using $TLS6$ (Schomaker & Trueblood, 1968). The principal axis of libration (m.s. amplitude 27·6°) is nearly parallel to the line C(11)–C(14), or roughly perpendicular to the mean plane of the tetramethylene bridge C(17)–C(20) and the benzene ring fused at C(21)–C(22). The r.m.s. difference between the observed and calculated U_{ij}'s is 0·0057 Å². * Corrections for librational motion to the

Fig. 1. *ORTEP* drawing of the molecule (Johnson, 1965), showing the numbering scheme used.

* See previous footnote.

bond distances ranged from 0·004–0·008 Å, and both the corrected and uncorrected distances are shown in the schematic diagram in Fig. 3. The bond angles involving carbon atoms (uncorrected) are given in Table 3. The experimental C—H distances average 0·99 Å with e.s.d.'s from 0·03 to 0·06 Å.

The existence of the *syn* form of the molecule is a consequence of the fusion of cyclooctatetraene and cyclododecadiene rings at the double bonds. An *anti* conformation would require the chair form of cyclooctatetraene and very large twists (~45°) in the ethylenic groups about the double bonds, although for an isolated cyclododecadiene ring this would give a more favorable conformation by reducing transannular interactions. Given the *syn* form of the molecule, only two general conformations are possible. The bond distances and bond angles of the molecule determine the magnitudes of the torsion angles of the bridges and, therefore, these are actually restricted within quite narrow limits. The two bridges could have the same torsion angles, which would relate them by a twofold axis, or one set could have the signs reversed compared with the other, giving rise to a mirror plane. This second possibility would bring one of the pairs C(15), C(19) or C(14), C(18) well within the van der Waals distance, and so the twofold axis is preferred.

From Fig. 2 it can be seen that of the six torsion angles of the bonds joining methylene groups, four are close to ±60°, the low-energy synclinal type (Klyne & Prelog, 1960) while the other two, C(14)—C(15) and C(18)—C(19), deviate by about 30° from these favorable values, introducing strain. However, as pointed out above, these angles are constrained, and could not be reduced without serious additional distortions of bond angles. In the tetramethylene bridge C(17)—C(20) the Newman projections of C(17)—C(18) and C(19)—C(20) show good staggered arrangements of the substituents, while C(18)—C(19) is about halfway between the staggered and eclipsed forms. The corresponding angles in the Newman projections of the bonds in the two bridges differ by only 5° on average.

The main source of strain in the molecule is the fact that the tetramethylene groups with normal bond

angles of ~112° (Dunitz, 1968) would be too short to bridge coplanar ethylenic groups. This strain is distributed in three ways. (1) The eight interior bond angles at the methylene groups are widened to an average angle of 116·5°. (2) The four angles C(11)—C(23)—C(24) *etc.* of the cyclooctatetraene ring average 117·1°, this narrowing serving to bring the ethylenic groups closer. (3) Atoms C(13), C(17), C(16) and C(20) are displaced inward from the mean planes of the other four atoms of each ethylenic group, as shown in Table 4.

Another source of strain arises from transannular repulsions between hydrogen atoms of separate tetramethylene bridges. These are illustrated in Fig. 2 where the experimental H···H distances are shown. However, since the centers of the hydrogen electron clouds, measured in the X-ray experiment, are perturbed by bonding forces towards the adjacent carbon atoms the proton–proton distances are probably even shorter. By moving the hydrogen atoms involved out along the C—H bonds to a distance of 1·08 Å, these distances are calculated to be: H(13,2)···H(17,1) 2·07; H(15,2)··· H(18,1) 2·05; H(16,2)···H(20,1) 2·15 Å. Using the non-bonded H···H potential function of Hendrickson (1967), we calculate that the total repulsive energy of these three interactions is 8·3 kJ mol⁻¹. While at these distances such calculations involve considerable uncertainty, it would appear that the repulsive energy, while certainly significant, is probably only a fraction of the total strain energy. However, the repulsions would be greater were it not for the widening of the four angles C(17)—C(11)—C(12) *etc.* to an average of 125·4°, which moves the two bridges away from each other.

The bond distances, bond angles and torsion angles (Table 5) not directly affected by strain-minimization considerations are in general normal, but there are some exceptions. The benzene ring fused at C(23)—C(24) shows distances very typical of a benzene ring joined to another system by two *ortho* single bonds, with the progressive decrease in length from C(23)—C(24) to C(8)—C(9) (Ardebili, Dougherty, Mislow,
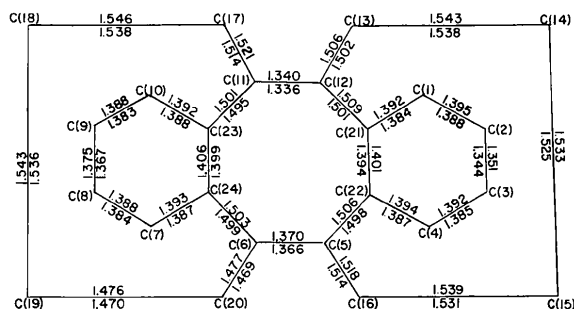

Fig. 3. Bond distances (Å) between the carbon atoms. The values shown above are corrected for molecular libration, those below are uncorrected. The e.s.d.'s range from 0·002–0·006 Å.

Table 4. *Deviations of atoms of the ethylenic groups from the least-squares mean planes of the sets of four atoms common to cyclooctatetraene (Å)*

Plane 1: C(5), C(6), C(22), C(24)

$$0.7431X - 0.6208Y - 0.2498Z = 3.3899$$

C(5)	0·026 (3)	C(24)	0·013 (2)
C(6)	−0·026 (2)	C(16)	−0·317 (3)
C(22)	−0·013 (2)	C(20)	−0·401 (3)

Plane 2: C(11), C(12), C(21), C(23)

$$-0.4587X + 0.4899Y - 0.7413Z = -3.7011$$

C(11)	0·004 (2)	C(23)	−0·002 (2)
C(12)	−0·004 (2)	C(13)	−0·224 (2)
C(21)	0·002 (2)	C(17)	−0·231 (3)

Table 5. *Torsion angles involving the carbon atoms* (°) *excluding those given in Fig. 2 and those within the benzene rings*

The e.s.d.'s are in the range 0·2–0·3°.

C(5)–C(6)–C(24)–C(23)	−78·4	C(14)–C(13)–C(12)–C(21)	−49·9
C(6)–C(24)–C(23)–C(11)	1·4	C(18)–C(17)–C(11)–C(23)	80·3
C(24)–C(23)–C(11)–C(12)	72·3	C(5)–C(6)–C(24)–C(7)	103·5
C(23)–C(11)–C(12)–C(21)	0·8	C(20)–C(6)–C(24)–C(7)	−89·0
C(11)–C(12)–C(21)–C(22)	−75·4	C(20)–C(6)–C(24)–C(23)	89·1
C(12)–C(21)–C(22)–C(5)	1·1	C(6)–C(5)–C(22)–C(4)	−113·6
C(21)–C(22)–C(5)–C(6)	68·6	C(16)–C(5)–C(22)–C(4)	84·6
C(22)–C(5)–C(6)–C(24)	5·6	C(16)–C(5)–C(22)–C(21)	−93·1
C(22)–C(5)–C(6)–C(20)	−160·5	C(11)–C(12)–C(21)–C(1)	108·1
C(24)–C(6)–C(5)–C(16)	165·3	C(13)–C(12)–C(21)–C(1)	−80·6
C(23)–C(11)–C(12)–C(13)	−169·3	C(13)–C(12)–C(21)–C(22)	96·0
C(21)–C(12)–C(11)–C(17)	169·1	C(12)–C(11)–C(23)–C(10)	−107·9
C(19)–C(20)–C(6)–C(24)	−39·5	C(17)–C(11)–C(23)–C(10)	82·5
C(15)–C(16)–C(5)–C(22)	80·5	C(17)–C(11)–C(23)–C(24)	−97·3

Schwartz & White, 1978). However, in the other benzene ring the C(2)–C(3) distance is abnormally short, 4σ less than that of C(8)–C(9). Owing to the position of the principal libration axis, C(2) and C(3) have large components of thermal vibration along the bond itself. Either because of incomplete correction for thermal-vibration effects, or the existence of other systematic errors, the e.s.d. of this bond must be considered low. The double bond C(11)=C(12) is close to the expected value, but C(5)=C(6) is significantly lengthened. Since this bond is in the ethylenic group more distorted from planarity, the lengthening is consistent with a weakening of the bond by reduction of π-bond orbital overlap. Omitting C(6)–C(20), the bonds joining the double bonds to the tetramethylene bridges average 1·515 Å, in excellent agreement with the value given by Dewar & Schmeising (1960) for Csp^2–Csp^3 bonds. All the distances of the sp^3–sp^3 bonds in the bridges themselves are normal with the exception of C(19)–C(20) which is 12σ less than the average of the other five. While a change in electronic distribution due to the non-planarity of the ethylenic group might conceivably shorten the bond C(6)–C(20), it seems implausible that such an effect would extend to the second neighbor of the double bond. The final electron-density map showed no evidence of disorder in this region, and in view of the rigidity of the system this would be unlikely. As indicated above the existence of systematic errors cannot be excluded, but they could hardly be expected to cause a difference of this magnitude. We have, therefore, been unable to find a satisfactory explanation for this bond distance.

The bond distances and angles of the cyclooctatetraene moiety may be compared with those found in cyclooctatetraene itself. The single-bond distances in cyclooctatetraene, average 1·456 Å (Bordner, Parker & Stanford, 1972),* are considerably shorter than our

---

* These authors acknowledge receipt of X-ray results on cyclooctatetraene from J. Bregman and B. Post.

average value of 1·505 Å, but a molecular-orbital calculation gives 1·491 Å for these bonds (Allinger, Sprague & Finder, 1973). A real lengthening may exist in our structure due to reduction of hyperconjugation on substitution of a benzenoid for a double bond. Our single-bond torsion angles average ±73·7° compared with ±55·7° in cyclooctatetraene, but this is a consequence of greater puckering due to strain, which reduces the interior ring angles from 126·8° in cyclooctatetraene to 117·9° on average.

References

ALLINGER, N. L., SPRAGUE, J. T. & FINDER, C. J. (1973). *Tetrahedron*, 29, 2519–2523.
ARDEBILI, M. H. P., DOUGHERTY, D. A., MISLOW, K., SCHWARTZ, L. H. & WHITE, J. G. (1978). *J. Am. Chem. Soc.* 100, 7994–7997.
BORDNER, J., PARKER, R. G. & STANFORD, R. H. (1972). *Acta Cryst.* B28, 1069–1075.
BUSING, W. R., MARTIN, K. O. & LEVY, H. A. (1962). *ORFLS.* Report ORNL-TM-305. Oak Ridge National Laboratory, Tennessee.
CROMER, D. T. & WABER, J. T. (1965). *Acta Cryst.* 18, 104–109.
DEWAR, M. J. S. & SCHMEISING, H. N. (1960). *Tetrahedron*, 11, 96–120.
DUNITZ, J. D. (1968). *Perspectives in Structural Chemistry*, Vol. II, pp. 9–10. New York: John Wiley.
GOLDMAN, N. L. & RUDEN, R. A. (1968). *Tetrahedron Lett.* pp. 3951–3954.
HENDRICKSON, J. B. (1967). *J. Am. Chem. Soc.* 89, 7036–7043.
JOHNSON, C. K. (1965). *ORTEP.* Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.
KLYNE, W. & PRELOG, V. (1960). *Experientia*, 16, 521–523.
LONG, R. E. (1965). PhD dissertation. Univ. of California at Los Angeles.
SCHOMAKER, V. & TRUEBLOOD, K. N. (1968). *Acta Cryst.* B24, 63–76.
STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* 42, 3175–3187.
WILLIS, B. T. M. & PRYOR, A. W. (1975). *Thermal Vibrations in Crystallography*, pp. 101–102. Cambridge Univ. Press.
ZIFFER, H. & LEVIN, L. W. (1969). *J. Org. Chem.* 34, 4056–4060.