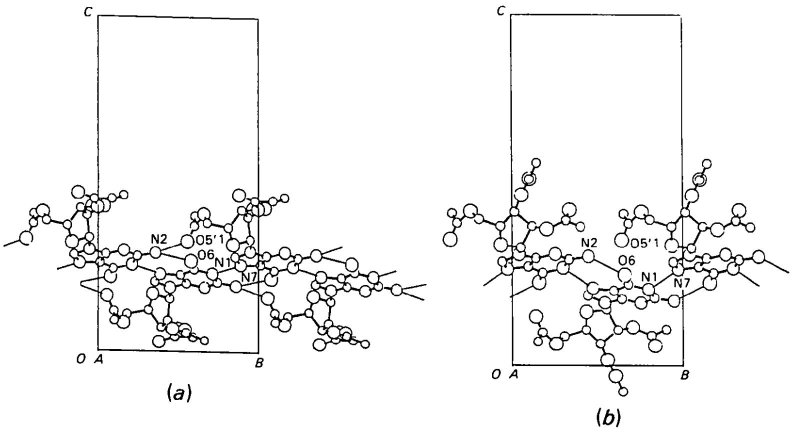

Fig. 2. Proposed hydrogen bonding of (*a*) molecule *A* and (*b*) molecule *B*.

detail (Wilson, Low & Tollin, to be published). In the structure of monoclinic TAG (Wilson, Low & Tollin, 1985), the acetyl O(2′)1 intercalates between two parallel bases, separated by 6·2 Å, with possible biological implications (Wilson & Tollin, 1985). However, while the 'scorpion-tail' shape of monoclinic TAG is repeated in molecule *A* of the present structure, O(2′)1 in this case does not intercalate. Instead, there is very strong stacking of O(4′) and acetyl O(5′)1 of molecule *A* above the base of *B*, on the opposite side of the base ring to O(2′)1. The existence of this second stacking contact involving these two O atoms with molecule *B* precludes the extended base–acetyl–base stacking structure noted in monoclinic TAG.

References

Bugg, C. E., Thomas, J. M., Sundaralingam, M. & Rao, S. T. (1971). *Biopolymers*, **10**, 175–219.

Dickerson, R. E. (1983). *J. Mol. Biol.* **166**, 419–441.

Gilmore, C. G. (1983). *MITHRIL*. A computer program for the automatic solution of crystal structures from X-ray data. Univ. of Glasgow.

Hoogsteen, K. (1959). *Acta Cryst.* **12**, 822–823.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

IUPAC–IUB Joint Commission on Biochemical Nomenclature (1983). *Pure Appl. Chem.* **55**, 1273–1280.

Low, J. N. & Wilson, C. C. (1983). *Acta Cryst.* C**39**, 1688–1690.

Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

Roberts, P. & Sheldrick, G. M. (1975). *XANADU*. Program for torsion-angle, mean-plane and libration-correction calculations. Univ. of Cambridge, England.

Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Wilson, C. C., Low, J. N. & Tollin, P. (1985). *Acta Cryst.* C**41**, 1123–1125.

Wilson, C. C. & Tollin, P. (1985). *Curr. Sci.* **54**, 1102–1104.

Acta Cryst. (1986). C**42**, 703–705

Structure of 1,2:7,8-Dibenzo[2.2]paracyclophane

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Abstract. $C_{24}H_{16}$, $M_r = 304{\cdot}39$, monoclinic, $P2_1/c$, $a = 7{\cdot}074$ (2), $b = 17{\cdot}761$ (4), $c = 12{\cdot}925$ (3) Å, $\beta = 98{\cdot}55$ (2)°, $V = 1605{\cdot}9$ (7) Å^3, $Z = 4$, $D_m = 1{\cdot}25$ (1), $D_x = 1{\cdot}259$ g cm^{-3}, Mo $K\alpha$, $\bar{\lambda} = 0{\cdot}71069$ Å, $\mu = 0{\cdot}66$ cm^{-1}, $F(000) = 639{\cdot}92$, $T = 295$ K, $R = 0{\cdot}093$ for 1064 observed data and 157 parameters. The two *para*-fused benzenoid rings are boat-shaped, and the innermost (bridging) bond of each *ortho*-fused ring is significantly lengthened to 1·431 (10) Å. The molecule departs very slightly from idealized D_{2h} symmetry.

Introduction. Compounds possessing orthogonal benzenoid rings are decidedly rare. Jacobson & Boekelheide (1978) reported the isolation and

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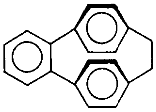
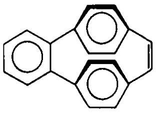
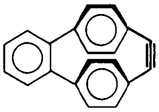
characterization of cyclophane (1) and cyclophene (2). Recently we succeeded in synthesizing 1,2:7,8-dibenzo-[2.2]paracyclophane* (formula given in Fig. 2; Chan & Wong, 1985) *via* trapping of the elusive cyclophyne intermediate (3). The present structural study was undertaken to establish the orthogonality of the two sets of *para-* and *ortho*-fused benzenoid rings and to measure the extent of distortion in this highly strained hydrocarbon.


(1)


(2)


(3)

Experimental. Only a small amount of the title compound was available, and much difficulty was encountered in growing crystals of acceptable quality (hence high R value, see below). Single crystal recrystallized from hexane; D_m measured by flotation in aqueous potassium iodide; colorless prism $0{\cdot}20 \times 0{\cdot}16 \times 0{\cdot}14$ mm; Nicolet $R3m$ diffractometer, graphite-monochromatized Mo $K\alpha$ radiation; unit-cell parameters by least-squares adjustment to setting angles of 25 reflections with $12 < 2\theta < 20°$ (Sparks, 1976); ω–2θ variable scan (2·02 to 8·37° min^{-1}), range 1° below $K\alpha_1$ to 1° above $K\alpha_2$, background counting for one-half of scan time on each side of peak; $2\theta_{\max} = 42°$, collection range $h = 0$ to 7, $k = 0$ to 17, $l = -12$ to 12; two standards (131 and $13\bar{1}$) monitored every 125 measurements, intensity variation <2%; 1544 unique reflections, $R_{\rm int} = 0{\cdot}008$, 1064 observed with $I > 1{\cdot}5\sigma(I)$; Lorentz and polarization factors applied, no absorption correction. Structure solution by direct phasing based on negative quartets (DeTitta, Edmonds, Langs & Hauptman, 1975); C atoms of *para*-type rings isotropic, C atoms of *ortho* rings anisotropic, C atoms of *ortho* rings anisotropic (to limit the number of parameters); H atoms generated geometrically (C—H fixed at 0·96 Å), assigned isotropic temperature factors, and allowed to ride on their respective parent C atoms; refinement of 157 parameters by blocked-cascade least squares (Schilling, 1970) with $\sum w(|F_o| - |F_c|)^2$ minimized, $w = [\sigma^2(|F_o|) + 0{\cdot}0012|F_o|^2]^{-1}$; final $R = 0{\cdot}093$, $wR = 0{\cdot}089$, $S = 1{\cdot}447$, max. $\Delta/\sigma = 0{\cdot}006$; residual extrema in final difference map +0·28 to −0·30 e Å^{-3}. *SHELXTL* program system (Sheldrick, 1982) on Data General Nova 3/12 minicomputer, Nicolet Zeta 1553 digital plotter; analytic expressions of atomic scattering factors, anomalous-dispersion corrections incorporated (*International Tables for X-ray Crystallography,* 1974).

Table 1. *Fractional atomic coordinates* (× 10^4) *and thermal parameters* (Å^2 × 10^3)

	x	y	z	$U_{\rm eq}$ or U
C(1)	5251 (9)	4589 (4)	3051 (6)	51 (3)*
C(2)	5185 (10)	4617 (4)	1945 (7)	50 (3)*
C(3)	3742 (10)	4132 (4)	1287 (5)	50 (2)
C(4)	4174 (10)	3388 (4)	1113 (5)	54 (2)
C(5)	2746 (9)	2847 (4)	981 (5)	50 (2)
C(6)	862 (9)	3037 (4)	1039 (5)	44 (2)
C(7)	412 (10)	3791 (4)	972 (5)	52 (2)
C(8)	1837 (10)	4339 (4)	1100 (5)	57 (2)
C(9)	−462 (9)	2490 (4)	1447 (5)	40 (3)*
C(10)	−1737 (10)	2022 (4)	837 (6)	53 (3)*
C(11)	−2927 (10)	1538 (4)	1261 (8)	63 (4)*
C(12)	−2857 (10)	1503 (4)	2330 (6)	57 (3)*
C(13)	−1582 (10)	1954 (4)	2966 (6)	53 (3)*
C(14)	−392 (9)	2456 (4)	2563 (6)	42 (3)*
C(15)	1035 (9)	2954 (4)	3214 (5)	40 (2)
C(16)	2941 (9)	2746 (4)	3402 (5)	51 (2)
C(17)	4364 (10)	3289 (4)	3513 (5)	49 (2)
C(18)	3927 (10)	4040 (4)	3450 (5)	45 (2)
C(19)	2043 (10)	4240 (4)	3505 (5)	59 (2)
C(20)	609 (10)	3696 (4)	3384 (5)	55 (2)
C(21)	6577 (10)	5035 (4)	3673 (7)	71 (4)*
C(22)	7768 (12)	5512 (5)	3250 (10)	89 (5)*
C(23)	7696 (13)	5552 (5)	2190 (10)	91 (5)*
C(24)	6398 (11)	5108 (4)	1529 (7)	70 (4)*

* Equivalent isotropic temperature factor $U_{\rm eq}$ defined as $\frac{1}{3}$ of the trace of the orthogonalized U matrix. The exponent of the isotropic temperature factor takes the form $-8\pi^2 U \sin^2\theta/\lambda^2$.

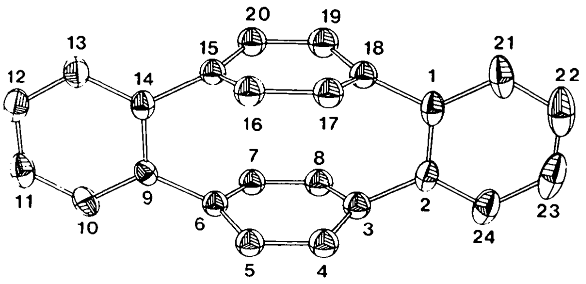


Fig. 1. Perspective view of the molecule with the atom numbering.

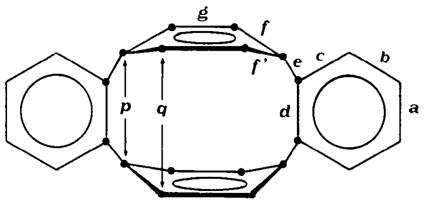


Fig. 2. Average dimensions of the molecule: a 1·371, b 1·380, c 1·385, d 1·431, e 1·499, f 1·381, g 1·390, p 2·80, q 3·10 Å, ab 120·0, bc 121·6, cd 118·4, ce 124·6, de 117·0, ef 120·4, fg 120·7, ff' 117·1°. Standard deviations are *ca* 0·009 Å, 0·6° and 0·01 Å for bond lengths, bond angles and transannular non-bonded distances, respectively.

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* The less descriptive IUPAC name for this compound is 5,8:13,16-diethenodibenzocyclododecene.

Discussion. The final atomic parameters for the title compound are listed in Table 1.* A perspective view of the molecule and the atom-numbering scheme are given in Fig. 1. Fig. 2 shows the structural formula, labels of C—C bonds, and dimensions averaged according to idealized D_{2h} molecular symmetry.

The sets of atoms (I) [C(4), C(5), C(7) and C(8)], (II) [C(16), C(17), C(19) and C(20)], (III) [C(1), C(2), and C(21) to C(24)] and (IV) [C(9) to C(14)] all conform to strict planarity, the maximum deviations from the corresponding best planes being 0·002, 0·003, 0·009 and 0·009 Å, respectively. The molecule, however, appears to deviate very slightly but perceptibly from rigorous D_{2h} symmetry. The dihedral angles ($\sigma \sim 0{\cdot}3°$) between pairs of planes are: (I) and (II), 0·7°; (I) and (III), 87·7°; (II) and (III), 87·8°; (I) and (IV), 87·9°; (II) and (IV), 88·0°; (III) and (IV), 1·3°.

The considerable molecular strain is reflected by the fact that each *para*-fused benzenoid ring is boat-shaped, with its bow and stern C atoms displaced inward towards the other ring by 0·15 (1) Å. The transannular non-bonded distances *p* and *q* (see Fig. 2) agree closely with the corresponding values of 2·78 and 3·09 Å found in [2.2]paracyclophane (Hope, Bernstein & Trueblood, 1972). The inter-ring bridging bond *e* is normal (see data compiled by Keehn, 1983) and in excellent accord with the measured distance of 1·488 (2) Å in 1,2:3,4:11,12:13,14-tetrabenzo[4.4]-metacyclophane (Irngartinger, 1976). An unusual feature in the present structure is the significant lengthening of bond *d* in relation to other C—C bonds in the *ortho*-fused ring. A precedent of this type of distortion in highly strained hydrocarbons exists in 5,6,11,12-tetradehydrodibenzo[*a,e*]cyclooctene, in which the inner C_{sp^2}—C_{sp^2} bond has a length of 1·435 (2) Å at 290 K (Destro, Pilati & Simonetta, 1977).

References

Chan, C. W. & Wong, H. N. C. (1985). *J. Am. Chem. Soc.* **107**, 4790–4791.

Destro, R., Pilati, T. & Simonetta, M. (1977). *Acta Cryst.* B**33**, 447–456.

DeTitta, G. T., Edmonds, J. W., Langs, D. A. & Hauptman, H. (1975). *Acta Cryst.* A**31**, 472–479.

Hope, H., Bernstein, J. & Trueblood, K. N. (1972). *Acta Cryst.* B**28**, 1733–1743.

International Tables for X-ray Crystallography (1974). Vol. IV, pp. 55, 99, 149. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

Irngartinger, H. (1976). *Acta Cryst.* B**32**, 696–702.

Jacobson, N. & Boekelheide, V. (1978). *Angew. Chem. Int. Ed. Engl.* **17**, 46–47.

Keehn, P. M. (1983). *Cyclophanes*, Vol. I, edited by P. M. Keehn & S. M. Rosenfeld, pp. 69–238. New York: Academic Press.

Schilling, J. W. (1970). *Crystallographic Computing*, edited by F. R. Ahmed, pp. 201–204. Copenhagen: Munksgaard.

Sheldrick, G. M. (1982). *Computational Crystallography*, edited by D. Sayre, pp. 506–514. Oxford Univ. Press.

Sparks, R. A. (1976). *Crystallographic Computing Techniques*, edited by F. R. Ahmed, pp. 452–467. Copenhagen: Munksgaard.

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* Lists of bond lengths and bond angles, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42735 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 4*β*,10*β*-Dimethyleudesma-5,11(13)-diene-7*β*-lactone (Alantolactone)*

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Abstract. $C_{15}H_{20}O_2$, $M_r = 232{\cdot}32$, orthorhombic, $P2_12_12_1$, $a = 6{\cdot}258\,(1)$, $b = 8{\cdot}156\,(1)$, $c = 25{\cdot}338\,(1)$ Å, $V = 1293{\cdot}3\,(2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}193$ Mg m^{-3}, Cu *Kα* radiation, $\lambda = 1{\cdot}5418$ Å, $\mu = 0{\cdot}53$ mm^{-1}, $F(000) = 504$, $T = 296$ K, final $R = 0{\cdot}042$ for 663 observed reflections. The naturally occurring sensitizing sesquiterpene lactone exhibits a distorted chair-shaped cyclohexane ring which is connected with a cyclohexane ring showing a deformed boat conformation. The 7,8-*cis*-fused lactone ring has a flattened envelope form. C(8) deviates −0·12 (1) Å, the *α*-methylene C(13) 0·11 (1) Å and the carbonyl oxygen O(2) −0·01 (1) Å from the best plane. Bond distances and angles are normal. The structure consists of discrete molecules.

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* IUPAC name: 3a*α*,5,6,7,8,8a,9,9a*α*-octahydro-5*β*,8a*β*-dimethyl-3-methylenenaphtho[2,3-*b*]furan-2(3*H*)-one.

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