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1,4,7,10-Tetramethyl-5,6-didehydrodibenzo[*a*,*e*]cyclooctene* (9,12,13,16-Tetramethyl-*sym*-dibenzo-1,3,5-cyclooctatrien-7-yne)

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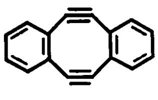
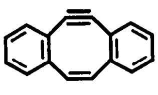
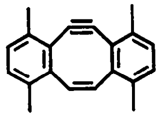
Abstract. $C_{20}H_{18}$, monoclinic, $P2_1$, $a = 10{\cdot}884\ (2)$, $b = 13{\cdot}290\ (3)$, $c = 4{\cdot}965\ (1)$ Å, $\beta = 95{\cdot}42\ (2)^\circ$, $Z = 2$, $D_m = 1{\cdot}17\ (1)$ by flotation in aqueous KI, $D_x = 1{\cdot}199$ Mg m^{-3}. To a very good approximation ($R = 0{\cdot}113$ for 761 observed Cu $K\alpha$ diffractometer data), the analysis was performed in space group $P2_1/n$ in accordance with a disordered structure in which the central ethylenic and acetylenic bridges are indistinguishable.

Introduction. The dibenz-annelated compounds *sym*-dibenzo-1,5-cyclooctadiene-3,7-diyne (I) and *sym*-dibenzo-1,3,5-cyclooctatrien-7-yne (II), which contain planar neutral conjugated eight-membered rings, were first reported by Wong, Garrett & Sondheimer (1974). The diacetylene (I) was comparatively stable, with only slight decomposition upon standing at room temperature for 2 d. Its crystal structure has been carefully studied at 290, 218 and 113 K (Destro, Pilati & Simonetta, 1977). On the other hand, the mono-acetylene (II) was very unstable and decomposed after standing for a few minutes.

Recently we synthesized the title compound (III), a tetramethyl derivative of (II), which turned out to be remarkably stable (Au, Siu, Mak & Chan, 1978). The present X-ray analysis was undertaken to provide the necessary structural proof.


(I)


(II)


(III)

Compound (III) was recrystallized from acetone as yellowish prisms elongated along *c*, m.p. 527–528 K. Using an approximately spherical crystal of diameter 0·4 mm, accurate unit-cell parameters and the intensities of 992 unique reflections up to $\sin\theta/\lambda = 0{\cdot}538$ Å^{-1} were measured on an automated diffractometer with Cu $K\alpha$ radiation ($\lambda = 1{\cdot}5418$ Å). The data were corrected for Lorentz and polarization factors but absorption was ignored ($\mu = 0{\cdot}517$ mm^{-1}). Reflections $h0l$ with $(h + l)$ odd are either very weak or unobservable.

To a very good approximation, the space group was taken as $P2_1/n$ in accordance with a disordered structure in which the molecule possesses an apparent centre of symmetry. In this idealized model, each molecule can take up either of two possible orientations related to each other by swapping the central ethylenic and acetylenic bridges; the two molecular orientations are assumed to occur randomly throughout the crystal lattice. The structure was solved with *MULTAN* (Germain, Main & Woolfson, 1971). All ten C atoms in the asymmetric unit (Fig. 1) were located in the resultant *E* map. Full-matrix anisotropic least-squares refinement yielded a final *R* of 0·113 for 761 observed reflections. A Fourier map (Fig. 1) showed that the electron densities around the bridging atoms C(9) and C(10) were very diffuse. Further refinement in the true space group $P2_1$ did not prove successful.

Scattering factors for carbon atoms were taken from *International Tables for X-ray Crystallography* (1974). Computations were performed on an ICL-1904A system with *ORFLS* (Busing, Martin & Levy, 1962). The function minimized was $\Sigma w(|F_o| - |F_c|)^2$, with $w = (8{\cdot}0 + |F_o| + |F_o|^2/125{\cdot}0)^{-1}$.

The final atomic parameters are listed in Table 1.‡ The mean values of the measured bond distances and

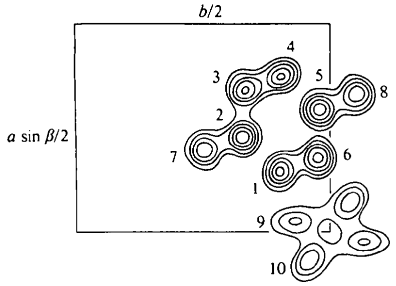


Fig. 1. Electron density projected along **c**. Contours are drawn at intervals of 1 e Å^{-2} starting with 3 e Å^{-2}. Atom numbering corresponds to the positional parameters in Table 1.

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* Systematic IUPAC name.

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‡ Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33923 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Atomic coordinates* ($\times 10^4$) *with e.s.d.'s in parentheses*

	x	*y*	*z*
C(1)	3537 (4)	4045 (4)	4065 (10)
C(2)	2704 (5)	3299 (4)	3019 (10)
C(3)	1539 (5)	3338 (4)	4003 (12)
C(4)	1218 (5)	4049 (4)	5881 (11)
C(5)	2065 (5)	4789 (4)	6939 (9)
C(6)	3226 (5)	4766 (4)	5975 (10)
C(7)	3025 (5)	2556 (4)	962 (11)
C(8)	1703 (5)	5520 (4)	8983 (11)
C(9)	4769 (7)	4308 (9)	3556 (17)
C(10)	5705 (8)	4650 (6)	3515 (18)

angles, with molecular symmetry *mmm* (D_{2h}) assumed, are shown in Fig. 2.

Discussion. The precision of the present analysis of (III) is limited by disorder. Nevertheless, the following structural features are worthy of note. (1) The molecule is planar within experimental error. (2) As far as molecular packing is concerned, the central ethylenic and acetylenic bridges are equivalent since they have virtually the same span between the benzene rings. Presumably the difference in length of the double and triple bonds is compensated by the different arching of the —C=C— and —C≡C— bridges. (3) The central eight-membered ring has an average span of 3·863 (7) Å which is significantly longer than the corresponding distance of 3·834 (3) Å in compound (I) (Destro, Pilati & Simonetta, 1975, 1977), and the difference gives a measure of the greater span of the ethylenic bridge.

We are grateful to Mr Masao Chiku of Jasco/Syntex Co. Ltd, Tokyo, for his intermediation in having the intensity data collected on a Syntex $P2_1$ diffractometer.

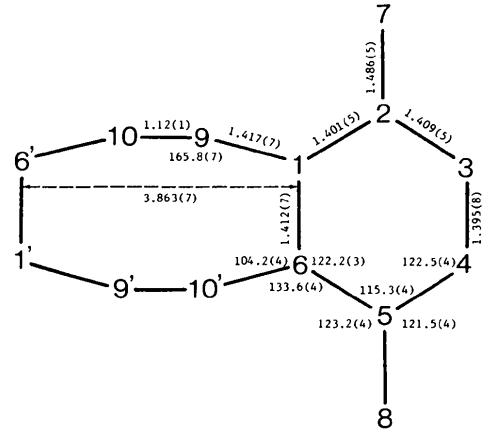

Fig. 2. Molecular dimensions of idealized model of (III) having symmetry *mmm*. Primed and unprimed atoms are related by the centre of symmetry at ($\frac{1}{2}, \frac{1}{2}, \frac{1}{2}$).

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Structure of β-6-Deoxyoxytetracycline Hydrochloride

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Abstract. $C_{22}H_{24}N_2O_8.HCl$, orthorhombic, $P2_12_12_1$, $a = 11{\cdot}638$ (4), $b = 12{\cdot}177$ (3), $c = 15{\cdot}840$ (4) Å. The unit cell contains four molecules; $D_m = 1{\cdot}42$, $D_x = 1{\cdot}426$ Mg m^{-3}. Full-matrix least-squares refinement of the structure resulted in an R index of 0·066. The absolute configuration has been determined.

Introduction. Tetracycline and some of its derivatives represent an important class of broad-spectrum antibiotics. Because of their importance, a number of crystal structure determinations have been reported on this class of compounds. At least five research groups have reported examples of this class of compounds: 7-

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