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Structure of 9,10-Dihydro-9,10-ethenoanthracene-11,12-dicarbonitrile

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Abstract. $C_{18}H_{10}N_2$, $M_r = 254·2$, monoclinic, $P2_1/c$, $a = 9·885$ (5), $b = 14·210$ (7), $c = 9·6066$ (5) Å, $\beta = 103·08$ (10)°, $U = 1314$ Å^3, $Z = 4$, $D_m = 1·28$ (1), $D_x = 1·28$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1·5418$ Å, $\mu = 5·21$ cm^{-1}, $F(000) = 528$, room temperature, $R = 0·046$ for 1052 observed reflections. The X-ray crystallographic structure is similar to that of 1-bromotriptycene [Palmer & Templeton (1968). *Acta Cryst.* B**24**, 1048–1052]. The short cyanide–ethane bond lengths [1·439 (5), 1·429 (5) Å] may furnish some evidence of electron delocalization, though the cyanide bond lengths [1·141 (4), 1·139 (4) Å] are of the expected magnitude. The other bond lengths and angles do not reveal any peculiarities.

Introduction. The Diels–Alder addition reaction of dicyanoacetylene with anthracene has been used for the preparation of the title compound (Weis, 1963). No doubt prompted by the similarity of the dicyano grouping in this compound to that of phthalonitrile, a commonly used precursor in the synthesis of phthalocyanines, the use of the former compound has been described (Kopranenkov & Rumyantseva, 1975). In order to gain some idea of the volume available in the axial positions of the metallobarrelenoporphoryrazine as well as pave the way to the interpretation of their X-ray crystallographic data the molecular structure of the title compound has been determined. In addition, such a study provides the opportunity of studying the structural effect of a single ethene group, which is not part of a peripheral benzene group, on the central ring system.

Experimental. A more convenient method for the preparation of the dicyano compound starts with the Diels–Alder addition of the dimethyl ester of dicarboxyacetylene to anthracene (Diels & Thiele, 1931; Holmes, 1949), followed by conversion of the diester product to the diamide by treatment with ammonia and final conversion to the dicyano form by reaction of the diamide with thionyl chloride in dimethylformamide. The final product after recrystallization from acetonitrile was characterized as follows: m.p. 540–541 K; composition: calculated: C 85·02, H 3·96, N 11·02%, found: C 85·38, H 4·20, N 10·72%; MS m/e (rel. int. %) 254 (M^+, 100%), 227 (38), 203 (12), 178 (38). ^{1}H NMR 7·54 (4H), 7·13 (4H), 6·03 (2H) p.p.m.

Crystal dimensions 0·2 × 0·1 × 0·1 mm. D_m by flotation. Cell dimensions determined from 24 reflections. 1948 reflections measured, $\theta = 3$–$60°$, Philips PW 1100 diffractometer, 1052 [$I \geq 3\sigma(I)$] used, index range h −11/10, k 0/15, l 0/10; Lorentz–polarization and absorption corrections (transmission coefficients max. 0·955, min. 0·924) applied; standard reflections measured every 4 h showed no reduction in intensity over the data-collection period; structure solved by direct methods; refined by full-matrix least squares using *SHELX76* (Sheldrick, 1976), F values, anisotropic temperature factors for non-hydrogen atoms and H atoms in geometrically calculated positions (riding model, C—H 1·08 Å) with a common isotropic temperature factor [U 0·071 (4) Å^2], $R = 0·046$, $wR = 0·047$, where $w = \sigma^{-2}(F)$; $\Delta/\sigma_{\max}$ 0·001 in final cycle; no correction for extinction; scattering factors taken from *International Tables for X-ray Crystallography*

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(1974); residual electron density in final ΔF map within $\pm0{\cdot}2\ \text{e}\ \text{Å}^{-3}$; computations performed on a VAX 11/780 computer.

Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors* (Å^2) *with e.s.d.'s in parentheses*

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

	x	y	z	U_{eq}
C(1)	0·7720 (4)	0·0962 (3)	0·1343 (4)	0·041 (1)
C(2)	0·8186 (4)	0·0727 (3)	0·0131 (4)	0·053 (2)
C(3)	0·7888 (4)	−0·0174 (3)	−0·0444 (4)	0·062 (2)
C(4)	0·7155 (4)	−0·0812 (3)	0·0177 (4)	0·057 (1)
C(5)	0·6688 (4)	−0·0572 (3)	0·1392 (4)	0·048 (1)
C(6)	0·6985 (3)	0·0313 (3)	0·1973 (4)	0·040 (1)
C(7)	0·8760 (4)	0·1621 (2)	0·3681 (4)	0·040 (1)
C(8)	1·0060 (3)	0·1933 (2)	0·4381 (5)	0·049 (1)
C(9)	1·0627 (4)	0·1589 (3)	0·5756 (5)	0·058 (1)
C(10)	0·9908 (4)	0·0942 (3)	0·6387 (4)	0·056 (2)
C(11)	0·8607 (4)	0·0618 (3)	0·5673 (4)	0·046 (1)
C(12)	0·8037 (4)	0·0959 (2)	0·4319 (4)	0·038 (1)
C(13)	0·6649 (3)	0·0670 (2)	0·3362 (4)	0·041 (1)
C(14)	0·7990 (3)	0·1891 (2)	0·2169 (4)	0·042 (1)
C(15)	0·6569 (4)	0·2216 (2)	0·2323 (4)	0·042 (1)
C(16)	0·5864 (3)	0·1593 (3)	0·2936 (4)	0·041 (1)
C(17)	0·4507 (4)	0·1756 (3)	0·3172 (4)	0·049 (1)
C(18)	0·6040 (4)	0·3132 (3)	0·1842 (4)	0·047 (1)
N(1)	0·5638 (4)	0·3860 (3)	0·1450 (4)	0·069 (1)
N(2)	0·3422 (4)	0·1889 (3)	0·3350 (4)	0·077 (1)

Table 2. *Bond lengths* (Å) *and bond angles* (°)

All e.s.d.'s in bond lengths 0·005 Å except where otherwise stated.

Benzene			
C(1)–C(2)	1·386	C(7)–C(8)	1·382
C(2)–C(3)	1·398	C(8)–C(9)	1·401
C(3)–C(4)	1·379	C(9)–C(10)	1·383
C(4)–C(5)	1·391	C(10)–C(11)	1·392
C(5)–C(6)	1·381	C(11)–C(12)	1·383
C(1)–C(6)	1·394	C(7)–C(12)	1·404
Barrelene		Nitrile	
C(1)–C(14)	1·532	C(15)–C(18)	1·439
C(6)–C(13)	1·533	C(16)–C(17)	1·429
C(7)–C(14)	1·528	N(1)–C(18)	1·141 (4)
C(12)–C(13)	1·525	N(2)–C(17)	1·139 (4)
C(14)–C(15)	1·518		
C(13)–C(16)	1·531		
C(15)–C(16)	1·343		
Benzene			
C(1)–C(2)–C(3)	118·2 (4)	C(7)–C(8)–C(9)	118·5 (4)
C(2)–C(3)–C(4)	121·0 (4)	C(8)–C(9)–C(10)	120·6 (4)
C(3)–C(4)–C(5)	120·5 (4)	C(9)–C(10)–C(11)	120·9 (4)
C(4)–C(5)–C(6)	118·8 (4)	C(10)–C(11)–C(12)	118·8 (4)
C(5)–C(6)–C(1)	120·7 (4)	C(11)–C(12)–C(7)	120·4 (4)
C(6)–C(1)–C(2)	120·7 (4)	C(12)–C(7)–C(8)	120·8 (4)
Barrelene		Benzene–barrelene	
C(1)–C(6)–C(13)	113·3 (3)	C(8)–C(7)–C(14)	126·2 (3)
C(6)–C(1)–C(14)	113·0 (3)	C(11)–C(12)–C(13)	126·4 (3)
C(7)–C(12)–C(13)	113·3 (3)	C(2)–C(1)–C(14)	126·2 (4)
C(12)–C(7)–C(14)	112·9 (3)	C(5)–C(6)–C(13)	125·8 (3)
C(1)–C(14)–C(7)	105·5 (3)		
C(6)–C(13)–C(12)	105·7 (3)	Ethene–nitrile	
C(1)–C(14)–C(15)	104·9 (3)	C(14)–C(15)–C(18)	121·5 (3)
C(7)–C(14)–C(15)	104·9 (3)	C(13)–C(16)–C(17)	122·2 (3)
C(12)–C(13)–C(16)	105·3 (3)	C(15)–C(16)–C(17)	124·1 (3)
C(6)–C(13)–C(16)	104·3 (3)	C(16)–C(15)–C(18)	123·4 (3)
C(14)–C(15)–C(16)	115·1 (3)	C(15)–C(18)–N(1)	178·8 (4)
C(15)–C(16)–C(13)	113·6 (3)	C(16)–C(17)–N(2)	179·5 (4)

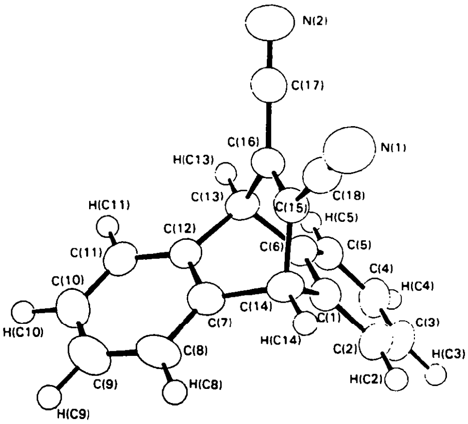

Fig. 1. *ORTEP* plot (Johnson, 1965) of the molecule with 50% probability thermal ellipsoids.

Discussion. Atomic parameters are listed in Table 1.*

The salient features of the X-ray crystallographic structure (Fig. 1) are similar to those of 1-bromotriptycene (Palmer & Templeton, 1968). Table 2 gives the bond lengths and angles. The cyanide–ethene bond distances [C(16)–C(17), C(15)–C(18)] are short, though not unexpectedly so for an sp^2–sp bond. The structure of the central cyclic system is conserved despite the loss of symmetry from the substitution of two cyano groups in the place of a peripheral benzene group as in 1-bromotriptycene.

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* Lists of structure factors, anisotropic thermal parameters and calculated H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42840 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.