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Structure of 1-(*trans*-2-Bromoethenyl)pyrene,* $C_{18}H_{11}Br$

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Abstract. $M_r = 307{\cdot}20$, monoclinic, $P2_1/c$, $a = 20{\cdot}506\ (3)$, $b = 3{\cdot}877\ (1)$, $c = 16{\cdot}236\ (6)$ Å, $\beta = 106{\cdot}79\ (2)°$, $V = 1235{\cdot}6\ (6)$ Å^3, $Z = 4$, $D_x = 1{\cdot}651$ g cm^{-3}, λ(Mo $K\alpha$) $= 0{\cdot}7107$ Å, $\mu = 32{\cdot}7$ cm^{-1}, $F(000) = 616$, $T = 115$ K, final $R = 0{\cdot}0423$ for 2265 observed reflections. The molecules are arranged herringbone fashion in parallel stacks separated by 3·487 (4) Å in the unit cell. The torsion angle between the bromoethenyl group and the pyrene ring is 24·9 (4)°. One ring of the pyrene portion is slightly twisted to allow the other three rings to be planar.

Introduction. 1-(*trans*-2-Bromoethenyl)pyrene is one of a series of compounds being studied as substrates for enzymes and as competitive and suicide inhibitors of cytochrome P-450-dependent benzo[*a*]pyrene hydroxylase activity in liver microsomes (Gan, Acebo & Alworth, 1984). Kinetic studies show that 1-(*trans*-2-bromoethenyl)pyrene binds to both benzoflavone-induced and phenobarbital-induced microsomes and can also function as a substrate. It serves as a competitive inhibitor of benzo[*a*]pyrene metabolism for some isozymes. It is a suicide substrate for both types of microsomes, but only inhibitits some of the isozymes present in phenobarbital-induced microsomes (Alworth, 1984). Olefinic bonds are known to undergo expoxidation due to the catalytic action of cytochrome P-450. Suicide inhibitors of the enzyme cause prosthetic heme alkylation through an intermediate in the oxidation sequence prior to or during formation of the epoxide (Ortiz de Montellano & Correia, 1983).

Experimental. Crystal obtained from Dr W. L. Alworth, Tulane University. Approximate dimensions: 0·45 × 0·65 × 0·40 mm; mounted on a glass fiber. Enraf–Nonius CAD-4 diffractometer, graphite-crystal monochromator, Mo $K\alpha$ radiation, low temperature (115 K). Lattice parameters from 25 reflections with $21 < \theta < 29°$. Systematic absences, $h0l$, $l = 2n + 1$, $0k0$, $k = 2n + 1$. Absorption as a function of ψ observed, and empirical absorption correction applied, relative transmission coefficients ranging from 0·676 to 0·998 with an average value of 0·808; reflections measured with ranges $1 < \theta < 30°$, $-28 \le h \le 28$, $-5 \le k \le 0$, $0 \le l \le 22$, ω:2θ scans. Intensities of three standard reflections decreased by 2·6% and were used to correct data. 3619 total reflections measured, 2988 unique, 2265 considered observed [$F > 3\sigma(F)$]. Corrected for Lorentz and polarization effects. Structure determined by direct methods using *MULTAN*80 [Main *et al.*, 1980; modified by Frenz (1982)]. Peaks corresponding to 19 non-H atoms located in E map and 11 H atoms from subsequent Fourier syntheses. Structure refined by full-matrix least squares; $\sum w(|F_o| - |F_c|)^2$ minimized. H positions and thermal parameters refined isotropically; other atoms refined anisotropically. Final $R = 0{\cdot}042$, $wR = 0{\cdot}069$ for 215 variables, $w = 1/\sigma(F)^2$, where $\sigma(F^2)^2 = \sigma_{cs}^2 +$

* (*E*)-1-(2-Bromovinyl)pyrene.

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$(0{\cdot}04F^2)^2$, $S = 2{\cdot}49$, $(\Delta/\sigma)_{max} = 0{\cdot}06$ [H(7)], maximum peak in final difference Fourier map: 1·1 (2) e Å^{-3}, located 0·90 Å from Br atom; minimum peak: 1·1 (1) e Å^{-3}, located 1·28 Å from Br atom. Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974), all computer programs from *SDP* system (Frenz, 1982).

Discussion. Fig. 1 shows an *ORTEP* plot (Johnson, 1976) of the molecule with atomic numbering. A stereoview of the unit cell is given in Fig. 2. Positional and thermal parameters are given in Table 1.* Interatomic bond distances and bond angles are listed in Table 2. The assignment of a *trans* configuration for the ethenyl group was confirmed by the structure determination.

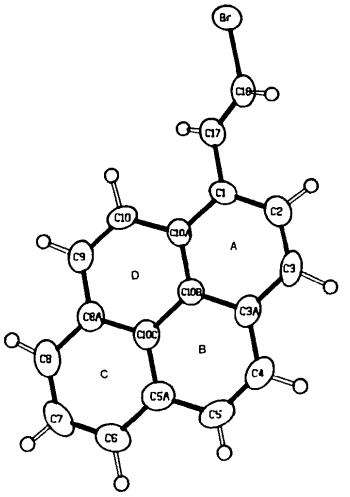

Fig. 1. Plot of the 1-(*trans*-2-bromoethenyl)pyrene molecule showing the atomic-labeling scheme. Thermal ellipsoids are plotted at the 50% probability level. H atoms have been plotted with arbitrary radii.

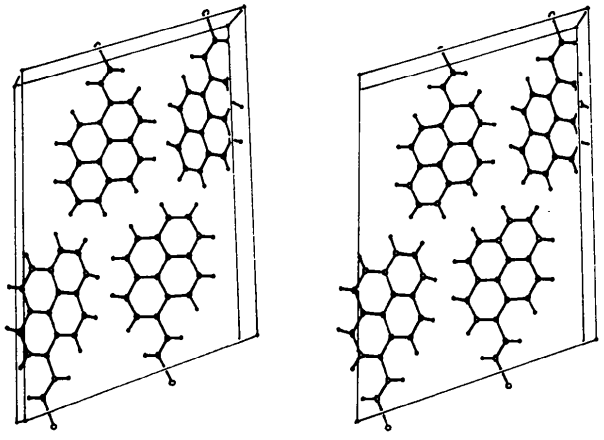

Fig. 2. Stereoview of the molecular packing in the unit cell of the 1-(*trans*-2-bromoethenyl)pyrene molecule.

* Lists of structure factors, anisotropic thermal parameters, coordinates and bond distances and angles involving H and least-squares-planes' data have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42150 (38 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Positional and thermal parameters with their e.s.d.'s*

Anisotropically refined atoms are given in the form of the equivalent isotropic thermal parameter defined as:
$\frac{4}{3}(a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + ab\cos\gamma\beta_{12} + ac\cos\beta\beta_{13} + bc\cos\alpha \times \beta_{23})$.

	x	y	z	B_{eq}(Å^2)
Br	0·95323 (2)	0·1754 (1)	0·36930 (3)	1·385 (9)
C(1)	1·1576 (2)	0·482 (1)	0·4079 (3)	1·05 (8)
C(2)	1·1793 (2)	0·643 (1)	0·4884 (3)	1·27 (9)
C(3a)	1·2955 (2)	0·681 (1)	0·4809 (3)	1·11 (9)
C(3)	1·2461 (3)	0·739 (1)	0·5244 (3)	1·26 (9)
C(4)	1·3650 (2)	0·778 (1)	0·5167 (3)	1·29 (9)
C(5)	1·4111 (2)	0·725 (1)	0·4732 (3)	1·33 (9)
C(5a)	1·3924 (2)	0·568 (1)	0·3897 (3)	1·21 (9)
C(6)	1·4396 (2)	0·512 (1)	0·3434 (3)	1·38 (9)
C(7)	1·4196 (2)	0·360 (1)	0·2636 (3)	1·40 (9)
C(8a)	1·3037 (2)	0·310 (1)	0·2706 (3)	1·04 (8)
C(8)	1·3520 (3)	0·256 (1)	0·2264 (3)	1·4 (1)
C(9)	1·2340 (2)	0·205 (1)	0·2352 (3)	1·22 (9)
C(17)	1·0855 (2)	0·378 (1)	0·3725 (3)	1·3 (1)
C(18)	1·0449 (3)	0·309 (1)	0·4184 (3)	1·52 (9)
C(10)	1·1879 (2)	0·255 (1)	0·2791 (3)	1·09 (9)
C(10a)	1·2052 (2)	0·422 (1)	0·3620 (3)	0·94 (8)
C(10b)	1·2743 (2)	0·522 (1)	0·3984 (3)	0·92 (8)
C(10c)	1·3236 (2)	0·467 (1)	0·3531 (3)	0·99 (8)

Table 2. *Bond distances* (Å) *and angles* (°)

Numbers in parentheses are e.s.d.'s in the least-significant digits.

Br–C(18)	1·891 (3)	C(5a)–C(10c)	1·418 (4)
C(1)–C(2)	1·400 (4)	C(6)–C(7)	1·372 (5)
C(1)–C(17)	1·479 (4)	C(7)–C(8)	1·401 (5)
C(1)–C(10a)	1·408 (4)	C(8a)–C(8)	1·399 (5)
C(2)–C(3)	1·377 (4)	C(8a)–C(9)	1·436 (4)
C(3a)–C(3)	1·410 (5)	C(8a)–C(10c)	1·418 (4)
C(3a)–C(4)	1·425 (4)	C(9)–C(10)	1·353 (5)
C(3a)–C(10b)	1·425 (4)	C(10)–C(10a)	1·443 (4)
C(4)–C(5)	1·350 (5)	C(10a)–C(10b)	1·423 (4)
C(5)–C(5a)	1·434 (5)	C(10b)–C(10c)	1·427 (4)
C(5a)–C(6)	1·404 (4)	C(17)–C(18)	1·295 (5)

C(2)–C(1)–C(17)	119·8 (3)	C(6)–C(7)–C(8)	121·3 (3)
C(2)–C(1)–C(10a)	119·1 (3)	C(7)–C(8)–C(8a)	119·6 (3)
C(17)–C(1)–C(10a)	121·2 (3)	C(9)–C(8a)–C(10c)	118·8 (3)
C(1)–C(2)–C(3)	121·8 (3)	C(8)–C(8a)–C(9)	121·5 (3)
C(3)–C(3a)–C(4)	122·1 (3)	C(8a)–C(9)–C(10)	121·0 (3)
C(3)–C(3a)–C(10b)	118·1 (3)	C(1)–C(10a)–C(10)	123·1 (3)
C(4)–C(3a)–C(10b)	119·7 (3)	C(10)–C(10a)–C(10b)	117·2 (3)
C(2)–C(3)–C(3a)	121·0 (3)	C(1)–C(10a)–C(10b)	119·7 (3)
C(3a)–C(4)–C(5)	121·0 (3)	C(3a)–C(10b)–C(10a)	120·4 (3)
C(4)–C(5)–C(5a)	121·5 (3)	C(3a)–C(10b)–C(10c)	118·7 (3)
C(5)–C(5a)–C(6)	122·3 (3)	C(10a)–C(10b)–C(10c)	121·0 (3)
C(5)–C(5a)–C(10c)	118·4 (3)	C(5a)–C(10c)–C(8a)	119·6 (3)
C(6)–C(5a)–C(10c)	119·2 (3)	C(5a)–C(10c)–C(10b)	120·6 (3)
C(5a)–C(6)–C(7)	120·5 (3)	C(8a)–C(10c)–C(10b)	119·8 (3)
Br–C(18)–C(17)	122·7 (3)	C(8)–C(8a)–C(10c)	119·7 (3)
C(1)–C(17)–C(18)	124·6 (3)		

Generally, the pyrene portion of the molecule shows no large deviations in bond lengths or bond angles compared to previous determinations of the structure of pyrene itself (Camerman & Trotter, 1965; Allmann, 1970; Hazell, Larsen & Lehmann, 1972; Kai, Hama, Yasuoka & Kasai, 1978). The C(1)–C(2) bond length [1·400 (4) Å], however, is slightly greater than in any of the pyrene structures [1·376 (12), 1·373 (2), 1·383 (3), 1·383 (2) Å, respectively].

The rings of the pyrene portion of the molecule are relatively planar with the exception of ring *D* (Fig. 1) which has a χ^2 value of 23, compared with a value of 15 expected on the basis of random errors. This ring is somewhat twisted to accommodate the planarity of the other three rings. The effect of this twisting is to give the entire molecule a slight fold along the line of atoms C(5) to C(10). The χ^2 value of the least-squares plane of the entire pyrene portion is 127, compared to a value of 28 expected on the basis of random errors. The torsion angle between the bromoethenyl group and the least-squares plane of the pyrene is 24·9 (4)°.

Layers of 1-(*trans*-2-bromoethenyl)pyrene are stacked herringbone fashion with 3·487 (4) Å between the layers. A stereoview of the contents of the unit cell is shown in Fig. 2 (Johnson, 1976).

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Structure of the Acetonitrile Solvate of the Antitumor Agent 4-Amino-*N*-(5-chloro-2-quinoxalinyl)benzenesulfonamide, $C_{14}H_{11}ClN_4O_2S.CH_3CN$

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Abstract. $M_r = 375·83$, monoclinic, $P2_1/c$, $a = 7·888$ (3), $b = 20·527$ (8), $c = 11·068$ (4) Å, $\beta = 98·64$ (3)°, $U = 1771$ (1) Å^3, $Z = 4$, $D_m = 1·41$, $D_x = 1·41$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 3·45$ cm^{-1}, $T = 298$ K, $F(000) = 776$, final $R = 0·076$ ($S = 2·32$) for 2107 observed reflections measured on a diffractometer. The structure of the title compound was determined to be the 5-chloro isomer, and thus the condensation reaction between 1,2-diamino-3-chlorobenzene and butyl glyoxalate gives 5-chloro-2-quinoxalinol. Bond distances and angles are normal. There is probably a hydrogen bond between the molecule and the solvate (NH···N 1·98 Å).

Introduction. The 2-hydroxyquinoxaline ring system is conveniently made by the reaction of 1,2-diaminobenzenes and butyl glyoxalate (Gowenlock, Newbold & Spring, 1945). When applied to substituted 1,2-diaminobenzenes, mixtures of isomers are often obtained. However, in the case of 1,2-diamino-3-chlorobenzene only one compound was produced (Wolf, Phister, Beutel, Wilson, Robinson & Stevens, 1949). In an investigation of compounds with seratonin-like activity (Lumma, Hartman, Saari & Engelhardt, 1981) the condensation product between 1,2-diamino-3-chlorobenzene and glyoxylic acid was presumed to be 5-chloro-2-quinoxalinol, but no reason for this conclusion was given. This material was converted to the title compound (I) for antimalarial testing (Wolf *et al.*, 1949) but whether it was the 5- or 8-chloro isomer was not known. Little further interest in compound (I) had been shown until recently, when it was discovered to

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