Enfin, il n'est pas étonnant à cause de la symétrie quasi-binaire constatée, que les spectres de RMN ^{13}C et de RMN ^1H manifestent des signaux correspondant à un environnement magnétique équivalent pour chacun des méthyles en 4b et 9b, et il nous faudra faire appel à une autre explication que la contrainte stérique pour rendre compte de la transposition acido-catalysée des composés (1) en (2).

Références

BARRETT, J. W. & LINSTEAD, R. P. (1935). *J. Chem. Soc.* pp. 436–442.
BARTON, D. H. R. & OVERTON, K. H. (1955). *J. Chem. Soc.* pp. 2639–2652.
CELIKEL, R., GEDDES, A. J. & SHELDRICK, B. (1976). *Cryst. Struct. Commun.* **5**, 43–46.
DHEKNE, V. V. & RAO, A. S. (1980). *Synthesis*, pp. 58–60.
International Tables for X-ray Crystallography (1974). Tome IV, p. 99. Birmingham: Kynoch Press.
JOHNSON, C. K. (1965). *ORTEP*. Rapport ORNL-3794. Oak Ridge National Laboratory, Tennessee.
MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. de York, Angleterre, et Louvain, Belgique.
RAMAH, M. & LAUDE, B. (1975). *Bull. Soc. Chim. Fr.* pp. 2655–2661.
URBAŃCZYK-LIPKOWSKA, Z., KRAJEWSKI, J. W., GLUZIŃSKI, P., KOZERSKI, L. & CZUGLER, M. (1982). *Cryst. Struct. Commun.* **11**, 1531–1536.

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Fluorene, $C_{13}H_{10}$

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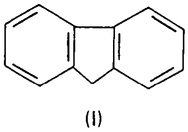
Abstract. $M_r = 166{\cdot}23$, orthorhombic, $Pnma$, $a = 8{\cdot}475\,(2)$, $b = 18{\cdot}917\,(3)$, $c = 5{\cdot}717\,(1)$ Å, $V = 916{\cdot}6\,(3)$ Å^3, $Z = 4$, $D_x = 1{\cdot}20$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu(\text{Mo}) = 0{\cdot}7$ cm^{-1}, $F(000) = 352$, $T = 295$ K, $R = 0{\cdot}056$ for 375 observed reflections $[I > 3\sigma(I)]$. The molecule is planar [maximum deviation 0·013 (6) Å for atom C(5)] and possesses C_{2v} symmetry. Intermolecular contacts correspond to normal van der Waals interactions. The angle C(6)—C(7)—C(6') is 102·7°.

Introduction. During the course of a systematic study of the fluorene-like heterocyclic compounds (with elements of Groups IV–VI) we have found that more or less precise data on the fluorene (I) structure are absent. The only data available are those published by Burns & Iball (1954) and Brown & Bortner (1954), with low accuracy. To obtain more reliable data we have undertaken an independent X-ray analysis of (I).



(I)

Experimental. Colourless transparent crystals 0·22 × 0·20 × 0·16 mm obtained by vacuum sublimation at 318 K. D_m not determined. Syntex $P\bar{1}$ diffractometer. Unit-cell parameters determined by least-squares refinement of 15 reflections. Data set ($\pm h$, $+k$, $+l$) up to $\theta = 23°$, $\theta/2\theta$ scan technique, graphite-monochromated Mo $K\alpha$ radiation. 723 reflections; 375 independent with $I > 3\sigma(I)$ used in the calculations. Three reference reflections (200, 040, 111) measured every 100 steps: intensities dropped about 40% (due to the volatility of the crystal, which became opaque); data corrected for the drift. Lorentz and polarization corrections, no absorption correction. Systematic absences ($hk0$ $h \neq 2n$, $0kl$ $k + l \neq 2n$) indicated space groups $Pnma$ (No. 62) and $Pn2_1a$ (No. 33). Structure solved in the centrosymmetric space group by direct methods and refined (on F) by full-matrix least squares with anisotropic thermal parameters for C atoms; final $R = 0{\cdot}056$, $R_w = 0{\cdot}068$, $w = 1/\sigma^2(F)$. Coordinates of H atoms calculated using geometric considerations; their positional and isotropic thermal parameters were refined. Atomic scattering factors for C from *International Tables for X-ray Crystallography* (1968), for H from Stewart, Davidson & Simpson (1965). No correction for secondary extinction. $(\Delta/\sigma)_{\max} = 0{\cdot}05$. $(\Delta\rho)_{\max} = |0{\cdot}20|$ e Å^{-3}. Calculations carried out with *SHELXTL* (Sheldrick, 1978) on the Nova-3 computer incorporated in the Nicolet R3 system.

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Table 1. *Atomic coordinates* (×10⁴ *for* C, ×10³ *for* H) *and equivalent isotropic thermal parameters* (×10³)

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

	x	y	z	$U_{\rm eq}$ or $U_{\rm iso}$ (Å^2)
C(1)	7499 (7)	3842 (3)	1696 (9)	73 (2)
C(2)	8194 (7)	4312 (3)	143 (12)	84 (3)
C(3)	9058 (7)	4084 (3)	−1765 (11)	82 (3)
C(4)	9255 (6)	3368 (3)	−2234 (11)	69 (2)
C(5)	8547 (5)	2894 (2)	−699 (9)	54 (2)
C(6)	7681 (5)	3121 (3)	1243 (9)	56 (2)
C(7)	7064 (8)	2500	2609 (14)	64 (3)
H(1)	691 (7)	397 (3)	335 (11)	116 (24)†
H(2)	811 (7)	483 (3)	59 (12)	124 (27)†
H(3)	949 (7)	444 (3)	−303 (13)	95 (24)†
H(4)	969 (6)	316 (3)	−391 (10)	99 (21)†
H(71)	748 (10)	250	452 (15)	104 (31)†
H(72)	580 (10)	250	282 (19)	101 (32)†

† $U_{\rm iso}$.

Discussion. The atomic coordinates are listed in Table 1,* interatomic distances and bond angles are presented in Fig. 1 (e.s.d.'s: C—C 0·006–0·009 Å, C—H 0·06–0·09 Å; C—C—C 0·4–0·5°, C—C—H 3–4°). The molecule is planar [maximum deviation from the mean plane is for C(5), 0·013 (6) Å] and possesses C_{2v} symmetry. The intermolecular contacts correspond to normal van der Waals interactions. The projection of the structure onto (001) is shown in Fig. 2. The most important difference between our results and those of Burns & Iball (1954) and Brown & Bortner (1954) involves the value of the angle C(6)—C(7)—C(6′), 102·7° against 105·6 and 106° respectively.

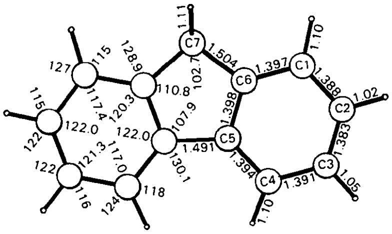

Fig. 1. Interatomic distances (Å) and bond angles (°).

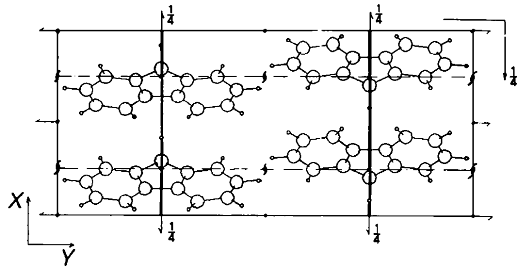

Fig. 2. Projection onto the (001) plane.

References

Brown, G. M. & Bortner, M. H. (1954). *Acta Cryst.* **7**, 139.
Burns, D. M. & Iball, J. (1954). *Nature (London)*, **173**, 635.
International Tables for X-ray Crystallography (1968). Vol. III, pp. 202–203. Birmingham: Kynoch Press.
Sheldrick, G. M. (1978). *SHELXTL. An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data.* Univ. of Göttingen.
Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

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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 39291 (5 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Polarized, Twisted, Ethylene: Structure of 3,3-Bis(methylthio)-2-nitro-2-propene-1-nitrile, $C_5H_6N_2O_2S_2$

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Abstract. $M_r = 190$, monoclinic, $P2_1/n$, $a = 4·123$ (1), $b = 13·096$ (3), $c = 15·126$ (3) Å, $\beta = 93·8$ (2)°, $V = 814·9$ Å^3, $Z = 4$, $D_m = 1·540$ (3), $D_x = 1·548$ (3) g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 53·64$ cm^{-1}, $F(000) = 392·0$, $T = 293$ K. Final $R = 0·054$ for 1119 significant reflections. The C=C bond length is 1·376 (4) Å which indicates only a moderate polarization, with a DNMR rotational barrier of 80·2 kJ mol^{-1}. The molecule is twisted about the C=C double bond by 12·8°. A reasonable correlation between rotational barrier (DNMR studies) and C=C bond lengths (X-ray measurements) has been obtained.

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