

The Structures of 9*H*-Heptadecachloro-9,9'-bifluorenyl and 9*H*,9'*H*-Hexadecachloro-9,9'-bifluorenyl

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Abstract

The crystal and molecular structures of 9*H*-heptadecachloro-9,9'-bifluorenyl (C₂₆HCl₁₇) and 9*H*,9'*H*-hexadecachloro-9,9'-bifluorenyl (C₂₆H₂Cl₁₆) have been determined by single-crystal X-ray techniques. The first compound is tetragonal, space group *P*4₂/*n*, with *a* = *b* = 29.003 (12), *c* = 7.768 (2) Å, *Z* = 8, and the second is monoclinic, space group *P*2₁/*c*, with *a* = 20.042 (9), *b* = 22.397 (5), *c* = 14.683 (8) Å, β = 111.25 (3)°, *Z* = 8. Both structures were solved with *MULTAN* and refined to *R* factors of 0.058 for 2395, and 0.053 for 5951 observed reflections, respectively. The most important differences between molecules of the first compound and molecules *A* and *B* of the second involve the torsion angles and the interatomic distances between the perchlorofluorenyl groups.

Introduction

A systematic structural investigation of overcrowded chlorinated aromatic compounds is in progress in our laboratories in order to obtain the conformations of these compounds. 9*H*-Heptadecachloro-9,9'-bifluorenyl (HPDF) and 9*H*,9'*H*-hexadecachloro-9,9'-bifluorenyl (HXDF) were synthesized by Ballester, Castañer & Camps (1980) in order to study the steric effects of the substituent at position 9 of the fluorenyl group. The two compounds have similar spectra (IR, UV, mass). We have undertaken an X-ray study to confirm the tentative structures that were assigned to these compounds, and to determine the conformations of these molecules.

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Experimental

Crystals of both compounds are colourless, with prismatic habit. Data collection was on a Syntex *P*2₁ four-circle diffractometer. A summary of the main crystallographic data is given in Table 1. Intensities were collected with Mo *K*α radiation, using the ω-scan technique, with a scan interval of −0.8 to 0.8°. Lorentz-polarization corrections were carried out.

Structure determination and refinement

The two structures were solved and refined in a similar way; the *MULTAN* system of computer programs (Main, Woolfson, Lessinger, Germain & Declercq, 1977) was used for structure determination; the *E* maps

Table 1. Crystallographic data

	9 <i>H</i> ,9' <i>H</i> - Hexadecachloro- 9,9'-bifluorenyl C ₂₆ H ₂ Cl ₁₆	9 <i>H</i> - Heptadecachloro- 9,9'-bifluorenyl C ₂₆ HCl ₁₇
<i>M</i> , <i>a</i> <i>b</i> <i>c</i> β <i>V</i> Space group <i>Z</i> <i>D</i> _h λ(Mo <i>K</i> α) μ(Mo <i>K</i> α) Number of measured reflections θ _{max} Observed reflections <i>I</i> > 2.5σ(<i>I</i>) <i>R</i>	881.5 20.042 (9) Å 22.397 (5) 14.683 (8) 111.25 (3)° 6143 (5) Å ³ <i>P</i> 2 ₁ / <i>c</i> 8 1.91 Mg m ⁻³ 0.71069 Å 1.44 mm ⁻¹ 9089 23.5° 5951 0.053	916.0 29.003 (12) Å 29.003 (12) 7.768 (2) 6534 (4) Å ³ <i>P</i> 4 ₂ / <i>n</i> 8 1.86 Mg m ⁻³ 0.71069 Å 1.43 mm ⁻¹ 4837 23.5° 2395 0.058

Table 2. Atomic parameters ($\times 10^5$, for H $\times 10^4$) for HPDF, with e.s.d.'s in parentheses

	x	y	z
C(1)	54350 (2)	17188 (2)	35143 (14)
C(2)	52419 (3)	20623 (3)	25160 (14)
C(3)	47590 (3)	20961 (2)	24508 (13)
C(4)	44670 (3)	17678 (3)	31751 (15)
C(5)	46763 (2)	13898 (2)	40328 (13)
C(6)	45015 (2)	9711 (2)	48624 (14)
C(7)	41094 (3)	6895 (3)	45588 (14)
C(8)	40329 (3)	2873 (3)	54598 (14)
C(9)	43765 (3)	1326 (3)	65547 (15)
C(10)	47951 (2)	3708 (3)	67243 (14)
C(11)	48408 (3)	7972 (2)	59370 (13)
C(12)	52578 (2)	11096 (3)	59149 (13)
C(13)	51536 (3)	14125 (2)	44051 (14)
C(14)	52846 (2)	14367 (2)	75619 (14)
C(15)	57414 (2)	17206 (2)	75336 (14)
C(16)	61838 (3)	15414 (2)	77615 (16)
C(17)	65492 (3)	18529 (3)	77183 (17)
C(18)	64616 (3)	23186 (3)	76355 (16)
C(19)	60181 (3)	24953 (3)	74369 (15)
C(20)	56576 (2)	21788 (2)	72802 (13)
C(21)	51427 (2)	22315 (2)	71198 (13)
C(22)	48721 (3)	26035 (2)	65589 (14)
C(23)	43846 (3)	25812 (3)	67946 (15)
C(24)	41879 (3)	21944 (3)	74948 (14)
C(25)	44575 (3)	17993 (3)	78480 (15)
C(26)	49568 (2)	18161 (3)	75753 (13)
Cl(27)	60289 (1)	16645 (1)	35750 (4)
Cl(28)	55734 (1)	24581 (1)	14650 (4)
Cl(29)	45085 (1)	25678 (1)	14336 (4)
Cl(30)	38845 (1)	18606 (1)	32572 (4)
Cl(31)	37470 (1)	8042 (1)	28053 (4)
Cl(32)	35505 (1)	-277 (1)	51246 (4)
Cl(33)	43002 (1)	3846 (1)	76032 (4)
Cl(34)	52509 (1)	1154 (1)	78119 (3)
Cl(35)	62862 (1)	9613 (1)	79766 (4)
Cl(36)	71036 (1)	16456 (1)	79923 (5)
Cl(37)	69208 (1)	26994 (1)	78452 (5)
Cl(38)	59365 (1)	30741 (1)	76633 (4)
Cl(39)	50758 (1)	30542 (1)	53438 (4)
Cl(40)	40428 (1)	30397 (1)	62470 (5)
Cl(41)	36058 (1)	21803 (1)	79507 (5)
Cl(42)	41956 (1)	13181 (1)	87044 (4)
Cl(43)	52618 (1)	11293 (1)	95816 (3)
H(C12)	5542 (4)	917 (4)	5836 (18)

The final atomic parameters of HPDF and HXDF are listed in Tables 2 and 3 respectively.* Figs. 1, 2 and 3 show views of the molecules and the numbering of the atoms (all drawn by ORTEP, Johnson, 1965).

Description of the structure

Figs. 4, 5 and 6 show the bond lengths, bond angles, and torsion angles less than 90° respectively.

The five-carbon-atom groups have envelope forms and C(12) and C(14) have the typical sp^3 configuration in the three molecules [HPDF, HXDF(A), and

* Lists of structure factors and anisotropic thermal parameters for both compounds have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35413 (52 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

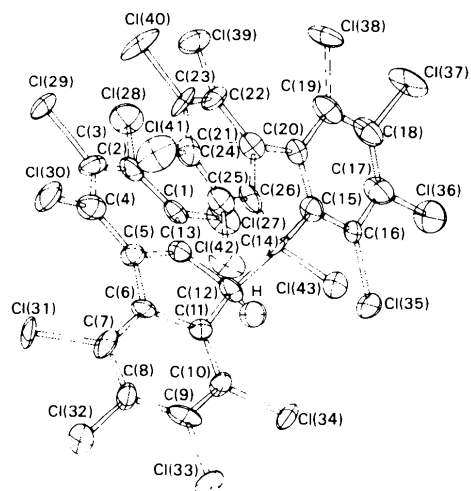


Fig. 1. Perspective (ORTEP) view of the HPDF molecule, and the numbering of the atoms.

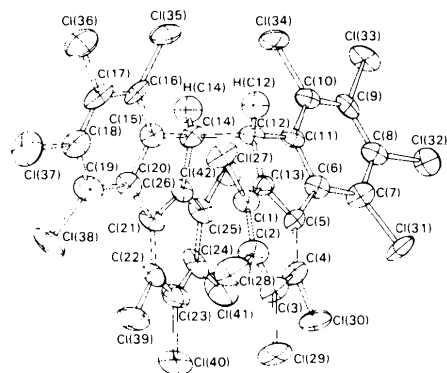


Fig. 2. Perspective (ORTEP) view of molecule A of HXDF, and the numbering of the atoms.

computed with the phases from the set with the highest combined figure of merit revealed all the non-hydrogen atoms.

The SFLS block-diagonal least-squares procedure (Ahmed, Hall, Pippy & Huber, 1966) was used in the refinement. The function minimized was $w\|F_o\| - \|F_c\|^2$, where $w = (p_1 + |F_o| + p_2|F_o|^2)^{-1/2}$. The values of p_1 and p_2 were, respectively, 40.82 and 0.002 in HPDF and 27.80 and 0.002 in HXDF. The H atoms were located from a difference synthesis and refinements were terminated at $R = 0.058$ for HPDF and at 0.053 for HXDF for all observed reflections, R being defined as $\sum \|F_o\| - \|F_c\| / \sum \|F_o\|$.

Table 3. Atomic parameters ($\times 10^5$, for H $\times 10^4$) for HXDF, with e.s.d.'s in parentheses

	Molecule A			Molecule B		
	x	y	z	x	y	z
C(1)	28159 (4)	-12453 (3)	31440 (5)	-20387 (4)	-12553 (4)	37062 (6)
C(2)	27020 (4)	-18401 (4)	33216 (6)	-21779 (4)	-18315 (4)	39667 (6)
C(3)	32558 (4)	-22557 (4)	35893 (6)	-16130 (4)	-22513 (3)	42074 (6)
C(4)	39555 (4)	-20709 (3)	38079 (5)	-9168 (4)	-20819 (3)	43376 (6)
C(5)	40929 (3)	-14540 (3)	37525 (5)	-7818 (4)	-14884 (3)	41766 (5)
C(6)	47566 (4)	-10719 (3)	40187 (5)	-1104 (4)	-11372 (3)	43233 (5)
C(7)	54632 (4)	-11561 (4)	46874 (6)	6105 (4)	-12085 (3)	48768 (6)
C(8)	59663 (4)	-6791 (3)	48230 (6)	11123 (4)	-7908 (4)	48259 (6)
C(9)	57552 (4)	-1378 (3)	43351 (5)	8833 (4)	-2494 (3)	43055 (6)
C(10)	50456 (4)	-587 (3)	37287 (5)	1488 (4)	-1557 (3)	38040 (5)
C(11)	45438 (4)	-5179 (3)	35816 (5)	-3306 (4)	-5881 (3)	38103 (5)
C(12)	37742 (4)	-4907 (3)	29749 (5)	-11285 (4)	-5649 (3)	32960 (5)
C(13)	35141 (4)	-10680 (3)	32864 (5)	-13627 (4)	-10959 (3)	37367 (5)
C(14)	36411 (4)	-5194 (3)	18151 (5)	-13307 (4)	-6581 (3)	21466 (5)
C(15)	28549 (4)	-5133 (3)	12342 (5)	-21329 (4)	-5825 (3)	16036 (5)
C(16)	24345 (4)	-155 (3)	10448 (6)	-25176 (4)	-650 (4)	14127 (6)
C(17)	17115 (4)	-662 (4)	4878 (6)	-32523 (4)	-696 (3)	8608 (6)
C(18)	14295 (4)	-6236 (4)	1324 (6)	-35757 (4)	-6070 (3)	4619 (6)
C(19)	18688 (4)	-11307 (4)	2604 (6)	-31818 (4)	-11362 (3)	6555 (5)
C(20)	25996 (4)	-10804 (3)	8565 (5)	-24628 (4)	-11319 (3)	12730 (5)
C(21)	32246 (4)	-14857 (3)	11516 (5)	-18971 (4)	-16006 (3)	16021 (5)
C(22)	33027 (4)	-21137 (3)	11841 (6)	-18962 (4)	-22368 (3)	16798 (6)
C(23)	40034 (5)	-23438 (4)	14147 (6)	-12632 (4)	-25467 (3)	18819 (6)
C(24)	45978 (4)	-19764 (4)	16482 (6)	-6514 (4)	-22373 (4)	19894 (6)
C(25)	45304 (4)	-13690 (4)	17670 (6)	-6100 (4)	-16042 (3)	20503 (5)
C(26)	38444 (4)	-11411 (3)	15732 (5)	-12381 (4)	-13089 (3)	19132 (5)
Cl(27)	21317 (1)	-7301 (1)	28307 (2)	-27323 (1)	-7366 (1)	33449 (2)
Cl(28)	18358 (1)	-20568 (1)	31427 (2)	-30087 (1)	-20209 (1)	39357 (2)
Cl(29)	30617 (1)	-29909 (1)	36652 (2)	-18180 (1)	-29815 (1)	43593 (2)
Cl(30)	46066 (1)	-26020 (1)	40362 (2)	-2925 (1)	-26368 (1)	45104 (2)
Cl(31)	57318 (1)	-17626 (1)	54186 (2)	9269 (1)	-17863 (1)	56880 (2)
Cl(32)	68148 (1)	-7636 (1)	56463 (2)	20004 (1)	-8740 (1)	54866 (2)
Cl(33)	63657 (1)	4252 (1)	45022 (2)	14935 (1)	2756 (1)	42617 (2)
Cl(34)	47642 (1)	6177 (1)	31328 (2)	-1481 (1)	5101 (1)	31963 (2)
Cl(35)	27898 (1)	6674 (1)	15066 (2)	-20940 (1)	6054 (1)	18767 (2)
Cl(36)	11537 (1)	5485 (1)	2856 (2)	-37380 (1)	5783 (1)	6798 (2)
Cl(37)	5354 (1)	-6870 (1)	-5858 (2)	-44516 (1)	-6095 (1)	-3021 (2)
Cl(38)	15167 (1)	-17543 (1)	-4042 (2)	-35809 (1)	-17584 (1)	-90 (2)
Cl(39)	26294 (1)	-26063 (1)	9616 (2)	-26580 (1)	-26140 (1)	16322 (2)
Cl(40)	41384 (1)	-31041 (1)	13858 (2)	-12668 (1)	-33057 (1)	19114 (2)
Cl(41)	54462 (1)	-22621 (1)	19279 (2)	1418 (1)	-26400 (1)	21985 (2)
Cl(42)	52572 (1)	-8988 (1)	20274 (2)	1739 (1)	-12388 (1)	21934 (2)
H(C14)	3939 (5)	-207 (4)	1652 (7)	-1093 (5)	-336 (4)	1885 (7)
H(C12)	3524 (5)	-141 (4)	3111 (7)	-1423 (5)	-840 (4)	3535 (7)

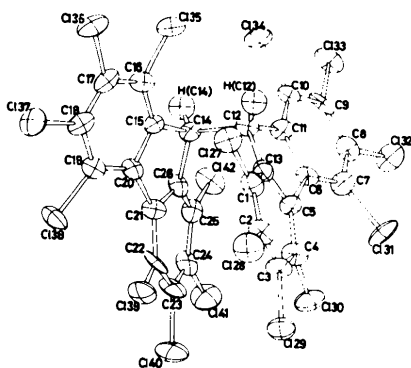


Fig. 3. Perspective (ORTEP) view of molecule B of HXDF, and the numbering of the atoms.

HXDF(B)]. The bond lengths [mean values: C(5)–C(6), 1.485 (2)–1.498 (2); C(12)–C(13), 1.506 (4)–1.515 (4); and C(5)–C(13), 1.389 (4)–1.408 (4) Å] and bond angles [C(13)–C(5)–C(6), 106.9 (4)–109.1 (4); C(12)–C(13)–C(5), 110.2 (4)–111.4 (4); and C(11)–C(12)–C(13), 100.4 (2)–101.1 (2)°] differ slightly from those found in other fluorene derivatives (Burns & Iball, 1955; Griffiths & Hine, 1970*a,b*; Luss & Smith, 1972; Dorset, Hybl & Ammon, 1972; Iball & Scrimgeour, 1977; Solans, Miravittles, Plana, Germain & Declercq, 1980). The main difference is in the C(11)–C(12)–C(13) angle; this is due to steric interaction between the two perchlorofluorenyl groups.

The molecule of HPDF, and molecules *A* and *B* of HXDF consist of the two planes of the perchlorofluorenyl groups, with a *gauche* C(12)–C(14) conformation [C(13)–C(12)–C(14)–C(26), range 36.8–42.0°]. This conformation in stacks produces short intramolecular distances between the atoms of different perchlorofluorenyl groups (Table 4). This,

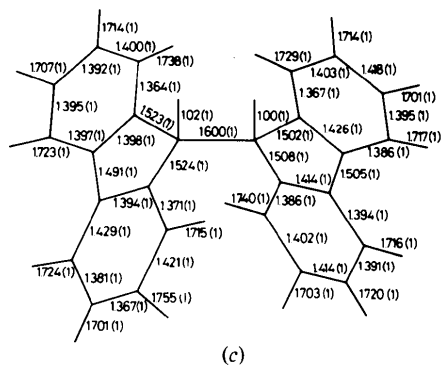
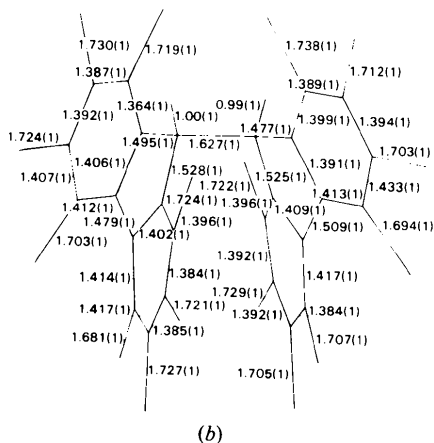
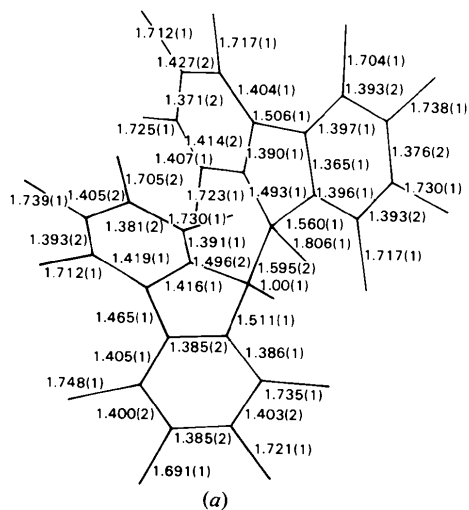


Fig. 4. Bond distances (Å) in (a) HPDF, (b) molecule *A* of HXDF, and (c) molecule *B* of HXDF.

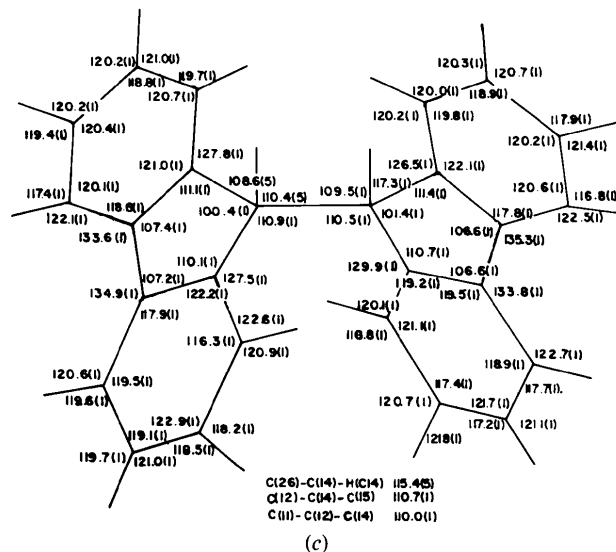
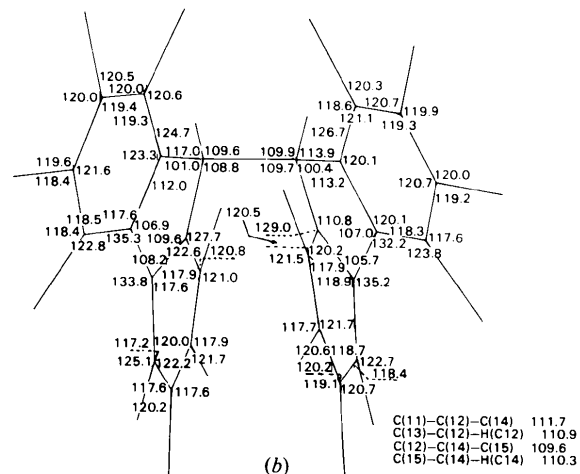
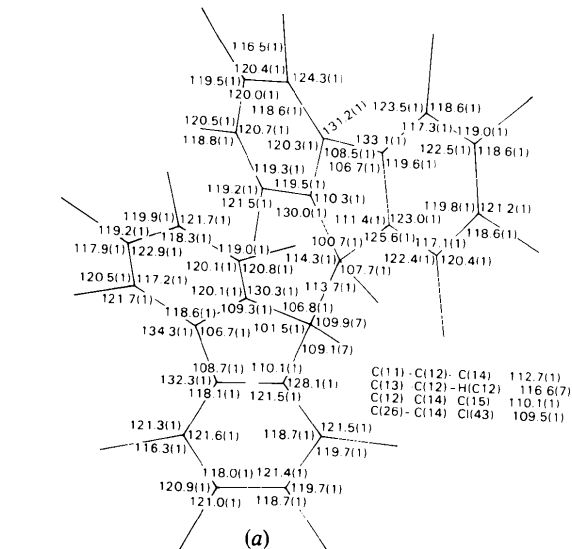


Fig. 5. Bond angles (°) in (a) HPDF, (b) molecule *A* of HXDF, (c) molecule *B* of HXDF.

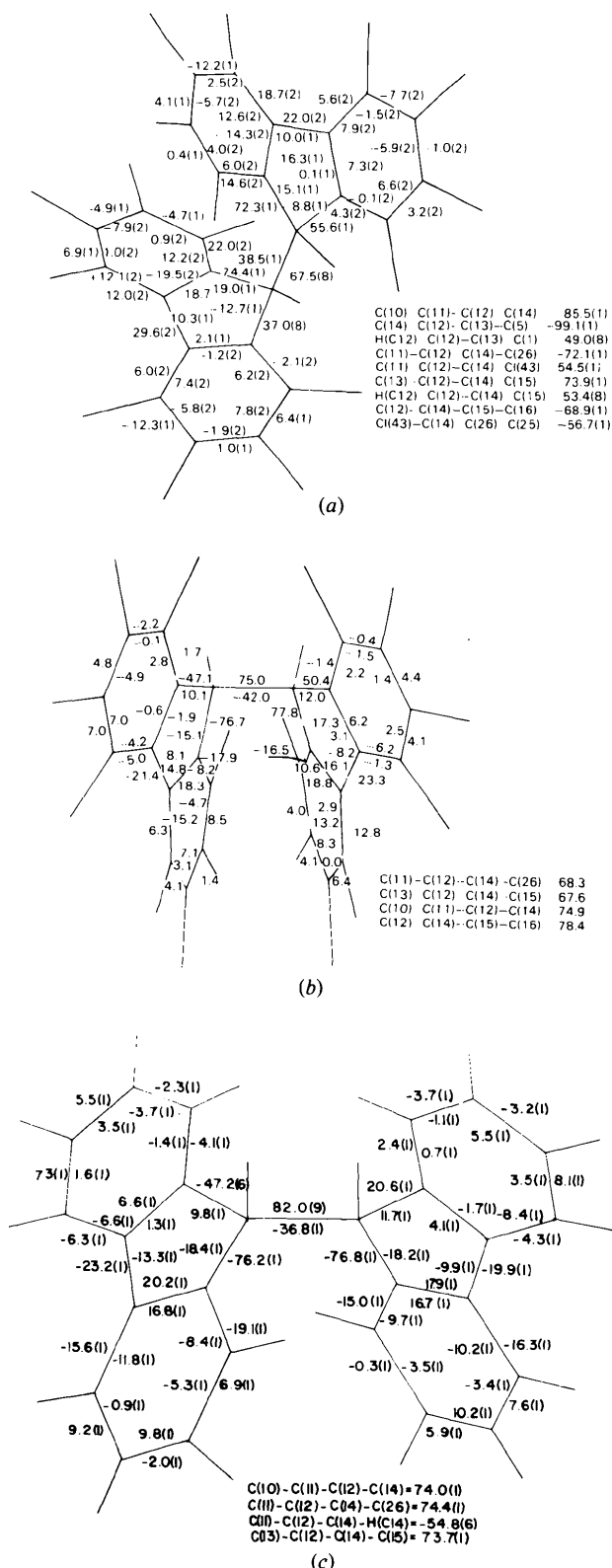


Fig. 6. Torsion angles ($^{\circ}$) less than 90° [e.s.d.'s: Cl(C)—C—C—Cl(C) 0.1° ; H—C—C—C 0.6° ; H—C—C—H 0.9°]. (a) HPDF, (b) molecule A of HXDF, (c) molecule B of HXDF.

together with the steric interaction between Cl(30)···Cl(31) and Cl(38)···Cl(39), causes the loss of planarity of the benzene groups and torsion in the C(5)—C(6) and C(20)—C(21) bonds, as well as the deviations from the typical values for the fluorenyl groups.

Comparison of the molecules

In order to compare the differences between the three molecules, the half-normal probability plot (Abrahams & Keve, 1971) was used. Fig. 7 shows the different plots between the HPDF molecule and the two molecules of HXDF. The analysis was carried out including all interatomic distances less than 4 \AA . The results of the first plots are shown in Table 5, where the number of distances included, the maximum δp value, the slope and intercept obtained by least squares, the agreement factor R between related distances, and the

Table 4. Shortest intramolecular distances (\AA) between the two perchlorofluorenyl groups (e.s.d.'s $\leq 0.002 \text{ \AA}$)

	HXDF(A)	HXDF(B)	HPDF
C(1)—C(14)	3.395	3.377	3.278
C(1)—C(15)	3.274	3.379	3.246
C(1)—C(20)	3.248	3.371	3.279
C(1)—C(21)	3.355	3.295	3.282
C(5)—C(25)	3.340	3.273	3.255
C(5)—C(26)	3.134	3.137	3.110
C(6)—C(25)	3.239	3.288	3.341
C(6)—Cl(42)	3.440	3.384	3.272
C(10)—C(14)	3.339	3.279	3.463
C(11)—C(25)	3.268	3.335	3.448
C(11)—C(26)	3.100	3.162	3.230
C(11)—Cl(42)	3.216	3.244	3.226
C(13)—C(15)	3.079	3.164	3.100
C(13)—C(21)	3.117	3.133	3.177
C(13)—C(26)	2.828	2.818	2.798
C(14)—Cl(35)	3.102	3.173	3.232
C(15)—Cl(27)	3.209	3.213	3.190

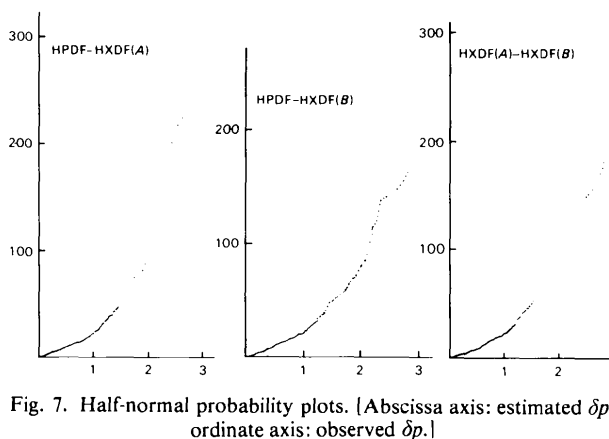


Fig. 7. Half-normal probability plots. [Abscissa axis: estimated δp ; ordinate axis: observed δp .]

Table 5. Results of the half-normal probability plot

Comparison	Number of points	Maximum δp	Slope	Intercept	R	χ^2	ρ
HXDF(A)-HPDF	308	127.0	58.0 (20)	-20.0 (20)	0.025	364.07	0.878
	203	20.0	9.0 (1)	1.0 (1)	0.007	1.33	0.978
	150	13.0	5.8 (1)	0.9 (1)	0.006	0.91	0.965
HXDF(B)-HPDF	304	116.0	48.0 (10)	-14.0 (10)	0.031	188.02	0.904
	195	20.0	9.2 (2)	1.1 (2)	0.007	1.88	0.970
	116	10.0	4.5 (1)	0.7 (1)	0.004	0.44	0.971
HXDF(A)-HXDF(B)	320	131.0	54.0 (10)	-17.0 (10)	0.022	201.84	0.916
	210	20.0	9.7 (2)	0.9 (2)	0.006	2.17	0.969
	158	14.0	6.2 (1)	0.9 (1)	0.005	.69	0.975

factor which indicates the linearity of the plot are indicated. Several trial plots were made with successive exclusion of the distances with largest δp , which were considered responsible for the distortions in the previous plot. An intermediate plot and the final plot are given in the same table. The slopes of the lines suggest that the e.s.d.'s are underestimated.

Discussion

The study of the distances excluded from the 'best' plot shows that the differences between the three molecules are found mainly in the interatomic distances between atoms of second or upper order neighbours belonging to different benzene groups. This indicates that the most important differences between the three molecules are in the torsion angles and the interatomic distances between the perchlorofluorenyl groups.

According to the results of the half-normal probability plots, the molecule of HPDF and molecule *B* of

HXDF have the same C(12)-C(14) torsion angle, while it is different in molecule *A* of HXDF. This, and the presence of Cl(43) in HPDF, produces the differences in the interatomic distances between the two perchlorofluorenyl groups and, consequently, the differences in several torsion angles.

The HPDF and HXDF packing is shown in Fig. 8. The two structures have similar packing because the differences in their molecular configurations are only slight. The two packings can be compared easily using the following cells: $a_1 = a$, $b_1 = b$ and $c_1 = 2c$ for HPDF and $a_2 = a + b$, $b_2 = b - a$, and $c_2 = c$ for HXDF. The parameters of the first cell are 29.003, 29.003, and 15.536 Å with $\alpha = \beta = \gamma = 90^\circ$, and those of the second are 30.055, 30.055, 14.683 Å, $\alpha = 76.01$, $\beta = 103.99$, and $\gamma = 83.65^\circ$. Molecules *A* and *B* of HXDF are related by the 'pseudo n symmetry plane'. Molecules *A* and *B* of HXDF are translated by $0.2c_1$ and $-0.1c_1$ respectively with respect to HPDF.

We thank the Chlorocarbon Group of the Instituto de Química Aplicada de Cataluña (CSIC) for valuable

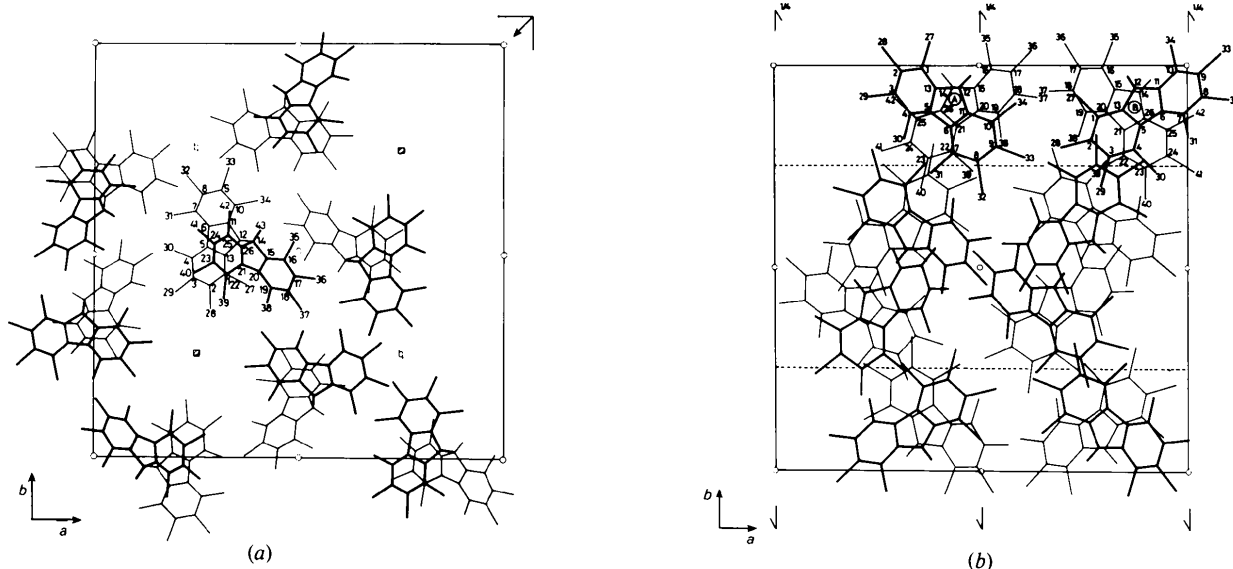


Fig. 8. Projections of the unit-cell contents down the c axis for (a) HPDF and (b) HXDF.

discussions and for providing samples of the compounds. One of us (CM) is grateful for the financial assistance provided by the University of Louvain and the CSIC (Consejo Superior de Investigaciones Científicas).

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Structure de la 5*H*-Dibenzo[*b,f*]azépine (Iminostilbène), Support Tricyclique d'Analogues Structuraux des Antidépresseurs Imipraminiques

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Abstract

The structure of iminostilbene, $C_{14}H_{11}N$, has been determined from 826 diffractometer data by direct methods and refined by least-squares calculations to a final *R* factor of 0.035. The space group is *Pnma* with $a = 8.226$ (3), $b = 20.413$ (6), $c = 6.035$ (2) Å, $D_x = 1.27$ Mg m⁻³, $V = 1013.4$ Å³. The four molecules in the unit cell are in special positions with N atoms on mirror planes. The iminostilbene molecule is not planar: the dihedral angle between the two planes of the benzene rings is 144.4 (1)°.

Introduction

Les travaux suscités par la découverte des propriétés antidépresseives de l'imipramine ont abouti à la préparation de nombreux dérivés qui, à des titres divers ont été retenus par la thérapeutique. Parmi eux, les dérivés de l'iminostilbène, dont le cycle central résulte

de la déshydrogénation de la liaison 10–11 de l'iminodibenzyle, ont contribué à enrichir le groupe des antidépresseurs dibenzazépiniques (Fig. 1).

Dans le cadre de travaux radiocristallographiques sur les amines psychotropes polycycliques et leurs

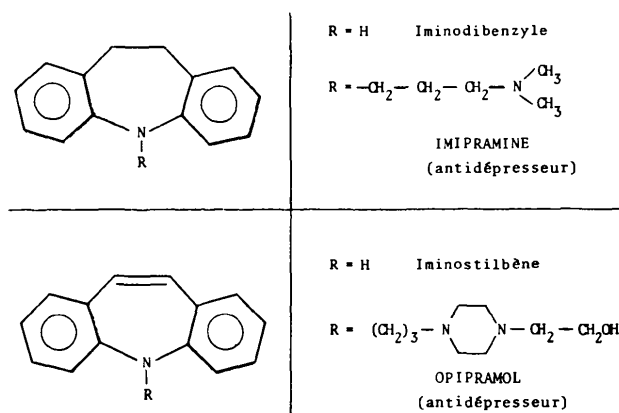


Fig. 1. Molécule étudiée et molécules apparentées.