triterpenes lying in different crystal fields, we supposed that the conformation of the ring exhibited in the crystalline state might correspond to the more stable of the two forms. It was revealed that the chair–chair form exists predominantly in a solution of (1), and the two forms coexist in equilibrium in a solution of (2) (Kamisako, Suwa, Honda, Isoi, Nakai, Shiro & Machida, 1987). This suggests that the difference in the stabilities of the two forms is large in the case of (1) and small in the case of (2).

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Acta Cryst. (1987). C**43**, 1782–1784

Fluorene-9-spiro-1′-cyanocyclopropane*

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(*Received* 16 *February* 1987; *accepted* 21 *April* 1987)

Abstract. $C_{16}H_{11}N$, $M_r = 217\cdot27$, orthorhombic, *Pbcn*, $a = 17\cdot831\,(17)$, $b = 11\cdot246\,(3)$, $c = 11\cdot359\,(4)$ Å, $V = 2278\,(2)$ Å^3, $Z = 8$, $D_x = 1\cdot27$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0\cdot71069$ Å, $\mu = 0\cdot80$ cm^{-1}, $F(000) = 912$, $T = 291$ K, $R = 0\cdot045$ for 859 observed reflections. The cyclopropane bond lengths agree with those predicted following additivity of the bond-length asymmetries of the cyano and biphenyl substituents.

Introduction. The cyclopropane ring undergoes drastic geometrical changes under the influence of electron-withdrawing and -donating substituents. These changes have been discussed in detail by Allen (1980). We have already analysed the ring bond-length pattern and the orientation of the aromatic groups in a series of *trans* and *cis* 1-cyano-1,2-diphenylcyclopropanes (Tinant, Deblock, Declercq, Germain, Van Meerssche, Leroy & Weiler, 1982; Tinant, Wu, Declercq, Van Meerssche, Leroy & Weiler, 1987). Continuing our studies in this field, we report here the structure of the title compound. The unique feature of this compound is the rigidly maintained bisected conformation of the π system, in this case a fluorenyl group.

Experimental. 5 g of 9-diazafluorene, prepared in the usual way from commercially available fluorenone, *via* fluorenone hydrazone, were dissolved in cold benzene and 3 g of acrylonitrile were added. Escape of N_2 was observed during the reaction. After 24 h the solvent was removed. Single crystals in the form of colourless needles were grown by slow evaporation of a petroleum-ether solution of the crude precipitate. Approximate dimensions of the crystal chosen for X-ray measurements $0\cdot35 \times 0\cdot2 \times 0\cdot15$ mm. D_m not measured. Lattice parameters refined using 15 reflections in the range $5 \leq 2\theta \leq 25°$. Syntex $P2_1$, graphite-monochromatized Mo $K\alpha$ radiation. 1682 *hkl* independent reflections with $(\sin\theta)/\lambda \leq 0\cdot561$ Å^{-1}; $0 \leq h \leq 20$, $0 \leq k \leq 12$, $0 \leq l \leq 12$; 859 with $I \geq 2\cdot5\sigma(I)$. Standard reflection (123) checked every 50 reflections: no

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* Spiro[cyclopropane-2,9′-fluorene]-1-carbonitrile.

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significant deviation. Structure solved by *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). H atoms from difference Fourier synthesis. Anisotropic least-squares refinement (*SHELX*76; Sheldrick, 1976) using F; H isotropic with common refined temperature factor. $w = 1/(\sigma^2 + 0{\cdot}00063F^2)$, $R = 0{\cdot}045$, $wR = 0{\cdot}043$ for 859 observed reflections. Final maximum shift-to-e.s.d. ratio = 0·71. $S = 1{\cdot}49$. Maximum and minimum heights in final difference Fourier synthesis = 0·15 and −0·18 e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Table 1. *Atomic coordinates* (× 10^4) *and equivalent isotropic temperature factors* (Å^2)

$$B_{eq} = \tfrac{8}{3}\pi^2 \textstyle\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
C1	3071 (2)	158 (4)	1029 (3)	3·31 (8)
C2	3477 (2)	−30 (5)	−158 (4)	4·07 (8)
C3	3585 (3)	−877 (4)	845 (4)	4·42 (10)
C4	4061 (3)	783 (5)	−503 (4)	4·53 (10)
N5	4515 (2)	1440 (4)	−804 (4)	6·73 (11)
C6	2240 (2)	69 (4)	1044 (3)	3·38 (7)
C7	1772 (3)	−719 (4)	470 (4)	4·24 (9)
C8	1007 (3)	−584 (5)	616 (4)	5·02 (11)
C9	720 (3)	310 (5)	1308 (5)	5·32 (12)
C10	1190 (2)	1093 (5)	1898 (4)	4·47 (9)
C11	1957 (2)	970 (4)	1758 (3)	3·47 (8)
C12	2582 (2)	1648 (4)	2258 (3)	3·51 (8)
C13	2601 (3)	2592 (4)	3039 (4)	4·47 (10)
C14	3284 (3)	3019 (5)	3406 (4)	5·18 (11)
C15	3944 (3)	2533 (5)	3007 (4)	5·17 (11)
C16	3942 (3)	1590 (4)	2210 (4)	4·11 (9)
C17	3252 (2)	1161 (3)	1838 (3)	3·32 (8)

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

C2—C1	1·545 (5)	C3—C1	1·497 (5)
C6—C1	1·485 (5)	C17—C1	1·490 (5)
C3—C2	1·498 (6)	C4—C2	1·440 (6)
N5—C4	1·147 (5)	C7—C6	1·381 (5)
C11—C6	1·393 (5)	C8—C7	1·382 (6)
C9—C8	1·375 (6)	C10—C9	1·388 (6)
C11—C10	1·384 (5)	C12—C11	1·465 (5)
C13—C12	1·384 (5)	C17—C12	1·399 (5)
C14—C13	1·374 (6)	C15—C14	1·375 (6)
C16—C15	1·394 (6)	C17—C16	1·387 (5)

C3—C1—C2	59·0 (3)	C6—C1—C2	117·9 (3)
C6—C1—C3	124·1 (4)	C17—C1—C2	122·7 (3)
C17—C1—C3	122·8 (3)	C17—C1—C6	105·1 (3)
C3—C2—C1	58·9 (2)	C4—C2—C1	119·3 (4)
C4—C2—C3	121·1 (4)	C2—C3—C1	62·1 (3)
N5—C4—C2	178·0 (5)	C7—C6—C1	129·9 (4)
C11—C6—C1	108·6 (3)	C11—C6—C7	121·5 (4)
C8—C7—C6	118·0 (5)	C9—C8—C7	121·1 (5)
C10—C9—C8	121·0 (4)	C11—C10—C9	118·6 (4)
C10—C11—C6	119·9 (4)	C12—C11—C6	109·2 (3)
C12—C11—C10	130·9 (4)	C13—C12—C11	131·8 (4)
C17—C12—C11	108·3 (3)	C17—C12—C13	119·9 (4)
C14—C13—C12	118·9 (4)	C15—C14—C13	121·4 (4)
C16—C15—C14	120·9 (5)	C17—C16—C15	117·7 (4)
C12—C17—C1	108·8 (3)	C16—C17—C1	130·1 (4)
C16—C17—C12	121·2 (4)		

Discussion. The atomic parameters are given in Table 1.* Fig. 1 is a stereoscopic view of the molecule, showing the numbering of the atoms (*PLUTO*; Motherwell & Clegg, 1978). Bond distances and angles are given in Table 2.

The orientations of the aromatic groups towards the cyclopropyl ring are given by the torsion angles $T_1 = \mathrm{C7{-}C6{-}C1{-}}M23 = 7{\cdot}4\ (8)$ and $T_2 = \mathrm{C16{-}C17{-}C1{-}}M23 = 10{\cdot}6\ (8)°$, $M23$ being the midpoint of the C2—C3 bond. These values indicate the expected bisected position for the biphenyl moiety.

From the collection of all geometrical data up to 1980, Allen has shown that π-acceptor substituents shorten the distal ring bond and lengthen the vicinal bonds (Allen, 1980). Moreover, the effect depends on the orientation of the π system towards the cyclopropyl ring; the maximum effect is observed when the π substituent bisects the ring. Ample evidence for this effect has been reported (Ramasubbu, Rajaram & Venkatesan, 1982; Korp, Bernal & Fuchs, 1983; Jason, Gallucci & Ibers, 1981).

Our cyclopropane ring-distances pattern is in complete agreement with the above observations. The ring bond lengths range from 1·497 (5) to 1·545 (5) Å with a mean value of 1·513 (5) Å; they can be thought of as the composite of the following two effects: (1) relative to the mean ring bond value, the cyano group shortens the distal bond C1—C3 by δCN and lengthens each of the vicinal bonds C1—C2 and C2—C3 by $\frac{1}{2}\delta$CN; (2) the biphenyl ring shortens C2—C3 by δb and lengthens C1—C2 and C1—C3 by $\frac{1}{2}\delta b$. According to Allen (1980), the value to expect for δCN is −0·017 Å. The structure of spiro[cyclopropane-1,9′-fluorene] has been reported, but unfortunately relatively large standard deviations on bond lengths were observed (Jason, Gallucci & Ibers, 1981). Nevertheless, these results

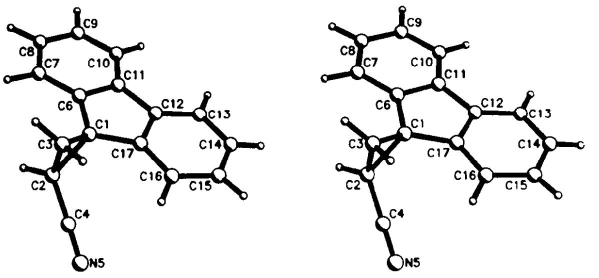

Fig. 1. Stereoscopic view of the molecule and atom numbering.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43992 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

show that the effect of the biphenyl group is approximately the same as that of a phenyl in bisected position *i.e.* δb −0·020 Å. The predicted bond lengths for C1—C2, C1—C3 and C2—C3 in the title compound would thus be respectively 1·532, 1·506 and 1·502 Å. They agree quite well with the observed values of 1·545, 1·497 and 1·498 Å. This demonstrates the validity of the additivity principle for π-acceptor substituents and confirms the simple model for charge transfer from cyclopropane to the π system (Hoffmann & Stohrer, 1971).

BT, JPD and MVM thank the SPPS Belgium for financial support.

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Acta Cryst. (1987). C**43**, 1784–1786

Intermolecular Interactions in the Structure of Methyl 4-Iodobenzoate

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(*Received* 30 *December* 1986; *accepted* 24 *April* 1987)

Abstract. $C_8H_7IO_2$, $M_r = 262{\cdot}05$, orthorhombic, *Pbca*, $a = 14{\cdot}378\ (2)$, $b = 5{\cdot}944\ (1)$, $c = 20{\cdot}361\ (6)$ Å, $V = 1740{\cdot}2\ (6)$ Å^3, $Z = 8$, $D_x = 2{\cdot}000$ g cm^{-3}, Mo $K\alpha$ (graphite monochromator), $\lambda = 0{\cdot}71073$ Å, $\mu = 35{\cdot}9$ cm^{-1}, $F(000) = 992$, $T = 295\ (1)$ K, $R = 0{\cdot}037$ for 1338 unique reflections with $(\sin\theta)/\lambda \le 0{\cdot}65$ Å^{-1}. Bond lengths and angles are normal. The structure contains chains of molecules linked by short —I···O=C— [3·203 (4) Å] interactions.

Introduction. A structure determination undertaken to confirm the stereochemistry of a supposed intermediate in a long synthetic scheme revealed instead that decomposition had taken place to yield methyl 4-iodobenzoate. We were surprised to discover that the structure of this simple material had never been reported even though several groups have been interested in short intermolecular I···I and I···O interactions.

Experimental. A large, colorless prism grown from an ethanol solution was cut to give a chunk with approximate dimensions 0·42 × 0·38 × 0·48 mm. Data collected on an Enraf–Nonius CAD-4F diffractometer in the ω–2θ mode; 22 reflections with $10{\cdot}7 \le \theta \le 19{\cdot}2°$ used to determine lattice parameters; $2 \le \theta \le 27{\cdot}5°$; octant of data with $0 \le h \le 18$, $0 \le k \le 7$, $0 \le l \le 26$; average intensity loss for three control reflections of 21·5% overall (corrected) appeared to result from sublimation; no absorption correction ($\mu = 35{\cdot}9$ cm^{-1}) since size and shape of crystal changed substantially during data collection; 1989 unique reflections measured; 1338 data with $I > 3\sigma(I)$ used for refinement; coordinates of I atom determined from solution of Patterson function, remaining non-H atoms located using *DIRDIF* (Beurskens, *et al.*, 1984); H atoms located in difference Fourier maps, idealized at a late point in the refinement [r(C—H) = 1·00 Å, B 1·0 Å^2 larger than B_{eq} of attached C atom]; extinction correction [final value $2{\cdot}29\ (23) \times 10^{-7}$] (Zachariasen, 1967) included; 101 variables; refinement on F magnitudes; R, $wR = 0{\cdot}037$, 0·049; error in an observation of unit weight = 2·25 e$^-$; $w = 4I/[\sigma^2(I)]$ where $[\sigma^2(I)]$ includes a term $(0{\cdot}03I)^2$; maximum Δ/σ in last cycle of 0·01; largest features in final difference Fourier synthesis have heights +1·0 and −1·5 e Å^{-3} (top nine

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