phenylethane is only marginally affected by packing requirements. Convenient parameters for the description of the molecular conformation are the torsion angles (φ_c) across the ethane C(1)—C(2) bond, and the angles of rotation (φ_r) of each phenyl ring around its corresponding C(Ph)—C(Et) bond. These angles, defined as in the case of 1,1,1-triphenylethane (Destro *et al.*, 1980), are here given a sign equal to that of the torsion angle across the C(Ph)—C(Et) bond, which involves the ring atoms labelled in Fig. 2. Owing to the similarity of the φ_c's found here to those of 1,1,1-tri- and pentaphenylethane, it can be stated that the staggered conformation of the ethane skeleton is substantially maintained in all three of these compounds. Furthermore, the φ_r values for rings R_1 to R_4 reported in Fig. 2 compare satisfactorily with the corresponding values of pentaphenylethane, −0·4, 53·1, 75·1 and −82·8°, respectively. This proves the minimal nature, for both crystal structures, of intermolecular effects on the molecular conformation.

We thank Professor G. Olah for supplying a sample of the substance.

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9-*tert*-Butyl-9-(9-fluorenyl)fluorene

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Abstract. $C_{30}H_{26}$, $M_r = 386·5$, triclinic, $P\bar{1}$, $a = 11·259\,(1)$, $b = 11·187\,(1)$, $c = 9·624\,(1)$ Å, $\alpha = 71·02\,(1)$, $\beta = 71·03\,(1)$, $\gamma = 78·55\,(1)°$, $Z = 2$, $D_m = 1·19$ (flotation in dilute K_2HgI_4 solution), $D_x = 1·191$ Mg m^{-3}. Data were collected on a computer-controlled four-circle diffractometer (Cu $K\alpha$ radiation, $\lambda = 1·5418$ Å, graphite monochromator), $F(000) = 412$, μ(Cu $K\alpha$) = 0·51 mm^{-1}, $T = 291 \pm 2$ K. Final $R = 0·042$ for 3957 observed $[I > 2\sigma(I)_{\rm c.s.}]$ reflexions, 0·046 including unobserved. The overcrowding in the molecule is reflected in the length of the bond connecting the two fluorenyl moieties, 1·585 (2) Å, in the severe spreading of the tetrahedral angles at C(9) and C(9′), 100·7 (1)–119·5 (1)°, and in the short intramolecular contacts. Both fluorenyl groups are nonplanar, the dihedral angles between the pertinent benzenoid rings amounting to 172·1 (2) and 168·1 (2)°.

Introduction. The crystal structure analysis of 9-*tert*-butyl-9-(9-fluorenyl)fluorene (TBFF) was undertaken as part of a continuing programme relating molecular deformations to overcrowding within a series of polyarylethanes. Previous investigations led to the complete characterization of 1,1,1-triphenylethane (Destro, Pilati & Simonetta, 1980*a*), 1,1,1,2-tetraphenylethane (Destro, Pilati & Simonetta, 1980*b*), and pentaphenylethane (Destro, Pilati & Simonetta, 1978).

Colourless crystals of TBFF were obtained by slow evaporation of a saturated solution in ethyl methyl ketone. Cell parameters were derived from a least-

Table 1. *Details of the data collection and refinement*

Crystal dimensions	0·45 × 0·30 × 0·17 mm
Radiation	Graphite-monochromatized Cu $K\alpha$ ($\lambda = 1·5418$ Å)
Scan rate	Variable in the range 2–12° min^{-1}
Scan range	0·8° below $K\alpha_1$ to 0·8° above $K\alpha_2$
Background counts	One-half of the scan time at both ends of the scan range
2θ range	4–148°
Independent reflexions scanned	4353
Reflexions retained $[I > 2\sigma(I)_{\rm c.s.}]$	3957
Final number of variables	376
R (on F, 4353 data)	0·046
R (on F, 3957 data)	0·042
R_w	0·048
Goodness-of-fit	2·49

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squares refinement of the setting angles of 60 reflexions that had been centered on a Syntex $P\bar{1}$ diffractometer. The same instrument was also used for the data collection, whose details are summarized in Table 1.

The structure was solved by direct methods using the program *MULTAN* (Germain, Main & Woolfson, 1971) and refined by least-squares techniques. The nine H atoms of the *tert*-butyl group were located in difference maps, while the remaining 17 H atoms were initially assumed at calculated positions. The quantity minimized during the refinement was $\sum w(|F_o| - k|F_c|)^2$, with weights $w = 4F_o^2/\sigma^2(F_o^2)$ for the 3957 reflexions classified as observed, $w = 0{\cdot}0$ for the 396 unobserved reflexions. The variances $\sigma^2(F_o^2)$ were derived from counting statistics plus a term $(0{\cdot}03S)^2$, where S is the scan count. Form factors for C were from Cromer & Waber (1965), and for H from Stewart, Davidson & Simpson (1965).

Table 2. *Atomic coordinates* ($\times 10^4$; *for* H $\times 10^3$)

	x	*y*	*z*
C(1)	3185 (1)	−1657 (1)	−1508 (1)
C(2)	4287 (2)	−1097 (2)	−2425 (2)
C(3)	5202 (2)	−1012 (2)	−1801 (2)
C(4)	5038 (1)	−1468 (2)	−244 (2)
C(5)	4040 (1)	−2515 (1)	3470 (2)
C(6)	3369 (2)	−2942 (2)	4999 (2)
C(7)	2176 (2)	−3309 (1)	5398 (2)
C(8)	1625 (1)	−3278 (1)	4284 (1)
C(9)	1918 (1)	−2784 (1)	1320 (1)
C(10)	3022 (1)	−2140 (1)	60 (1)
C(11)	3943 (1)	−2022 (1)	682 (1)
C(12)	3489 (1)	−2482 (1)	2351 (1)
C(13)	2291 (1)	−2882 (1)	2752 (1)
C(1′)	−1353 (1)	−3115 (1)	2479 (2)
C(2′)	−2512 (1)	−3247 (1)	3595 (2)
C(3′)	−2925 (1)	−2496 (1)	4587 (2)
C(4′)	−2212 (1)	−1571 (1)	4446 (2)
C(5′)	−348 (1)	601 (1)	3365 (2)
C(6′)	529 (1)	1471 (1)	2627 (2)
C(7′)	1486 (1)	1327 (1)	1346 (2)
C(8′)	1613 (1)	294 (1)	783 (2)
C(9′)	675 (1)	−1843 (1)	1219 (1)
C(10′)	−592 (1)	−2228 (1)	2361 (1)
C(11′)	−1065 (1)	−1427 (1)	3316 (1)
C(12′)	−223 (1)	−434 (1)	2814 (1)
C(13′)	760 (1)	−611 (1)	1541 (1)
C(14)	1943 (1)	−4171 (1)	1170 (1)
C(15)	1187 (2)	−5067 (1)	2643 (2)
C(16)	1461 (2)	−4096 (2)	−178 (2)
C(17)	3310 (1)	−4813 (1)	861 (2)
H(1)	250 (2)	−167 (1)	−196 (2)
H(2)	436 (2)	−77 (2)	−352 (2)
H(3)	597 (2)	−58 (2)	−248 (2)
H(4)	565 (2)	−136 (2)	25 (2)
H(5)	488 (2)	−221 (2)	311 (2)
H(6)	377 (2)	−299 (2)	582 (2)
H(7)	168 (2)	−362 (2)	656 (2)
H(8)	76 (2)	−352 (1)	461 (2)
H(1′)	−109 (1)	−365 (1)	178 (2)
H(2′)	−305 (2)	−391 (2)	369 (2)
H(3′)	−374 (2)	−264 (2)	538 (2)
H(4′)	−253 (2)	−100 (2)	515 (2)
H(5′)	−109 (2)	71 (1)	428 (2)
H(6′)	48 (2)	221 (2)	304 (2)
H(7′)	209 (2)	197 (2)	83 (2)
H(8′)	228 (1)	21 (1)	−16 (1)
H(9′)	62 (1)	−166 (1)	15 (1)
H(151)	31 (2)	−468 (2)	308 (2)
H(152)	163 (2)	−533 (2)	348 (2)
H(153)	112 (2)	−588 (2)	242 (2)
H(161)	54 (2)	−367 (2)	−11 (2)
H(162)	202 (2)	−364 (2)	−118 (2)
H(163)	146 (2)	−498 (2)	−25 (2)
H(171)	371 (2)	−483 (2)	172 (2)
H(172)	386 (2)	−437 (2)	−17 (2)
H(173)	328 (2)	−571 (2)	87 (2)

In the final least-squares cycles, 376 parameters were adjusted: coordinates and anisotropic temperature factors for the 30 C atoms, coordinates and isotropic B's for the 26 H atoms, a scale factor, and a secondary-extinction parameter g (Larson, 1967). These parameters were divided into two matrices and refinement was considered complete when parameter shifts were less than $0{\cdot}3\sigma$. The secondary-extinction coefficient assumed a final value of 45 (3) $\times 10^{-6}$. The final difference map was essentially featureless with the highest peak equal to 0·17 e Å^{-3}. Coordinates obtained from the refinement are given in Table 2.* The estimated standard deviations in bond distances and angles involving C atoms are in the range 0·0016–0·0033 Å and 0·10–0·21°, respectively.

Discussion. A perspective view of the molecule is given in Fig. 1, which also contains bond distances and angles involving C atoms. Fig. 2, a projection down the C(9)–C(9′) bond, clearly shows that the conformation of TBFF in the crystal is *gauche*; pertinent torsion angles are listed in Table 3. The same conformation was found in 9,9′-bifluorenyl, and has been predicted for other polyarylethanes containing two fluorenyl moieties by Dougherty, Llort, Mislow & Blount (1978). Bond distances and angles of TBFF compare well with the corresponding ones of 9,9′-bifluorenyl, except for those involving the two atoms C(9) and C(9′). Rather surprisingly, and contrary to expectation (Hounshell, Dougherty, Hummel & Mislow, 1977), in the latter molecule the length of the central C(9)–C(9′) bond, 1·542 (3) Å, is not significantly larger than that quoted as standard for a paraffinic single bond, 1·541 (3) Å (*International Tables for X-ray Crystallography*, 1962). By contrast, a value as large as 1·585 (2) Å is found for this distance in TBFF, where a significant lengthening of the C(sp^3)–C(sp^2) bonds is also observed [mean 1·532 (2) *vs* 1·515 (3) Å in 9,9′-bifluorenyl]. Furthermore, the spreading of the C—C—C tetrahedral bond angles, already severe in 9,9′-bifluorenyl (102·1 to 116·5°), is even greater in TBFF (100·7 to 119·5°).

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* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35389 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

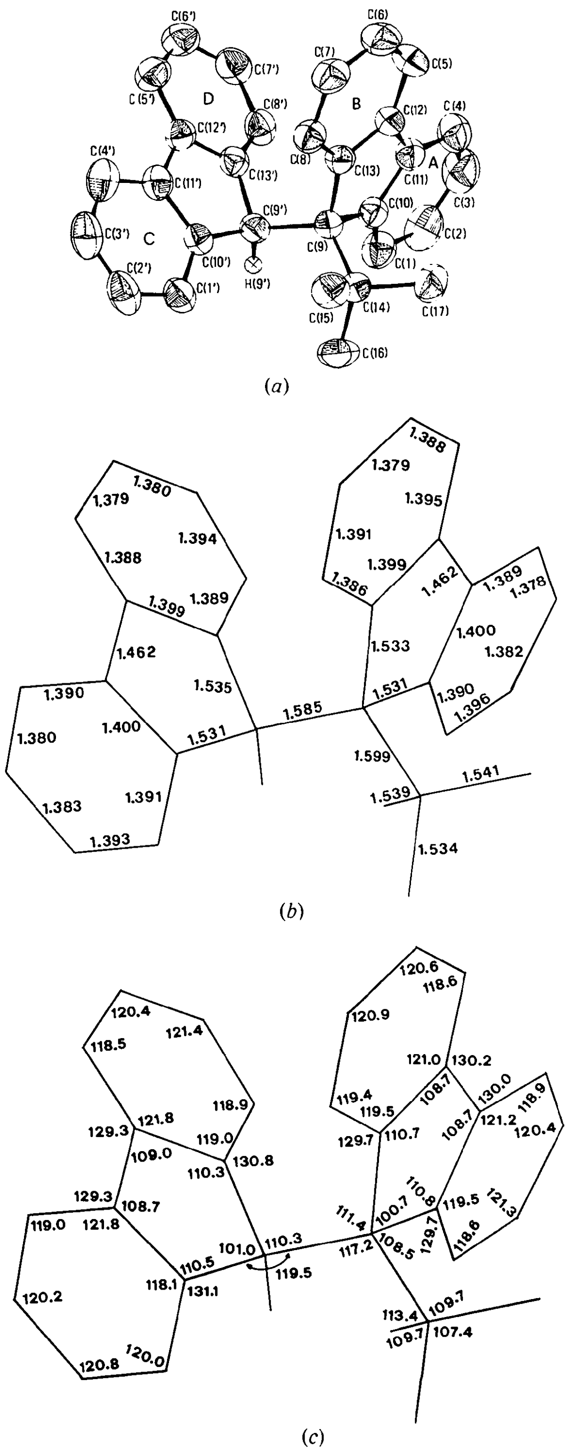

Fig. 1. (*a*) A view of a molecule of TBFF with the numbering scheme. All H atoms, except H(9′), have been omitted for the sake of clarity; they are numbered (see Table 2) according to the C atom to which they are bonded. Thermal ellipsoids are drawn at the 50% probability level. (*b*) Bond distances (Å) involving C atoms. E.s.d.'s are in the range 0·0016–0·0033 Å. (*c*) C–C–C bond angles (°). E.s.d.'s are in the range 0·1–0·2°. The four missing values are: C(13)–C(9)–C(14), 110·2; C(10)–C(9)–C(9′), 107·5; C(9)–C(14)–C(16), 110·9; and C(15)–C(14)–C(17), 105·6°.

Evidently, these deformations help to relieve the strong interactions between the fluorenyl moieties and the *tert*-butyl group, which is connected to the ethane framework by a rather long bond, 1·599 (3) Å. This value, however, is not unexpected: similar lengths were found for the bonds to the *tert*-butyl groups in other overcrowded molecules, *e.g.* 2,2-di-*tert*-butyl-3,3-diphenylthiirane (1·587 and 1·598 Å; Mugnoli & Simonetta, 1976), and tetra-*tert*-butylacetone (1·59–1·61 Å; Lepicard, Berthou, Delettré, Laurent & Mornon, 1973).

Further evidence for the steric hindrance in the present compound is given by the short intramolecular contacts listed in Table 4. Some of these may be responsible for the significant deviation from planarity of the ring labelled *C* in Fig. 1, where the largest displacement from the mean plane, 0·025 Å (~10σ), is observed for atom C(10′). The maximum deviations from the least-squares planes of the other three

Table 3. *Torsion angles* (°) *of interest, with e.s.d.'s in parentheses*

C(9)–C(9′)–C(10′)–C(1′)	−72·9 (3)
C(9)–C(9′)–C(13′)–C(8′)	60·7 (2)
C(13)–C(9)–C(9′)–C(10′)	−69·8 (2)
C(10)–C(9)–C(9′)–C(10′)	−179·3 (2)
C(13)–C(9)–C(9′)–C(13′)	46·4 (2)
C(10)–C(9) C(9′) C(13′)	−63·1 (2)
C(14)–C(9)–C(9′)–H(9′)	−66 (1)
C(10)–C(9)–C(9′)–H(9′)	56 (1)

Table 4. *Some short intramolecular contacts* (Å)

E.s.d.'s are about 0·003, 0·025 and 0·035 Å for C···C, C···H and H···H, respectively.

C(1)···H(8′)	2·67	C(13)···H(171)	2·69
C(1)···H(162)	2·68	C(1′)···C(15)	3·250
C(8)···C(15)	3·113	C(1′)···H(151)	2·38
C(8)···H(152)	2·65	C(9′)···H(161)	2·79
C(10)···C(8′)	3·035	C(10′)···H(151)	2·68
C(10)···H(8′)	2·56	C(16)···H(9′)	2·79
C(10)···H(172)	2·54	H(1)···H(162)	2·20
C(11)···C(17)	3·277	H(1′)···H(161)	2·13
C(13)···H(152)	2·79		

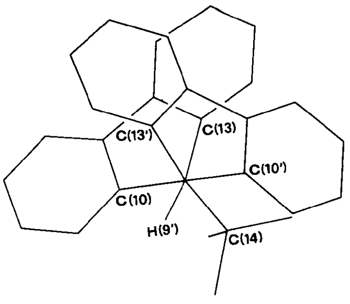

Fig. 2. A projection of TBFF down the C(9)–C(9′) bond.

benzenoid fragments amount to 0·010 (2), 0·011 (3) and 0·014 (2) Å for rings *A*, *B* and *D*, respectively.

Both fluorenyl systems are significantly nonplanar: the dihedral angle between the least-squares planes through rings *A* and *B* is 172·2 (2)°, while that between the planes of rings *C* and *D* amounts to 168·1 (2)°. We may confidently ascribe this conformational feature to intramolecular effects, since there are only two intermolecular contacts shorter by 0·1 Å or more than the sum of van der Waals radii (C 1·7, H 1·2 Å); they are: C(5′)···C(5′) (at $-x$, $-y$, $1-z$), 3·281 (3), and H(6′)···H(153) (at x, $1+y$, z), 2·23 (2) Å.

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Structure of 7-Methoxy-6-(2-methyl-3-indolyl)-2*H*-1,4-benzoxazin-3(4*H*)-one

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Abstract. $C_{18}H_{16}N_2O_3$, monoclinic, space group $P2_1/a$, $a = 20·182$ (10), $b = 7·323$ (4), $c = 10·656$ (5) Å, $\beta = 90·74$ (1)°, $Z = 4$. The final R value is 0·08 including H atoms. The compound is obtained by an unusual reaction of 4-acetoxy-7-methoxy-2*H*-1,4-benzoxazin-3(4*H*)-one with 2-methylindole and the present study has established its structure.

Introduction. Derivatives of 4-hydroxybenzoxazine found in graminaceous plants (Hofman & Hofmanoa', 1969) as biologically active substances (Hashimoto, Shudo, Okamoto, Nagao, Takahashi & Sugimura, 1978) have some quite novel reactivities and several unexpected reaction products are obtained (Hashimoto, Ohta, Shudo & Okamoto, 1979). The present paper describes the X-ray diffraction study of a product obtained by an unusual reaction of 4-acetoxy-7-methoxybenzoxazine with 2-methylindole [Fig. 1(III)].

The lattice constants and intensity data were obtained on a Philips PW 1100 diffractometer using graphite-monochromated Cu $K\alpha$ radiation by the θ–2θ scan method. The scans were repeated twice when the total counts during the first scan were less than 2000. The background was measured at each end of the scan range for half the total scan time. A total of 2389 reflexions were measured within the 2θ angle of 130°. Intensities were corrected for Lorentz and polarization

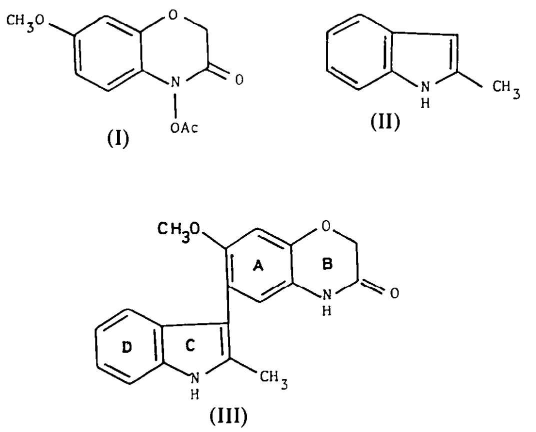

Fig. 1. The chemical structures of 4-acetoxy-7-methoxy-2*H*-1,4-benzoxazin-3(4*H*)-one (I), 2-methylindole (II) and 7-methoxy-6-(2-methyl-3-indolyl)-2*H*-1,4-benzoxazin-3(4*H*)-one (III).