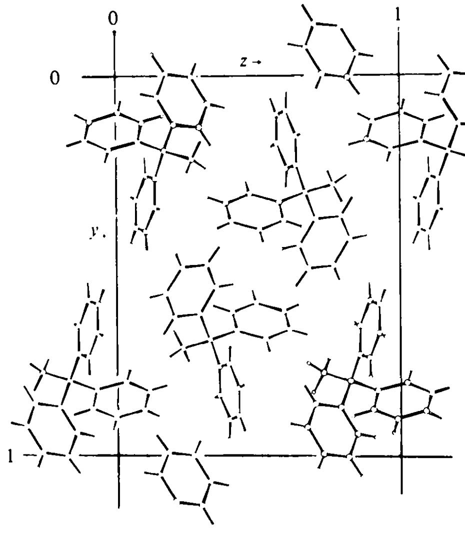

Fig. 2. A projection of the structure down the *a* axis.

this effect is less evident. (ii) The diameters of the phenyl rings passing through C(Ph) [*e.g.* C(6)···C(3)] are not collinear with the C(1)—C(Ph) bonds, owing to a difference (in the range 1·6—3·5°) between the exterior angles at each C(Ph). In addition, atom C(1) lies significantly out of the least-squares planes of the phenyl rings, at distances 0·035 (1), 0·071 (1) and 0·055 (1) Å, respectively. These displacements correspond to a slight bending of all three rings away from the methyl group, individual atoms of each ring remaining substantially coplanar [maximum deviation 0·004 (2) Å for C(7)].

The mean of the three C(2)—C(1)—C(Ph) angles, 109·0 (1)°, is very close to the mean of the other three angles at C(1), 109·9 (1)°. In contrast, much larger differences between these two average values (*e.g.* 106 *vs* 112°, respectively) occur in crystals of triphenylmethane, chlorotriphenylmethane, bromotriphenylmethane, and chlorotris(*p*-methoxyphenyl)methane (Dunand & Gerdil, 1976). While in this series the values of the angles at C(1) seem to be dictated exclusively by steric interference between the phenyl groups, in the present case the bulkiness and the chemical nature of the substituent at C(1) (*i.e.* the methyl group) require a different geometry.

A drawing of the molecular packing as viewed down the *a* axis is shown in Fig. 2. The absence of strong intermolecular contacts proves that the conformation of the molecule mainly results from intramolecular effects. The shortest distances – only slightly less than the sum of van der Waals radii – include C(5)···H(19)$_{1+x,y,z}$, 2·90 (2); C(19)···H(10)$_{-1+x,y,z}$, 2·89 (2); and C(12)···H(6)$_{\frac{3}{2}-x,-\frac{1}{2}+y,\frac{3}{2}-z}$, 2·84 (2) Å.

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1,1,1,2-Tetraphenylethane

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Abstract. $C_{26}H_{22}$, $M_r = 334·5$, monoclinic, $P2_1/c$, $a = 9·814\,(1)$, $b = 10·774\,(1)$, $c = 18·032\,(1)$ Å, $\beta = 97·69(1)°$, $Z = 4$, $D_m = 1.17$ (flotation in dilute K_2HgI_4 solution), $D_x = 1·176$ Mg m^{-3}. Computer-controlled four-circle diffractometer data (Cu $K\alpha$ radiation, $\lambda = 1·5418$ Å, graphite monochromator), $F(000) = 712$, $\mu(\mathrm{Cu}\,K\alpha) = 0·51$ mm^{-1}, $T = 291 \pm 2$ K. The structure has been solved by direct methods and refined to an R of 0·037 for 3071 reflexions with $I > 2\sigma(I)_{\mathrm{c.s.}}$. Strong interactions between the phenyl rings result in marked deviations of some bond distances and angles from their standard values. Intramolecular forces, rather than packing effects, control the conformation of the molecule in the crystal structure.

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Introduction. In line with our continuing interest in the geometries and conformations of highly strained molecules, we have undertaken the study of the crystal structure of the title compound, the middle term of the series that includes 1,1,1-triphenylethane (Destro, Pilati & Simonetta, 1980) and pentaphenylethane (Destro, Pilati & Simonetta, 1978). Colourless bladed crystals were obtained by slow evaporation of a saturated solution of 1,1,1,2-tetraphenylethane in THF. The unit-cell dimensions (see *Abstract*) were obtained by the least-squares fitting of the $\sin^2\theta$ values of 60 reflexions. Systematic absences $h0l$ with l odd and $0k0$ with k odd indicate the space group $P2_1/c$. Intensities were collected by the variable-rate θ–2θ scan technique on a Syntex $P\bar{1}$ diffractometer to a maximum 2θ value of 140°, using a crystal of approximate dimensions 0·35 × 0·25 × 0·20 mm. Background measurements were taken at both ends of the scan range, each for a time equal to one-half the scan time. No sublimation or decay was observed during the data collection, monitored by the periodic measurement of two standard reflexions. Out of 3515 measured reflexions, 444 with $I < 2\sigma(I)_{\text{c.s.}}$ were given zero weight; all other reflexions were assigned variances, $\sigma^2(I)$, based on counting statistics plus the additional term (0·03 scan count)^2. The data were corrected for Lorentz and polarization factors but not for absorption.

Table 1. *Atomic coordinates* ($\times 10^4$; *for* H $\times 10^3$)

	x	y	z
C(1)	2628 (1)	7575 (1)	4076 (1)
C(2)	3616 (1)	8708 (1)	4264 (1)
C(3)	1270 (1)	7991 (1)	3602 (1)
C(4)	322 (1)	7082 (1)	3330 (1)
C(5)	−917 (1)	7380 (1)	2909 (1)
C(6)	−1249 (1)	8610 (2)	2753 (1)
C(7)	−337 (2)	9512 (1)	3021 (1)
C(8)	916 (1)	9216 (1)	3443 (1)
C(9)	2173 (1)	7010 (1)	4792 (1)
C(10)	1795 (1)	7796 (1)	5346 (1)
C(11)	1333 (1)	7325 (2)	5981 (1)
C(12)	1204 (1)	6067 (2)	6079 (1)
C(13)	1519 (1)	5282 (1)	5530 (1)
C(14)	1993 (1)	5743 (1)	4891 (1)
C(15)	3412 (1)	6678 (1)	3620 (1)
C(16)	3322 (1)	6816 (1)	2847 (1)
C(17)	4108 (2)	6099 (2)	2430 (1)
C(18)	5002 (2)	5227 (2)	2772 (1)
C(19)	5116 (1)	5078 (1)	3533 (1)
C(20)	4347 (1)	5805 (1)	3953 (1)
C(21)	5027 (1)	8405 (1)	4673 (1)
C(22)	5224 (1)	8104 (1)	5425 (1)
C(23)	6518 (1)	7826 (2)	5791 (1)
C(24)	7641 (1)	7870 (2)	5411 (1)
C(25)	7475 (1)	8196 (2)	4674 (1)
C(26)	6176 (1)	8456 (1)	4301 (1)
H(2*A*)	316 (1)	934 (1)	455 (1)
H(2*B*)	374 (1)	910 (1)	377 (1)
H(4)	56 (1)	621 (1)	342 (1)
H(5)	−160 (2)	670 (1)	271 (1)
H(6)	−212 (1)	881 (1)	245 (1)
H(7)	−56 (2)	1038 (2)	293 (1)
H(8)	155 (1)	991 (1)	363 (1)
H(10)	181 (1)	870 (1)	526 (1)
H(11)	112 (2)	792 (1)	636 (1)
H(12)	87 (2)	573 (2)	656 (1)
H(13)	142 (2)	438 (2)	559 (1)
H(14)	220 (1)	513 (1)	450 (1)
H(16)	269 (1)	743 (1)	259 (1)
H(17)	404 (2)	622 (2)	190 (1)
H(18)	553 (2)	472 (2)	247 (1)
H(19)	576 (2)	446 (1)	379 (1)
H(20)	447 (1)	569 (1)	451 (1)
H(22)	441 (2)	809 (1)	571 (1)
H(23)	662 (2)	757 (2)	632 (1)
H(24)	855 (2)	766 (2)	567 (1)
H(25)	829 (2)	824 (2)	440 (1)
H(26)	604 (2)	870 (1)	375 (1)

MULTAN (Germain, Main & Woolfson, 1971) gave the solution of the structure and a map that revealed the positions of all 26 C atoms. The 22 H atoms were initially given calculated positions. A modified version of *ORFLS* (Busing, Martin & Levy, 1962) was used for the full-matrix least-squares refinement by minimization of the quantity $\sum w(\Delta F)^2$, with weights $w = 4F_o^2/\sigma^2(F_o^2)$ for the 3071 reflexions classified as observed. Scattering factors for C were taken from Cromer & Waber (1965), and for H from Stewart, Davidson & Simpson (1965). Because of computer-core size limitation and the high number of parameters (324), isotropic H and anisotropic C atoms were refined in separate cycles; a scale factor and a secondary-extinction coefficient g (Larson, 1967) were included in each cycle. Refinement was considered complete when parameter shifts were less than 0·33σ for C atoms, and less than 0·7σ for H. The final values of R are 0·043 for all measured reflexions and 0·037 for the 3071 reflexions of non-zero weight, while R_w is 0·048; the secondary-extinction parameter, g, assumed the final value of 7 (1) × 10^{-6}. A final difference map showed no peaks higher than 0·15 e Å^{-3}. Final values for the parameters of C and H atoms are given in Table 1.*

Discussion. Fig. 1 shows the numbering scheme and the geometry of 1,1,1,2-tetraphenylethane. The formal e.s.d.'s in the C—C bond lengths, as evaluated directly from the coordinate uncertainties in Table 1, range from 0·0014 to 0·0028 Å, those in the C—C—C bond angles from 0·08 to 0·15°. The average value of the C—H distances is 0·99 Å, with an e.s.d. of about 0·015 Å. Only one of the five tetrahedral angles involving H atoms [C(1)—C(2)—H(2*B*), 105·9 (7)°] deviates by more than 3σ from the theoretical value. The phenyl C—C—H angles are in the range 118–122° (mean 119·7°) with e.s.d.'s 0·8–1·0°.

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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 35388 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

While bond distances and angles involving H atoms are in the usual range, some of those involving only C atoms deviate markedly from their standard values. As expected, the magnitudes of these deformations are larger than those found in the less crowded 1,1,1-triphenylethane (Destro, Pilati & Simonetta, 1980), and smaller than in the very strained pentaphenylethane (Destro, Pilati & Simonetta, 1978). Of particular interest are: (i) the ethane C(1)—C(2) bond length, 1·567 (2) Å [to be compared with 1·553 (2) Å in 1,1,1-triphenylethane and 1·606 (3) Å in pentaphenylethane]; (ii) the mean value of the three C(1)—C(Ph) bonds, 1·545 (2) Å [*vs* 1·538 (2) and 1·552 (2) Å, respectively, in the tri- and pentasubstituted ethanes]; (iii) the closure of the C(3)—C(1)—C(9) bond angle to 104·5 (1)° and the expansion of C(9)—C(1)—C(15) and C(1)—C(2)—C(21) to 114·8 (1) and 115·9 (1)°, respectively. [Bond angles at the central atoms C(1) and C(2) are in the range 107·8 (1)–110·6 (1)° in 1,1,1-triphenylethane, and in the range 102·2 (2)–118·6 (2)° in pentaphenylethane.]

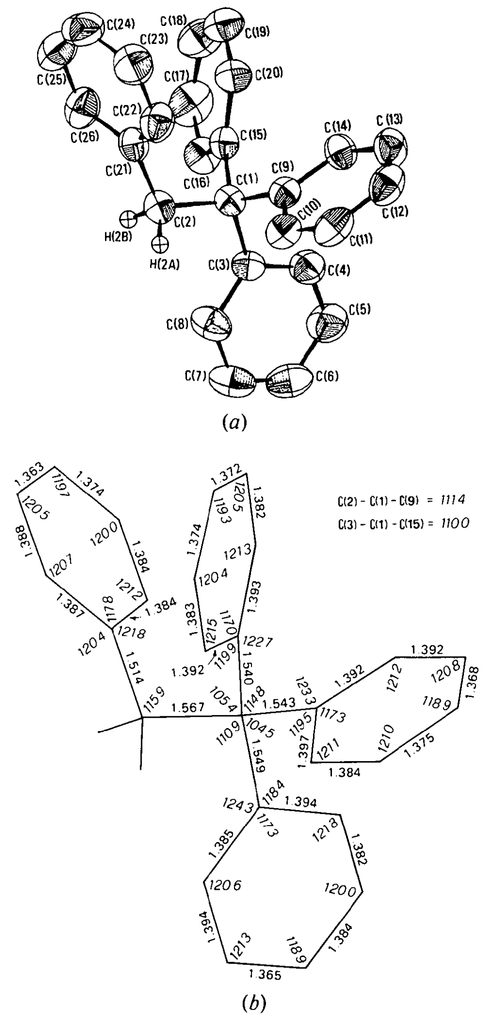

Fig. 1. (*a*) Thermal-ellipsoid plot (Johnson, 1965) and numbering scheme. The phenyl H atoms, omitted for the sake of clarity, are numbered (see Table 1) according to the C atom to which they are attached. (*b*) Bond distances (Å) and angles (°) involving the C atoms.

Other features which are common to all three crystal structures so far compared are the closure of the phenyl rings at the atom, C(Ph), directly bonded to the ethane atoms, and the large differences between the two exterior angles at C(Ph). For the molecule of tetraphenylethane here described, the closure amounts to about 3°, and the differences are 5·9° at C(3) (ring R_1), 3·8° at C(9) (ring R_2), 2·8° at C(15) (ring R_3), and 1·4° at C(21) (ring R_4). As a result of these differences, atoms C(1) and C(2) do not lie on the lines defined by the diameters of the phenyl rings passing through each C(Ph).

While the phenyl ring R_1 is planar within experimental uncertainty [maximum deviation 0·004 (1) Å], the other three rings are significantly nonplanar, with the largest deviations, up to 0·016 (1) Å, occurring for ring R_2. Atom C(2) is displaced from the mean plane of ring R_4 by 0·005 (1) Å, whereas atom C(1) lies out of the least-squares planes of the adjacent phenyl rings at distances 0·016 (1), 0·059 (1) and 0·127 (1) Å from rings R_1, R_2 and R_3, respectively.

There are only five intermolecular contacts (see Table 2) less than the sum of van der Waals radii (1·7 and 1·2 Å for C and H atoms, respectively). Since none of them is particularly short, it can be argued that the conformation of this unsymmetrically substituted tetra-

Table 2. *Intermolecular contact distances* (Å)

For each pair of atoms, the coordinates of the second atom are transformed as shown.

C(13)···C(13)	$-x, 1-y, 1-z$	3·374 (2)
C(11)···H(24)	$-1+x, y, z$	2·74 (2)
C(13)···H(19)	$1-x, 1-y, 1-z$	2·80 (1)
H(7)···H(11)	$-x, 2-y, 1-z$	2·34 (2)
H(17)···H(22)	$x, \frac{1}{2}-y, -\frac{1}{2}+z$	2·35 (2)

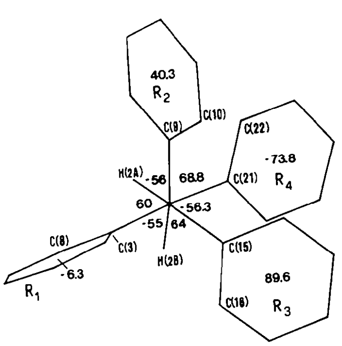

Fig. 2. A projection of 1,1,1,2-tetraphenylethane down the C(1)–C(2) bond, showing torsion angles (°) across the ethane bond and angles of rotation of the rings (°). Estimated standard deviations are in the range 0·1–0·2° when only C atoms are involved, and about 1° for angles involving H atoms.

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.

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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, α = 71·02 (1), β = 71·03 (1), γ = 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, λ = 1·5418 Å, graphite monochromator), $F(000)$ = 412, μ(Cu $K\alpha$) = 0·51 mm^{-1}, T = 291 ± 2 K. Final R = 0·042 for 3957 observed $[I > 2\sigma(I)_{\mathrm{c.s.}}]$ 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 $K\alpha$ (λ = 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 $[I > 2\sigma(I)_{\mathrm{c.s.}}]$	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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