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1,1,1-Triphenylethane

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Abstract. $C_{20}H_{18}$, $M_r = 258 \cdot 4$, monoclinic, $P2_1/n$, $a = 7 \cdot 208$ (1), $b = 16 \cdot 734$ (1), $c = 12 \cdot 622$ (1) Å, $\beta = 98 \cdot 17$ (1)°, $Z = 4$, $D_m = 1 \cdot 134$ (flotation), $D_x = 1 \cdot 138$ Mg m^{-3}, μ(Cu $K\alpha$) $= 0 \cdot 49$ mm^{-1}, $F(000) = 552$. Counter data collected with Cu $K\alpha$ radiation ($\lambda = 1 \cdot 5418$ Å) were used to derive the structure by direct methods. Full-matrix least-squares refinement based on 2389 reflexions with $I > 2\sigma(I)_{c.s.}$ led to $R = 0 \cdot 043$ and $R_w = 0 \cdot 054$. The molecule shows the expected propeller-like conformation, with slight but significant deformations from ideal tetrahedral and trigonal geometry.

Introduction. The present work is part of a systematic investigation of the effects of overcrowding on the conformation of substituted ethanes; the molecular geometry of pentaphenylethane has been previously reported (Destro, Pilati & Simonetta, 1978). A sample of 1,1,1-triphenylethane, in the form of colourless crystals, was kindly supplied by Professor G. Olah. Cell dimensions were obtained by a least-squares fit to the $\sin^2 \theta$ values of 59 reflexions measured on a diffractometer. The space group $P2_1/n$ was indicated by the absences $h0l$ with $h + l$ odd and $0k0$ with k odd. The density was measured by flotation in a dilute K_2HgI_4 solution. For the data collection a crystal of dimensions $0 \cdot 40 \times 0 \cdot 25 \times 0 \cdot 20$ mm was mounted on a computer-controlled four-circle diffractometer using graphite-monochromatized Cu $K\alpha$ radiation and a variable θ–2θ scan technique. The background was counted for half the total scanning time at each end of the scan range. Two standard reflexions were checked after every 48 intensity measurements; they showed no appreciable trend. In the range of measurements ($2\theta_{max}$

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

	x	y	z
C(1)	2404 (2)	8049 (1)	8289 (1)
C(2)	916 (3)	7831 (2)	7319 (1)
C(3)	3588 (2)	8743 (1)	7947 (1)
C(4)	3874 (2)	9449 (1)	8518 (1)
C(5)	4981 (3)	10055 (1)	8202 (2)
C(6)	5827 (3)	9968 (1)	7310 (2)
C(7)	5584 (3)	9275 (1)	6728 (2)
C(8)	4464 (3)	8665 (1)	7038 (1)
C(9)	3668 (2)	7326 (1)	8618 (1)
C(10)	5567 (3)	7406 (1)	8963 (1)
C(11)	6668 (4)	6754 (2)	9306 (1)
C(12)	5899 (6)	6011 (2)	9307 (2)
C(13)	4017 (7)	5916 (1)	8968 (2)
C(14)	2905 (4)	6563 (1)	8625 (2)
C(15)	1405 (2)	8299 (1)	9235 (1)
C(16)	2050 (3)	8079 (1)	10280 (1)
C(17)	1169 (3)	8338 (1)	11122 (1)
C(18)	−377 (3)	8814 (1)	10945 (2)
C(19)	−1043 (3)	9037 (1)	9922 (2)
C(20)	−168 (2)	8786 (1)	9077 (1)
H(2A)	1 (3)	744 (1)	753 (2)
H(2B)	23 (3)	835 (1)	696 (2)
H(2C)	152 (3)	757 (1)	678 (2)
H(4)	332 (2)	953 (1)	918 (1)
H(5)	520 (3)	1055 (1)	867 (2)
H(6)	671 (3)	1037 (1)	708 (2)
H(7)	611 (3)	917 (1)	608 (2)
H(8)	431 (3)	815 (1)	661 (1)
H(10)	611 (3)	793 (1)	899 (1)
H(11)	801 (3)	686 (1)	954 (2)
H(12)	668 (3)	557 (1)	953 (2)
H(13)	341 (3)	539 (2)	894 (2)
H(14)	156 (3)	650 (1)	841 (2)
H(16)	315 (2)	772 (1)	1043 (1)
H(17)	162 (3)	817 (1)	1180 (2)
H(18)	−106 (3)	902 (1)	1148 (2)
H(19)	−212 (3)	940 (1)	979 (2)
H(20)	−63 (3)	897 (1)	835 (2)

= 144°) 3015 reflexions were monitored, of which 2389 with $I > 2\sigma(I)$ were treated as observed. The variance, $\sigma^2(I)$, was calculated as $[S + B + (0.03S)^2]v^2$, where S = scan count, B = total background, v = scan rate. Intensities and their standard deviations were corrected for Lorentz and polarization effects, but not for absorption.

The structure was solved by direct methods (*MULTAN*, Germain, Main & Woolfson, 1971). The E map corresponding to the solution with the highest combined figure of merit clearly revealed the entire structure with essentially no extraneous detail. After preliminary refinement with isotropic thermal parameters, a difference map showed all the H atoms. The last cycles of least-squares refinement were carried out by simultaneously adjusting in a single matrix 254 parameters: coordinates for all the atoms, anisotropic b_{ij}'s for C atoms, isotropic B's for H atoms, a scale factor, and a secondary-extinction coefficient g (Larson, 1967). Refinement was by minimization of the quantity $\sum w(|F_o| - |F_c|)^2$, with weights $w = 4F_o^2/\sigma^2(F_o^2)$ for the 2389 reflexions classified as observed, and $w = 0.0$ for the 626 unobserved. Form factors for C were from Cromer & Waber (1965), and for H from Stewart, Davidson & Simpson (1965). The final values of the residuals are $R = 0.043$ and $R_w = 0.054$, and the goodness-of-fit, based on 2389 weighted reflexions and 254 parameters, is 1.98. Final values for the atomic parameters are given in Table 1; the final value for the extinction parameter g is $8(2) \times 10^{-6}$.*

Discussion. The most salient details of the molecular geometry of 1,1,1-triphenylethane in the crystal structure are reported in Fig. 1, together with the numbering scheme adopted. The conformation of the molecule can be conveniently described by the torsion angles across the central ethane bond, φ_c, and the twist angles, φ_r, defined as the dihedral angle between the mean plane of a given phenyl ring and the plane through the C(2)—C(1)—C(Ph) fragments [where C(Ph) is the ring atom bonded to C(1)]. The values of these parameters [see Fig. 1(a)] indicate that 1,1,1-triphenylethane adopts a propeller-like conformation similar to that found in triphenylmethane (Riche & Pascard-Billy, 1974) and in chlorotris(p-methoxyphenyl)methane (Dunand & Gerdil, 1976). Minimization of steric interference between the twisted phenyl rings and the H atoms at C(2) is achieved mainly through a rotation [11 (1)°] of the methyl group with respect to the staggered conformation of the ethane skeleton, and, to a lesser extent, through a lengthening of the C(1)—C(2)

bond [1.553 (2) Å *vs* the standard value of 1.541 (3) Å for a paraffinic single bond].

Intramolecular forces are presumably responsible for other distortions from ideal tetrahedral and trigonal geometry: (i) As shown in Fig. 1(b), there is a closure of the phenyl rings at C(Ph) [C(3), C(9) and C(15), respectively] relative to the mean of the other trigonal angles. A similar feature is observed in triphenylmethane (Riche & Pascard-Billy, 1974) and in tetraphenylmethane (Robbins, Jeffrey, Chesick, Donohue, Cotton, Frenz & Murillo, 1975), while in chlorotris(p-methoxyphenyl)methane (Dunand & Gerdil, 1976)
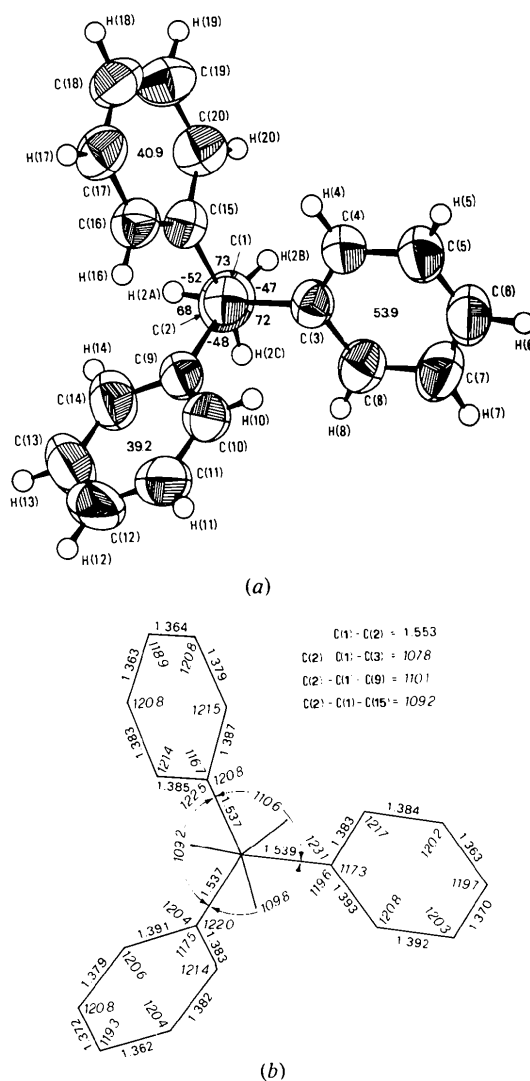

Fig. 1. The molecule of 1,1,1-triphenylethane viewed down the C(1)—C(2) bond. (a) Thermal-ellipsoid plot with numbering scheme, torsion angles, φ_c, (°) across the ethane bond, and, inside the phenyl rings, twist angles φ_r (°) (see text). E.s.d.'s are around 1.3° for torsion angles and 0.2° for twist angles. (b) Bond distances (Å) and angles (°) involving C atoms. E.s.d.'s are in the range 0.002–0.006 Å in bond lengths and 0.1–0.3° in bond angles.
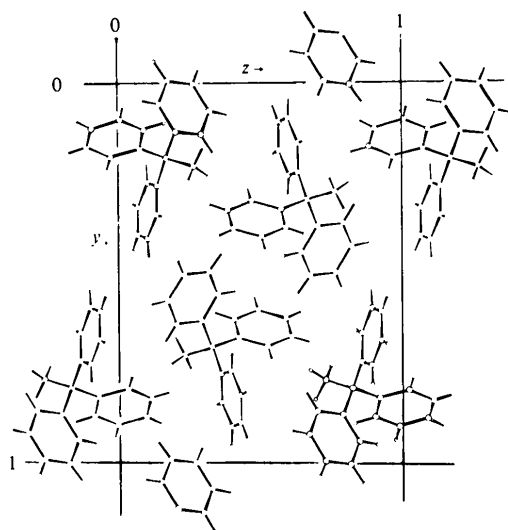
Fig. 2. A projection of the structure down the a axis.

differences between these two average values (e.g. 106 vs 112°, respectively) occur in crystals of triphenylmethane, chlorotriphenylmethane, bromo-triphenylmethane, 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 $C(5) \cdots H(19)_{1+x,y,z}$, 2·90 (2); $C(19) \cdots H(10)_{-1+x,y,z}$, 2·89 (2); and $C(12) \cdots H(6)_{\frac{1}{2}-x,-\frac{1}{2}+y,\frac{1}{2}-z}$, 2·84 (2) Å.

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

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1,1,1,2-Tetraphenylethane

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Abstract. $C_{26}H_{22}$, $M_r = 334\cdot5$, monoclinic, $P2_1/c$, $a = 9\cdot814$ (1), $b = 10\cdot774$ (1), $c = 18\cdot032$ (1) Å, $\beta = 97\cdot69(1)°$, $Z = 4$, $D_m = 1.17$ (flotation in dilute K_2HgI_4 solution), $D_x = 1\cdot176$ Mg m^{-3}. Computer-controlled four-circle diffractometer data (Cu $K\alpha$ radiation, $\lambda = 1\cdot5418$ Å, graphite monochromator), $F(000) = 712$, μ(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 $I > 2\sigma(I)_{c.s.}$. 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.