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1,1,2,2-Tetrachloro-1,2-diphenylethane

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.02 Å
R factor = 0.026
wR factor = 0.067
Data-to-parameter ratio = 17.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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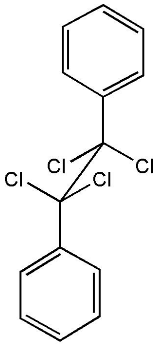
The title compound, $C_{14}H_{10}Cl_4$, possesses C_i symmetry and crystallizes with one half-molecule in the asymmetric unit. The two phenyl rings are antiperiplanar and inclined to one another by 180° by symmetry. The central C—C bond distance and the Cl—C—Cl bond angle [1.5887 (18) Å and 106.02 (7)°, respectively] are significantly different from ideal sp^3 values. In the crystal, a supramolecular structure is achieved by soft parallel-displaced π–π stacking and C—H$\cdots\pi$ intermolecular interactions.

Comment

Halogenated hydrocarbons are known for their toxic effects, and some of these compounds are suspected of having human carcinogenic activity (Cornia *et al.*, 1993). The title compound, (I), and its derivatives constitute an important class of halogenated hydrocarbons whose internal rotation processes and conformational preferences have been studied by NMR and X-ray methods (Antolini *et al.*, 1995). In this context the crystal structure of (I) is reported here.



(I)

The molecular conformation and dimensions of (I), illustrated in Fig. 1, are very similar to those reported for several p-substituted diaryl derivatives of 1,1,2,2-tetrachloroethane (Antolini *et al.*, 1994). Selected interatomic distances and bond and torsion angles are listed in Table 1.

The asymmetric unit of (I) contains one half-molecule, with the other half generated by a center of inversion, which lies at the midpoint of the C1—C1^i bond [symmetry code: (i) $-x + 1$, $-y$, $-z + 1$]. Thereby, perfect staggering around the C1—C1^i bond is a consequence, with the aromatic rings in an antiperiplanar position [C2—C1—C1^i—C2^i = 180°]. The same molecular conformation and symmetry requirements ($\bar{1}$) were observed in other 4,4-(1,1,2,2-tetrachloroethane-1,2-diyl)dichlorobenzenes (Hovmöller *et al.*, 1978; Antolini *et al*, 1994). Good agreement of the corresponding values of bond

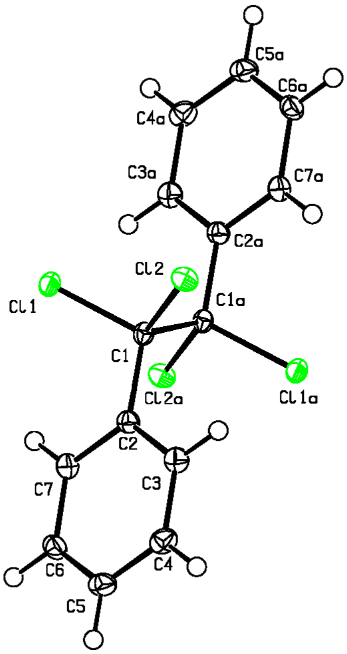


Figure 1
The molecular structure of compound (I), showing displacement ellipsoids drawn at the 30% probability level [Symmetry code: (*a*) $-x + 1, -y, -z + 1$.]

distances and angles with compound (I) is found. Nevertheless, some of these geometric parameters are significantly different from those reported for many DDT-type derivatives of known structures and from mean values retrieved from the Cambridge Structural Database (Version of April 2004; Allen, 2002).

The C1—C1^{i} distance of 1.5887 (18) Å is significantly longer than the mean value of 1.513 Å for a C—C single bond (Allen *et al.*, 1987). Such bond lengthening is accompanied by a substantial decrease of the bond angle [Cl1—C1—Cl2 = 106.02 (7)°] in comparison with the ideal sp^3 angle of 109.5 Å. In contrast, the Cl1—C1 and Cl2—C1 bond distances, of 1.7969 (13) and 1.7882 (14) Å, respectively, are similar to the value of 1.792 Å found in DDT-type derivatives.

The supramolecular structure of (I), shown in Fig. 2, is achieved by parallel displaced π-stacking interactions [symmetry code: $-x, -y, 1 - z$] between the aromatic rings, with interplanar and intercentroid distances of 3.374 and 3.8916 (10) Å, respectively (Singh & Thornton, 1990). T-shaped (Umezawa *et al.*, 1998) C4—H4···Ph^{iii} [symmetry code: (iii) $x, \frac{1}{2} - y, -\frac{1}{2} + z$] intermolecular interactions of 2.8267 Å [C4···Ph = 3.6971 (16) Å and C4—H4···Ph = 152.74°] also contribute to the crystal packing.

Experimental

α,α,α-Trichlorotoluene (1.02 ml, 7.15 mmol) and triethylamine (3.8 ml, 27.24 mmol) were heated at 393 K in a sealed ampoule for 24 h. The resulting mixture was dissolved in $CHCl_3$ (50 ml) and extracted with three portions of distilled water (20 ml). After drying with Na_2SO_4 and concentrating, the organic phase was chromatographed on silica gel to obtain 0.914 g (80% yield) of (I) as a white solid. Crystals suitable for X-ray analysis were obtained by crystallization from a chloroform solution. ^{1}H NMR ($CDCl_3$): δ 7.44 (*br*, 2H, H_p), 7.35 (*t*, 2H, H_m), 7.21 (*br*, 2H, H_o); ^{13}C NMR ($CDCl_3$): δ 136.4 (C_i), 131.0 (C_p), 130.0 (C_m), 126.87 (C_o), 96.8 (CCl_2).

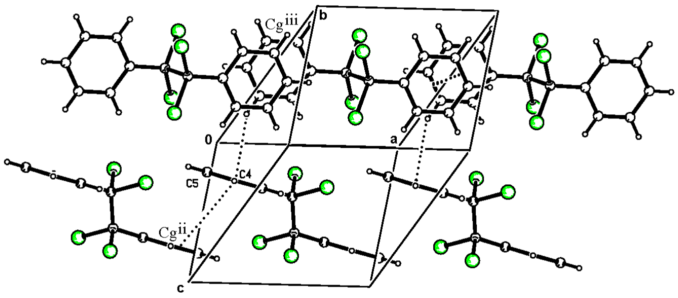


Figure 2
The crystal packing of compound (I). Parallel displaced π–π stacking and C4—H4···π^{iii} intermolecular interactions are shown as dotted lines. [Symmetry codes: (ii) $-x, -y, 1 - z$; (iii) $x, \frac{1}{2} - y, -\frac{1}{2} + z$.]

Crystal data

$C_{14}H_{10}Cl_4$
$M_r = 320.02$
Monoclinic, $P2_1/c$
$a = 8.5925$ (10) Å
$b = 10.6129$ (13) Å
$c = 7.7558$ (9) Å
$\beta = 114.315$ (2)°
$V = 644.52$ (13) Å^3
$Z = 2$
$D_x = 1.649$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 600 reflections
$\theta = 20$–25°
$\mu = 0.89$ mm^{-1}
$T = 100$ (2) K
Block, colorless
0.52 × 0.49 × 0.42 mm

Data collection

Bruker SMART area-detector diffractometer
φ and ω scans
6982 measured reflections
1461 independent reflections
1451 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 27.6°$
$h = -10 \rightarrow 10$
$k = -13 \rightarrow 13$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.026$
$wR(F^2) = 0.067$
$S = 1.12$
1461 reflections
82 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0354P)^2 + 0.3344P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.41$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—C1	1.7969 (13)	C1—C2	1.523 (2)
Cl2—C1	1.7882 (14)	C1—C1^{i}	1.5887 (18)
Cl1—C1—Cl2	106.02 (7)	Cl2—C1—C2	110.37 (9)
Cl1—C1—C2	110.43 (9)	Cl2—C1—C1^{i}	107.69 (9)
Cl1—C1—C1^{i}	107.00 (9)		

Symmetry code: (i) $-x + 1, -y, -z + 1$.

All the H atoms could be located in difference Fourier maps, and were refined as riding atoms, with C—H = 0.95 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2000); program(s) used to solve

Acta Cryst. (2005). E**61**, o678–o680 Garcia-Sanchez *et al.* • $C_{14}H_{10}Cl_4$ **o679**

structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXL97* and *WinGX2003* (Farrugia, 1999).

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o680 Garcia-Sanchez *et al.* • $C_{14}H_{10}Cl_4$ *Acta Cryst.* (2005). E**61**, o678–o680