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(*E*)-2,3-Dichloro-1,1,4,4-tetrakis(4-chlorophenyl)-but-2-ene

Hisashi Shimakoshi, Isao Aritome, Mami Tokunaga and Yoshio Hisaeda*

Department of Chemistry and Biochemistry, Kyushu University, Hakozaki Higashi-ku, Fukuoka 812-8581, Japan

Correspondence e-mail: yhisatcm@mbox.nc.kyushu-u.ac.jp

Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.003 Å
R factor = 0.038
wR factor = 0.099
Data-to-parameter ratio = 16.2

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

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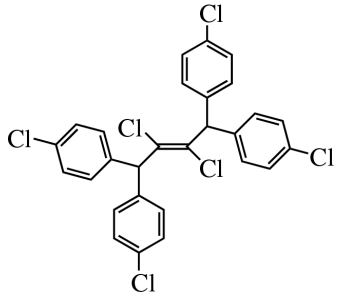
The title compound, $C_{28}H_{18}Cl_6$, was obtained by the electrolysis of 2,2,2-trichloro-1,1-bis(4-chlorophenyl)ethane (DDT) in the presence of a catalytic amount of a cobalamin derivative. The molecule has a centre of symmetry, and the alkene moiety has an *E* configuration.

Comment

1,1-Bis(4-chlorophenyl)-2,2,2-trichloroethane (DDT) is characterized by a pronounced insecticidal property and has been used world-wide for several decades, despite its known hazardous effects on human health and wildlife (Fellenberg, 2000). Therefore, the degradation of such a pollutant has been carried out extensively using several methods (Alonso *et al.*, 2002; Häggblom & Bossert, 2003).

Recently, we reported the partial dechlorination of DDT by catalysis of a cobalamin derivative as electrochemical mediator; various dechlorinated products were obtained, such as 1,1-bis(4-chlorophenyl)-2,2-dichloroethane (DDD), 1,1-bis(4-chlorophenyl)-2,2-dichloroethylene (DDE) and 1,1,4,4-tetrakis(4-chlorophenyl)-2,3-dichloro-2-butene (TTDB) (Shimakoshi *et al.*, 2004). Structural data for these DDT analogues have been reported and discussed from the point of view of their toxicity (Kennard *et al.*, 1984). In this paper, the crystal structure of the title compound, (I), TTDB (*E* form), is reported, in order to confirm the geometry and to obtain detailed information on the molecular conformation.



(I)

The molecular structure of (I) is shown in Fig. 1, with the atom-numbering scheme. The centrosymmetric molecule contains four benzene rings, each substituted by one Cl atom. The alkene moiety of the molecule has the *E* configuration. Deviations from ideal bond-angle geometry around the Csp^2 atom (C14) of the double bond are observed (Table 1). The dihedral angles between the plane of atoms C1/C13/C7 and the planes of the two benzene rings are 88.6 (1) and 8.9 (3)°. These angles are considerably different from those of DDT (84.2 and 47.4°; DeLacy & Kennard, 1972). The dihedral angle

between the benzene rings is 88.51 (6)° in (I), which is larger than that in DDT (64.9°).

Experimental

The title compound, (I), was obtained using the method of Shimakoshi *et al.* (2004), *i.e.* by the electrolysis of DDT in the presence of a catalytic amount of a cobalamin derivative, and was isolated by preparative thin-layer chromatography (eluent *n*-hexane–$CHCl_3$, 10:1, R_f = 0.82). Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of a saturated benzene/ethanol solution.

Crystal data

$C_{28}H_{18}Cl_6$	Z = 1
M_r = 567.12	D_x = 1.538 Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
a = 7.6756 (6) Å	Cell parameters from 2309 reflections
b = 9.2556 (7) Å	θ = 2.2–30.4°
c = 10.2911 (8) Å	μ = 0.72 mm^{-1}
α = 66.459 (1)°	T = 100 (2) K
β = 88.165 (2)°	Block, colourless
γ = 67.361 (2)°	0.18 × 0.15 × 0.09 mm
V = 612.18 (8) Å^3	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	2494 independent reflections
	2229 reflections with $I > 2\sigma(I)$
φ and ω scans	R_{int} = 0.016
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	θ_{max} = 26.4°
	$h = -9 \rightarrow 9$
T_{min} = 0.882, T_{max} = 0.938	$k = -11 \rightarrow 11$
4002 measured reflections	$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.054P)^2 + 0.3132P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.038	
$wR(F^2)$ = 0.099	
S = 1.05	$(\Delta/\sigma)_{max}$ = 0.003
2494 reflections	$\Delta\rho_{max}$ = 0.47 e Å^{-3}
154 parameters	$\Delta\rho_{min}$ = −0.23 e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

C4—Cl1	1.743 (2)	C14—C14^{i}	1.330 (4)
C10—Cl2	1.747 (2)	C14—Cl3	1.7454 (19)
C13—C14	1.517 (3)		
C14^{i}—C14—C13	126.4 (2)	C13—C14—Cl3	115.60 (14)
C14^{i}—C14—Cl3	118.0 (2)		

Symmetry code: (i) $-x, 2-y, 1-z$.

H atoms were located geometrically and allowed to ride on their parent atoms, with C—H distances in the range 0.95–1.00 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT* (Bruker, 2001); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

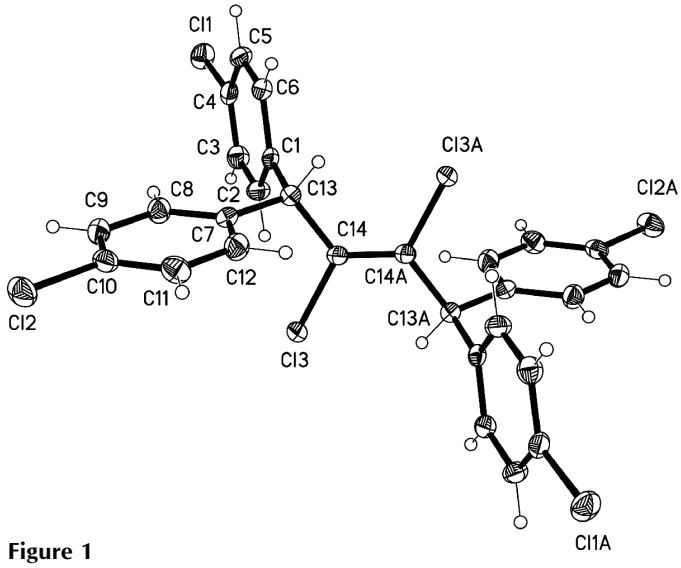


Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. Atoms labelled with the suffix A are at the symmetry position $(-x, 2-y, 1-z)$.

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