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## Key indicators

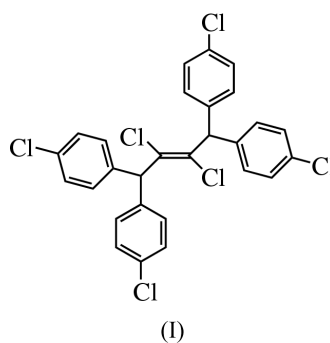
Single-crystal X-ray study  
 $T = 100$  K  
Mean  $\sigma(C-C) = 0.003$  Å  
 $R$  factor = 0.038  
 $wR$  factor = 0.099  
Data-to-parameter ratio = 16.2For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.**(E)-2,3-Dichloro-1,1,4,4-tetrakis(4-chlorophenyl)-  
but-2-ene**

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.



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

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between the benzene rings is  $88.51(6)^\circ$  in (I), which is larger than that in DDT ( $64.9^\circ$ ).

## 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– $\text{CHCl}_3$ , 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

$\text{C}_{28}\text{H}_{18}\text{Cl}_6$	$Z = 1$
$M_r = 567.12$	$D_x = 1.538 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 7.6756(6) \text{ \AA}$	Cell parameters from 2309 reflections
$b = 9.2556(7) \text{ \AA}$	$\theta = 2.2\text{--}30.4^\circ$
$c = 10.2911(8) \text{ \AA}$	$\mu = 0.72 \text{ mm}^{-1}$
$\alpha = 66.459(1)^\circ$	$T = 100(2) \text{ K}$
$\beta = 88.165(2)^\circ$	Block, colourless
$\gamma = 67.361(2)^\circ$	$0.18 \times 0.15 \times 0.09 \text{ mm}$
$V = 612.18(8) \text{ \AA}^3$	

### Data collection

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

### Refinement

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

**Table 1**

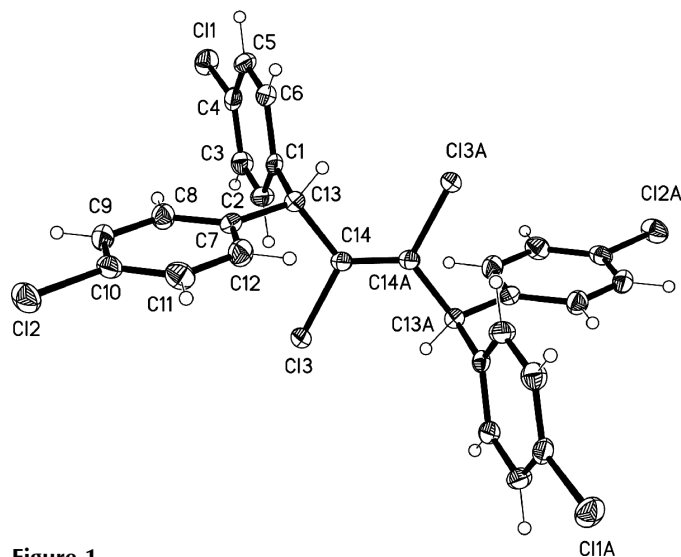
Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

C4–C11	1.743 (2)	C14–C14 <sup>i</sup>	1.330 (4)
C10–C12	1.747 (2)	C14–Cl3	1.7454 (19)
C13–C14	1.517 (3)		
C14 <sup>i</sup> –C14–C13	126.4 (2)	C13–C14–Cl3	115.60 (14)
C14 <sup>i</sup> –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  $0.95\text{--}1.00 \text{ \AA}$  and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

Data collection: SMART (Bruker, 2001); cell refinement: SAINT (Bruker, 2001); data reduction: SAINT (Bruker, 2001); program(s)



**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 ( $-x, 2 - y, 1 - z$ ).

used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 2001); software used to prepare material for publication: SHELXTL.

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