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Key indicators

Single-crystal X-ray study
 $T = 223$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.050
 wR factor = 0.128
Data-to-parameter ratio = 16.8

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

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

The title compound, $\text{C}_{28}\text{H}_{18}\text{Cl}_6$, 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 the cobalamin derivative heptamethyl cobyrinate perchlorate. The alkene group of the molecule has a *Z* configuration.

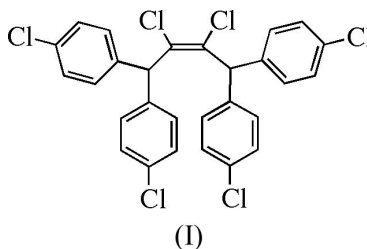
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Comment

Dechlorination of organic halides, ubiquitous pollutants such as polychlorinated alkenes and alkanes, has increased their importance over the past few decades (Hitchman *et al.*, 1995; Alonso *et al.*, 2002). Among organic halides, 2,2,2-trichloro-1,1-bis(4-chlorophenyl)ethane (DDT) is characterized by a pronounced insecticidal property and has been used worldwide for several decades, despite its known hazardous effects on human health and wildlife (Fellenberg, 2000). Therefore, the degradation of DDT has been carried out extensively using electrochemical methods (Schweizer *et al.*, 1994; Merica *et al.*, 1999).



Recently, we also reported the partial dechlorination of DDT by catalysis with 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, Tokunaga & Hisaeda, 2004). Structural data for these DDT analogues have been reported and discussed from the point of view of their toxicity (Kennard *et al.*, 1984). Previously, we reported the crystal structure of TTDB (*E* form) (Shimakoshi, Aritome *et al.*, 2004). In the present paper, the crystal structure of the title compound, (I) (TTDB) (*Z* 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 molecule contains four benzene rings, each substituted by one Cl atom. The alkene group of the molecule has a *Z* configuration. Deviations from ideal bond-angle geometry around the Csp^2 atoms (C26 and C27) of the double bond are observed (Table 1). The dihedral angles

between the C1/C25/C7 and C13/C28/C19 planes and the planes of the related benzene rings [A (C1–C6) and B (C7–C12), and C (C13–C18) and D (C19–C24)] are 34.4 (2) and 42.7 (2)°, and 41.4 (2) and 75.6 (1)°, respectively. These angles are considerably different from those of the *E* form [88.6 (1) and 8.9 (3)°; Shimakoshi Aritome *et al.*, 2004]. The dihedral angles between the benzene rings are *A/B* = 67.5 (1)° and *C/D* = 87.9 (1)° in (I), compared with 88.51 (6)° in the *E* form.

Experimental

The title compound, (I), was obtained using the method of Shimakoshi, Tokunaga & Hisaeda (2004), *i.e.* by the electrolysis of DDT in the presence of a catalytic amount of the cobalamin derivative heptamethyl cobyrinate perchlorate, and was isolated by preparative thin-layer chromatography (eluant, *n*-hexane–CHCl₃, 10:1, *R_f* = 0.74). 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	<i>Z</i> = 2
<i>M_r</i> = 567.12	<i>D_x</i> = 1.479 Mg m ⁻³
Triclinic, <i>P</i> 1̄	Mo <i>K</i> α radiation
<i>a</i> = 7.1993 (5) Å	Cell parameters from 2294 reflections
<i>b</i> = 9.2058 (7) Å	<i>θ</i> = 2.7–26.6°
<i>c</i> = 19.9448 (16) Å	<i>μ</i> = 0.69 mm ⁻¹
<i>α</i> = 101.133 (2)°	<i>T</i> = 223 (2) K
<i>β</i> = 100.209 (2)°	Plate, colorless
<i>γ</i> = 91.704 (2)°	0.24 × 0.23 × 0.08 mm
<i>V</i> = 1273.64 (17) Å ³	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	5163 independent reflections
<i>φ</i> and <i>ω</i> scans	3898 reflections with <i>I</i> > 2σ(<i>I</i>)
Absorption correction: multi-scan (<i>SADABS</i> ; Sheldrick, 1996)	<i>R_{int}</i> = 0.020
<i>T_{min}</i> = 0.852, <i>T_{max}</i> = 0.947	<i>θ_{max}</i> = 26.4°
8266 measured reflections	<i>h</i> = -7 → 8
	<i>k</i> = -11 → 11
	<i>l</i> = -22 → 24

Refinement

Refinement on <i>F</i> ²	$w = 1/[\sigma^2(F_o^2) + (0.0618P)^2 + 0.236P]$
$R[F^2 > 2\sigma(F^2)] = 0.050$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.128$	(Δ/σ) _{max} < 0.001
<i>S</i> = 1.02	$\Delta\rho_{max} = 0.45 \text{ e } \text{Å}^{-3}$
5163 reflections	$\Delta\rho_{min} = -0.21 \text{ e } \text{Å}^{-3}$
307 parameters	
H-atom parameters constrained	

Table 1

Selected geometric parameters (Å, °).

C4–C11	1.744 (3)	C26–C27	1.323 (4)
C10–C12	1.736 (3)	C26–C15	1.737 (3)
C16–C13	1.748 (3)	C27–C28	1.521 (4)
C22–C14	1.743 (3)	C27–C16	1.736 (3)
C25–C26	1.517 (4)		
C27–C26–C25	124.4 (2)	C26–C27–C28	123.3 (2)
C27–C26–C15	121.0 (2)	C26–C27–C16	120.4 (2)
C25–C26–C15	114.63 (18)	C28–C27–C16	116.22 (18)

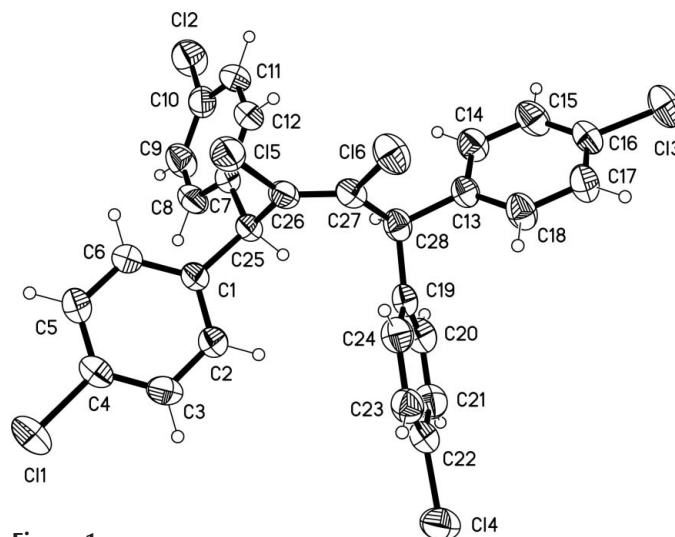


Figure 1

A view of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

H atoms were positioned geometrically at distances of 0.94 and 0.99 Å from the parent C atoms; a riding model was used during the refinement process, with *U_{iso}*(H) values set at 1.2*U_{eq}*(carrier atom).

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