

Bis(pentachlorophenyl) disulfide

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$
R factor = 0.036
wR factor = 0.099
Data-to-parameter ratio = 16.3

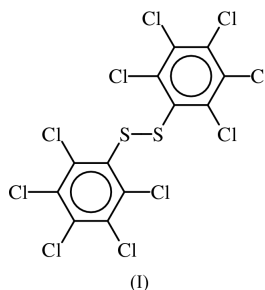
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The molecule of bis(pentachlorophenyl) disulfide, $(\text{Cl}_5\text{C}_6)_2\text{S}_2$, lies on a twofold axis; the phenyl rings are twisted by $19.2 (1)^\circ$ and the C—S—S—C torsion angle is $-82.8 (2)^\circ$. The crystal packing is dominated by weak $\text{Cl}\cdots\text{Cl}$ contacts of 3.5–3.7 Å.

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Comment

Polychloroaromatic hydrocarbons undergo nucleophilic substitution with thiolate ions in polar aprotic solution (Baird *et al.*, 1988), as exemplified by the reaction of perchlorocoronene, $\text{C}_{24}\text{Cl}_{12}$, with $\text{CH}_3\text{O}-4-\text{C}_6\text{H}_4\text{SNa}$, in which all Cl atoms are replaced by the $\text{CH}_3\text{O}-4-\text{C}_6\text{H}_4\text{S}$ groups. On the other hand, hexachlorobenzene reacts with sodium phenylthiolate to form hexa(phenylsulfido)benzene (MacNicol *et al.*, 1982). In the present study, the reaction of C_6Cl_6 with $(\text{O}_2\text{CCH}_2\text{S})^{2-}$ in DMF afforded, instead, a disulfide, $\text{C}_6\text{Cl}_5\text{SSC}_6\text{Cl}_5$ (Fig. 1), (I).



The molecule of (I) occupies a special position on a twofold axis. The $\text{S1}-\text{S1}^i$ bond distance (symmetry code as in Table 1), $2.063 (2) \text{ \AA}$, is similar to that found in 2-nitrophenyl 4-nitrophenyl disulfide (Glidewell *et al.*, 2002). The aromatic ring is planar, and the Cl substituents lie close to its plane, the largest deviation being $0.085 (4) \text{ \AA}$ for atom Cl2. The two rings are twisted by $19.2 (1)^\circ$ and the $\text{C1}-\text{S1}-\text{S1}^i-\text{Cl1}^i$ torsion angle is $-82.8 (2)^\circ$. The crystal packing is dominated by $\text{Cl}\cdots\text{Cl}$ contacts of 3.5–3.7 Å.

Experimental

Hexachlorobenzene (0.28 g, 1 mmol) and an excess of disodium thioglycollate (1.34 g, 10 mmol) were refluxed in a DMF–water (1/1) mixture for 5 h. The reaction mixture was cooled, and the product was extracted with toluene. The toluene solution was washed with water and then dried over magnesium sulfate. Evaporation of the

solvent afforded the crude product, which was then recrystallized from toluene.

Crystal data

$C_{12}Cl_{10}S_2$
 $M_r = 562.74$
 Monoclinic, $C2/c$
 $a = 15.188 (4) \text{ \AA}$
 $b = 8.685 (3) \text{ \AA}$
 $c = 14.645 (3) \text{ \AA}$
 $\beta = 111.12 (1)^\circ$
 $V = 1802.1 (8) \text{ \AA}^3$
 $Z = 4$

$D_x = 2.074 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 Cell parameters from 25 reflections
 $\theta = 13.0\text{--}15.0^\circ$
 $\mu = 1.77 \text{ mm}^{-1}$
 $T = 298 (2) \text{ K}$
 Block, yellow
 $0.35 \times 0.33 \times 0.15 \text{ mm}$

Data collection

Enraf–Nonius CAD-4 diffractometer
 ω scans
 Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.459, T_{\max} = 0.767$
 3538 measured reflections
 1774 independent reflections
 1469 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.039$
 $\theta_{\text{max}} = 26.0^\circ$
 $h = -18 \rightarrow 18$
 $k = -10 \rightarrow 0$
 $l = -18 \rightarrow 18$
 2 standard reflections
 frequency: 60 min
 intensity decay: none

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.099$
 $S = 1.08$
 1774 reflections
 109 parameters

$w = 1/[\sigma^2(F_o^2) + (0.0476P)^2 + 1.1173P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.43 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.32 \text{ e \AA}^{-3}$

Table 1

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

C1–C2	1.722 (3)	C1–C2	1.397 (4)
C2–C3	1.712 (3)	C1–C6	1.398 (4)
C3–C4	1.710 (3)	C2–C3	1.385 (4)
C4–C5	1.717 (3)	C3–C4	1.398 (4)
C5–C6	1.715 (3)	C4–C5	1.386 (4)
S1–C1	1.771 (3)	C5–C6	1.384 (4)
S1–S1 ⁱ	2.063 (2)		
C1–S1–S1 ⁱ	100.3 (1)	C5–C4–C3	120.0 (3)
C2–C1–C6	118.4 (3)	C5–C4–Cl3	120.1 (2)
C2–C1–S1	120.5 (2)	C3–C4–Cl3	119.9 (2)
C6–C1–S1	121.0 (2)	C6–C5–C4	120.1 (3)
C3–C2–C1	121.1 (3)	C6–C5–Cl4	119.9 (2)
C3–C2–Cl1	118.4 (2)	C4–C5–Cl4	119.9 (2)
C1–C2–Cl1	120.4 (2)	C5–C6–C1	120.8 (3)
C2–C3–C4	119.5 (3)	C5–C6–Cl5	119.7 (2)
C2–C3–Cl2	120.9 (2)	C1–C6–Cl5	119.5 (2)
C4–C3–Cl2	119.6 (2)		

Symmetry code: (i) $1 - x, y, \frac{1}{2} - z$.

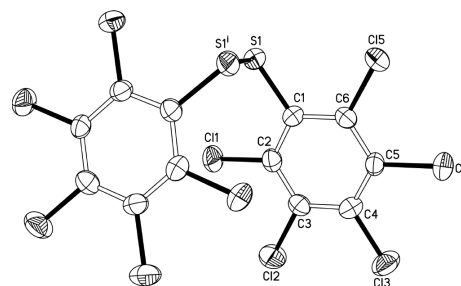


Figure 1
 ORTEP II (Johnson, 1976) plot of (I), with displacement ellipsoids drawn at the 50% probability level. [Symmetry code: (i) $1 - x, y, \frac{1}{2} - z$.]

Data collection: *CAD-4 Software* (Enraf–Nonius, 1988); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP II* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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