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# Bis(pentachlorophenyl) disulfide

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**Key indicators**

Single-crystal X-ray study
*T* = 298 K
Mean σ(C–C) = 0.004 Å
*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.

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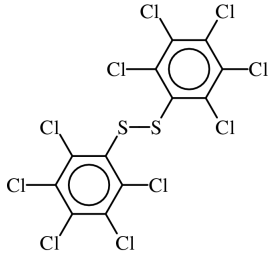
The molecule of bis(pentachlorophenyl) disulfide, $(Cl_5C_6)_2S_2$, lies on a twofold axis; the phenyl rings are twisted by 19.2 (1)° and the C—S—S—C torsion angle is −82.8 (2)°. The crystal packing is dominated by weak Cl···Cl contacts of 3.5–3.7 Å.

## Comment

Polychloroaromatic hydrocarbons undergo nucleophilic substitution with thiolate ions in polar aprotic solution (Baird *et al.*, 1988), as exemplified by the reaction of perchlorocoronene, $C_{24}Cl_{12}$, with $CH_3O$-4-$C_6H_4SNa$, in which all Cl atoms are replaced by the $CH_3O$-4-$C_6H_4S$ 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 $(O_2CCH_2S)^{2-}$ in DMF afforded, instead, a disulfide, $C_6Cl_5SSC_6Cl_5$ (Fig. 1), (I).



(I)

The molecule of (I) occupies a special position on a twofold axis. The S1—S1$^{i}$ bond distance (symmetry code as in Table 1), 2.063 (2) Å, 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) Å for atom Cl2. The two rings are twisted by 19.2 (1)° and the C1—S1—S1$^{i}$–C1$^{i}$ torsion angle is −82.8 (2)°. The crystal packing is dominated by Cl···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*

| | |
|---|---|
| $C_{12}Cl_{10}S_2$ | $D_x$ = 2.074 Mg m$^{-3}$ |
| $M_r$ = 562.74 | Mo $K\alpha$ radiation |
| Monoclinic, $C2/c$ | Cell parameters from 25 reflections |
| $a$ = 15.188 (4) Å | $\theta$ = 13.0–15.0° |
| $b$ = 8.685 (3) Å | $\mu$ = 1.77 mm$^{-1}$ |
| $c$ = 14.645 (3) Å | $T$ = 298 (2) K |
| $\beta$ = 111.12 (1)° | Block, yellow |
| $V$ = 1802.1 (8) Å$^3$ | 0.35 × 0.33 × 0.15 mm |
| $Z$ = 4 | |

*Data collection*

| | |
|---|---|
| Enraf–Nonius CAD-4 diffractometer | $R_{int}$ = 0.039 |
| $\omega$ scans | $\theta_{max}$ = 26.0° |
| Absorption correction: $\psi$ scan (North *et al.*, 1968) | $h = -18 \rightarrow 18$ |
| $T_{min}$ = 0.459, $T_{max}$ = 0.767 | $k = -10 \rightarrow 0$ |
| 3538 measured reflections | $l = -18 \rightarrow 18$ |
| 1774 independent reflections | 2 standard reflections |
| 1469 reflections with $I > 2\sigma(I)$ | frequency: 60 min |
| | intensity decay: none |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | $w = 1/[\sigma^2(F_o^2) + (0.0476P)^2 + 1.1173P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $R[F^2 > 2\sigma(F^2)]$ = 0.036 | $(\Delta/\sigma)_{max} < 0.001$ |
| $wR(F^2)$ = 0.099 | $\Delta\rho_{max}$ = 0.43 e Å$^{-3}$ |
| $S$ = 1.08 | $\Delta\rho_{min}$ = −0.32 e Å$^{-3}$ |
| 1774 reflections | |
| 109 parameters | |

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| Cl1—C2 | 1.722 (3) | C1—C2 | 1.397 (4) |
| Cl2—C3 | 1.712 (3) | C1—C6 | 1.398 (4) |
| Cl3—C4 | 1.710 (3) | C2—C3 | 1.385 (4) |
| Cl4—C5 | 1.717 (3) | C3—C4 | 1.398 (4) |
| Cl5—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) $1-x, y, \frac{1}{2}-z$.

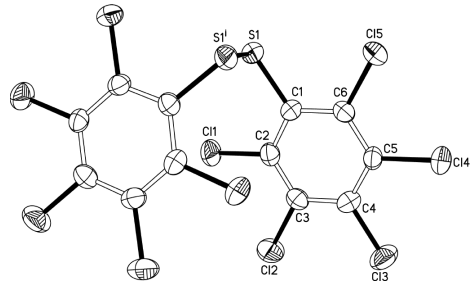



**Figure 1**
*ORTEP*II (Johnson, 1976) plot of (I), with displacement ellipsoids drawn at the 50% probability level. [Symmetry code: (i) $1-x, y, \frac{1}{2}-z$.]

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1988); cell refinement: *CAD*-4 *Software*; data reduction: *XCAD*4 (Harms, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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