

## 1,2-Bis[4-(4-chlorophenyl)-1,3-thiazol-2-yl]disulfane

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## Key indicators

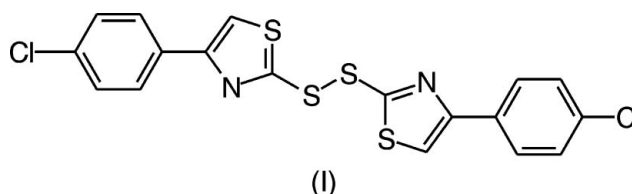
Single-crystal X-ray study  
 $T = 301$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.007$  Å  
 $R$  factor = 0.039  
 $wR$  factor = 0.118  
Data-to-parameter ratio = 11.2For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.In the title compound,  $\text{C}_{18}\text{H}_{10}\text{Cl}_2\text{N}_2\text{S}_4$ , a 1,2-substituted  
disulfane, the torsion angle between the *p*-chlorophenyl and  
the 1,3-thiazole rings is  $-2.0$  ( $6$ )° for the group attached to the  
the first disulfane S atom, and  $14.6$  ( $7$ )° for the substituent  
located at the second.

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## Comment

The title compound, (I) (Runge, 1963), was isolated as a side-  
product in reactions starting from *N*-hydroxy-4-(*p*-chloro-  
phenyl)thiazole-2(3*H*)-thione, tetraethylammonium 2-thioxo-  
2,3-dihydro-4-(*p*-chlorophenyl)thiazole-1-olate and *N*-(pival-  
oxyloxy)-4-(*p*-chlorophenyl)thiazole-2(3*H*)-thione (Hartung *et al.*,  
1999, 2003; Adam *et al.*, 2000). Compound (I) separates  
from a mixture of petroleum ether–EtOH as colourless  
needles, and these were investigated by X-ray diffraction in  
order to establish its structural details in the solid state.The S—S bond in (I) is a stereogenic axis (Fig. 1). Since (I)  
crystallizes in the monoclinic space group  $P2_1/c$ , the structure  
is that of a racemate. The disulfane dihedral angle (C12—S3—  
S2—C2) is  $89.1$  ( $2$ )°. The *p*-chlorophenyl and thiazyl rings in  
this stereoisomer are inclined by  $-2.0$  ( $6$ )° for the substituent  
attached at S2, and by  $14.6$  ( $7$ )° for the substituent at S3, as  
measured by appropriate torsion angles (Table 1). The bond  
lengths and angles in the 4-(*p*-chlorophenyl)-2-thiazyl sub-  
units at S2 and S3 are identical to within experimental error  
(Table 1). The exocyclic S—C bond distances (Table 1) are  
close to the mean value found for the  $\text{Csp}^2\text{—S}(2)$  bond  
[ $1.73$  ( $1$ ) Å; Allen *et al.*, 1987]. The S2—S3 bond length  
[ $2.040$  ( $2$ ) Å] agrees with values reported for structurally  
related 1,2-bis(heteroaryl)disulfanes (Furberg & Solbakk,  
1973; Bodige *et al.*, 1997).In the unit cell of (I), enantiomers of each hand are packed  
to form columns along [010] (Fig. 2). Along [100], compound  
(I) packs in sheets, forming alternating sequences of enanti-  
omers. Selected  $\text{Cl}\cdots\text{Cl}$  distances fall below the sum of the van  
der Waals radii for two Cl atoms (Bondi, 1964), thus leading to  
close contacts [ $\text{Cl}2\cdots\text{Cl}2^i = 3.210$  ( $7$ ) Å; symmetry code: (i)  
 $-x + 1, -y + 1, -z - 1$ ].

Experimental

1,2-Bis[4-(*p*-chlorophenyl)-1,3-thiaz-2-yl]disulfane, (I), was prepared as described previously (Hartung *et al.*, 1999, 2003; Adam *et al.*, 2000). Crystals suitable for X-ray diffraction were obtained by gradual concentration of a saturated solution of (I) in petroleum ether–EtOH at 298 K.

Crystal data

C<sub>18</sub>H<sub>10</sub>Cl<sub>2</sub>N<sub>2</sub>S<sub>4</sub>  $D_x = 1.587 \text{ Mg m}^{-3}$   
 $M_r = 453.44$  Mo  $K\alpha$  radiation  
 Monoclinic,  $P2_1/c$  Cell parameters from 25 reflections  
 $a = 22.643(2) \text{ \AA}$   $\theta = 4.4\text{--}11.8^\circ$   
 $b = 5.702(2) \text{ \AA}$   $\mu = 0.79 \text{ mm}^{-1}$   
 $c = 15.315(3) \text{ \AA}$   $T = 301(2) \text{ K}$   
 $\beta = 106.28(1)^\circ$  Needle, colourless  
 $V = 1898.0(8) \text{ \AA}^3$   $0.75 \times 0.08 \times 0.03 \text{ mm}$   
 $Z = 4$

Data collection

Enraf–Nonius CAD-4 diffractometer  $R_{\text{int}} = 0.031$   
 $\omega/2\theta$  scans  $\theta_{\text{max}} = 23.0^\circ$   
 Absorption correction:  $\psi$  scan (North *et al.*, 1968)  $h = -23 \rightarrow 24$   
 $T_{\text{min}} = 0.617$ ,  $T_{\text{max}} = 0.982$   $k = -6 \rightarrow 0$   
 2751 measured reflections  $l = -16 \rightarrow 1$   
 2626 independent reflections 3 standard reflections  
 1362 reflections with  $I > 2\sigma(I)$  frequency: 120 min  
 intensity decay: 0.7%

Refinement

Refinement on  $F^2$  H-atom parameters constrained  
 $R[F^2 > 2\sigma(F^2)] = 0.039$   $w = 1/[\sigma^2(F_o^2) + (0.0594P)^2]$   
 $wR(F^2) = 0.118$  where  $P = (F_o^2 + 2F_c^2)/3$   
 $S = 0.97$   $(\Delta/\sigma)_{\text{max}} < 0.001$   
 2626 reflections  $\Delta\rho_{\text{max}} = 0.22 \text{ e \AA}^{-3}$   
 235 parameters  $\Delta\rho_{\text{min}} = -0.26 \text{ e \AA}^{-3}$

Table 1 Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

C2–N3	1.285 (5)	C12–N13	1.293 (6)
C2–S1	1.735 (5)	C12–S4	1.734 (5)
C2–S2	1.756 (5)	C12–S3	1.758 (5)
C4–C5	1.352 (6)	C14–C15	1.354 (7)
C4–N3	1.389 (6)	C14–N13	1.396 (6)
C4–C6	1.463 (6)	C14–C16	1.460 (7)
C5–S1	1.703 (5)	C15–S4	1.696 (6)
C9–C11	1.739 (5)	S2–S3	2.040 (2)
N3–C2–S1	115.7 (4)	C14–C15–S4	112.7 (4)
N3–C2–S2	121.1 (4)	C2–N3–C4	110.9 (4)
C5–C4–N3	113.3 (4)	C5–S1–C2	87.7 (2)
C4–C5–S1	112.4 (4)	C2–S2–S3	102.0 (2)
N13–C12–S4	115.3 (4)	C12–S3–S2	101.7 (2)
N13–C12–S3	121.2 (4)	C15–S4–C12	88.1 (3)
C15–C14–N13	112.9 (5)		
N3–C4–C6–C11	–2.0 (6)	S4–C12–S3–S2	–15.5 (3)
N13–C14–C16–C17	14.6 (7)	C2–S2–S3–C12	–89.1 (2)

All H atoms were positioned geometrically and treated as riding atoms, with C–H = 0.93  $\text{\AA}$  and with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

Data collection: CAD-4 Diffractometer Control Software (Enraf–Nonius, 1993); cell refinement: CAD-4 Diffractometer Control Software; data reduction: CAD-4 Diffractometer Control Software; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997);

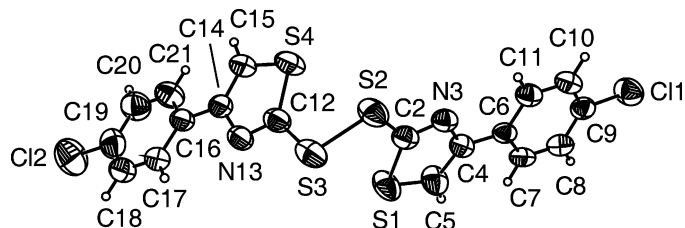


Figure 1 The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level.

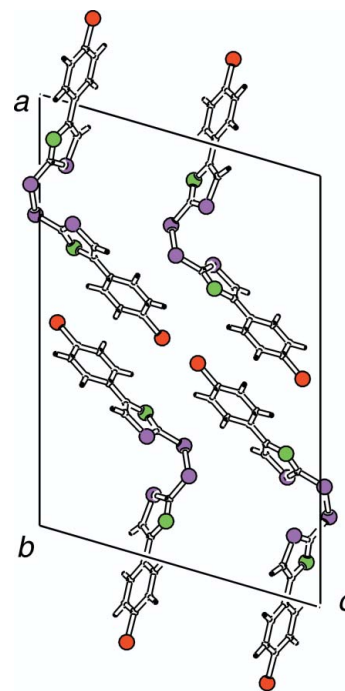


Figure 2 The packing of (I) in the solid state, viewed along [010]. S atoms are depicted in purple, Cl in orange and N in green.

molecular graphics: PLATON (Spek, 2003) and ORTEP3 (Farrugia, 1997); software used to prepare material for publication: SHELXL97.

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