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A second polymorph of 1,2-bis[4-(4-chlorophenyl)-1,3-thiazol-2-yl]disulfane

Jens Hartung,[a] Kristina Daniel,[a] Philipp Schmidt,[b] Stefanie Laug,[b] Ingrid Svoboda[c] and Hartmut Fuess[c]*

[a]Fachbereich Chemie, Organische Chemie, Technische Universität Kaiserslautern, Erwin-Schrödinger-Strasse, D-67663 Kaiserslautern, Germany, [b]Institut für Organische Chemie, Universität Würzburg, Am Hubland, D-97074 Würzburg, Germany, and [c]Strukturforschung, FB11 Material- und Geowissenschaften, Technische Universität Darmstadt, Petersenstrasse 23, D-64287 Darmstadt, Germany

Correspondence e-mail: hartung@chemie.uni-kl.de

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Key indicators

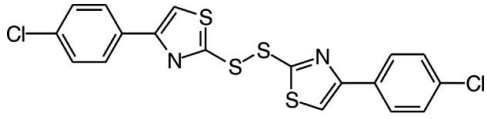
Single-crystal X-ray study
T = 299 K
Mean σ(C–C) = 0.007 Å
R factor = 0.039
wR factor = 0.123
Data-to-parameter ratio = 11.3

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

The asymmetric unit of the title compound, $C_{18}H_{10}Cl_2N_2S_4$, consists of one full molecule and two half molecules, which are completed by twofold rotation symmetry.

Comment

The title racemate of (I) has been isolated from photochemical transformations of, for example, 1-hydroxy- or 1-pivalyloxy-4-(*p*-chlorophenyl)thiazole-2(3*H*)-thione (Hartung *et al.*, 2005) and its structure is reported here.



(I)

Compound (I) (Fig. 1), which crystallizes in the space group $C2/c$, is a polymorph of the first monoclinic modification ($P2_1/c$) reported in the preceding paper (Hartung *et al.*, 2005). The asymmetric units consists of one full molecule and two half molecules, which are completed by twofold rotation symmetry. This arrangement gives rise to three different disulfane torsion angles and ligand configurations at the S atoms (Fig. 2). Different interplanar angles between the 1,3-thiaz-2-yl group and the *p*-chlorophenyl plane are found in the two S-bound substituents for molecule 1 (Fig. 2). By symmetry, identical interplanar angles are present in each half of molecules 2 and 3, although the absolute values differ between the two molecules. The bond lengths and angles (Table 1) are similar but not identical to the values reported for (I) in its first monoclinic modification (Hartung *et al.*, 2005).

The structure comprises a racemic mixture of *P*- and *M*-enantiomers (Figs. 2 and 3). The *P*-(I) and *M*-(I) enantiomers form separate stacks, as seen in the view along [010]. Packing within these stacks occurs with an approximate 90° twist between the *p*-chlorophenyl entities of adjacent layers (Fig. 3). The distance between stacks of enantiomers falls below the the sum of the van der Waals radii of two Cl atoms [Cl1$\cdots$Cl2^{i} = 3.357 (6) Å and Cl3$\cdots$Cl4^{i} = 3.389 (7) Å; Bondi, 1964; symmetry code: (i) $x-\frac{1}{2}, -y+\frac{1}{2}, z+\frac{1}{2}$; (ii) $x-1, y-1, z$].

Experimental

The title compound, (I), was prepared as described previously (Hartung *et al.*, 1999, 2003; Adam *et al.*, 2000). The compound (Runge, 1963) crystallizes as colourless needles by the gradual concentration of a saturated solution of (I) in petroleum ether–Et_2O at 298 K.

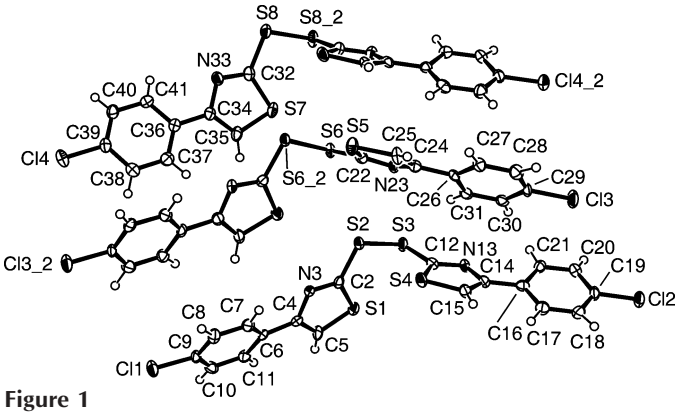
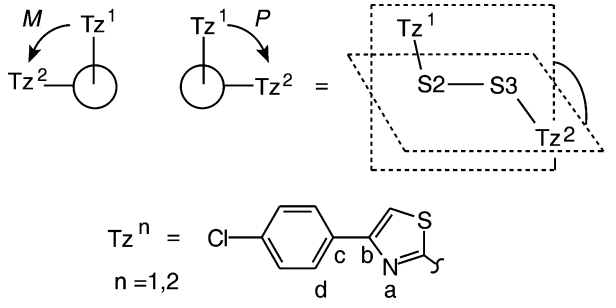


Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level. In the top molecule _2 indicates the symmetry position1 $-x, y, \frac{3}{2}-z$ and in the middle molecule _2 indicates the symmetry position $-x, y, \frac{1}{2}-z$



	Tz1–S–S–Tz2 (°)	a–b–c–d (°)
molecule 1	92.2 (2)	17.7 (6) for Tz1 –15.0 (6) for Tz2
molecule 2	102.6 (3)	18.4 (6) for Tz1 and Tz2
molecule 3	85.3 (2)	–21.4 (6) for Tz1 and Tz2

Figure 2
Conformational aspects of independent molecules of *P*-(I).

Crystal data

$C_{18}H_{10}Cl_2N_2S_4$
M_r = 453.44
Monoclinic, $C2/c$
a = 20.293 (3) Å
b = 9.999 (3) Å
c = 37.594 (3) Å
β = 91.53 (1)°
V = 7625 (3) Å^3
Z = 16
D_x = 1.580 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 4.3–12.3°
μ = 0.78 mm^{-1}
T = 299 (2) K
Needle, colourless
0.55 × 0.22 × 0.05 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.696, T_{max} = 0.965
7300 measured reflections
5282 independent reflections
3152 reflections with $I > 2\sigma(I)$
R_{int} = 0.048
θ_{max} = 23.0°
$h = -22 \rightarrow 6$
$k = -10 \rightarrow 0$
$l = -41 \rightarrow 41$
3 standard reflections
frequency: 120 min
intensity decay: 3.1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.123
S = 0.99
5282 reflections
469 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.057P)^2 + 7.2189P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.005
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C2—N3	1.306 (5)	C22—S6	1.756 (5)
C2—S1	1.725 (4)	C24—C25	1.360 (6)
C2—S2	1.750 (4)	C24—N23	1.381 (6)
C4—C5	1.364 (6)	C24—C26	1.468 (6)
C4—N3	1.388 (5)	C25—S5	1.705 (5)
C4—C6	1.466 (6)	C29—Cl3	1.733 (5)
C5—S1	1.707 (5)	C32—N33	1.296 (5)
C9—Cl1	1.739 (5)	C32—S7	1.722 (4)
C12—N13	1.298 (5)	C32—S8	1.761 (4)
C12—S4	1.731 (4)	C34—C35	1.355 (6)
C12—S3	1.759 (5)	C34—N33	1.383 (5)
C14—C15	1.359 (6)	C34—C36	1.471 (6)
C14—N13	1.379 (5)	C35—S7	1.702 (5)
C14—C16	1.474 (6)	C39—Cl4	1.745 (5)
C15—S4	1.692 (5)	S2—S3	2.028 (2)
C19—Cl2	1.749 (5)	S6—S6^{i}	2.026 (2)
C22—N23	1.297 (5)	S8—S8^{ii}	2.031 (2)
C22—S5	1.725 (5)		
N3—C2—S1	115.3 (3)	N33—C32—S8	120.6 (3)
N3—C2—S2	120.7 (3)	S7—C32—S8	122.8 (2)
S1—C2—S2	124.0 (2)	C35—C34—N33	113.6 (4)
C5—C4—N3	113.9 (4)	C34—C35—S7	112.3 (4)
C4—C5—S1	111.4 (4)	C2—N3—C4	110.6 (3)
N13—C12—S4	115.1 (3)	C12—N13—C14	110.8 (3)
N13—C12—S3	120.0 (3)	C22—N23—C24	110.8 (4)
S4—C12—S3	124.7 (2)	C32—N33—C34	110.2 (3)
C15—C14—N13	113.6 (4)	C5—S1—C2	88.8 (2)
C14—C15—S4	112.1 (3)	C2—S2—S3	101.9 (1)
N23—C22—S5	115.4 (4)	C12—S3—S2	102.9 (1)
N23—C22—S6	119.9 (4)	C15—S4—C12	88.3 (2)
S5—C22—S6	124.7 (3)	C25—S5—C22	88.4 (2)
C25—C24—N23	113.8 (4)	C22—S6—S6^{i}	102.8 (2)
C24—C25—S5	111.6 (4)	C35—S7—C32	87.8 (2)
N33—C32—S7	116.2 (3)	C32—S8—S8^{ii}	101.7 (1)
N3—C4—C6—C7	−15.0 (6)	N33—C34—C36—C41	−18.4 (6)
N13—C14—C16—C21	17.7 (6)	C2—S2—S3—C12	92.2 (2)
N23—C24—C26—C31	−21.4 (6)		

Symmetry codes: (i) $-x, y, -z+\frac{1}{2}$; (ii) $-x+1, y, -z+\frac{3}{2}$.

All H atoms were positioned geometrically and treated as riding atoms, with C—H = 0.93 Å and with U_{iso}(H) = 1.2U_{eq}(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); molecular graphics: *PLATON* (Spek, 2003) and *ORTEP3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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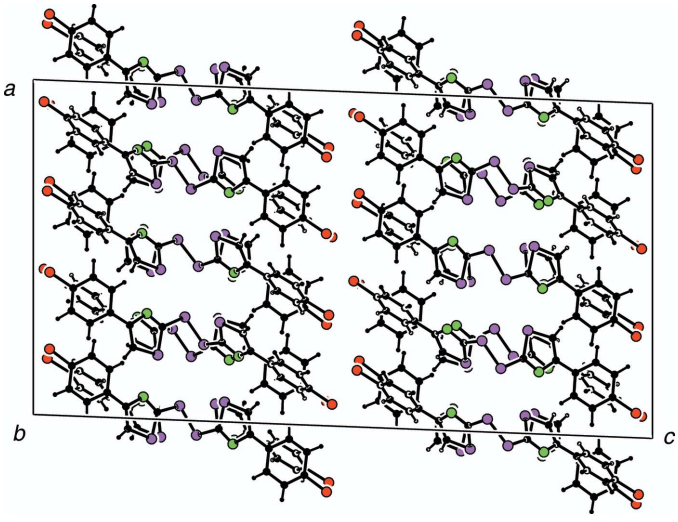

Figure 3
The packing of (I) in the unit cell, viewed along [010]. S atoms are depicted in purple, Cl in orange and N in green.

Reaktionen Metall-aktivierter Moleküle; Graduiertenkolleg 690: Elektronendichte – Theorie und Experiment).

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