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Bis(4-amino-2-chlorophenyl) disulfide

 Jun-Mei Tang,^{a*} Zhi-Qiang Feng^b and Wei Cheng^a

^aDepartment of Biological and Chemical Engineering, Chien-shiung Institute of Technology, Taicang 215411, Suzhou, People's Republic of China, and ^bSchool of Materials Engineering, Jinling Institute of Technology, Nanjing 211169, People's Republic of China

Correspondence e-mail: Tangjm_83@126.com

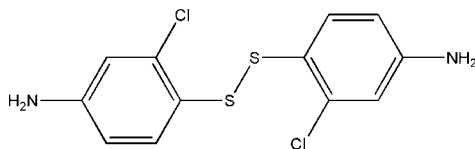
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.006$ Å; R factor = 0.031; wR factor = 0.088; data-to-parameter ratio = 8.2.

The title compound, $\text{C}_{12}\text{H}_{10}\text{Cl}_2\text{N}_2\text{S}_2$, features an S—S bond [2.0671 (16) Å] that bridges two 4-amino-2-chlorophenyl rings with a C—S—S—C torsion angle of -84.2 (2)°. The two benzene rings are twisted with respect to each other at a dihedral angle of 39.9 (2)°. Intermolecular N—H...S hydrogen bonding is present in the crystal structure.

Related literature

For the application of the title compound, see: Crowley (1964). For S—S bond distances, see: Allen *et al.* (1991). For similar C—S—S—C torsion angles in disulfide compounds, see: Korp & Bernal (1984); Poveteva & Zvonkova (1975).



Experimental

Crystal data

 $\text{C}_{12}\text{H}_{10}\text{Cl}_2\text{N}_2\text{S}_2$
 $M_r = 317.24$

 Monoclinic, Cc
 $a = 6.6360$ (13) Å

 $b = 14.907$ (3) Å

 $c = 13.588$ (3) Å

 $\beta = 95.09$ (3)°

 $V = 1338.9$ (5) Å³
 $Z = 4$

 Mo $K\alpha$ radiation

 $\mu = 0.78$ mm⁻¹
 $T = 296$ K

 $0.30 \times 0.20 \times 0.10$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.800$, $T_{\max} = 0.940$
2606 measured reflections

1331 independent reflections
1221 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.026$
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.031$
 $wR(F^2) = 0.088$
 $S = 1.00$
1331 reflections
163 parameters
2 restraints

H-atom parameters constrained
 $\Delta\rho_{\max} = 0.19$ e Å⁻³
 $\Delta\rho_{\min} = -0.22$ e Å⁻³
Absolute structure: Flack (1983), 110 Friedel pairs
Flack parameter: 0.09 (11)

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1A}\cdots\text{S1}^{\text{i}}$	0.86	2.80	3.611 (5)	158
$\text{N2}-\text{H2A}\cdots\text{S2}^{\text{ii}}$	0.86	2.86	3.684 (5)	162

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1985); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

The authors thank the Center of Testing and Analysis, Nanjing University, for data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5174).

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supporting information

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Bis(4-amino-2-chlorophenyl) disulfide

Jun-Mei Tang, Zhi-Qiang Feng and Wei Cheng

S1. Comment

The title compound has been used as fungicide and mildew-proofing agent (Crowley, 1964). We herein report its crystal structure. The S-S distance, 2.0670 (13) Å, is normal and falls within the range of 2.018–2.099 Å found for the acyclic disulfides in the Cambridge Structural Database (Allen *et al.*, 1991). The torsion angle C-S-S-C of 84.2 (2)° is close to the 85.0° found in diphenyldisulfide (Korp & Bernal, 1984) and lower than the 101.7° found in 4-amino-4'-nitrodiphenyl disulfide (Poveteva & Zvonkova, 1975). The intermolecular N–H···S hydrogen bonds may be effective in the stabilization of the crystal structure.

S2. Experimental

The aqueous solution (20 ml) of 3,4-dichloronitrobenzene (19.2 g, 0.1 mol) and sodium sulfhydrate (28.5 g, 0.22 mol) was refluxed for 16 h, and then filtered. The title compound was obtained from the filtrate. The single crystals were obtained by recrystallization from an ethanol solution after 5 d.

S3. Refinement

H atoms were positioned geometrically with N–H = 0.86 and C–H = 0.93 Å, and constrained to ride on their parent atoms with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$. As a half of reciprocal space diffraction data were collected only using a four-circle diffractometer, Friedel pair coverage is low in this determination.

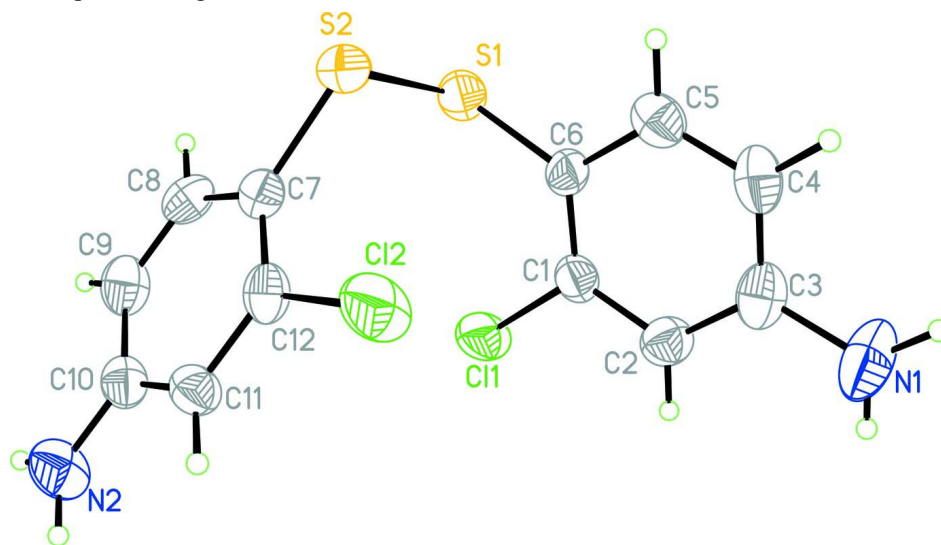


Figure 1

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

4-[(4-amino-2-chlorophenyl)disulfanyl]-3-chloroaniline

Crystal data

C₁₂H₁₀Cl₂N₂S₂ $M_r = 317.24$ Monoclinic, *Cc*

Hall symbol: C -2yc

 $a = 6.6360$ (13) Å $b = 14.907$ (3) Å $c = 13.588$ (3) Å $\beta = 95.09$ (3)° $V = 1338.9$ (5) Å³ $Z = 4$ $F(000) = 648$ $D_x = 1.574$ Mg m⁻³Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 25 reflections

 $\theta = 10\text{--}14^\circ$ $\mu = 0.78$ mm⁻¹ $T = 296$ K

Block, yellow

 $0.30 \times 0.20 \times 0.10$ mm

Data collection

Enraf–Nonius CAD-4

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 $\omega/2\theta$ scansAbsorption correction: ψ scan
(North *et al.*, 1968) $T_{\min} = 0.800$, $T_{\max} = 0.940$

2606 measured reflections

1331 independent reflections

1221 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.026$ $\theta_{\max} = 25.4^\circ$, $\theta_{\min} = 2.7^\circ$ $h = 0 \rightarrow 7$ $k = -17 \rightarrow 17$ $l = -16 \rightarrow 16$

3 standard reflections every 200 reflections

intensity decay: 1%

Refinement

Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.031$ $wR(F^2) = 0.088$ $S = 1.00$

1331 reflections

163 parameters

2 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.066P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.19$ e Å⁻³ $\Delta\rho_{\min} = -0.22$ e Å⁻³Absolute structure: Flack (1983), 110 Friedel
pairs

Absolute structure parameter: 0.09 (11)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2 , conventional R-factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F , and R-factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.28662 (15)	0.23231 (7)	0.80302 (8)	0.0442 (3)
Cl1	-0.1503 (2)	0.18083 (8)	0.87818 (12)	0.0609 (4)

N1	-0.3017 (9)	0.5116 (3)	0.9000 (3)	0.0681 (14)
H1A	-0.2710	0.5674	0.8950	0.082*
H1B	-0.4135	0.4965	0.9229	0.082*
C1	-0.0811 (7)	0.2917 (3)	0.8603 (3)	0.0380 (9)
Cl2	-0.2000 (2)	0.26890 (7)	0.57856 (11)	0.0586 (4)
S2	0.26156 (17)	0.21357 (8)	0.65170 (9)	0.0441 (3)
N2	-0.3804 (7)	-0.0596 (3)	0.5629 (3)	0.0525 (10)
H2A	-0.3514	-0.1157	0.5684	0.063*
H2B	-0.5012	-0.0431	0.5426	0.063*
C2	-0.2194 (8)	0.3561 (3)	0.8835 (3)	0.0431 (10)
H2C	-0.3415	0.3397	0.9070	0.052*
C3	-0.1716 (8)	0.4459 (3)	0.8708 (3)	0.0453 (11)
C4	0.0098 (8)	0.4693 (3)	0.8332 (3)	0.0455 (11)
H4A	0.0401	0.5292	0.8226	0.055*
C5	0.1417 (8)	0.4043 (3)	0.8121 (3)	0.0425 (10)
H5A	0.2634	0.4208	0.7881	0.051*
C6	0.1012 (6)	0.3126 (3)	0.8252 (3)	0.0358 (9)
C7	0.0675 (7)	0.1343 (3)	0.6287 (3)	0.0364 (9)
C8	0.1075 (7)	0.0430 (3)	0.6402 (3)	0.0418 (10)
H8A	0.2376	0.0250	0.6627	0.050*
C9	-0.0379 (8)	-0.0212 (3)	0.6193 (3)	0.0464 (11)
H9A	-0.0047	-0.0815	0.6277	0.056*
C10	-0.2349 (7)	0.0029 (3)	0.5857 (3)	0.0379 (10)
C11	-0.2776 (7)	0.0940 (3)	0.5730 (3)	0.0371 (9)
H11A	-0.4068	0.1122	0.5490	0.044*
C12	-0.1295 (7)	0.1569 (3)	0.5957 (3)	0.0377 (10)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0341 (6)	0.0425 (6)	0.0543 (6)	0.0054 (5)	-0.0052 (5)	-0.0064 (5)
Cl1	0.0460 (6)	0.0332 (5)	0.1040 (10)	-0.0052 (5)	0.0090 (6)	0.0041 (6)
N1	0.088 (4)	0.049 (2)	0.071 (3)	0.026 (3)	0.027 (3)	0.007 (2)
C1	0.036 (2)	0.031 (2)	0.046 (2)	0.0004 (18)	-0.0038 (19)	-0.0030 (17)
Cl2	0.0485 (7)	0.0276 (5)	0.0974 (10)	0.0017 (5)	-0.0065 (6)	0.0012 (6)
S2	0.0369 (6)	0.0427 (6)	0.0538 (6)	-0.0042 (5)	0.0115 (5)	-0.0047 (5)
N2	0.057 (2)	0.0300 (19)	0.069 (3)	-0.0092 (18)	-0.001 (2)	0.0023 (18)
C2	0.038 (2)	0.044 (2)	0.048 (2)	0.003 (2)	0.0039 (19)	0.003 (2)
C3	0.060 (3)	0.039 (2)	0.036 (2)	0.011 (2)	0.000 (2)	0.0018 (19)
C4	0.065 (3)	0.030 (2)	0.042 (2)	0.002 (2)	0.003 (2)	0.0003 (18)
C5	0.046 (3)	0.041 (2)	0.040 (2)	-0.007 (2)	0.003 (2)	0.0019 (18)
C6	0.030 (2)	0.033 (2)	0.043 (2)	0.0057 (17)	-0.0024 (17)	-0.0062 (16)
C7	0.037 (2)	0.035 (2)	0.038 (2)	0.0007 (18)	0.0072 (17)	-0.0044 (17)
C8	0.036 (2)	0.044 (2)	0.047 (2)	0.012 (2)	0.0091 (19)	0.0015 (19)
C9	0.060 (3)	0.028 (2)	0.051 (3)	0.005 (2)	0.006 (2)	-0.0003 (17)
C10	0.043 (3)	0.036 (2)	0.034 (2)	-0.0029 (18)	0.005 (2)	-0.0024 (17)
C11	0.035 (2)	0.034 (2)	0.042 (2)	-0.0049 (17)	0.0024 (18)	-0.0014 (17)
C12	0.048 (3)	0.0219 (19)	0.043 (2)	0.0055 (19)	0.0043 (19)	-0.0025 (16)

Geometric parameters (Å, °)

S1—C6	1.762 (4)	C3—C4	1.393 (7)
S1—S2	2.0671 (16)	C4—C5	1.354 (7)
C11—C1	1.738 (4)	C4—H4A	0.9300
N1—C3	1.387 (6)	C5—C6	1.407 (6)
N1—H1A	0.8601	C5—H5A	0.9300
N1—H1B	0.8599	C7—C12	1.385 (7)
C1—C6	1.375 (7)	C7—C8	1.393 (6)
C1—C2	1.384 (6)	C8—C9	1.371 (7)
C12—C12	1.744 (4)	C8—H8A	0.9300
S2—C7	1.755 (4)	C9—C10	1.393 (7)
N2—C10	1.357 (6)	C9—H9A	0.9300
N2—H2A	0.8599	C10—C11	1.396 (6)
N2—H2B	0.8600	C11—C12	1.374 (7)
C2—C3	1.390 (6)	C11—H11A	0.9300
C2—H2C	0.9300		
C6—S1—S2	105.43 (14)	C6—C5—H5A	118.9
C3—N1—H1A	120.2	C1—C6—C5	116.6 (4)
C3—N1—H1B	119.8	C1—C6—S1	123.7 (3)
H1A—N1—H1B	120.0	C5—C6—S1	119.6 (4)
C6—C1—C2	122.9 (4)	C12—C7—C8	116.0 (4)
C6—C1—C11	121.0 (3)	C12—C7—S2	123.4 (3)
C2—C1—C11	116.1 (4)	C8—C7—S2	120.5 (4)
C7—S2—S1	105.13 (15)	C9—C8—C7	122.4 (4)
C10—N2—H2A	119.9	C9—C8—H8A	118.8
C10—N2—H2B	120.1	C7—C8—H8A	118.8
H2A—N2—H2B	120.0	C8—C9—C10	120.7 (4)
C1—C2—C3	118.5 (5)	C8—C9—H9A	119.7
C1—C2—H2C	120.8	C10—C9—H9A	119.7
C3—C2—H2C	120.8	N2—C10—C9	121.7 (4)
N1—C3—C2	119.4 (5)	N2—C10—C11	120.5 (4)
N1—C3—C4	120.6 (4)	C9—C10—C11	117.8 (4)
C2—C3—C4	120.0 (4)	C12—C11—C10	120.2 (4)
C5—C4—C3	119.7 (4)	C12—C11—H11A	119.9
C5—C4—H4A	120.2	C10—C11—H11A	119.9
C3—C4—H4A	120.2	C11—C12—C7	122.9 (4)
C4—C5—C6	122.3 (5)	C11—C12—C12	116.4 (4)
C4—C5—H5A	118.9	C7—C12—C12	120.7 (3)
C6—S1—S2—C7	-84.2 (2)	S1—S2—C7—C12	99.7 (4)
C6—C1—C2—C3	-0.1 (7)	S1—S2—C7—C8	-82.5 (3)
C11—C1—C2—C3	179.9 (4)	C12—C7—C8—C9	0.6 (6)
C1—C2—C3—N1	-175.6 (4)	S2—C7—C8—C9	-177.4 (3)
C1—C2—C3—C4	1.6 (7)	C7—C8—C9—C10	-0.2 (6)
N1—C3—C4—C5	175.0 (4)	C8—C9—C10—N2	178.8 (4)
C2—C3—C4—C5	-2.1 (7)	C8—C9—C10—C11	0.8 (6)

C3—C4—C5—C6	1.1 (6)	N2—C10—C11—C12	-179.7 (4)
C2—C1—C6—C5	-0.8 (6)	C9—C10—C11—C12	-1.7 (6)
C11—C1—C6—C5	179.2 (3)	C10—C11—C12—C7	2.1 (6)
C2—C1—C6—S1	175.6 (3)	C10—C11—C12—C12	-179.2 (3)
C11—C1—C6—S1	-4.4 (5)	C8—C7—C12—C11	-1.5 (6)
C4—C5—C6—C1	0.3 (6)	S2—C7—C12—C11	176.4 (3)
C4—C5—C6—S1	-176.3 (3)	C8—C7—C12—C12	179.8 (3)
S2—S1—C6—C1	102.4 (3)	S2—C7—C12—C12	-2.2 (5)
S2—S1—C6—C5	-81.3 (3)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1 <i>A</i> ...S1 ⁱ	0.86	2.80	3.611 (5)	158
N2—H2 <i>A</i> ...S2 ⁱⁱ	0.86	2.86	3.684 (5)	162

Symmetry codes: (i) $x-1/2, y+1/2, z$; (ii) $x-1/2, y-1/2, z$.