

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

2,4-Disulfanyl-6-[(*E*)-(2-sulfanylbenzyl)-iminomethyl]phenol

Yong-Ming Cui, Xi-Bin Dai, Lei Lei and Qing-Fu Zeng*

Engineering Research Center for Clean Production of Textile Dyeing and Printing, Ministry of Education, Wuhan 430073, People's Republic of China
Correspondence e-mail: qfzeng@wuse.edu.cn

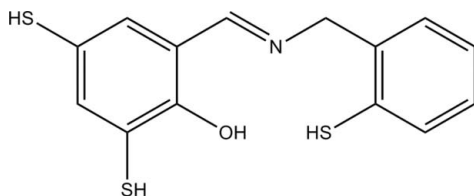
Received 12 August 2009; accepted 13 August 2009

Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.047; wR factor = 0.140; data-to-parameter ratio = 13.9.

In the title compound, $\text{C}_{14}\text{H}_{13}\text{NOS}_3$, the dihedral angle between the benzene rings is $73.26(5)^\circ$ and an intramolecular $\text{O}-\text{H}\cdots\text{N}$ hydrogen bond occurs.

Related literature

For background, see: Shi *et al.* (2007). For reference structural data, see: Allen *et al.* (1987);



Experimental

Crystal data

$\text{C}_{14}\text{H}_{13}\text{NOS}_3$
 $M_r = 307.43$
Monoclinic, $P2_1/c$

$a = 11.9763(13)$ Å
 $b = 8.2333(13)$ Å
 $c = 14.2213(13)$ Å

$\beta = 98.723(3)^\circ$
 $V = 1386.1(3)$ Å³
 $Z = 4$
Mo $K\alpha$ radiation

$\mu = 0.52$ mm⁻¹
 $T = 296$ K
 $0.28 \times 0.25 \times 0.25$ mm

Data collection

Enraf-Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.867$, $T_{\max} = 0.880$

7137 measured reflections
2443 independent reflections
1929 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.047$
 $wR(F^2) = 0.140$
 $S = 1.06$
2443 reflections

176 parameters
H-atom parameters constrained
 $\Delta\rho_{\max} = 0.28$ e Å⁻³
 $\Delta\rho_{\min} = -0.39$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O1}-\text{H1}\cdots\text{N1}$	0.82	1.87	2.591 (3)	147

Data collection: *CAD-4 Software* (Enraf-Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5045).

References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Enraf-Nonius (1989). *CAD-4 Software*. Enraf-Nonius, Delft, The Netherlands.
Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
Shi, L., Ge, H.-M., Tan, S.-H., Li, H.-Q., Song, Y.-C., Zhu, H.-L. & Tan, R.-X. (2007). *Eur. J. Med. Chem.* **42**, 558–564.

supplementary materials

Acta Cryst. (2009). E65, o2179 [doi:10.1107/S160053680903205X]

2,4-Disulfanyl-6-[(*E*)-(2-sulfanylbenzyl)iminomethyl]phenol

Y.-M. Cui, X.-B. Dai, L. Lei and Q.-F. Zeng

Comment

There has been much research interest in Schiff base compounds due to their biological activities (Shi *et al.*, 2007). In this work, we report here the crystal structure of the title compound, (I). In (I), all bond lengths are within normal ranges (Allen *et al.*, 1987) (Fig. 1). There is an intramolecular O—H···N hydrogen bond (Table 1) in (I). The dihedral angle between the two benzene rings is 73.26 (0.05) °.

Experimental

A mixture of 2-hydroxy-3,5-disulfanylbenzaldehyde (186 mg, 1 mmol) and 2-(aminomethyl)benzenethiol (139 mg, 1 mmol) in methanol (10 ml) was stirred for 2 h. After keeping the filtrate in air for 6 d, yellow blocks of (I) were formed.

Refinement

All H atoms were positioned geometrically (C—H = 0.93–0.97 Å, S—H = 1.20 Å) and refined as riding with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{carrier})$ or $1.5U_{\text{eq}}(\text{methyl C})$.

Figures

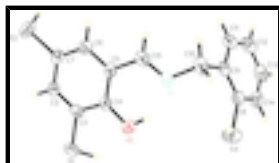


Fig. 1. The molecular structure of (I) showing 30% probability displacement ellipsoids.

2,4-Disulfanyl-6-[(*E*)-(2-sulfanylbenzyl)iminomethyl]phenol

Crystal data

C₁₄H₁₃NOS₃

$M_r = 307.43$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 11.9763$ (13) Å

$b = 8.2333$ (13) Å

$c = 14.2213$ (13) Å

$\beta = 98.723$ (3)°

$V = 1386.1$ (3) Å³

$Z = 4$

$F_{000} = 640$

$D_x = 1.473$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 25 reflections

$\theta = 9$ –12°

$\mu = 0.52$ mm⁻¹

$T = 296$ K

Block, yellow

0.28 × 0.25 × 0.25 mm

Data collection

Enraf–Nonius CAD-4 diffractometer	2443 independent reflections
Radiation source: fine-focus sealed tube	1929 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.025$
$T = 296$ K	$\theta_{\text{max}} = 25.0^\circ$
$\omega/2\theta$ scans	$\theta_{\text{min}} = 1.7^\circ$
Absorption correction: ψ scan (North <i>et al.</i> , 1968)	$h = -14 \rightarrow 10$
$T_{\text{min}} = 0.867$, $T_{\text{max}} = 0.880$	$k = -9 \rightarrow 9$
7137 measured reflections	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.047$	H-atom parameters constrained
$wR(F^2) = 0.140$	$w = 1/[\sigma^2(F_o^2) + (0.0746P)^2 + 0.5508P]$
$S = 1.06$	where $P = (F_o^2 + 2F_c^2)/3$
2443 reflections	$(\Delta/\sigma)_{\text{max}} = 0.002$
176 parameters	$\Delta\rho_{\text{max}} = 0.28 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.39 \text{ e } \text{\AA}^{-3}$
	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C2	0.3001 (2)	0.0386 (3)	-0.0207 (2)	0.0502 (6)
H2	0.2415	-0.0310	-0.0118	0.060*
C3	0.3723 (2)	0.0988 (3)	0.05602 (18)	0.0468 (6)
C4	0.46151 (19)	0.2023 (3)	0.04542 (18)	0.0441 (6)
C5	0.4777 (2)	0.2435 (3)	-0.04744 (18)	0.0448 (6)

C6	0.4044 (2)	0.1839 (3)	-0.1250 (2)	0.0532 (7)
H6	0.4148	0.2118	-0.1864	0.064*
C7	0.3166 (2)	0.0837 (3)	-0.1110 (2)	0.0538 (7)
C8	0.9011 (2)	0.3750 (3)	0.1048 (2)	0.0551 (7)
C9	0.8496 (2)	0.3907 (3)	0.0113 (2)	0.0478 (6)
C10	0.7427 (2)	0.4868 (3)	-0.0169 (3)	0.0605 (8)
H10A	0.7476	0.5890	0.0174	0.073*
H10B	0.7333	0.5106	-0.0844	0.073*
C11	0.8989 (2)	0.3091 (3)	-0.0574 (2)	0.0594 (7)
H11	0.8660	0.3151	-0.1209	0.071*
C12	0.9966 (3)	0.2189 (4)	-0.0324 (3)	0.0718 (9)
H12	1.0297	0.1668	-0.0792	0.086*
C13	1.0442 (3)	0.2064 (4)	0.0608 (3)	0.0726 (9)
H13	1.1092	0.1445	0.0772	0.087*
C14	0.9971 (3)	0.2840 (4)	0.1300 (2)	0.0690 (8)
H14	1.0297	0.2753	0.1934	0.083*
C15	0.5736 (2)	0.3436 (3)	-0.0638 (2)	0.0523 (7)
H15	0.5821	0.3710	-0.1257	0.063*
N1	0.64557 (17)	0.3939 (3)	0.00480 (18)	0.0541 (6)
O1	0.52860 (16)	0.2588 (2)	0.12252 (13)	0.0597 (5)
H1	0.5820	0.3078	0.1060	0.090*
S1	0.35256 (7)	0.04078 (11)	0.16895 (5)	0.0677 (3)
H1A	0.4408	-0.0037	0.2128	0.102*
S2	0.22437 (8)	0.01029 (13)	-0.20692 (7)	0.0860 (4)
H2A	0.2734	-0.0823	-0.2515	0.129*
S3	0.84366 (10)	0.47293 (14)	0.19417 (7)	0.0939 (4)
H3A	0.8005	0.5977	0.1640	0.141*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C2	0.0407 (14)	0.0441 (14)	0.0657 (18)	0.0040 (11)	0.0080 (12)	-0.0050 (12)
C3	0.0450 (14)	0.0445 (14)	0.0537 (15)	0.0098 (11)	0.0161 (11)	-0.0037 (11)
C4	0.0379 (13)	0.0410 (13)	0.0531 (15)	0.0074 (10)	0.0060 (11)	-0.0070 (11)
C5	0.0394 (13)	0.0392 (13)	0.0555 (15)	0.0099 (10)	0.0059 (11)	0.0044 (11)
C6	0.0543 (16)	0.0523 (15)	0.0514 (15)	0.0073 (12)	0.0029 (12)	0.0093 (12)
C7	0.0475 (15)	0.0527 (15)	0.0575 (17)	0.0041 (12)	-0.0037 (12)	-0.0010 (13)
C8	0.0483 (15)	0.0544 (16)	0.0643 (18)	-0.0054 (13)	0.0144 (13)	-0.0064 (13)
C9	0.0399 (13)	0.0369 (13)	0.0683 (17)	-0.0032 (10)	0.0136 (12)	0.0039 (11)
C10	0.0440 (15)	0.0444 (15)	0.095 (2)	0.0030 (11)	0.0151 (14)	0.0117 (14)
C11	0.0616 (17)	0.0542 (16)	0.0662 (18)	0.0002 (13)	0.0223 (14)	0.0053 (13)
C12	0.072 (2)	0.0588 (18)	0.094 (2)	0.0109 (15)	0.0409 (19)	-0.0026 (17)
C13	0.0478 (17)	0.067 (2)	0.105 (3)	0.0124 (15)	0.0171 (17)	0.0114 (19)
C14	0.0527 (17)	0.076 (2)	0.075 (2)	-0.0016 (15)	-0.0010 (15)	0.0072 (17)
C15	0.0477 (15)	0.0453 (14)	0.0651 (17)	0.0099 (12)	0.0121 (13)	0.0103 (12)
N1	0.0395 (12)	0.0460 (12)	0.0771 (16)	0.0029 (9)	0.0100 (11)	0.0029 (11)
O1	0.0542 (11)	0.0678 (13)	0.0566 (11)	-0.0068 (9)	0.0063 (9)	-0.0133 (9)
S1	0.0733 (5)	0.0826 (6)	0.0530 (5)	-0.0086 (4)	0.0280 (4)	-0.0057 (4)

supplementary materials

S2	0.0784 (6)	0.1025 (7)	0.0678 (6)	-0.0198 (5)	-0.0192 (4)	-0.0078 (5)
S3	0.1002 (8)	0.1088 (8)	0.0779 (6)	0.0057 (6)	0.0300 (5)	-0.0335 (5)

Geometric parameters (Å, °)

C2—C3	1.377 (4)	C10—N1	1.464 (3)
C2—C7	1.380 (4)	C10—H10A	0.9700
C2—H2	0.9300	C10—H10B	0.9700
C3—C4	1.392 (4)	C11—C12	1.386 (4)
C3—S1	1.726 (3)	C11—H11	0.9300
C4—O1	1.341 (3)	C12—C13	1.364 (5)
C4—C5	1.405 (4)	C12—H12	0.9300
C5—C6	1.391 (4)	C13—C14	1.365 (5)
C5—C15	1.461 (4)	C13—H13	0.9300
C6—C7	1.374 (4)	C14—H14	0.9300
C6—H6	0.9300	C15—N1	1.269 (3)
C7—S2	1.729 (3)	C15—H15	0.9300
C8—C14	1.374 (4)	O1—H1	0.8200
C8—C9	1.384 (4)	S1—H1A	1.2000
C8—S3	1.733 (3)	S2—H2A	1.2000
C9—C11	1.390 (4)	S3—H3A	1.2000
C9—C10	1.507 (4)		
C3—C2—C7	118.7 (2)	N1—C10—H10A	109.7
C3—C2—H2	120.7	C9—C10—H10A	109.7
C7—C2—H2	120.7	N1—C10—H10B	109.7
C2—C3—C4	122.3 (2)	C9—C10—H10B	109.7
C2—C3—S1	118.6 (2)	H10A—C10—H10B	108.2
C4—C3—S1	119.1 (2)	C12—C11—C9	120.7 (3)
O1—C4—C3	119.9 (2)	C12—C11—H11	119.7
O1—C4—C5	122.3 (2)	C9—C11—H11	119.7
C3—C4—C5	117.9 (2)	C13—C12—C11	120.1 (3)
C6—C5—C4	119.9 (2)	C13—C12—H12	119.9
C6—C5—C15	119.4 (2)	C11—C12—H12	119.9
C4—C5—C15	120.7 (2)	C12—C13—C14	120.5 (3)
C7—C6—C5	120.2 (3)	C12—C13—H13	119.7
C7—C6—H6	119.9	C14—C13—H13	119.7
C5—C6—H6	119.9	C13—C14—C8	119.3 (3)
C6—C7—C2	121.1 (2)	C13—C14—H14	120.4
C6—C7—S2	120.5 (2)	C8—C14—H14	120.4
C2—C7—S2	118.4 (2)	N1—C15—C5	121.4 (3)
C14—C8—C9	122.3 (3)	N1—C15—H15	119.3
C14—C8—S3	118.2 (2)	C5—C15—H15	119.3
C9—C8—S3	119.5 (2)	C15—N1—C10	118.5 (3)
C8—C9—C11	117.1 (2)	C4—O1—H1	109.5
C8—C9—C10	122.8 (3)	C3—S1—H1A	109.5
C11—C9—C10	120.0 (3)	C7—S2—H2A	109.5
N1—C10—C9	109.9 (2)	C8—S3—H3A	109.5

Hydrogen-bond geometry (Å, °)

<i>D—H···A</i>	<i>D—H</i>	<i>H···A</i>	<i>D···A</i>	<i>D—H···A</i>
O1—H1···N1	0.82	1.87	2.591 (3)	147

Fig. 1

