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7-Amino-3-(3-aminophenyl)-1*H*-quinazoline-2,4-dithione

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Hydrothermal reaction of *m*-phenylenediamine and carbon disulfide in the molar ratio 1:2 produces the title compound, $C_{14}H_{12}N_4S_2$. X-ray crystal structure determination shows that the compound is chiral by virtue of restricted rotation and crystallizes in a non-centrosymmetric but achiral space group, giving a racemic structure.

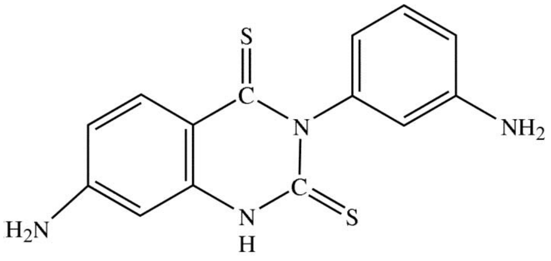
Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.007 Å
R factor = 0.066
wR factor = 0.164
Data-to-parameter ratio = 15.9

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

Comment

Hydrothermal reactions of *m*-phenylenediamine and carbon disulfide in different molar ratios give different products, for example, for 1:1, 5-amino-1,3-benzothiazole-2(3*H*)-thione (Zhong *et al.*, 2003); for 1:2, 7-amino-1*H*-3,1-benzothiazine-2,4-dithione (Yu *et al.*, 2005), and for 2:1, the title compound, (I).



(I)

The crystal structure analysis shows that the molecule of (I) is chiral, because the aminophenyl ring is twisted out of the plane of the ring to which is is attached (Fig. 1). The quinazoline ring system is planar, with a small mean deviation of 0.034 Å from the least-squares plane. The aminobenzene ring is almost perpendicular to that plane (74.2°) The molecule features C═S double bonds of nearly the same length [S1—C1 = 1.663 (4) Å and S2—C2 = 1.691 (5) Å]. The molecules pack in the solid state in a non-centrosymmetric but achiral space group, as a racemate.

Experimental

An ethanol solution (20 ml) containing *m*-phenylenediamine dihydrochloride (10 mmol) and carbon disulfide (5 mmol) was placed in a 25 ml autoclave with a Teflon liner. The pH of the solution was adjusted to 7–8 with sodium hydroxide solution. The autoclave was heated to 373 K, kept at that temperature for 96 h, and then cooled to room temperature at a rate of 0.5 K min^{-1}. Orange prism-shaped crystals were obtained for X-ray diffraction. Elemental analysis calculated for $C_{14}H_{12}N_4S_2$: C 55.97, H 4.03, N 18.65, S 42.50%; found: C 55.18, H 4.34, N 18.67%. MS: M^+/Z, 300. IR(KBr): ν_{max} 3424, 3317, 3208, 1618, 1545, 1491, 1401, 1385, 1281, 1196, 1149, 827, 772, 694 cm^{-1}. UV–vis (CH_3CN): λ_{max} 400 nm (ε = 31000 dm^3 mol^{-1} cm^{-1}), 292 nm (ε = 42000 dm^3 mol^{-1} cm^{-1}).

Crystal data

$C_{14}H_{12}N_4S_2$
$M_r = 300.40$
Orthorhombic, $Pca2_1$
$a = 21.883\ (2)$ Å
$b = 8.7246\ (6)$ Å
$c = 7.2407\ (6)$ Å
$V = 1382.4\ (2)$ Å^3
$Z = 4$
$D_x = 1.443$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4278 reflections
$\theta = 2.0\text{–}27.0°$
$\mu = 0.38$ mm^{-1}
$T = 273\ (2)$ K
Block, pale orange
0.32 × 0.20 × 0.18 mm

Data collection

Bruker SMART APEX CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.888$, $T_{\max} = 0.935$
7640 measured reflections
2884 independent reflections
2080 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.068$
$\theta_{\max} = 27.0°$
$h = -10 \rightarrow 27$
$k = -10 \rightarrow 10$
$l = -9 \rightarrow 9$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.066$
$wR(F^2) = 0.164$
$S = 0.99$
2884 reflections
181 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0886P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.32$ e Å^{-3}
$\Delta\rho_{\min} = -0.30$ e Å^{-3}
Absolute structure: Flack (1983)1247 Friedel pairs
Flack parameter: −0.08 (15)

Table 1
Selected geometric parameters (Å, °).

S1—C1	1.691 (5)	N2—C2	1.426 (5)
S2—C2	1.663 (4)	N2—C9	1.443 (5)
N1—C1	1.348 (5)	N4—C13	1.381 (7)
N1—C7	1.397 (6)	C2—C8	1.459 (6)
N2—C1	1.405 (5)		
C1—N1—C7	124.3 (4)	N1—C1—S1	119.7 (3)
C1—N2—C2	123.4 (3)	N2—C1—S1	123.5 (3)
C1—N2—C9	117.5 (4)	N2—C2—C8	115.7 (4)
C2—N2—C9	118.9 (3)	N2—C2—S2	120.0 (3)
N1—C1—N2	116.9 (4)	C8—C2—S2	124.3 (3)

All H atoms were placed geometrically, with C—H = 0.93 Å and N—H = 0.86 Å, and refined using a riding model with isotropic displacement parameters $U_{\rm iso}$(H) fixed at 1.2 times $U_{\rm eq}$ of the parent C or N atom.

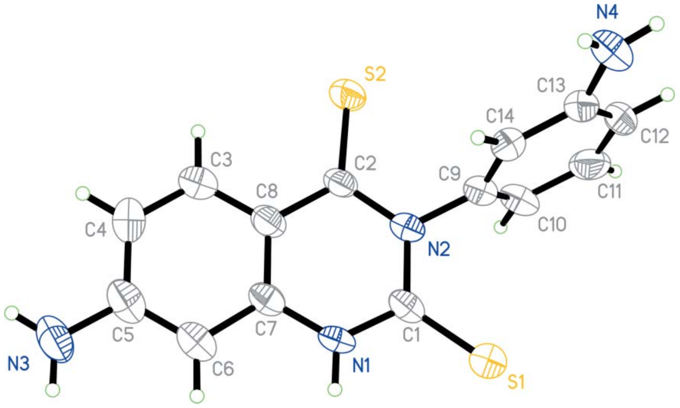


Figure 1
A view of the molecule of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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