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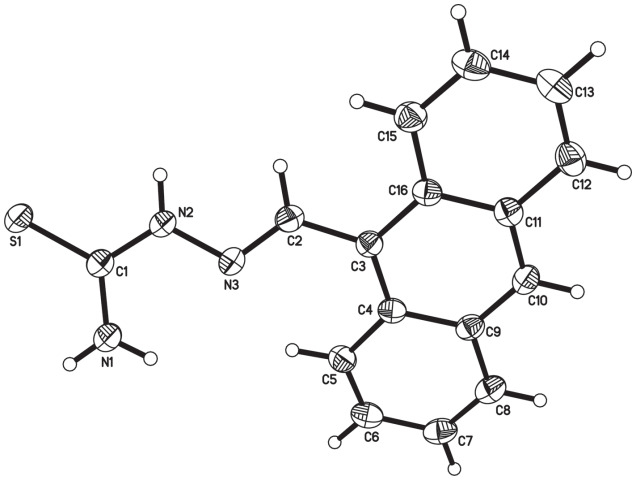
Crystal structure of (*E*)-2-(anthracen-9-ylmethylene)hydrazine-1-carbothioamide, $C_{16}H_{13}N_3S$



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Abstract

$C_{16}H_{13}N_3S$, monoclinic, $P2_1/c$ (no. 14), $a = 19.0479(17)$ Å, $b = 4.7282(4)$ Å, $c = 16.7554(15)$ Å, $\beta = 114.309(1)°$, $V = 1375.2(2)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0412$, $wR_{ref}(F^2) = 0.1436$, $T = 293(2)$ K.

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The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Brown block
Size:	0.25 × 0.23 × 0.20 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.23 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω-scans
θ_{max}, completeness:	25.1°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	6869, 2441, 0.033
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1724
$N(param)_{refined}$:	181
Programs:	Bruker programs [1], SHELX [2]

Source of material

The title compound was synthesized by the reaction of hydrazinecarbothioamide (1 mmol, 91.1 mg) with anthracene-9-carbaldehyde (1 mmol, 206.2 mg) in ethanol (20 mL) containing a few droups of glacial acetic acid under reflux conditions (353 K) for 5 h. When cooled to room temperature, the solution was filtrated and then stand at room temperature. After about 4 days, brown, block-shaped crystals suitable for X-ray diffraction study were obtained.

Experimental details

All H atoms were placed in calculated positions (C—H = 0.93 Å, N—H = 0.86 Å) and refined as riding atoms. The U_{iso} values were constrained to be $1.2U_{eq}$ of the carrier atom for all H atoms.

Discussion

Synthesis of Schiff-bases and their derivatives have been investigated for many years due to their higher bioactivity and applications in metal coordination chemistry. Among the Schiff bases, thiosemicarbazones containing S, N donor ligands are of particular interest because of their coordination chemistry [3–7] and broad spectrum of biological activities [8–11].

In order to search for new thiosemicarbazones with higher bioactivity, the title compound was synthesized and its crystal structure is reported here.

The C = N bond length in the molecule of 1.273(3) Å (C9 = N2) shows a typical double-bond character. The C = N—N angle of 115.4(2)° (C2 = N3—N2) is much smaller than the ideal value of 120° expected for sp^2 N atoms. This is probably a consequence of repulsion between the nitrogen lone pairs and the adjacent N bonds. The dihedral angle formed by the anthracenyl moiety and the mean plane of thiosemicarbazone

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
S1	0.46809(4)	0.11323(17)	0.85501(5)	0.0643(3)
N1	0.57922(14)	0.3045(6)	1.00050(14)	0.0786(9)
H1A	0.6197	0.4048	1.0281	0.094*
H1B	0.5594	0.2064	1.0292	0.094*
N2	0.58195(11)	0.4597(5)	0.87381(13)	0.0504(6)
H2	0.5615	0.4747	0.8177	0.061*
N3	0.64992(11)	0.5984(5)	0.92255(13)	0.0487(6)
C1	0.54755(13)	0.3010(6)	0.91403(16)	0.0497(7)
C2	0.67894(13)	0.7378(5)	0.87853(16)	0.0433(6)
H2A	0.6503	0.7491	0.8183	0.052*
C3	0.75326(12)	0.8814(5)	0.91456(14)	0.0365(5)
C4	0.81327(13)	0.8102(5)	0.99696(14)	0.0368(5)
C5	0.80709(14)	0.5985(5)	1.05470(16)	0.0442(6)
H5	0.7608	0.5022	1.0393	0.053*
C6	0.86704(15)	0.5349(6)	1.13121(16)	0.0492(6)
H6	0.8611	0.3942	1.1667	0.059*
C7	0.93798(15)	0.6756(6)	1.15832(17)	0.0529(7)
H7	0.9782	0.6317	1.2117	0.063*
C8	0.94707(14)	0.8762(5)	1.10576(16)	0.0496(7)
H8	0.9940	0.9695	1.1235	0.060*
C9	0.88646(13)	0.9474(5)	1.02407(15)	0.0394(6)
C10	0.89780(14)	1.1483(5)	0.96999(15)	0.0430(6)
H10	0.9454	1.2376	0.9887	0.052*
C11	0.84073(13)	1.2203(5)	0.88915(15)	0.0395(6)
C12	0.85456(15)	1.4245(5)	0.83458(18)	0.0501(7)
H12	0.9024	1.5123	0.8540	0.060*
C13	0.79941(16)	1.4925(6)	0.75531(18)	0.0554(7)
H13	0.8093	1.6262	0.7205	0.066*
C14	0.72702(16)	1.3602(6)	0.72562(18)	0.0551(7)
H14	0.6893	1.4066	0.6708	0.066*
C15	0.71105(14)	1.1664(5)	0.77536(16)	0.0493(7)
H15	0.6624	1.0845	0.7540	0.059*
C16	0.76701(13)	1.0842(5)	0.85986(15)	0.0377(6)

unit is 26°. In the crystal structure, A dimer structure is formed by the N—H$\cdots$S hydrogen bonds.

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