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Crystal structure of (*E*)-2-(1-(2,4-dihydroxyphenyl)ethylidene)hydrazinecarbothioamide, $C_9H_{11}N_3O_2S$

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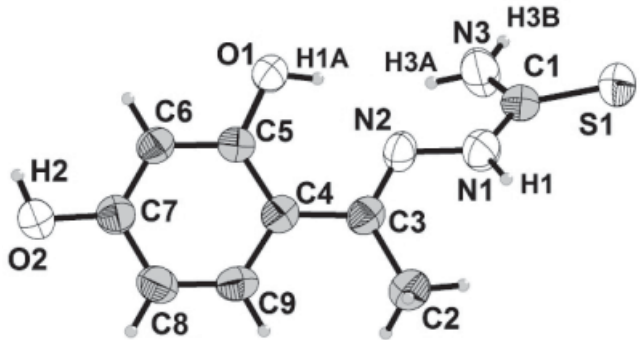

Abstract

$C_9H_{11}N_3O_2S$, monoclinic, *C*2/*c* (no. 15), a = 20.900(2) Å, b = 7.9290(8) Å, c = 13.248(1) Å, β = 100.209(1)°, V = 2160.7 Å^3, Z = 8, $R_{gt}(F)$ = 0.0460, $wR_{ref}(F^2)$ = 0.1669, T = 298 K.

Table 1. Data collection and handling.

Crystal:	yellow blocks, size 0.11×0.21×0.30 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.84 cm^{-1}
Diffractometer, scan mode:	CCD area detector, φ and ω
$2\theta_{max}$:	50.04°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5267, 1905
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1172
$N(param)_{refined}$:	137
Programs:	SHELX [7]

Source of material

2,4-Dihydroxyacetophenone (10 mmol) and an equimolar amount of aminothiourea were mixed and microwave radiated for 5 min at 200W. The yellow powder was dissolved in ethanol (95%) and stood still in air for 5 days. Large block crystals were precipitated. They were filtered and washed with ethanol (95%) and dried in air (yield 80%).

Discussion

The hydroxyacetopheno-aminothiourea are important chelating ligand [1-6]. The title compound, (*E*)-2-(1-(2,4-dihydroxyphenyl)ethylidene)hydrazinecarbothioamide was synthesized by a microwave solid phase reaction of 2,4-dihydroxyacetophenone and aminothiourea. The N1–C1, N1–N2, N2–C3, N3–C1and S1–C1 bonds are 1.339(4) Å, 1.391(3) Å, 1.288(4) Å, 1.327(4) Å and 1.697(4) Å, respectively. The bond angles of C1–N1–N2, C3–N2–N1, N3–C1–N1, N3–C1–S1 and N1–C1–S1 are 119.5(3)°, 117.6(3)°, 117.0(3)°, 122.4(3)° and 120.6(3)°, respectively. The dihedral angles between benzene ring plane I (C4/C5/C6/C7/C8/C9) and plane II (C1/N1/N3/S1) are 49.6°. X-ray analysis reveals that intermolecular N1–H1···S1 (d(N1–S1) = 3.471(8) Å), O1–H1A···N2 [$\frac{1}{2}$-x, $\frac{5}{2}$-y, 1-z] (d(O1–N2) = 2.599(6) Å), O2–H2···S1 (d(O2–S1) = 3.183(3) Å), N3–H3A···N2 [-$\frac{1}{2}$+x, -$\frac{1}{2}$+y, z] (d(N3–N2) = 2.642(4) Å), N3–H3A···O1 [-x, y, $\frac{1}{2}$-z] (d(N3–O1) = 3.150(4) Å), N3–H3B···O2 [-x, 1+y, $\frac{1}{2}$-z] (d(N3–O2) = 2.953(3) Å) and C2–H2A···N1 [-x, 1+y, $\frac{1}{2}$-z] (d(C2–N1) = 2.764(5) Å) hydrogen bond in the crystal lattice.

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	8*f*	0.7028	0.6181	0.4323	0.073
H(3A)	8*f*	0.6021	0.7635	0.2363	0.087
H(3B)	8*f*	0.6406	0.9110	0.2115	0.087
H(1A)	8*f*	0.5232	0.6559	0.3646	0.077
H(2)	8*f*	0.3308	0.1983	0.3748	0.081
H(2A)	8*f*	0.7040	0.3729	0.4063	0.126
H(2B)	8*f*	0.6604	0.2119	0.3825	0.126
H(2C)	8*f*	0.6727	0.2892	0.4931	0.126
H(6)	8*f*	0.3875	0.4482	0.3656	0.056
H(8)	8*f*	0.4693	–0.0118	0.3941	0.067
H(9)	8*f*	0.5691	0.1070	0.3997	0.064

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Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	8*f*	0.6715(1)	0.6582(4)	0.3878(2)	0.047(2)	0.058(2)	0.072(2)	–0.001(2)	–0.003(2)	0.007(2)
N(2)	8*f*	0.6102(1)	0.5847(4)	0.3742(2)	0.036(2)	0.056(2)	0.051(2)	–0.003(1)	0.001(1)	0.004(1)
N(3)	8*f*	0.6361(1)	0.8262(4)	0.2501(2)	0.060(2)	0.066(2)	0.083(2)	–0.014(2)	–0.013(2)	0.027(2)
O(1)	8*f*	0.4856(1)	0.6242(3)	0.3628(2)	0.042(1)	0.049(2)	0.062(2)	0.001(1)	0.008(1)	0.005(1)
O(2)	8*f*	0.3598(1)	0.1276(3)	0.3795(2)	0.053(2)	0.052(2)	0.099(2)	–0.008(1)	0.019(1)	–0.011(1)
S(1)	8*f*	0.75004(5)	0.9098(1)	0.36186(9)	0.0479(6)	0.0619(7)	0.0973(9)	–0.0059(5)	0.0021(5)	0.0005(5)
C(1)	8*f*	0.6816(2)	0.7920(4)	0.3309(3)	0.051(2)	0.045(2)	0.070(2)	0.003(2)	0.009(2)	0.004(2)
C(2)	8*f*	0.6665(2)	0.3147(5)	0.4212(4)	0.052(3)	0.062(3)	0.132(4)	0.004(2)	0.000(2)	–0.015(3)
C(3)	8*f*	0.6070(2)	0.4257(4)	0.3924(2)	0.045(2)	0.056(2)	0.043(2)	0.003(2)	0.004(1)	–0.005(2)
C(4)	8*f*	0.5416(2)	0.3527(4)	0.3859(2)	0.045(2)	0.052(2)	0.035(2)	0.003(2)	0.004(1)	–0.002(2)
C(5)	8*f*	0.4847(2)	0.4531(4)	0.3737(2)	0.047(2)	0.043(2)	0.035(2)	0.002(2)	0.006(1)	0.001(1)
C(6)	8*f*	0.4243(2)	0.3802(4)	0.3723(2)	0.045(2)	0.050(2)	0.044(2)	0.005(2)	0.010(2)	–0.003(2)
C(7)	8*f*	0.4181(2)	0.2065(4)	0.3808(2)	0.053(2)	0.052(2)	0.049(2)	–0.001(2)	0.011(2)	–0.004(2)
C(8)	8*f*	0.4732(2)	0.1047(4)	0.3900(3)	0.062(2)	0.044(2)	0.061(2)	–0.000(2)	0.008(2)	0.000(2)
C(9)	8*f*	0.5328(2)	0.1767(4)	0.3929(2)	0.056(2)	0.047(2)	0.055(2)	0.011(2)	0.006(2)	0.001(2)

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