Redetermination of the crystal structure of 2-(*o*-hydroxyphenyl)-Δ^2-1,3-benzo[4,5]thiazoline, $C_{13}H_9NOS$

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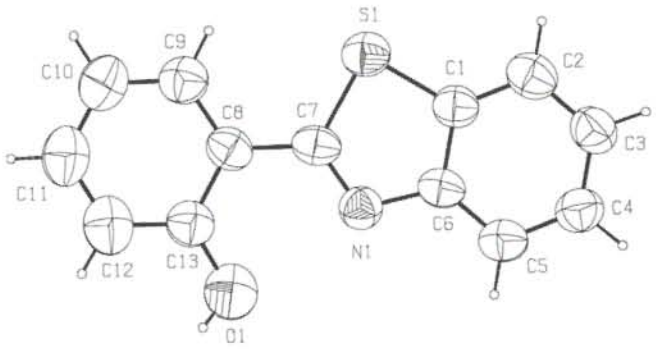

Abstract

$C_{13}H_9NOS$, monoclinic, $P12_1/c1$ (No. 14), $a = 12.394(1)$ Å, $b = 5.853(1)$ Å, $c = 15.632(1)$ Å, $\beta = 111.620(2)°$, $V = 1054.2$ Å^3, $Z = 4$, $R_{gt}(F) = 0.047$, $wR(F^2) = 0.121$, $T = 295$ K.

Source of material

2-hydroxybenzaldehyde (0.25 g; 2 mmol) was added to a solution of 2-aminothiophenol (0.20 g; 2 mmol) in ethanol (100 ml). The mixture was heated and waited for two days. Suitable crystals were obtained from ethyl alcohol solution as brown plates. In all stages of the refinement, N1 was refined isotropically. In opposite case, the displacement parameter of N1 was none-positive defined.

Discussion

The crystal structure of this compound had already been determined by Stenson [1] using integrated Weissenberg photographs. However, the *R* factor was rather high (0.088), some atoms had negative thermal factors and some problems obtained with the choice of the space group. So we performed a new data collection on an automatic diffractometer in order to obtain better accuracy on the atomic positions. This redetermination gives e.s.d.'s of an order magnitude lower than for the original study [1]. The C1–S1 and C9–S1 distances in the thiazole ring are 1.741(3) Å and 1.743(4) Å respectively. These values are comparable with those in 3-(2-benzothiazolyl)-7-(diethylamino)coumarin (1.754(2) Å and 1.730(2) Å) [2], and in 2-(4-hydroxyphenyl)benzothiazole [1.747(2) Å and 1.732(2) Å] [3]. In the benzothiazole moiety, the angle between the planar benzene ($\chi^2 = 15.8$ Å) and thiazole ($\chi^2 = 2.9$ Å) rings is 1.1(2)°, establishing an essentially planar arrangement, supported by a short intramolecular hydrogen bond between H9 (on C9) an S1, with H···S 2.69(6) Å. From the structural formula and the close contact O1···N1, it would be natural to assume an intramolecular H bond O1–H1···N1. This would also be a better explanation for the coplanarity of the rings than the C–H···S interaction mentioned in the discussion. The displacement of all atoms in the molecule, except H atoms, is less than 0.972(3) Å from the least-squares plane. Therefore the whole molecule, including the hydroxyl group, is a π-conjugated system. Two short contacts (O1...N1 2.605(5) Å, O1···H4 (2–*x*,–1–*y*,1–*z*) 2.68(5) Å) determine the intermolecular packing.

Table 1. Data collection and handling.

Crystal:	brown plate, size 0.20 × 0.25 × 0.35 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.80 cm^{-1}
Diffractometer, scan mode:	Enraf-Nonius CAD-4, ω/2θ
$2\theta_{max}$:	56.12°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2302, 2301
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1838
$N(param)_{refined}$:	154
Programs:	MolEN [4], SIR-92 [5], SHELXL-97 [6], PLATON [7]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
N(1)	4*e*	0.9342(2)	–0.1698(5)	0.3726(2)	0.0555
H(1)	4*e*	0.6779	–0.4326	0.3222	0.122
H(2)	4*e*	1.231	0.292	0.432	0.081
H(3)	4*e*	1.353	0.060	0.547	0.081
H(4)	4*e*	1.288	–0.277	0.584	0.080
H(5)	4*e*	1.099	–0.400	0.506	0.075
H(9)	4*e*	0.767	0.280	0.184	0.081
H(10)	4*e*	0.578	0.273	0.085	0.097
H(11)	4*e*	0.459	–0.027	0.091	0.104
H(12)	4*e*	0.528	–0.311	0.198	0.098

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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}
S(1)	4*e*	0.97785(8)	0.2219(2)	0.31584(6)	0.0682(5)	0.0488(5)	0.0690(5)	0.0057(3)	0.0305(4)	–0.0024(3)
O(1)	4*e*	0.7325(3)	–0.3709(5)	0.3136(2)	0.088(3)	0.065(3)	0.085(3)	0.006(3)	0.027(3)	–0.022(3)
C(1)	4*e*	1.0901(3)	0.0830(6)	0.4021(2)	0.063(2)	0.059(2)	0.058(2)	–0.002(1)	0.030(1)	0.002(1)
C(2)	4*e*	1.2044(4)	0.1554(7)	0.4471(3)	0.073(2)	0.057(2)	0.080(2)	–0.005(2)	0.038(2)	–0.007(2)
C(3)	4*e*	1.2766(3)	0.0158(8)	0.5155(3)	0.060(2)	0.071(2)	0.075(2)	–0.007(2)	0.028(2)	0.000(2)
C(4)	4*e*	1.2373(3)	–0.1868(7)	0.5375(3)	0.066(2)	0.066(2)	0.072(2)	0.006(2)	0.031(2)	0.012(2)
C(5)	4*e*	1.1241(4)	–0.2612(7)	0.4917(3)	0.070(2)	0.058(2)	0.067(2)	–0.001(2)	0.033(2)	0.005(2)
C(6)	4*e*	1.0508(3)	–0.1241(6)	0.4242(2)	0.064(2)	0.050(2)	0.062(2)	–0.005(1)	0.033(1)	0.001(1)
C(7)	4*e*	0.8876(3)	–0.0070(6)	0.3151(2)	0.069(2)	0.045(2)	0.063(2)	–0.003(1)	0.035(2)	–0.003(1)
C(8)	4*e*	0.7661(3)	–0.0118(6)	0.2510(2)	0.062(2)	0.058(2)	0.059(2)	–0.006(1)	0.028(1)	–0.004(1)
C(9)	4*e*	0.7200(4)	0.1593(7)	0.1866(3)	0.070(2)	0.061(2)	0.074(2)	0.005(2)	0.031(2)	–0.000(2)
C(10)	4*e*	0.6073(4)	0.1563(9)	0.1276(3)	0.075(2)	0.086(3)	0.077(2)	0.014(2)	0.024(2)	0.004(2)
C(11)	4*e*	0.5363(4)	–0.023(1)	0.1314(4)	0.069(2)	0.099(3)	0.086(3)	0.011(2)	0.021(2)	–0.007(2)
C(12)	4*e*	0.5777(4)	–0.1951(9)	0.1947(3)	0.075(2)	0.084(3)	0.083(3)	0.004(2)	0.024(2)	–0.019(2)
C(13)	4*e*	0.6930(4)	–0.1975(7)	0.2542(3)	0.070(2)	0.060(2)	0.064(3)	–0.002(3)	0.026(3)	–0.005(3)

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