Crystal structure of 4-(2-oxobenzothiazolin-3-yl)butanoic acid, $C_{11}H_{11}NO_3S$

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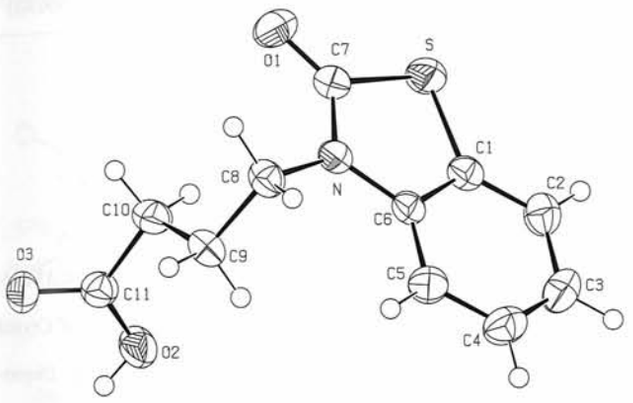

Abstract

$C_{11}H_{11}NO_3S$, triclinic, $P\bar{1}$ (No. 2), $a = 7.240(1)$ Å, $b = 7.495(2)$ Å, $c = 10.427(1)$ Å, $\alpha = 83.89(1)°$, $\beta = 85.74(1)°$, $\gamma = 71.16(1)°$, $V = 532.0$ Å^3, $Z = 2$, $R_{gt}(F) = 0.035$, $wR_{ref}(F^2) = 0.096$, $T = 293$ K.

Source of material

For the synthesis, 10.0 mmol ethyl 4-(2-oxobenzothiazolin-3-yl)-butanoate in concentrated hydrochloric acid (50 ml) was stirred at room temperature for 2 hours, then refluxed for 4 hours. The reaction mixture was cooled, poured into 100 g ice-water, and stirred for 1 hour. The precipitate was collected by filtration, washed with water, dried and crystallised from water.

Discussion

The benzene ring defined by C1–C2–C3–C4–C5–C6 atoms is planar. Torsion angles of C1–C2–C3–C4, C6–C5–C4–C3 and C1–C6–C5–C4 are 0.4(3)°, 0.1(3)° and 0.3(3)°, respectively. The thiazolon ring defined by C1–C6–N–C7–S atoms is planar. Torsion angles of C7–S–C1–C6 and C6–N–C7–S are 0.6(1)° and 1.2(2)°, respectively. The dihedral angle between these two planes is 0.99(8)° implying that they are co-planar. The torsion angles of C7–N–C6–C5, N–C6–C1–C2, C7–S–C1–C2 and S2–C1–C2–C3 are 178.8(2)°, 179.2(2)°, –178.6(2)° and 179.1(2)°, respectively. O1 and C8 atoms are also at this plane. Torsion angles of C6–N–C7–O1, C1–S–C7–O1, C8–N–C6–C1 and C8–N–C7–O1 are –179.0(2)°, 179.2(2)°, –179.0(2)° and –0.7(3)°, respectively. C9 atom lies below 1.351(3) Å from thiazolon plane. Torsion angles of C7–N–C8–C9, C6–N–C8–C9 and N–C8–C9–C10 are 103.8(2)°, –78.0(2)° and –68.5(2)°, respectively. The bond lengths and angles in the 4-(2-oxobenzothiazolin-3-yl)butanoic acid are all in accord with similar structures in the literature [1-3]. The bond lengths of C—C are between 1.378(3) Å – 1.522(3) Å. The bond lengths of C7=O1, C11—O2 and C11=O3 are 1.214(2) Å, 1.262(2) Å and 1.263(2) Å, respectively. The bond lengths of C7—S and C1—S are 1.782(2) Å and 1.743(2) Å, respectively. The bond lengths of C6—N, C7—N and C8—N are 1.395(2) Å, 1.368(2) Å and 1.464(2) Å, respectively.

Table 1. Data collection and handling.

Crystal:	white needle, size 0.06 × 0.27 × 0.48 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.94 cm^{-1}
Diffractometer, scan mode:	Enraf-Nonius CAD-4, $\omega/2\theta$
$2\theta_{max}$:	52.58°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2300, 2130
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1786
$N(param)_{refined}$:	147
Programs:	SHELXS-97 [4] SHELXL-97 [5], ORTEP-III [6]

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

Atom	Site	x	y	z	U_{iso}
H(2)	2*i*	–0.4244	–0.0414	0.5929	0.070
H(8A)	2*i*	–0.1139	0.5419	0.6207	0.041
H(8B)	2*i*	–0.2521	0.5945	0.7436	0.041
H(5)	2*i*	–0.3345	0.4424	0.9429	0.046
H(4)	2*i*	–0.3352	0.3370	1.1597	0.053
H(9A)	2*i*	–0.4118	0.4711	0.6134	0.042
H(9B)	2*i*	–0.3442	0.3186	0.7301	0.042
H(10A)	2*i*	–0.1531	0.2997	0.4823	0.048
H(10B)	2*i*	–0.0717	0.1516	0.5994	0.048
H(2A)	2*i*	0.2404	0.0690	1.1354	0.048
H(3)	2*i*	–0.0519	0.1549	1.2552	0.054

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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	2*i*	0.32516(6)	0.16590(7)	0.85628(5)	0.0272(2)	0.0507(3)	0.0421(3)	–0.0099(2)	–0.0009(2)	–0.0008(2)
O(2)	2*i*	–0.3456(2)	0.0043(2)	0.6171(1)	0.0589(9)	0.0509(9)	0.0424(8)	–0.0339(7)	–0.0150(6)	0.0080(6)
O(3)	2*i*	–0.3910(2)	0.1564(2)	0.4210(1)	0.0590(9)	0.0510(8)	0.0337(7)	–0.0311(7)	–0.0082(6)	0.0022(6)
O(1)	2*i*	0.2322(2)	0.3345(2)	0.6211(1)	0.0448(8)	0.069(1)	0.0399(8)	–0.0219(7)	0.0058(6)	0.0044(7)
N	2*i*	–0.0148(2)	0.3698(2)	0.7785(1)	0.0288(7)	0.0354(8)	0.0317(8)	–0.0138(6)	–0.0040(6)	–0.0008(6)
C(6)	2*i*	–0.0443(2)	0.3184(2)	0.9091(2)	0.0309(8)	0.0297(8)	0.0310(8)	–0.0143(7)	–0.0032(7)	–0.0042(7)
C(1)	2*i*	0.1269(2)	0.2062(2)	0.9673(2)	0.0297(8)	0.0310(9)	0.0354(9)	–0.0108(7)	–0.0014(7)	–0.0053(7)
C(11)	2*i*	–0.3165(3)	0.1242(3)	0.5303(2)	0.0350(9)	0.0347(9)	0.0354(9)	–0.0138(7)	0.0006(7)	–0.0047(7)
C(8)	2*i*	–0.1711(3)	0.4898(2)	0.6966(2)	0.0362(9)	0.0313(9)	0.0382(9)	–0.0133(7)	–0.0091(7)	0.0015(7)
C(5)	2*i*	–0.2193(3)	0.3681(3)	0.9809(2)	0.0314(9)	0.043(1)	0.039(1)	–0.0094(8)	0.0010(7)	–0.0052(8)
C(4)	2*i*	–0.2187(3)	0.3048(3)	1.1104(2)	0.043(1)	0.049(1)	0.042(1)	–0.0148(9)	0.0107(8)	–0.0088(9)
C(9)	2*i*	–0.2984(2)	0.3820(3)	0.6545(2)	0.0300(9)	0.036(1)	0.041(1)	–0.0119(7)	–0.0069(7)	–0.0038(8)
C(10)	2*i*	–0.1901(3)	0.2362(3)	0.5611(2)	0.039(1)	0.048(1)	0.042(1)	–0.0243(9)	0.0025(8)	–0.0088(9)
C(7)	2*i*	0.1740(3)	0.3046(3)	0.7310(2)	0.0339(9)	0.040(1)	0.0361(9)	–0.0184(8)	–0.0007(7)	–0.0032(7)
C(2)	2*i*	0.1258(3)	0.1438(3)	1.0969(2)	0.042(1)	0.040(1)	0.037(1)	–0.0109(8)	–0.0083(8)	0.0006(8)
C(3)	2*i*	–0.0487(3)	0.1949(3)	1.1678(2)	0.058(1)	0.047(1)	0.0299(9)	–0.018(1)	0.0018(8)	–0.0003(8)

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