Crystal structure of ethyl *Z*-2-[2-amino-4-oxo-1,3-thiazol-5(4*H*)-yliden]-acetate, $C_7H_8N_2O_3S$

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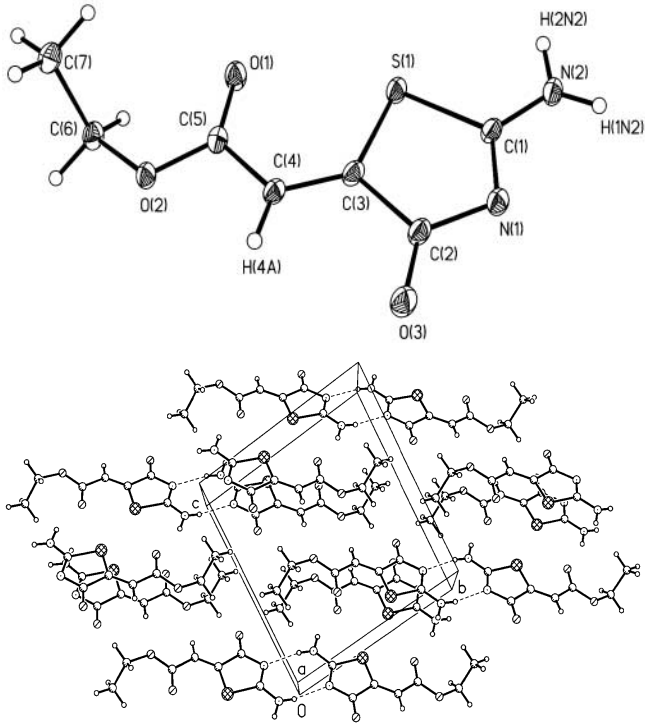
Received October 1, 2002, accepted and available on-line December 14, 2002; CCDC-No. 1267/946



Abstract

$C_7H_8N_2O_3S$, triclinic, $P\bar{1}$ (No. 2), a = 4.1057(9) Å, b = 10.394(2) Å, c = 11.000(2) Å, α = 84.455(4)°, β = 72.175(2)°, γ = 67.277(2)°, V = 412.1 Å^3, Z = 2, $R_{gt}(F)$ = 0.047, $wR_{ref}(F^2)$ = 0.132, T = 110 K.

Source of material

Ethyl *Z*-2-[2-amino-4-oxo-1,3-thiazol-5(4*H*)-yliden]acetate was synthesized in accordance with our new procedure [1]. The solution of title compound (0.35 g) in methanol (20 ml) left in room temperature for five days. The colorless crystals were filtered off, washed with cold methanol (5 ml) and dried in vacuum over P_4O_{10} (mp 512 K (dec.); yield 80%). Elemental analyses were consistent with the composition $C_7H_8N_2O_3S$ (found: C, 41.87%; H, 4.10%; N, 14.17%; calc. : C, 41.99%; H, 4.03%; N, 13.99%). Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected. Elemental analyses were performed using a Heraeus CHN-O-Rapid analyzer.

Discussion

Organosulfur chemistry has provided organic chemists with a wealth of reactions, many of which have found general application in organic synthesis. Thiazole skeleton compounds are important heterocycles in bio-organic chemistry and are present in many pharmaceuticals. There have been reported many studies on the synthesis of the thiazole ring structure [1].

In the structure of title molecule (upper figure), the covalent bonds are found to be normal (relevant bond lengths : d(S1—C3) =1.730(2) Å, d(S1—C1) = 1.773(2) Å, d(O1—C5) = 1.218(2) Å, d(O2—C5) = 1.335(2) Å, d(O2—C6) = 1.463(2) Å, d(O3—C2) = 1.216(2) Å, d(N1—C1) = 1.320(2)Å, d(N1—C2) = 1.384(2) Å, d(N2—C1) = 1.310(2) Å, d(C2—C3) = 1.517(2) Å, d(C3—C4) = 1.344(2) Å, d(C4—C5) = 1.469(2) Å, d(C6—C7) = 1.507(2) Å; angles: 116.19(12)° at O2, 88.34(7)° at S1, and 110.81(13)° at N1) and almost the same as in the previous reports [2-4]. The ∠ C6–O2–C5–O1 = 4.4(2)°, agrees well with a participation of O2 in the electron donation to the carbonyl group (C5=O1). The torsion angle ∠C3–C4–C5–O1 = –7.9(2)°, shows clearly that the carbonyl group (C5—O1) and C=C double bond (C3=C4) share approximately a common plane with *S-cis* conformation, probably is as a result of π-systems conjugations. Intermolecular π-systems stacking are observed within the crystals, probably are as a result of the presence of parallel π-systems of the molecules in the crystal network. Intermolecular hydrogen bondigs between N1 and H(1N2) atoms (d(N1···H(1N2) = 2.108 Å and ∠ N1–H(1N2)–N2 = 168.02°) are observed within the crystals, probably is as a result of the presence of electron-poor (H(1N2)) and electron-rich (N1) atoms in suitable positions of the crystal network (lower figure). These intermolecular interactions appear to be plausible factors in the stabilization of the crystal packing.

Table 1. Data collection and handling.

Crystal:	colorless plate, size 0.20 × 0.45 × 0.60 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	3.66 cm^{-1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, φ/ω
$2\theta_{max}$:	60.1°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4841, 2352
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2012
$N(param)_{refined}$:	126
Programs:	SHELXTL-97 [5], SADABS [6]

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

Atom	Site	x	y	z	U_{iso}
H(4A)	2*i*	–0.4477	0.5800	0.3843	0.019
H(6A)	2*i*	0.3539	0.2171	0.5123	0.021
H(6B)	2*i*	0.5860	0.2450	0.3735	0.021
H(7A)	2*i*	0.5594	0.0237	0.3760	0.031
H(7B)	2*i*	0.3478	0.1200	0.2799	0.031
H(7C)	2*i*	0.1177	0.0917	0.4189	0.031
H(1N2)	2*i*	–0.147(5)	0.928(2)	–0.098(2)	0.015(5)
H(2N2)	2*i*	0.163(7)	0.795(3)	–0.129(2)	0.035(6)

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)	2*i*	0.1068(1)	0.62997(4)	0.07207(3)	0.0160(2)	0.0117(2)	0.0173(2)	–0.0010(2)	–0.0034(2)	0.0015(1)
O(1)	2*i*	0.3378(3)	0.3821(1)	0.2088(1)	0.0186(5)	0.0156(5)	0.0192(5)	–0.0008(4)	–0.0003(4)	0.0032(4)
O(2)	2*i*	0.0398(3)	0.3596(1)	0.4148(1)	0.0168(5)	0.0125(5)	0.0161(5)	–0.0015(4)	–0.0036(4)	0.0030(4)
O(3)	2*i*	–0.8180(3)	0.8286(1)	0.3159(1)	0.0186(6)	0.0164(5)	0.0217(6)	–0.0010(5)	0.0001(4)	0.0009(4)
N(1)	2*i*	–0.4750(4)	0.8704(1)	0.1178(1)	0.0156(6)	0.0136(6)	0.0171(6)	–0.0014(5)	–0.0038(5)	0.0015(4)
N(2)	2*i*	–0.0255(4)	0.8521(2)	–0.0749(1)	0.0201(6)	0.0131(6)	0.0169(6)	–0.0011(5)	–0.0030(5)	0.0029(5)
C(1)	2*i*	–0.1559(4)	0.8008(2)	0.0343(1)	0.0169(7)	0.0104(6)	0.0181(7)	–0.0024(5)	–0.0067(6)	0.0000(5)
C(2)	2*i*	–0.5437(4)	0.7914(2)	0.2243(2)	0.0155(7)	0.0117(6)	0.0193(7)	–0.0017(5)	–0.0059(6)	0.0000(5)
C(3)	2*i*	–0.2343(4)	0.6497(2)	0.2160(1)	0.0135(6)	0.0107(6)	0.0184(7)	–0.0009(5)	–0.0036(5)	–0.0004(5)
C(4)	2*i*	–0.2380(4)	0.5582(2)	0.3110(1)	0.0151(7)	0.0114(6)	0.0177(7)	–0.0016(5)	–0.0033(5)	0.0002(5)
C(5)	2*i*	0.0753(4)	0.4252(2)	0.3039(1)	0.0179(7)	0.0128(6)	0.0176(7)	–0.0056(6)	–0.0062(6)	0.0029(5)
C(6)	2*i*	0.3540(4)	0.2325(2)	0.4221(2)	0.0162(7)	0.0140(7)	0.0204(7)	–0.0021(6)	–0.0079(6)	0.0037(5)
C(7)	2*i*	0.3438(4)	0.1059(2)	0.3696(2)	0.0205(7)	0.0147(7)	0.0249(8)	–0.0039(6)	–0.0083(6)	0.0027(6)

Acknowledgment. Support of this investigation by Zanjan University Research Council (ZURC4950) is gratefully acknowledged.

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