Crystal structure of ethyl 4-(2-oxobenzothiazolin-3-yl)butanoate, $C_{13}H_{15}NO_3S$

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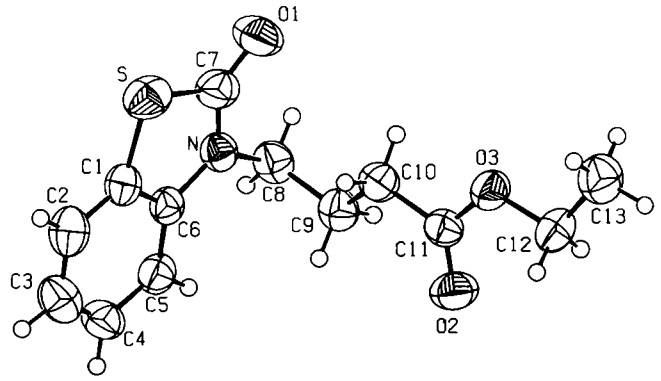

Abstract

$C_{13}H_{15}NO_3S$, monoclinic, $P12_1/n1$ (No. 14), $a = 4.952(1)$ Å, $b = 22.001(3)$ Å, $c = 11.961(2)$ Å, $\beta = 94.03(2)°$, $V = 1300.0$ Å^3, $Z = 4$, $R_{gt}(F) = 0.030$, $wR_{ref}(F^2) = 0.081$, $T = 293$ K.

Source of material

0.002 Mole of sodium was dissolved in 30 ml of absolute ethanol and 0.002 mole of 2-oxobenzothiazoline was added to this solution. After 2-oxobenzothiazoline had dissolved, stirred for one hour and then evaporate to dryness. The solid residue was dissolved in dimethylformamide and 0.002 mole of ethyl 4-chlorobutanoate was added. The final solution was refluxed for 4 hours. The solution was cooled to 273 K and then 100 g of ice-water was added and stirring continued for 1 hour at between 273 K and 283 K. The precipitate thus formed was collected by filtration, washed with water, dried and crystallised from hexane.

Experimental details

The ratio $N(hkl)_{gt}/N(param) = 4.228$ is very low. Main reason is very poor crystal quality. But refinement results (R values) are encouraging and parameters of refine_diff_density_max (0.117) and refine_ diff_density_min (–0.138) are consistent with refinement values of these type of compounds.

Discussion

The benzene ring defined by C1–C2–C3–C4–C5–C6 atoms is planar. Torsion angles of C1–C2–C3–C4, C1–C6–C5–C4 and C5–C4–C3–C2 are –1.4(5)°, –0.4(4)° and 0.5(5)°, respectively. The thiazolon ring defined by C1–C6–N–C7–S atoms is planar. Torsion angles of C1–S–C7–N and C7–N–C6–C1 are –0.6(2)° and –0.3(3)°, respectively. The dihedral angle between these two planes is 0.6(2)° implying that these planes are essentialy planar. Torsion angles of C7–N–C6–C5, N–C6–C5–C4 and C7–S–C1–C2 are –179.9(2)°, 179.2(2)° and –179.6(3)°, 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 180.0(2)°, –179.9(2)°, 177.8(2)° and 1.8(4)°, respectively. The bond lengths and angles in the ethyl 4-(2-oxobenzothiazolin-3-yl) butanoate are all in accord with similar structures in the literature [1-3]. The bond lengths of C—C are between 1.369(4) Å – 1.516(4) Å. The bond lengths of C11—O3, C12–O3, C7=O1 and C11=O2 are 1.338(3) Å, 1.456(3) Å, 1.217(3) Å and 1.200(3) Å, respectively. The bond lengths of C1—S and C7—S are 1.743(3) Å and 1.772(3) Å, respectively. The bond lengths of C6—N, C7—N and C8—N are 1.395(3) Å, 1.375(3) Å and 1.457(4) Å, respectively.

Table 1. Data collection and handling.

Crystal:	white needle, size 0.12 × 0.24 × 0.30 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.49 cm^{-1}
Diffractometer, scan mode:	Enraf-Nonius CAD-4, $\omega/2\theta$
$2\theta_{max}$:	70.22°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	1681, 1560
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 947
$N(param)_{refined}$:	224
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(9A)	4e	0.723(5)	0.345(1)	1.168(2)	0.065(9)
H(9B)	4e	0.501(5)	0.297(1)	1.148(2)	0.063(8)
H(10A)	4e	0.744(6)	0.353(1)	0.970(2)	0.073(9)
H(12A)	4e	0.232(5)	0.497(1)	0.958(2)	0.061(9)
H(10B)	4e	0.508(5)	0.307(1)	0.954(2)	0.062(9)
H(4)	4e	0.190(5)	0.114(1)	1.242(2)	0.067(9)
H(12B)	4e	0.019(6)	0.455(1)	0.889(2)	0.077(9)
H(8A)	4e	1.024(5)	0.278(1)	1.095(2)	0.059(8)
H(2)	4e	0.337(4)	0.052(1)	0.943(2)	0.050(9)
H(3)	4e	0.110(6)	0.042(1)	1.095(2)	0.08(1)
H(13A)	4e	0.221(6)	0.479(2)	0.722(3)	0.11(1)
H(13B)	4e	0.125(7)	0.540(2)	0.778(2)	0.10(1)
H(13C)	4e	0.438(6)	0.521(1)	0.788(2)	0.08(1)
H(8B)	4e	0.899(5)	0.244(1)	1.199(2)	0.073(9)
H(5)	4e	0.497(4)	0.193(1)	1.223(2)	0.044(7)

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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	4*e*	0.7336(2)	0.14268(4)	0.86967(7)	0.0798(5)	0.0838(8)	0.0495(6)	0.0112(5)	0.0101(4)	–0.0018(4)
O(3)	4*e*	0.3982(3)	0.42275(8)	0.9059(2)	0.065(1)	0.057(2)	0.062(2)	0.015(1)	0.0127(9)	0.009(1)
N	4*e*	0.7852(4)	0.2122(1)	1.0436(2)	0.051(1)	0.048(2)	0.051(2)	0.001(1)	0.002(1)	0.003(1)
C(11)	4*e*	0.3897(5)	0.3894(1)	0.9990(3)	0.044(1)	0.051(3)	0.062(3)	–0.005(1)	0.004(1)	0.001(2)
O(1)	4*e*	1.0497(4)	0.2399(1)	0.9007(2)	0.074(1)	0.091(2)	0.084(2)	–0.004(1)	0.027(1)	0.022(1)
C(6)	4*e*	0.5974(5)	0.1680(1)	1.0697(2)	0.044(1)	0.042(2)	0.049(2)	0.004(1)	–0.001(1)	0.005(1)
C(7)	4*e*	0.8850(5)	0.2064(1)	0.9397(3)	0.055(2)	0.070(3)	0.063(2)	0.011(2)	0.009(2)	0.013(2)
C(9)	4*e*	0.6581(6)	0.3129(2)	1.1169(3)	0.054(2)	0.054(3)	0.059(2)	–0.005(2)	–0.002(1)	–0.003(2)
C(5)	4*e*	0.4662(5)	0.1633(2)	1.1679(3)	0.061(2)	0.050(3)	0.053(3)	0.008(2)	0.005(1)	0.001(2)
C(10)	4*e*	0.5912(6)	0.3389(2)	1.0018(3)	0.052(2)	0.057(3)	0.061(3)	0.001(2)	0.006(1)	0.007(2)
C(4)	4*e*	0.2838(6)	0.1167(2)	1.1774(3)	0.064(2)	0.068(3)	0.064(3)	0.001(2)	0.016(2)	0.016(2)
C(12)	4*e*	0.2066(7)	0.4729(2)	0.8931(3)	0.065(2)	0.056(3)	0.070(3)	0.016(2)	0.005(2)	–0.002(2)
C(8)	4*e*	0.8653(6)	0.2621(2)	1.1187(3)	0.049(2)	0.058(3)	0.067(3)	–0.005(2)	–0.005(1)	0.006(2)
C(1)	4*e*	0.5461(5)	0.1263(1)	0.9839(2)	0.056(2)	0.049(3)	0.050(2)	0.005(2)	–0.003(1)	0.003(2)
C(2)	4*e*	0.3660(6)	0.0797(2)	0.9962(3)	0.079(2)	0.056(3)	0.066(3)	–0.003(2)	–0.007(2)	–0.010(2)
C(3)	4*e*	0.2334(6)	0.0755(2)	1.0925(3)	0.069(2)	0.070(3)	0.082(3)	–0.015(2)	0.000(2)	0.007(2)
C(13)	4*e*	0.2536(9)	0.5049(2)	0.7879(4)	0.088(3)	0.072(3)	0.077(4)	0.010(2)	0.005(2)	0.012(2)
O(2)	4*e*	0.2352(4)	0.39953(9)	1.0697(2)	0.068(1)	0.085(2)	0.069(2)	0.013(1)	0.025(1)	0.009(1)

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