Crystal structure of 1-[2(3*H*)-benzothiazolone-3-yl]propanoyl-morpholine, $C_{14}H_{16}N_2O_3S$

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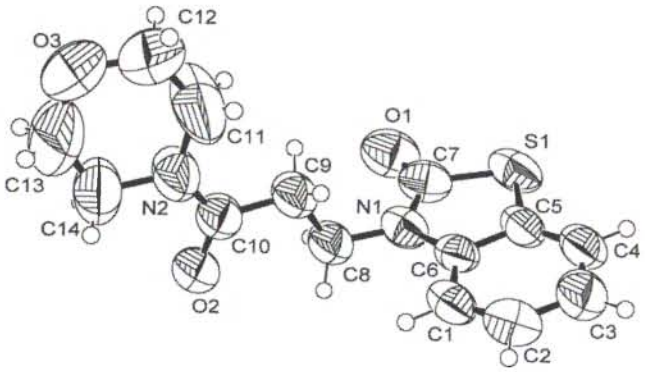

Abstract

$C_{14}H_{16}N_2O_3S$, orthorhombic, *Pnaa* (No. 56), $a = 8.933(1)$ Å, $b = 11.391(1)$ Å, $c = 27.983(1)$ Å, $V = 2847.4$ Å^3, $Z = 8$, $R_{gt}(F) = 0.080$, $wR_{ref}(F^2) = 0.228$, $T = 293$ K.

Source of material

0.01 mol metallic sodium was dissolved in 30 ml of ethanol. Ethanol was evaporated. The residue was dissolved in 20 ml of *N*,*N*-dimethylformamide, 0.01 mol 2-oxobenzothiazoline and 0.01 mol of corresponding chloroamide were added, the eventual solution was refluxed and the reaction p rogression was monitored through TLC on silicagel using methanol-chloroform mixture (1:4) as the solvent system. When reaction mixture was poured in 50 ml of ice-cold water and stirred. The oil separated was taken into 50 ml of acetone and treated with activated charcoal. The acetone was evaporated and the residue was crystallized from alcohol-water mixture.

Discussion

The bond lengths and angles in the benzothiazolinone are all in accord with similar structures in the literature [1,2,3,4]. C11—N2 and C14—N2 bonds are clearly longer than the normal N—C bond and similar results have been observed in related compounds containing piperazin [1.44(2) Å and 1.46(2) Å]. The average C=O distances are the same in the other similar structures [1,4]. The bonds lengths S1—C5 and S1—C7 are 1.73(2) Å and 1.78(1) Å, respectively. In similar studies, those lengths have been reported to be 1.77(3) Å and 1.73(2) Å in 4-(2-carboxybenzoyl)-2(3*H*)-benzothiazolone [1] 1.71(1) Å and 1.72(2) Å in the structure of styrylbenzothiazole platinum (II) complex: $[NEt_4][PtBr_3(asb)]$ [3], 1.74(2) Å and 1.79(3)Å in the structure of 6-benzoyl-2,3-dihydro-1,3-benzothiazol-2-one [4]. C9 atom lies above 1.48(1) Å from plane 3*H*-benzothiazolone. Torsion angles C6–N1–C8–C9, N1–C8–C9–C10, C14–N2–C10–O2 and C8–C9–C10–O2 are 84.6(9)°, −170.7(8)°, −3.7(15)° and 3.9(13)°, respectively. O2 atom lies 0.33(2) Å below the benzene plane. The dihedral angle between the planes defined by C1-C6 and thiazolone plane is 1.81(5)°, implying that the 3*H*-benzothiazolone ring is essentialy planar. In other similar structures the thiazolone ring and phenyl ring are coplanar [2, 4]. The dihedral angle between the 3*H*-benzothiazolone and defined by N2-C11-C12-O3-C13-C14 plane is 77.45(6) . Similar high values for the diplacement parameters of C10-C13 have been found in lignocaine tetrabromozincate [5].

Table 1. Data collection and handling.

Crystal:	white parallelepiped, size 0.30 × 0.35 × 0.40 mm
Wavelength:	Cu K_α radiation (1.54184 Å)
μ:	21.06 cm^{-1}
Diffractometer, scan mode:	Enraf-Nonius CAD-4, ω/2θ
$2\theta_{max}$:	103.52°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	1687, 1532
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1210
$N(param)_{refined}$:	182
Programs:	SHELXS-97 [6], SHELXL [7], MoEN [8], ORTEP [9]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	8*e*	−0.3026	0.4475	0.4874	0.102
H(2)	8*e*	−0.3571	0.4922	0.5637	0.114
H(3)	8*e*	−0.2424	0.6458	0.6032	0.127
H(4)	8*e*	−0.0663	0.7569	0.5623	0.114
H(8A)	8*e*	−0.1924	0.4086	0.4131	0.104
H(8B)	8*e*	−0.0966	0.4644	0.3719	0.104
H(9A)	8*e*	−0.3754	0.5582	0.3987	0.109
H(9B)	8*e*	−0.2839	0.5932	0.3531	0.109
H(11A)	8*e*	−0.6028	0.6138	0.3472	0.337
H(11B)	8*e*	−0.4805	0.6378	0.3087	0.337
H(12A)	8*e*	−0.6188	0.6701	0.2668	0.232
H(12B)	8*e*	−0.7401	0.6499	0.3055	0.232
H(13A)	8*e*	−0.7775	0.3895	0.2710	0.450
H(13B)	8*e*	−0.6593	0.4093	0.2316	0.450
H(14A)	8*e*	−0.5156	0.3491	0.2696	0.169
H(14B)	8*e*	−0.6361	0.3269	0.3092	0.169

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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)	8*e*	0.0390(3)	0.7444(2)	0.4632(1)	0.085(2)	0.061(2)	0.149(3)	–0.012(1)	–0.009(1)	0.004(1)
N(1)	8*e*	–0.1072(7)	0.5671(5)	0.4306(2)	0.070(4)	0.059(4)	0.100(5)	–0.005(3)	0.001(4)	0.001(3)
N(2)	8*e*	–0.512(1)	0.4680(7)	0.3211(3)	0.106(6)	0.090(6)	0.143(7)	0.015(4)	–0.048(6)	–0.025(5)
C(1)	8*e*	–0.255(1)	0.5105(7)	0.5022(4)	0.086(5)	0.058(5)	0.110(7)	0.001(5)	–0.006(6)	–0.005(5)
C(2)	8*e*	–0.287(1)	0.5373(9)	0.5475(4)	0.094(7)	0.077(6)	0.114(8)	0.001(5)	0.011(5)	0.009(5)
C(3)	8*e*	–0.219(1)	0.629(1)	0.5716(4)	0.128(9)	0.083(7)	0.107(7)	0.018(6)	–0.019(7)	–0.008(6)
C(4)	8*e*	–0.115(1)	0.6953(8)	0.5470(4)	0.110(7)	0.059(5)	0.116(8)	0.009(5)	–0.031(6)	–0.009(5)
C(5)	8*e*	–0.0831(9)	0.6711(7)	0.5007(3)	0.084(5)	0.053(5)	0.097(6)	0.000(4)	–0.011(4)	–0.006(4)
C(6)	8*e*	–0.1511(9)	0.5779(6)	0.4772(3)	0.072(5)	0.050(4)	0.089(5)	0.001(4)	–0.009(4)	0.005(4)
C(7)	8*e*	–0.007(1)	0.6490(8)	0.4149(4)	0.083(6)	0.081(6)	0.106(6)	0.010(5)	0.011(5)	0.011(5)
C(8)	8*e*	–0.170(1)	0.4813(7)	0.3965(3)	0.094(6)	0.069(5)	0.098(6)	0.010(4)	–0.013(5)	–0.006(4)
C(9)	8*e*	–0.310(1)	0.5283(7)	0.3740(4)	0.094(6)	0.068(5)	0.111(7)	0.011(4)	–0.024(5)	–0.010(5)
C(10)	8*e*	–0.391(1)	0.4368(8)	0.3457(3)	0.086(6)	0.072(6)	0.085(5)	0.009(5)	–0.005(5)	–0.007(4)
C(11)	8*e*	–0.566(3)	0.589(1)	0.316(1)	0.31(3)	0.11(1)	0.42(3)	0.11(2)	–0.27(3)	–0.12(2)
C(12)	8*e*	–0.662(3)	0.611(1)	0.2874(6)	0.25(2)	0.14(1)	0.19(2)	0.05(1)	–0.10(2)	0.02(1)
C(13)	8*e*	–0.687(4)	0.433(2)	0.264(1)	0.51(5)	0.17(2)	0.44(4)	0.00(3)	–0.41(5)	0.00(2)
C(14)	8*e*	–0.590(2)	0.387(1)	0.2898(5)	0.15(1)	0.13(1)	0.147(9)	0.007(8)	–0.057(9)	–0.037(8)
O(1)	8*e*	0.0411(8)	0.6585(6)	0.3751(3)	0.119(6)	0.103(5)	0.135(6)	–0.001(4)	0.032(5)	0.013(4)
O(2)	8*e*	–0.3466(8)	0.3360(5)	0.3444(2)	0.126(5)	0.072(4)	0.103(4)	0.007(4)	–0.021(4)	–0.005(3)
O(3)	8*e*	–0.733(1)	0.539(1)	0.2585(4)	0.171(9)	0.17(1)	0.169(8)	0.022(8)	–0.095(7)	0.020(7)

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