Crystal structure of (2*Z*)-*N*,3-ditosylthiazol-2(3*H*)-imine, $C_{17}H_{16}N_2O_4S_3$

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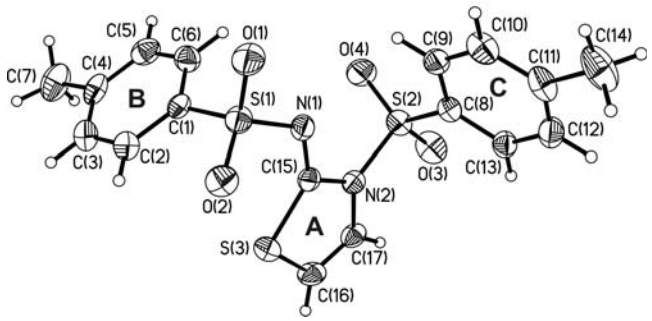

Abstract

$C_{17}H_{16}N_2O_4S_3$, monoclinic, $P2_1/n$ (no. 14), a = 8.312(2) Å, b = 12.343(3) Å, c = 18.077(4) Å, β = 101.35(3)°, V = 1818.4 Å^3, Z = 4, $R_{gt}(F)$ = 0.057, $wR_{ref}(F^2)$ = 0.192, T = 200 K.

Source of material

2-Aminothiazole (5 mmol) was added to a solution of 4-toluenesulfonyl chloride (5 mmol) in CH_2Cl_2 (30 mL). The pH of resulting mixture was adjusted to 8 with an aqueous solution of sodium carbonate. The reaction mixture was stirred at room temperature. The progress of reaction was monitored by thin layer chromatography (TLC) using ethyl acetate and normal hexane in 2 : 1 ratio as eluent. After completion of the reaction (10 h), water and ethyl acetate (50 mL, 1:1, *v*/*v*) was added, and the organic layer was then separated. The solvent was evaporated and solid residue was filtered, washed with cold CH_2Cl_2 (10 mL) and recrystallized from CH_2Cl_2.

Discussion

Sulfonamides, an important class of pharmaceutical compounds, actually exhibit a wide spectrum of biological activities as antibacterials, diuretics, hypoglycemics and HIV protease inhibitors [1]. Aromatic sulfonamides are strong inhibitors of carbonic anhydrase and are of pharmacological value because of their effects on various physiological reactions ultimately involving bicarbonate. Sulfonamides have been useful in studies of the physicochemistry and mechanism of carbonic anhydrase due to their highly specific interaction with the active site [2]. The practice of synthesizing numerous structurally related compounds in an effort to find some that are more efficient or have fewer side effects than those already available is very important to the pharmaceutical industry. Moreover, sulfonamide containing different donor atoms also finds use in coordination chemistry [3-5]. Recently we reported on synthesis of new sulfonamide compounds [6,7].

The bond lengths S—O (d(S1—O1) = 1.432(3) Å, d(S1—O2) = 1.441(3) Å, d(S2—O3) = 1.426(3) Å, d(S2—O4) = 1.418(3) Å) and S—N (d(S1—N1) = 1.618(3) Å, d(S2—N2) = 1.711(3) Å) are in accordance with the values from the literature [8,9].

The molecular structure is stabilized by some intramolecular C–H···O hydrogen bonds (d(C2–H2B···O2) = 2.899(5) Å, ∠C2–H2B···O2 = 104°; d(C2–H13B···O3) = 2.920(5) Å, ∠C2–H13B···O3 = 102°) and intermolecular hydrogen bonds (d(C7–H7B···O2) = 3.531(6) Å, ∠C7–H7B···O2 = 164° and d(C14–H14C···O3) = 3.501(6) Å, ∠C14–H14C···O3 = 162°). The basic rings skeletons are: **A** (S3/C15/N2/C17/C16), **B** (C1-C6) and **C** (C8-C13). The dihedral angles formed by the planes **A/B**, **A/C** and **B/C** are 84.1(2)°, 72.4(2)° and 71.3(2)°, respectively. The other intermolecular C–H···π interactions involve the hydrogen atoms of the –CH from thiazolo ring in one molecule and the aromatic ring of neighboring molecule (d(C17–H17A···Cg2) = 2.690 Å, Cg2 is centroid of ring **B**). The title sulfunamide shows imido-amido tautomerism, which has been reported for some sulfunamide compounds [7,8].

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.30 × 0.30 × 0.50 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	4.34 cm^{-1}
Diffractometer, scan mode:	Stoe Stadi 4, ω
$2\theta_{max}$:	63.7°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	8169, 4181
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2475
$N(param)_{refined}$:	237
Programs:	SHELXS-97, SHELXL-97 [10]

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

Atom	Site	x	y	z	U_{iso}
H(2B)	4*e*	0.3183	0.6754	0.2161	0.045
H(3B)	4*e*	0.5851	0.7300	0.2650	0.048
H(5A)	4*e*	0.7366	0.4189	0.2723	0.047
H(6A)	4*e*	0.4716	0.3635	0.2218	0.041
H(7A)	4*e*	0.8621	0.6899	0.3241	0.085
H(7B)	4*e*	0.9219	0.6193	0.2603	0.085
H(7C)	4*e*	0.9169	0.5666	0.3407	0.085
H(9A)	4*e*	0.1413	0.1887	0.0915	0.039
H(10A)	4*e*	–0.1104	0.1033	0.0823	0.047
H(12A)	4*e*	–0.1978	0.1338	–0.1446	0.046
H(13A)	4*e*	0.0513	0.2217	–0.1368	0.037
H(14A)	4*e*	–0.3591	0.0353	0.0126	0.089
H(14B)	4*e*	–0.3415	–0.0133	–0.0675	0.089
H(14C)	4*e*	–0.4297	0.1011	–0.0631	0.089
H(16)	4*e*	0.3049	0.6349	–0.0934	0.044
H(17A)	4*e*	0.3387	0.4496	–0.1192	0.038

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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*	0.1706(1)	0.47164(8)	0.17290(5)	0.0328(5)	0.0349(5)	0.0290(5)	–0.0051(4)	0.0087(4)	0.0010(4)
S(2)	4*e*	0.3099(1)	0.27040(8)	–0.01859(6)	0.0258(5)	0.0300(5)	0.0388(6)	0.0004(4)	0.0078(4)	0.0000(4)
S(3)	4*e*	0.2208(1)	0.59860(8)	0.02317(6)	0.0398(6)	0.0275(5)	0.0360(6)	–0.0017(4)	0.0070(4)	0.0034(4)
O(1)	4*e*	0.1172(4)	0.3929(2)	0.2210(2)	0.051(2)	0.051(2)	0.035(2)	–0.014(2)	0.015(1)	0.005(1)
O(2)	4*e*	0.0721(3)	0.5674(2)	0.1535(2)	0.032(2)	0.044(2)	0.042(2)	0.006(1)	0.008(1)	0.001(1)
O(3)	4*e*	0.3604(4)	0.2545(2)	–0.0888(2)	0.045(2)	0.045(2)	0.052(2)	0.002(1)	0.028(2)	–0.005(1)
O(4)	4*e*	0.4164(3)	0.2439(2)	0.0504(2)	0.029(2)	0.042(2)	0.054(2)	0.005(1)	–0.007(1)	0.007(1)
N(1)	4*e*	0.1983(4)	0.4060(3)	0.0989(2)	0.039(2)	0.031(2)	0.025(2)	–0.007(2)	0.005(1)	–0.001(1)
N(2)	4*e*	0.2749(4)	0.4069(2)	–0.0177(2)	0.026(2)	0.030(2)	0.025(2)	–0.002(1)	0.004(1)	0.001(1)
C(1)	4*e*	0.3703(5)	0.5142(3)	0.2139(2)	0.035(2)	0.032(2)	0.018(2)	0.000(2)	0.006(2)	–0.001(1)
C(2)	4*e*	0.4038(6)	0.6232(3)	0.2271(2)	0.043(2)	0.034(2)	0.033(2)	0.000(2)	0.006(2)	–0.003(2)
C(3)	4*e*	0.5621(5)	0.6552(4)	0.2562(2)	0.043(3)	0.039(2)	0.038(2)	–0.005(2)	0.005(2)	–0.009(2)
C(4)	4*e*	0.6884(5)	0.5810(4)	0.2730(2)	0.034(2)	0.055(3)	0.022(2)	–0.007(2)	0.006(2)	–0.007(2)
C(5)	4*e*	0.6517(5)	0.4713(4)	0.2603(2)	0.040(2)	0.047(2)	0.031(2)	0.010(2)	0.009(2)	0.002(2)
C(6)	4*e*	0.4948(5)	0.4383(3)	0.2308(2)	0.038(2)	0.032(2)	0.034(2)	0.001(2)	0.010(2)	–0.001(2)
C(7)	4*e*	0.8626(6)	0.6174(5)	0.3021(3)	0.036(3)	0.083(4)	0.047(3)	–0.009(3)	0.001(2)	–0.011(3)
C(8)	4*e*	0.1186(4)	0.2115(3)	–0.0224(2)	0.025(2)	0.025(2)	0.030(2)	0.001(2)	0.009(2)	–0.003(2)
C(9)	4*e*	0.0720(5)	0.1774(3)	0.0437(2)	0.042(2)	0.030(2)	0.027(2)	–0.002(2)	0.009(2)	0.000(2)
C(10)	4*e*	–0.0764(6)	0.1271(3)	0.0378(2)	0.042(2)	0.035(2)	0.045(2)	–0.004(2)	0.023(2)	–0.002(2)
C(11)	4*e*	–0.1790(5)	0.1099(3)	–0.0319(3)	0.027(2)	0.029(2)	0.061(3)	–0.000(2)	0.013(2)	–0.010(2)
C(12)	4*e*	–0.1289(5)	0.1453(3)	–0.0967(2)	0.034(2)	0.037(2)	0.040(2)	–0.001(2)	0.000(2)	–0.007(2)
C(13)	4*e*	0.0183(5)	0.1966(3)	–0.0924(2)	0.033(2)	0.031(2)	0.029(2)	–0.003(2)	0.007(2)	–0.002(2)
C(14)	4*e*	–0.3415(6)	0.0533(4)	–0.0380(4)	0.035(3)	0.043(3)	0.106(4)	–0.010(2)	0.025(3)	–0.011(3)
C(15)	4*e*	0.2285(4)	0.4602(3)	0.0413(2)	0.019(2)	0.028(2)	0.028(2)	–0.003(2)	–0.000(1)	0.000(2)
C(16)	4*e*	0.2858(5)	0.5780(3)	–0.0608(2)	0.038(2)	0.038(2)	0.033(2)	–0.007(2)	0.006(2)	0.012(2)
C(17)	4*e*	0.3061(5)	0.4751(3)	–0.0748(2)	0.031(2)	0.040(2)	0.024(2)	–0.006(2)	0.006(2)	0.005(2)

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