Crystal structure of 4-[4-(dimethylamino)phenyl]-5-(2,4-dimethyl-1,3-thiazol-5-yl)-2,4-dihydro-3*H*-1,2,4-triazol-3-thione, $C_{15}H_{17}N_5S_2$

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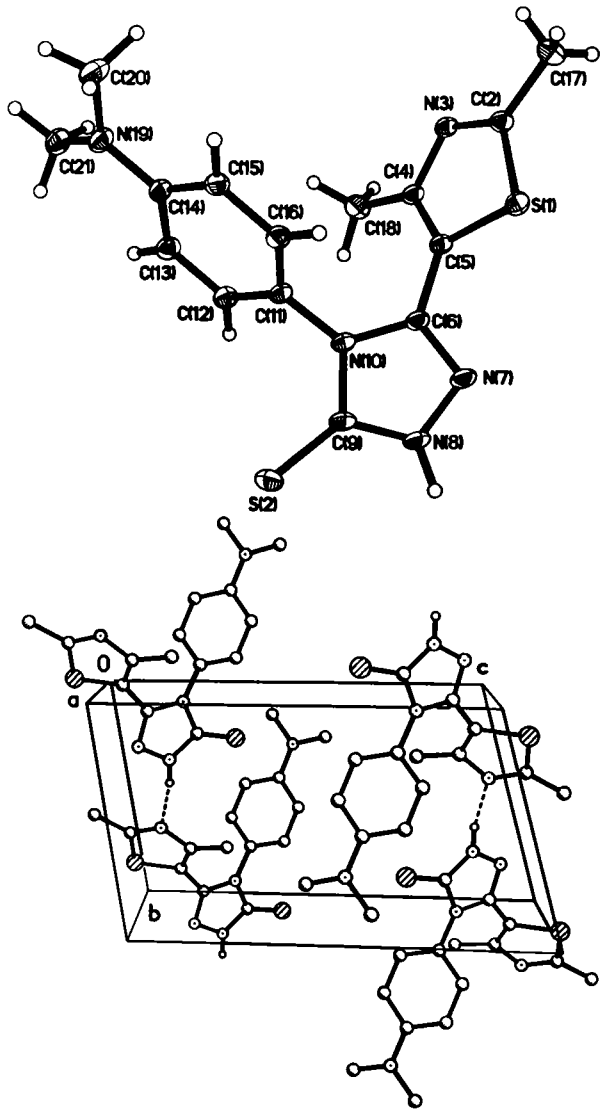

Abstract

$C_{15}H_{17}N_5S_2$, triclinic, $P\bar{1}$ (No. 2), $a = 7.822(1)$ Å, $b = 8.355(1)$ Å, $c = 13.925(2)$ Å, $\alpha = 75.651(4)°$, $\beta = 82.732(3)°$, $\gamma = 62.609(3)°$, $V = 782.7$ Å^3, $Z = 2$, $R_{gt}(F) = 0.054$, $wR_{ref}(F^2) = 0.155$, $T = 110$ K.

Source of material

4-[4-(Dimethylamino)phenyl]-5-(2,4-dimethyl-1,3-thiazol-5-yl)-2,4-d hydro-3*H*-1,2,4-triazol-3-thione was synthesized in accordance with a published procedure [1]. The solution of title compound (0.5 g) in acetonitrile-benzene (1:1, 20 ml) left in room temperature for three days. The colorless crystals were filtered off, washed with cold acetonitrile-benzene (1:1, 5 ml) and dried in vacuum over P_4O_{10} (mp 498 K; yield 55%). Elemental analyses were consistent with the stiochiometry $C_{15}H_{17}N_5S_2$ (Found: C, 54.31%; H, 5.21%; N, 21.18%. Calc.: C, 54.36%; H, 5.17%; N, 21.13%).

Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected. Elemental analyses were performed using a Heraeus CHN-O-Rapid analyzer.

Discussion

Thiazolo-1,2,4-triazole derivatives exhibit antibacterial and antifungal activity [1]. In the recent years there has been increasing interest in the determination of X-ray crystal structures of biologically active compounds [2-10].

In the crystal structure of title compound, the S—C distance of 1.6738(14) Å agrees well with the values in the literature of 1.687(3) Å, 1.681(5) Å, 1.679(4) Å and 1.678(3) Å, being intermediate between the values of 1.82 Å for a S—C single bond and 1.56 Å for S=C double bond [10]. The single bonds N10—C6 (1.389(2) Å), N10—C9 (1.389 (2) Å) and N19—C14 (1.375(2) Å) are slightly short. π-Systems conjugations and banana characteristic of these bonds appears to be a plausible factors in the reduction of these single bonds lengths. The N10—C11 bond length (1.436(2) Å) show that there is no π-system conjugation between benzene and triazole rings [10]. The bond angles C14–N19–C20 and C21–N19–C20 (120.0(1)° and 118.3(1)°, respectively) agree well with a participation of N19 in the electron donation to the benzene ring. The C9–N10–C11–C16 and C4–C5–C6–N7 (115.2(2)° and 131.4(2)°, respectively) torsion angles show clearly that the three rings do not share a common plane probably is as a result of steric intractions. Because of the fairly high acidity of H8, the N8-atom has the potential to act as a hydrogen donor in the intermolecular hydrogen bonding. Also, because of the presence fairly high electron-rich atom (N3) in the thiazole ring of the molecule, it able to act as a hydrogen acceptor in intermolecular hydrogen bonding that stabilize the crystal packing. No ring stacking is observed within the crystals probably is as a result of the presence of different plans for the three rings in the molecule.

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Table 1. Data collection and handling.

Crystal:	colorless, prism, size 0.3 × 0.5 × 0.5 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	3.44 cm^{-1}
Diffractometer, scan mode:	Bruker SMARTCCD, φ, ω
$2\theta_{max}$:	60.04°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6206, 4434
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3985
$N(param)_{refined}$:	199
Programs:	SHELXTL [11] SHEXTL-97 [12], SADABS [13]

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

Atom	Site	x	y	z	U_{iso}
H(8)	2*i*	–0.7124	0.4483	0.1252	0.05
H(12)	2*i*	–0.3725	–0.0793	0.4009	0.05
H(13)	2*i*	–0.2854	–0.3706	0.4994	0.05
H(15)	2*i*	–0.3900	–0.5431	0.2686	0.05
H(16)	2*i*	–0.4761	–0.2475	0.1699	0.05
H(17A)	2*i*	0.0487	–0.4015	–0.1291	0.05
H(17B)	2*i*	0.2058	–0.3220	–0.1349	0.05
H(17C)	2*i*	0.1865	–0.4870	–0.0730	0.05
H(18A)	2*i*	0.1513	–0.2265	0.2253	0.05
H(18B)	2*i*	–0.0645	–0.1135	0.2611	0.05
H(18C)	2*i*	0.0126	–0.3195	0.2733	0.05
H(20A)	2*i*	–0.2659	–0.6250	0.5887	0.05
H(20B)	2*i*	–0.0577	–0.7164	0.5395	0.05
H(20C)	2*i*	–0.1810	–0.8347	0.5824	0.05
H(21A)	2*i*	–0.3903	–0.7805	0.3878	0.05
H(21B)	2*i*	–0.1635	–0.8478	0.3627	0.05
H(21C)	2*i*	–0.2477	–0.9111	0.4676	0.05

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*	–0.21185(4)	–0.08756(4)	–0.03669(2)	0.0131(2)	0.0168(2)	0.0174(2)	–0.00233	–0.0039(1)	–0.0009(1)
S(2)	2*i*	–0.79212(5)	0.24633(5)	0.31664(3)	0.0173(2)	0.0212(2)	0.0241(2)	–0.0008(2)	0.0025(1)	–0.0060(2)
N(3)	2*i*	0.0842(2)	–0.3073(2)	0.06947(8)	0.0127(5)	0.0136(5)	0.0180(5)	–0.0022(4)	–0.0016(4)	–0.0018(4)
N(7)	2*i*	–0.4703(2)	0.2506(2)	0.07774(9)	0.0123(5)	0.0130(5)	0.0234(6)	–0.0008(4)	–0.0021(4)	–0.0015(4)
N(8)	2*i*	–0.6198(2)	0.3285(2)	0.14131(9)	0.0132(5)	0.0117(5)	0.0242(6)	0.0009(4)	–0.0019(4)	–0.0024(4)
N(10)	2*i*	–0.4738(2)	0.0417(2)	0.21243(9)	0.0116(5)	0.0120(5)	0.0185(5)	–0.0002(4)	–0.0020(4)	–0.0014(4)
N(19)	2*i*	–0.2725(2)	–0.6649(2)	0.45438(9)	0.0251(6)	0.0145(5)	0.0184(5)	–0.0069(5)	–0.0051(4)	0.0007(4)
C(2)	2*i*	0.0158(2)	–0.2691(2)	–0.0190(1)	0.0122(5)	0.0148(6)	0.0191(6)	–0.0033(5)	–0.0004(4)	–0.0029(5)
C(4)	2*i*	–0.0432(2)	–0.1900(2)	0.1283(1)	0.0118(5)	0.0125(5)	0.0178(6)	–0.0021(4)	–0.0014(4)	–0.0011(4)
C(5)	2*i*	–0.2142(2)	–0.0622(2)	0.0832(1)	0.0118(5)	0.0125(5)	0.0173(6)	–0.0019(4)	–0.0018(4)	–0.0016(4)
C(6)	2*i*	–0.3828(2)	0.0771(2)	0.1231(1)	0.0107(5)	0.0122(6)	0.0192(6)	–0.0016(4)	–0.0017(4)	–0.0013(4)
C(9)	2*i*	–0.6295(2)	0.2070(2)	0.2239(1)	0.0110(5)	0.0132(6)	0.0223(6)	0.0001(4)	–0.0022(4)	–0.0039(5)
C(11)	2*i*	–0.4294(2)	–0.1364(2)	0.2756(1)	0.0130(5)	0.0118(5)	0.0173(6)	–0.0018(4)	–0.0024(4)	–0.0008(4)
C(12)	2*i*	–0.3751(2)	–0.1745(2)	0.3734(1)	0.0184(6)	0.0158(6)	0.0191(6)	–0.0058(5)	–0.0026(5)	–0.0038(5)
C(13)	2*i*	–0.3205(2)	–0.3492(2)	0.4323(1)	0.0184(6)	0.0163(6)	0.0172(6)	–0.0055(5)	–0.0032(5)	–0.0023(5)
C(14)	2*i*	–0.3231(2)	–0.4916(2)	0.3956(1)	0.0149(6)	0.0140(6)	0.0157(6)	–0.0043(5)	–0.0009(4)	–0.0006(5)
C(15)	2*i*	–0.3834(2)	–0.4485(2)	0.2969(1)	0.0184(6)	0.0148(6)	0.0167(6)	–0.0046(5)	–0.0017(5)	–0.0037(5)
C(16)	2*i*	–0.4355(2)	–0.2731(2)	0.2378(1)	0.0145(6)	0.0157(6)	0.0173(6)	–0.0035(5)	–0.0025(4)	–0.0023(5)
C(17)	2*i*	0.1231(2)	–0.3694(2)	–0.0991(1)	0.0199(6)	0.0245(7)	0.0230(7)	–0.0080(6)	0.0029(5)	–0.0096(5)
C(18)	2*i*	0.0185(2)	–0.2112(2)	0.2295(1)	0.0151(6)	0.0241(7)	0.0183(6)	–0.0028(5)	–0.0040(5)	–0.0044(5)
C(20)	2*i*	–0.1840(2)	–0.7141(2)	0.5495(1)	0.0216(7)	0.0194(6)	0.0196(6)	–0.0053(5)	–0.0054(5)	0.0022(5)
C(21)	2*i*	–0.2653(2)	–0.8113(2)	0.4136(1)	0.0299(8)	0.0132(6)	0.0266(7)	–0.0063(5)	–0.0058(6)	–0.0016(5)

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