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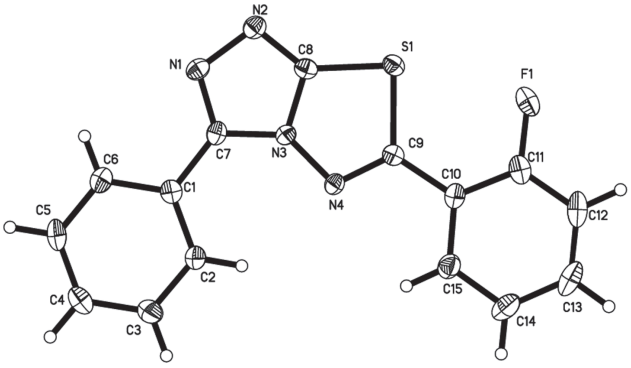
Crystal structure of 6-(2-fluorophenyl)-3-phenyl-[1,2,4]-triazolo[3,4-*b*][1,3,4]thiadiazole, $C_{15}H_9FN_4S$



DOI 10.1515/ncrs-2015-0236
Received January 25, 2016; accepted February 9, 2016; available online February 26, 2016

Abstract

$C_{15}H_9FN_4S$, orthorhombic, $Pna2_1$ (no. 33), $a = 18.9361(2)$ Å, $b = 11.5248(1)$ Å, $c = 6.0142(1)$ Å, $V = 1312.52(3)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0263$, $wR_{ref}(F^2) = 0.0706$, $T = 100$ K.

CCDC no.: 1416454

The crystal structure is shown in the figure. Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Colourless, prism, size 0.1467×0.1714×0.3504 mm
Wavelength:	Cu $K\alpha$ radiation (1.54184 Å)
μ:	22.9 cm^{-1}
Diffractometer, scan mode:	Xcalibur, Ruby, Gemini, ω scans
$2\theta_{max}$:	140.94°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6872, 2510
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2044
$N(param)_{refined}$:	190
Programs:	CrysAlis^{PRO} [16], SHELX [17]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(12)	4*a*	0.2635	0.6295	−0.5280	0.042
H(5)	4*a*	0.5152	0.0662	0.9684	0.041
H(13)	4*a*	0.3812	0.6383	−0.6289	0.047
H(15)	4*a*	0.4320	0.4358	−0.1120	0.037
H(2)	4*a*	0.4707	0.2752	0.3255	0.036
H(6)	4*a*	0.4001	0.1159	0.8790	0.036
H(3)	4*a*	0.5853	0.2271	0.4230	0.042
H(4)	4*a*	0.6076	0.1239	0.7454	0.040
H(14)	4*a*	0.4649	0.5392	−0.4229	0.047

Source of material

A mixture of 4-amino-5-phenyl-4*H*-1,2,4-triazole-3-thiol (1.92 g, 0.01 mol), 2-fluorobenzoyl chloride (1.40 g, 0.01 mol) and phosphorous oxychloride (10 mL) was heated under reflux for four hours. On cooling, the reaction mixture was cautiously poured onto crushed ice (50 gm) and the precipitated solid product was filtered, washed with saturated sodium hydrogen carbonate solution and then with water, dried, crystallized from ethanol to yield 2.16 g (73%) of the title compound. M.p. 517–519 K. Colourless prismatic crystals were obtained by slow evaporation of chloroform-ethanol solution (1:1) at room temperature. ^{1}H NMR ($CDCl_3$, 500.13 MHz): δ 7.16–7.33 (m, 6H, aromatic-H), 7.55–7.77 (m, 3H, aromatic-H). ^{13}C NMR ($CDCl_3$, 125.76 MHz): δ = 115.11, 120.73, 124.27, 127.54, 127.61, 127.77, 133.45, 133.52, 144.57, 158.43 (aromatic-C), 160.05 (C-3), 160.46 (C-8), 163.96 (C-6).

Table 3: Fractional coordinates and atomic displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S(1)	4*a*	0.22196(2)	0.39409(3)	0.17939(9)	0.0203(2)	0.0243(2)	0.0278(2)	0.0004(1)	−0.0020(2)	−0.0002(2)
F(1)	4*a*	0.20979(6)	0.5248(1)	−0.2014(2)	0.0355(6)	0.0347(6)	0.0354(7)	0.0103(5)	−0.0101(5)	−0.0024(5)
N(3)	4*a*	0.32725(7)	0.2993(1)	0.3665(3)	0.0216(7)	0.0203(6)	0.0201(8)	−0.0016(6)	0.0010(7)	0.0000(6)
N(4)	4*a*	0.35789(7)	0.3513(1)	0.1858(3)	0.0241(7)	0.0211(6)	0.0184(7)	−0.0016(5)	0.0000(8)	0.0000(7)
N(1)	4*a*	0.29497(7)	0.2115(1)	0.6688(3)	0.0273(7)	0.0224(6)	0.0250(8)	−0.0034(5)	0.0043(8)	0.0000(7)
N(2)	4*a*	0.23441(8)	0.2597(1)	0.5748(3)	0.0275(8)	0.0250(7)	0.0252(9)	−0.0038(6)	0.0004(8)	−0.0001(7)
C(12)	4*a*	0.2976(1)	0.5912(2)	−0.4444(4)	0.062(1)	0.0189(8)	0.023(1)	0.0032(9)	−0.014(1)	−0.0028(8)
C(10)	4*a*	0.3273(1)	0.4703(1)	−0.1265(3)	0.031(1)	0.0186(7)	0.021(1)	−0.0038(7)	−0.0054(8)	−0.0013(7)
C(5)	4*a*	0.5062(1)	0.1091(2)	0.8405(4)	0.045(1)	0.030(1)	0.029(1)	0.0049(8)	−0.013(1)	0.0051(8)
C(13)	4*a*	0.3675(1)	0.5957(2)	−0.5047(4)	0.068(2)	0.027(1)	0.023(1)	−0.015(1)	−0.003(1)	0.0035(8)
C(15)	4*a*	0.3979(1)	0.4750(2)	−0.1939(4)	0.032(1)	0.034(1)	0.026(1)	−0.0076(8)	−0.0042(8)	0.0050(8)
C(11)	4*a*	0.2789(1)	0.5289(2)	−0.2584(4)	0.037(1)	0.0195(8)	0.027(1)	0.0018(7)	−0.0095(9)	−0.0057(7)
C(7)	4*a*	0.3502(1)	0.2355(2)	0.5453(3)	0.030(1)	0.0175(8)	0.0209(9)	−0.0016(7)	−0.0012(8)	0.0001(7)
C(2)	4*a*	0.4793(1)	0.2341(2)	0.4558(4)	0.028(1)	0.0327(9)	0.030(1)	0.0030(8)	−0.0001(9)	0.0091(8)
C(8)	4*a*	0.25608(9)	0.3114(2)	0.3954(3)	0.0207(8)	0.0206(8)	0.028(1)	−0.0015(7)	0.0002(8)	−0.0028(8)
C(6)	4*a*	0.4371(1)	0.1384(2)	0.7867(4)	0.036(1)	0.0278(9)	0.026(1)	−0.0001(8)	0.0013(9)	0.0035(8)
C(1)	4*a*	0.4233(1)	0.2018(1)	0.5933(4)	0.0277(9)	0.0201(8)	0.0250(9)	−0.0004(7)	−0.0024(8)	−0.0022(7)
C(3)	4*a*	0.5480(1)	0.2048(2)	0.5141(4)	0.026(1)	0.038(1)	0.040(1)	0.0023(8)	−0.001(1)	0.004(1)
C(4)	4*a*	0.5615(1)	0.1427(2)	0.7067(4)	0.0296(9)	0.0313(9)	0.040(1)	0.0044(7)	−0.010(1)	−0.0024(9)
C(14)	4*a*	0.4177(1)	0.5370(2)	−0.3806(4)	0.045(1)	0.044(1)	0.030(1)	−0.016(1)	0.004(1)	0.0048(9)
C(9)	4*a*	0.30898(9)	0.4043(1)	0.0730(3)	0.0220(9)	0.0191(7)	0.024(1)	−0.0004(6)	−0.0004(8)	−0.0031(7)

Experimental details

Cell refinement and data reduction were carried out by CrysAlis PRO [16]. The coordinates of the aromatic H atoms were idealized and refined using a riding model (AFIX 43 option of the SHELX program [17]).

Discussion

1,2,4-Triazole derivatives and their fused heterocyclic analogues are well known for their different biological activities over 30 years ago, and 1,2,4-triazole rings have been incorporated into ligands used in coordination compounds and polymers. Thus, various 1,2,4-triazole derivatives and their *N*-bridged heterocyclic analogues have been extensively studied [1–6]. Several 3,6-disubstituted[1,2,4]triazolo[3,4-*b*][1,3,4] thiadiazoles were reported to exhibit significant antibacterial [7–10], pesticidal [11], anticancer [12], anti-inflammatory, analgesic and anti-oxidant activities [13]. In continuation to a previous interest in the chemical synthesis of [1,2,4]-triazolo[3,4-*b*][1,3,4]thiadiazole derivatives [14, 15], we report herein the synthesis and the crystal structure of the title compound as potential bioactive agent.

One independent molecule comprises the asymmetric unit. The compound is nearly planer, with respect to the [1,2,4]-triazolo[3,4-*b*][1,3,4]thiadiazole system (N1/N2/C8/S1/C9/N4/N3/C7), the fluorophenyl ring (C10—C15) form dihedral angles of 2.55 (6)° and the phenyl ring (C1—C6) form dihedral angles of 3.83 (3)°. In the crystal structure, the packing is stabilized by one non-classical intermolecular hydrogen bond, of which the N1 acts as hydrogen bond acceptor and the C12 acts as hydrogen bond donor. The distance of the interaction between C12—H12···N1 is 2.33 Å and the angle is 161°.

Acknowledgements: The authors would like to extend their sincere appreciation to the Deanship of Scientific Research at King Saud University for funding this work through the Research Group Project No. PRG-1436–23. We also acknowledge the financial support from Spanish Ministerio de Economía y Competitividad (MINECO-13-MAT2013–40950-R, FPI grant BES-2011–046948 to MSM-A).

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