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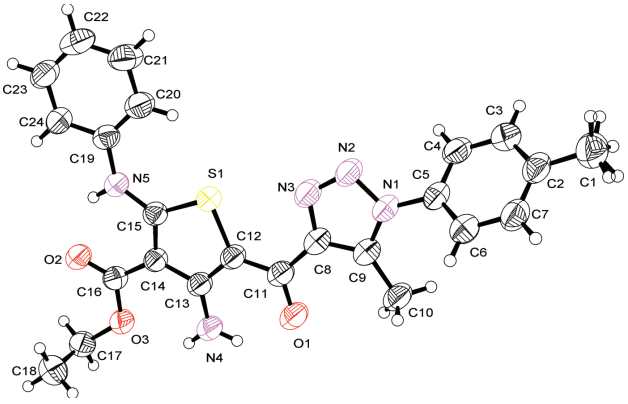
Crystal structure of ethyl 4-amino-5-(5-methyl-1-(4-tolyl)-1*H*-1,2,3-triazole-4-carbonyl)-2-(phenylamino)thiophene-3-carboxylate, $C_{24}H_{23}N_5O_3S$



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Abstract

$C_{24}H_{23}N_5O_3S$, triclinic, $P\bar{1}$ (no. 2), $a = 9.1704(9)$ Å, $b = 10.1253(11)$ Å, $c = 12.2182(14)$ Å, $\alpha = 83.686(10)°$, $\beta = 89.542(9)°$, $\gamma = 76.982(9)°$, $V = 1098.5(2)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0551$, $wR_{ref}(F^2) = 0.1510$, $T = 296(2)$ K.

CCDC no.: 1844847

Source of material

The title compound was synthesized from reaction of an equimolar mixture of ethyl 2-cyano-2-(4-hydroxy-4-(5-methyl-1-(4-tolyl)-1*H*-1,2,3-triazol-4-yl)-3-phenylthiazolidin-2-ylidene)acetate, hydroxylamine hydrochloride and anhydrous potassium carbonate in anhydrous ethanol under

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Table 1: Data collection and handling.

Crystal:	Needle, yellow
Size:	0.37 × 0.21 × 0.13 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	1.85 mm^{-1}
Diffractometer, scan mode:	SuperNova φ and ω-scans
θ_{max}, completeness:	29.99°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	9769, 5171, 0.032
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3341
$N(param)_{refined}$:	300
Programs:	CrysAlis^{Pro} [1], SHELX [2, 3], WinGX and Ortep [11]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.1586(3)	−0.1973(3)	0.5590(3)	0.0885(9)
H1A	0.117670	−0.240273	0.504099	0.133
H1B	0.241182	−0.261384	0.596358	0.133
H1C	0.082841	−0.167440	0.611197	0.133
H1D	0.176792	−0.205792	0.637004	0.133
H1E	0.053280	−0.184680	0.544744	0.133
H1F	0.211621	−0.278625	0.529906	0.133
C2	0.2120(3)	−0.0766(3)	0.5048(2)	0.0643(7)
C3	0.2024(3)	−0.0452(3)	0.3915(2)	0.0692(7)
H3	0.159406	−0.098227	0.349584	0.083
C4	0.2544(3)	0.0619(3)	0.3393(2)	0.0616(6)
H4	0.245519	0.081672	0.263131	0.074
C5	0.3197(2)	0.1395(3)	0.40043(18)	0.0538(6)
C6	0.3314(3)	0.1108(3)	0.51376(19)	0.0627(6)
H6	0.376190	0.162912	0.555366	0.075
C7	0.2761(3)	0.0043(3)	0.5643(2)	0.0668(7)
H7	0.282234	−0.013530	0.640638	0.080
C8	0.4566(2)	0.4286(3)	0.28995(18)	0.0542(6)
C9	0.3712(2)	0.3756(3)	0.36957(17)	0.0538(6)
C10	0.2778(3)	0.4372(3)	0.4594(2)	0.0682(7)
H10A	0.196194	0.392898	0.473156	0.102
H10B	0.238963	0.532683	0.437652	0.102
H10C	0.337917	0.425725	0.525161	0.102
C11	0.4792(2)	0.5688(3)	0.27327(18)	0.0557(6)
C12	0.5619(2)	0.6132(3)	0.18264(18)	0.0523(6)
C13	0.5799(2)	0.7468(3)	0.15920(18)	0.0517(6)
C14	0.6680(2)	0.7656(2)	0.06442(17)	0.0490(5)
C15	0.7184(2)	0.6425(2)	0.01817(17)	0.0491(5)
C16	0.7118(2)	0.8890(3)	0.02047(19)	0.0533(6)

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C17	0.7086(3)	1.1197(3)	0.0473(2)	0.0644(7)
H17A	0.632652	1.198032	0.064235	0.077
H17B	0.726857	1.130091	−0.031099	0.077
C18	0.8490(3)	1.1136(3)	0.1097(2)	0.0778(8)
H18A	0.831615	1.099620	0.187221	0.117
H18B	0.880319	1.197818	0.092837	0.117
H18C	0.925835	1.039572	0.089198	0.117
C19	0.8743(2)	0.5255(3)	−0.12913(18)	0.0526(6)
C20	0.8436(3)	0.3976(3)	−0.1163(2)	0.0673(7)
H20	0.772925	0.378557	−0.065559	0.081
C21	0.9173(3)	0.2974(3)	−0.1785(2)	0.0792(8)
H21	0.897219	0.210909	−0.168209	0.095
C22	1.0195(3)	0.3244(3)	−0.2550(2)	0.0783(8)
H22	1.069031	0.256887	−0.296611	0.094
C23	1.0481(3)	0.4525(4)	−0.2696(2)	0.0756(8)
H23	1.116341	0.472029	−0.322156	0.091
C24	0.9774(3)	0.5513(3)	−0.2078(2)	0.0638(7)
H24	0.998551	0.637401	−0.218484	0.077
N1	0.38313(19)	0.2453(2)	0.34652(14)	0.0545(5)
N2	0.4711(2)	0.2189(2)	0.25693(16)	0.0622(5)
N3	0.5141(2)	0.3305(2)	0.22421(16)	0.0607(5)
N4	0.5175(2)	0.8463(2)	0.22077(16)	0.0643(6)
H4A	0.464554	0.828679	0.276588	0.077
H4B	0.530521	0.927610	0.204329	0.077
N5	0.80860(19)	0.6336(2)	−0.06973(14)	0.0546(5)
H5	0.830101	0.709702	−0.094230	0.066
O1	0.4214(2)	0.64891(19)	0.34080(14)	0.0713(5)
O2	0.79200(18)	0.89881(18)	−0.05869(13)	0.0640(5)
O3	0.65593(18)	0.99586(18)	0.07665(14)	0.0653(5)
S1	0.65561(6)	0.50883(6)	0.08608(5)	0.05279(19)

reflux for 4 h. The mixture was allowed to cool to room temperature and poured into ice-water. The solid obtained was collected by filtration, dried and recrystallized from DMF to give pale-yellow crystals (Mp. 211−212 °C; lit. Mp. 210 °C [4]) of the title compound (68%).

Experimental details

All hydrogen atoms were placed in calculated positions and refined using a riding model. Difference Fourier calculation indicated disorder in the tolyl methyl group and the group was refined with two positions rotated by 60° from each other (AFIX 123 instruction in SHELXL [3]) The other methyl groups (AFIX 137) were allowed to rotate about the C-C bond and U_{iso} values for all methyl groups were set to $1.5U_{eq}$(C). The U_{iso} values for the amine (AFIX 93), methylene (AFIX 23), aromatic hydrogens (AFIX 43) were set to $1.2U_{eq}$(C, N).

Comment

Thiophene derivatives have been used as building blocks in drugs since they show various pharmacological activities such as antibacterial, anti-inflammatory, anticancer, and antiviral [5–7] properties. Compounds containing the 1,2,3-triazole moiety have also shown a variety of biological activities [8–10].

The asymmetric unit consists of one molecule. The methyltriazole-carbonyl-aminothiophene-carboxylate group (**A**) is planar with a maximum deviation of 0.124(2) Å from the least squares plane. With a twist of just 8.3(1)°, the phenylamine group is also almost co-planar with **A**, whereas the angle is 48.0(1)° for the tolyl group. The ethoxy group also deviates from the plane with a C18-C17-O3-C16 torsion angle of 86.4(3)°. In the crystal, the molecules pack in layers parallel to (101). For adjacent layers, the planes through **A** and the phenylamine groups are separated by about 3.65 Å.

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