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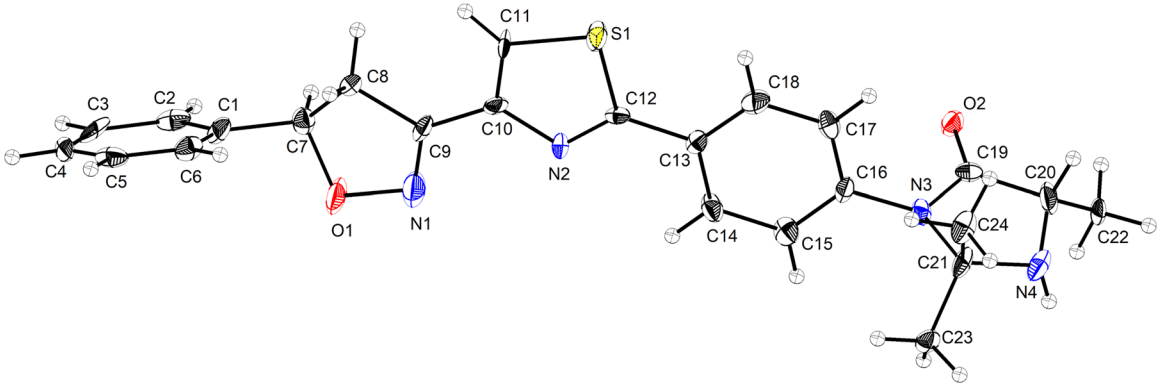
The crystal structure of 2,2,5-trimethyl-3-(4-(4-(5-phenyl-4,5-dihydroisoxazol-3-yl)thiazol-2-yl)phenyl)imidazolidin-4-one, $C_{24}H_{24}N_4O_2S$



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Abstract

$C_{24}H_{24}N_4O_2S$, orthorhombic, $P2_12_12_1$ (no. 19) a = 5.6357(3) Å, b = 18.8798(11) Å, c = 20.0148(11) Å, V = 2129.6(2) Å^3, Z = 4, $R_{gt}(F)$ = 0.0668, $wR_{ref}(F^2)$ = 0.1724, T = 100(2) K.

CCDC no.: 2311780

The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless needle
Size:	0.16 × 0.02 × 0.01 mm
Wavelength:	Ga *K*α radiation (1.34139 Å)
μ:	1.03 mm^{-1}
Diffractometer, scan mode:	Bruker PHOTON III, φ and ω
θ_{max}, completeness:	57.4°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	18646, 4355, 0.065
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3,833
$N(param)_{refined}$:	286
Programs:	Bruker,[1] Olex,[2] SHELX[3]

1 Source of materials

The 4-(4-(5-phenyl-4,5-dihydroisoxazol-3-yl)thiazol-2-yl) aniline (0.321 g 1 mmol) and Boc–L-alanine (0.100 g 1.05 mmol) in pyridine (5 mL) were stirred in a 25 mL flask. The reaction progress was monitored by TLC. The mixture was poured into iced water, and the solution was filtered to obtain intermediate. After, the intermediate was dissolved in dichloromethane (10 mL), then trifluoroacetic acid (2 mL) was slowly added at room temperature. The mixture was evaporated and then saturated aqueous $NaHCO_3$ was added. The crude product was purified by column chromatography (EA: MeOH = 10:1, v/v) to give 2-amino-*N*-(4-(4-(5-phenyl-4,5-dihydroisoxazol-3-yl)thiazol-2-yl)phenyl)propanamide. Next, 2-amino-*N*-(4-(4-(5-phenyl-4,5-dihydroisoxazol-3-yl)thiazol-2-yl)phenyl)propanamide was dissolved in acetone (10 mL) and heated at 60 °C for 30 min. At last, the reaction mixture was allowed to recrystallize from acetone to obtain 2,2,5-trimethyl-3-(4-(4-(5-phenyl-4,5-dihydroisoxazol-3-yl)thiazol-2-yl)phenyl)imidazolidin-4-one. ^{1}H NMR (400 MHz, $CDCl_3$) d 8.04–8.00 (m, 2H), 7.80 (s, 1H), 7.45–7.37 (m, 4H), 7.35–7.31 (m, 1H), 7.30–7.26 (m, 2H), 5.78 (dd, J = 11.1, 8.3 Hz, 1H), 5.30 (s, 1H), 3.95 (dd, J = 17.3, 11.1 Hz, 1H), 3.76 (q, J = 6.9 Hz, 1H), 3.54 (dd, J = 17.3, 8.3 Hz, 1H), 1.49–1.46 (m, 6H), 1.45 (s, 3H).

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

Atom	x	y	z	U_{iso}*/U_{eq}
C1	0.4311 (10)	1.1195 (2)	0.3217 (2)	0.0173 (11)
C2	0.6204 (10)	1.1622 (3)	0.3371 (2)	0.0234 (12)
H2	0.740322	1.145678	0.366558	0.028*
C3	0.6372 (10)	1.2309 (3)	0.3089 (3)	0.0270 (13)
H3	0.768584	1.260612	0.318911	0.032*
C4	0.4581 (11)	1.2543 (2)	0.2664 (2)	0.0232 (13)
H4	0.467631	1.299804	0.246362	0.028*
C5	0.2688 (11)	1.2113 (3)	0.2537 (2)	0.0249 (13)
H5	0.144505	1.227967	0.225752	0.030*
C6	0.2538 (10)	1.1439 (3)	0.2806 (2)	0.0225 (11)
H6	0.121309	1.114628	0.270617	0.027*
C7	0.4334 (10)	1.0454 (2)	0.3530 (2)	0.0213 (11)
H7	0.546777	1.045103	0.391456	0.026*
C8	0.1992 (10)	1.0136 (2)	0.3755 (2)	0.0138 (10)
H8A	0.168503	1.022007	0.423583	0.017*
H8B	0.063837	1.031093	0.348706	0.017*
C9	0.2553 (8)	0.9366 (2)	0.36102 (19)	0.0097 (9)
C10	0.1170 (8)	0.8775 (2)	0.38746 (18)	0.0090 (9)
C11	−0.0812 (9)	0.8869 (2)	0.4259 (2)	0.0126 (10)
H11	−0.142978	0.931484	0.439437	0.015*
C12	0.0279 (8)	0.7650 (2)	0.40236 (18)	0.0075 (9)
C13	0.0446 (8)	0.6863 (2)	0.40106 (19)	0.0087 (9)
C14	0.2236 (9)	0.6541 (2)	0.3632 (2)	0.0116 (9)
H14	0.328622	0.682251	0.337295	0.014*
C15	0.2474 (8)	0.5806 (2)	0.3637 (2)	0.0121 (10)
H15	0.367135	0.558693	0.337496	0.015*
C16	0.0970 (8)	0.5396 (2)	0.40232 (19)	0.0088 (9)
C17	−0.0822 (9)	0.5719 (2)	0.4397 (2)	0.0129 (9)
H17	−0.185851	0.543902	0.466239	0.015*
C18	−0.1085 (9)	0.6446 (2)	0.4382 (2)	0.0143 (9)
H18	−0.233031	0.666242	0.462805	0.017*
C19	0.1983 (9)	0.4307 (2)	0.46081 (18)	0.0113 (9)
C20	0.1384 (9)	0.3519 (2)	0.4527 (2)	0.0141 (10)
H20	−0.002594	0.341258	0.481312	0.017*
C21	−0.0059 (8)	0.4147 (2)	0.3582 (2)	0.0112 (10)
C22	0.3357 (10)	0.3024 (2)	0.4728 (2)	0.0206 (11)
H22A	0.281883	0.253254	0.468178	0.031*
H22B	0.379716	0.311399	0.519423	0.031*
H22C	0.473652	0.310261	0.443975	0.031*
C23	0.0771 (10)	0.4253 (3)	0.2862 (2)	0.0198 (11)
H23A	0.024416	0.471780	0.270229	0.030*
H23B	0.009487	0.388154	0.257860	0.030*
H23C	0.250685	0.422849	0.284498	0.030*
C24	−0.2718 (10)	0.4249 (3)	0.3649 (3)	0.0240 (12)
H24A	−0.354007	0.393055	0.333870	0.036*
H24B	−0.312653	0.474077	0.354277	0.036*
H24C	−0.320571	0.414113	0.410746	0.036*
N1	0.4243 (9)	0.9276 (2)	0.31985 (19)	0.0195 (9)
N2	0.1782 (7)	0.80797 (17)	0.37417 (16)	0.0100 (8)
N3	0.1221 (7)	0.46412 (18)	0.40504 (16)	0.0090 (8)
N4	0.0668 (8)	0.34418 (19)	0.38278 (19)	0.0134 (8)
H4A	0.161 (11)	0.325 (3)	0.362 (3)	0.016*
O1	0.5175 (7)	0.99552 (16)	0.30212 (17)	0.0238 (9)
O2	0.2935 (7)	0.45759 (15)	0.50931 (14)	0.0198 (8)
S1	−0.2015 (2)	0.80629 (5)	0.44617 (5)	0.0155 (3)

2 Experimental details

All hydrogen atoms were placed in the calculated positions and constrained to ride on their parent atoms. Using Olex,[2] the structure was solved with the XM[3] structure solution program using Dual Space and refined with the XL.[3]

3 Comment

Nitrogen-containing heterocycles are widely found in natural products and pharmaceuticals.[4,5] Isoxazoline is an important scaffold of pesticide active molecules. Most of its derivatives have high-efficiency and broad-spectrum agricultural activities such as insecticidal,[6,7] bactericidal[8,9] and herbicide.[10,11] The thiazole rings exhibit a wide range of biological activities and have found extensive applications in the development of environmentally friendly pesticides.[12,13] The investigation of single crystal structures of isoxazoline-thiazoles is crucial in elucidating their structure-activity relationships, given their wide range of physiological activities and significance in drug development.

The title compound crystallizes in the orthorhombic $P2_12_12_1$ space group. In the crystal structure (figure), the presence of chirality of C7 in the title molecule exhibits an *R* configuration. The bond length of C11=S1 and C12=S1 are 1.715 and 1.746 Å, respectively. The dihedral angle between the isoxazoline ring and the thiazole ring is 10.01(18)°. The torsion angles of C1–C7–O1–N1, C9–N1–O1–C7, C16–N3–C19–C20 and C16–N3–C21–N4 are −155.3(4)°, 17.2(5)°, 164.6(4)° and −177.2(4)°, respectively. All bond lengths and angles are within a reasonable range.[14,15]

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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