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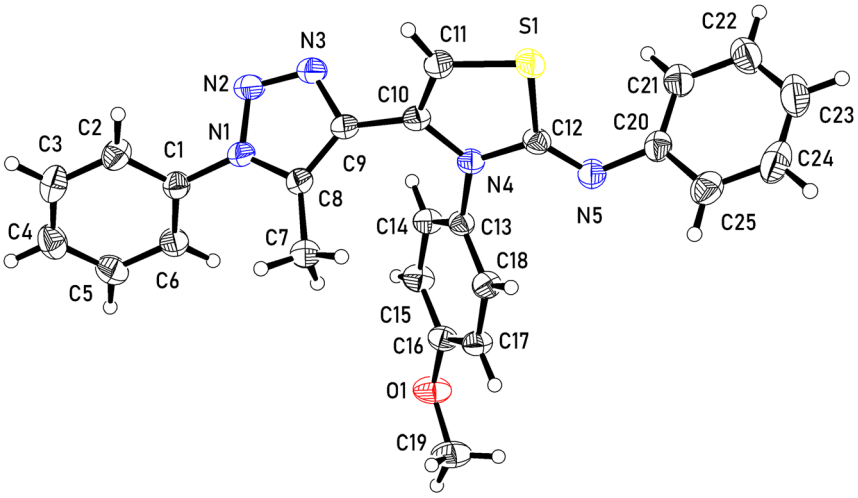
Crystal structure of (*Z*)-3-(4-methoxyphenyl)-4-(5-methyl-1-phenyl-1*H*-1,2,3-triazol-4-yl)-*N*-phenylthiazol-2(3*H*)-imine, $C_{25}H_{21}N_5OS$



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Abstract

$C_{25}H_{21}N_5OS$, monoclinic, $P2_1/n$ (no. 14), a = 12.9303(5) Å, b = 9.1111(4) Å, c = 18.7111(8) Å, β = 97.879(4)°, V = 2183.53(16) Å^3, Z = 4, $R_{gt}(F)$ = 0.0487, $wR_{ref}(F^2)$ = 0.1327, T = 293(2) K.

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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.

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.17 × 0.10 × 0.10 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	6.0 mm^{-1}
Diffractometer, scan mode:	Bruker APEXII, φ and ω
θ_{max}, completeness:	56.6°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	13,239, 9148, 0.025
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 7183
$N(param)_{refined}$:	686
Programs:	*CrystAlis*^{PRO} [1], SHELX [2], WinGX/ORTEP [3], CHEMDRAW [4]

1 Source of material

A mixture of 4-methoxyaniline (0.62 g, 5 mmol) and phenyl isothiocyanate (0.68 g, 5 mmol) in anhydrous EtOH (15 ml), was refluxed for 15 min, followed by the addition of 2-bromo-1-(5-methyl-1-phenyl-1*H*-1,2,3-triazol-4-yl)ethan-1-one (1.2 g, 5 mmol). The mixture was refluxed for 4 h. The solid produced on standing overnight was filtered, dried, and recrystallized from DMF to give the title heterocycle in 80 % yield, mp 179–181 °C. **IR** (KBr): 3121, 1610, 1517 cm^{-1}. **^{1}H NMR** (DMSO-d_6): 2.17 (*s*, 3H, Me), 3.69 (*s*, 3H, OMe), 6.56 (*s*, 1H, thiazolyl), 6.88 (*d*, 8.2 Hz, 2H, Ar), 6.93 (*d*, 8.2 Hz, 2H, Ar), 7.00 (*t*, 7.7 Hz, 1H, Ar), 7.23 (*d*, 9.0 Hz, 2H, Ar), 7.29 (*t*, 7.7 Hz, 2H, Ar), 7.45–7.58 (*m*, 5H, Ar). **^{13}C NMR** (DMSO-d_6):

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.03708 (14)	0.8202 (2)	0.58167 (10)	0.0381 (4)
C2	0.02149 (17)	0.8323 (2)	0.65315 (11)	0.0465 (5)
H2	0.078034	0.840490	0.689431	0.056*
C3	−0.07936 (18)	0.8319 (2)	0.66978 (12)	0.0527 (5)
H3	−0.090937	0.840711	0.717563	0.063*
C4	−0.16260 (17)	0.8186 (2)	0.61607 (14)	0.0534 (5)
H4	−0.230188	0.816764	0.627804	0.064*
C5	−0.14671 (16)	0.8080 (3)	0.54508 (13)	0.0533 (5)
H5	−0.203530	0.799827	0.509007	0.064*
C6	−0.04606 (15)	0.8095 (3)	0.52713 (11)	0.0472 (5)
H6	−0.034824	0.803380	0.479181	0.057*
C7	0.14718 (17)	0.5873 (2)	0.49655 (13)	0.0520 (5)
H7A	0.100601	0.549008	0.527739	0.078*
H7B	0.202285	0.518070	0.493239	0.078*
H7C	0.109399	0.603970	0.449407	0.078*
C8	0.19250 (14)	0.7281 (2)	0.52637 (10)	0.0370 (4)
C9	0.29014 (14)	0.7883 (2)	0.52875 (10)	0.0370 (4)
C10	0.38571 (14)	0.7371 (2)	0.50277 (10)	0.0374 (4)
C11	0.47860 (15)	0.7321 (3)	0.54423 (10)	0.0452 (5)
H11	0.488089	0.753554	0.593280	0.054*
C12	0.49111 (13)	0.6638 (2)	0.41659 (10)	0.0360 (4)
C13	0.30245 (13)	0.6816 (2)	0.37593 (9)	0.0333 (4)
C14	0.23351 (14)	0.7963 (2)	0.35908 (10)	0.0403 (4)
H14	0.245273	0.886080	0.382397	0.048*
C15	0.14671 (15)	0.7776 (2)	0.30741 (11)	0.0438 (5)
H15	0.099693	0.854345	0.296742	0.053*
C16	0.13007 (14)	0.6446 (2)	0.27167 (10)	0.0394 (4)
C17	0.19949 (14)	0.5302 (2)	0.28807 (10)	0.0408 (4)
H17	0.188671	0.440945	0.264112	0.049*
C18	0.28517 (14)	0.5492 (2)	0.34031 (10)	0.0384 (4)
H18	0.331599	0.471952	0.351557	0.046*
C19	0.01870 (18)	0.4975 (3)	0.18771 (13)	0.0612 (6)
H19A	0.071844	0.470723	0.158993	0.092*
H19B	−0.047461	0.503426	0.157511	0.092*
H19C	0.015101	0.424652	0.224382	0.092*
C20	0.61751 (14)	0.6008 (2)	0.34534 (9)	0.0401 (4)
C21	0.69189 (16)	0.7120 (3)	0.35746 (11)	0.0470 (5)
H21	0.673378	0.802522	0.374875	0.056*
C22	0.79308 (17)	0.6887 (3)	0.34376 (13)	0.0587 (6)
H22	0.842110	0.763633	0.351820	0.070*
C23	0.82140 (19)	0.5553 (4)	0.31831 (14)	0.0706 (8)
H23	0.889682	0.539351	0.309744	0.085*
C24	0.7486 (2)	0.4456 (3)	0.30558 (13)	0.0670 (7)
H24	0.767881	0.355497	0.288194	0.080*
C25	0.64659 (18)	0.4672 (3)	0.31826 (11)	0.0520 (5)
H25	0.597676	0.392473	0.308669	0.062*
N1	0.14152 (12)	0.82403 (18)	0.56448 (8)	0.0383 (4)
N2	0.20444 (13)	0.9371 (2)	0.59012 (10)	0.0481 (4)
N3	0.29442 (12)	0.9153 (2)	0.56804 (9)	0.0465 (4)
N4	0.39117 (11)	0.69774 (17)	0.43072 (8)	0.0358 (3)
N5	0.51235 (12)	0.6236 (2)	0.35497 (9)	0.0436 (4)
O1	0.04339 (11)	0.63654 (17)	0.22080 (8)	0.0553 (4)
S1	0.57902 (4)	0.68154 (7)	0.49731 (3)	0.04634 (16)

9.5, 55.8, 100.6, 114.4, 121.5, 122.9, 125.2, 130.1, 130.2, 130.3, 130.5, 130.7, 130.9, 134.2, 135.8, 136.7, 152.1, 158.9, 159.4. **Anal. Calcd.** for $C_{25}H_{21}N_5OS$ (439.54): C, 68.32; H, 4.82; N, 15.93. Found: C, 68.43; H, 4.84; N, 16.08 %.

2 Experimental details

Hydrogen atoms were located in the difference Fourier map. Idealized geometries were subsequently used for the initial positions and a riding model applied during refinement. Methyl C–H bonds were fixed at 0.96 Å, with displacement parameters 1.5 times U_{iso}(C), and were allowed to spin about the C–C bond. C–H distances were set to 0.93 Å for sp^2 hydrogen atoms and their *U*(iso) set to 1.2 times the U_{iso}(C). Crystal data, data collection and structure refinement details are summarized in Table 1.

3 Comment

Heterocycles containing the thiazol-2-imine moiety have been found to possess interesting biological activities [5–7]. In addition, triazoles show a wide range of activities against pathogens [8–10]. Therefore, synthesizing heterocycles containing *N*-phenylthiazol-2(3*H*)-imines and 1,2,3-triazoles has received attention [11, 12]. In previous work, we have reported the X-ray crystal structures of related heterocycles [13, 14].

The crystal structure is monoclinic, with an asymmetric unit comprising one molecule of $C_{25}H_{21}N_5OS$ (Figure 1) which is composed of the following five planar groups: phenyl (**A**: C1–C6), methyltriazole (**B**: C7–C9, N1–N3), thiazole (**C**: C10–C12, N4, S1), methoxybenzene (**D**:C13–C19, O1) and aniline (**E**: C20–C25, N5). The planes of adjacent groups in the molecule are significantly twisted relative to each other. The twist angles between the groups are: **A**/**B** = 55.21(7)°, **B**/**C** = 51.60(7)°, **C**/**D** = 55.87(7)° **D**/**E** = 45.69(5)°.

In the crystal structure, neighbouring molecules are related by inversion symmetry to form pairs with parallel thiazole groups with a **C**···**C** centroid separated distance of 3.86 Å. The pairs of molecules are stacked to form columns parallel to the **b**-axis of the crystal. Adjoining columns are linked by C–H···O (C11···O1 = 3.506(2), C11–H11···O1 = 166.1) and C–H···N (C19···N3 = 3.504(3), C19–H19B···N3 = 165.0) interactions.

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

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

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