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Crystal structure of 2-((4-phenyl-5-(pyridin-4-yl)-4*H*-1,2,4-triazol-3-yl)thio)acetonitrile, C₁₅H₁₁N₅S

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Abstract

C₁₅H₁₁N₅S, orthorhombic, *Pbca* (no. 61), $a = 9.7864(2)$ Å, $b = 15.5709(3)$ Å, $c = 18.7774(4)$ Å, $V = 2861.36(10)$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.0485$, $wR_{\text{ref}}(F^2) = 0.1390$, $T = 296.15$ 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:	Colorless block
Size:	0.26 × 0.24 × 0.22 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.23 mm ⁻¹
Diffractometer, scan mode:	Bruker P4, ω
θ_{max} , completeness:	27.5°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	14,104, 3291, 0.064
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1834
$N(\text{param})_{\text{refined}}$:	190
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

Source of material

A mixture of isonicotinohydrazide (1.37 g, 10 mmol) and isothiocyanatobenzene (1.35 g, 10 mmol) in ethanol (50 mL) was refluxed for 5 h. After cooling, the 2-isonicotinoyl-*N*-phenylhydrazine-1-carbothioamide were obtained from the solution of ethanol. Then the 2-isonicotinoyl-*N*-phenylhydrazine-1-carbothioamide was dissolved in NaOH solution (5 mL, 2 mol/L) and refluxed for 4 h. After cooling, a hydrochloric acid solution (4 mol/L) was added dropwise. A large amount of solid (4-phenyl-5-(pyridin-4-yl)-4*H*-1,2,4-triazole-3-thiol) was obtained. Finally, *N,N*-dimethylformamide (DMF) (5 mL), a mixture of 4-phenyl-5-(pyridin-4-yl)-4*H*-1,2,4-triazole-3-thiol (0.25 g, 1 mmol), 2-chloroacetonitrile (1.1 mmol) and NaOH (0.05 g, 1.2 mmol) was stirred overnight. The mixture was poured into ice (40 mL) and the product precipitated. The crude product was recrystallized from EtOH to give colorless crystals after three days [5].

Experimental details

Using Olex2 [2], the structure was solved with the ShelXS [3] structure solution program using Direct Methods and refined with the ShelXL [4] refinement package U_{iso} values of the hydrogen atoms were set to 1.2 U_{eq} of the parent atoms.

Comment

1,2,4-Triazole compounds are attractive because of their excellent biological activity [6–8]. A pyridinyl

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
S1	0.54434 (7)	0.12148 (5)	0.40264 (3)	0.0688 (3)
N1	0.3033 (3)	0.1055 (3)	0.25373 (14)	0.1320 (13)
N2	0.32293 (19)	0.02297 (11)	0.43785 (10)	0.0515 (5)
N3	0.22525 (19)	0.02069 (11)	0.49174 (10)	0.0504 (5)
N4	0.37085 (17)	0.12470 (10)	0.51578 (9)	0.0429 (4)
N5	0.0041 (3)	0.12568 (15)	0.72022 (13)	0.0803 (7)
C1	0.3958 (3)	0.0839 (2)	0.28496 (15)	0.0885 (10)
C2	0.5152 (3)	0.0572 (2)	0.32430 (14)	0.0807 (9)
H2A	0.5946	0.0609	0.2935	0.097*
H2B	0.5044	−0.0024	0.3383	0.097*
C3	0.4067 (2)	0.08598 (14)	0.45343 (11)	0.0454 (5)
C4	0.2550 (2)	0.08174 (13)	0.53737 (11)	0.0430 (5)
C5	0.1721 (2)	0.09991 (13)	0.60097 (10)	0.0447 (5)
C6	0.0341 (2)	0.07890 (15)	0.59960 (12)	0.0530 (6)
H6	−0.0052	0.0562	0.5586	0.064*
C7	−0.0432 (3)	0.09219 (17)	0.65951 (14)	0.0681 (7)
H7	−0.1350	0.0768	0.6578	0.082*
C8	0.1367 (3)	0.14596 (19)	0.71986 (14)	0.0786 (8)
H8	0.1728	0.1701	0.7611	0.094*
C9	0.2244 (3)	0.13394 (15)	0.66338 (12)	0.0608 (7)
H9	0.3163	0.1483	0.6671	0.073*
C10	0.4384 (2)	0.19786 (13)	0.54776 (10)	0.0410 (5)
C11	0.3701 (2)	0.27510 (14)	0.55115 (12)	0.0520 (6)
H11	0.2827	0.2805	0.5322	0.062*
C12	0.4333 (3)	0.34446 (15)	0.58314 (13)	0.0591 (6)
H12	0.3879	0.3968	0.5864	0.071*
C13	0.5623 (3)	0.33623 (15)	0.60991 (12)	0.0559 (6)
H13	0.6039	0.3830	0.6319	0.067*
C14	0.6316 (2)	0.25930 (15)	0.60474 (12)	0.0546 (6)
H14	0.7205	0.2549	0.6219	0.066*
C15	0.5691 (2)	0.18888 (14)	0.57408 (11)	0.0467 (5)
H15	0.6144	0.1365	0.5712	0.056*

substituted 1,2,4-triazole possessed diversity biological activities, such as antimicrobial activity [9] and there are related compounds that show fungicidal activity [10].

There is One molecule in the asymmetric unit (see the Figure). The bond angles and bond lengths of the title compound (pyridine ring, 1,2,4-triazole ring and phenyl ring) are all in the normal ranges [11–13]. The C–N bond length in the cyano group is 1.130(4) Å. The bond angle of C2–S1–C3 is 98.53(12)°, while the bond angle of N1–C1–C2 is 179.2(4)°. The torsion angle of the thioether group C1–C2–S1–C3 is 70.0(3)°. The plane of the triazole ring makes dihedral angles of 28.1° and 114.5° to the two other rings.

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