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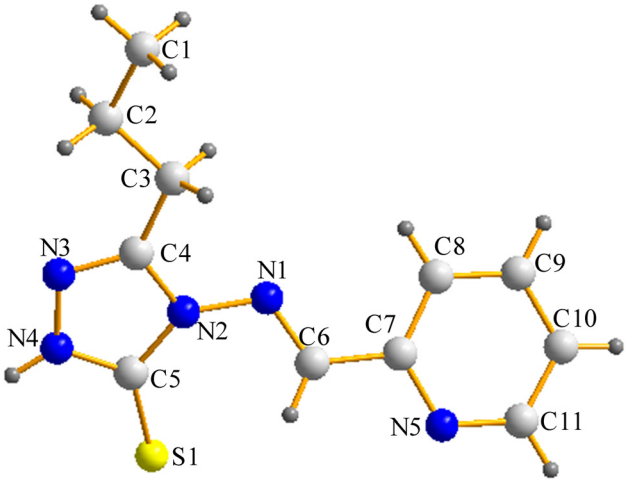
Crystal structure of (*E*)-5-propyl-4-((pyridin-2-ylmethylene)amino)-2,4-dihydro-*3H*-1,2,4-triazole-3-thione – methanol (1/1), $C_{11}H_{13}N_5S$



https://doi.org/10.1515/ncrs-2022-0041
Received January 22, 2022; accepted March 7, 2022;
published online March 16, 2022

Abstract

$C_{11}H_{13}N_5S$, monoclinic, $P2_1/c$ (no. 14), a = 7.2720(14) Å, b = 11.631(2) Å, c = 17.031(3) Å, β = 95.12(3)°, V = 1434.7(5) Å^3, Z = 4, $R_{gt}(F)$ = 0.0353, $wR_{ref}(F^2)$ = 0.0953, T = 293 K.

CCDC no.: 2142563

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:	Yellow block
Size:	0.28 × 0.26 × 0.23 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.23 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, ω
θ_{max}, completeness:	30.8°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	7771, 4368, 0.016
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 4127
$N(param)_{refined}$:	174
Programs:	Bruker [1], SHELX [2, 3]

Source of material

About 5 mmol of 4-amino-3-ethyl-1,2,4-triazole-5-thione solid was completely dissolved in 10 mL methanol solution in a 50 mL round-bottom flask, and then 5 mmol of yellowish pyridine formaldehyde solution was gradually added to the above mixed solution. After the mixture was stirred and refluxed for 3.5 h, and cooled to room temperature, the yellowish precipitate was collected by vacuum filtration, and recrystallized from methanol, and finally dried in vacuo. Yield 89%. In addition, the yellow crystal was precipitated from the filtrate that had been left standing for two weeks. **Elemental analysis** calculated for $C_{11}H_{13}N_5S$: Calcd(%) C 53.42; H 5.30; N 28.32; Found(%) C 53.28; H 5.23; N 28.21; **IR**(cm^{-1}): 3429, 1589, 1413, 1315, 999, 758; **UV**(nm): 333, 244, 206.

Experimental details

The structures were solved by the direct method. Hydrogen atoms attached to C, N, and O atoms were constrained using a riding model approximation, with C–H = 0.93–0.97 Å, O–H = 0.82 Å, and U_{iso}(H) = 1.2U_{eq}(C).

Comment

Aromatic heterocycles such as 1,2,4-triazole, imidazole, pyridine, pyrazine, quinoline as well as their derivatives are arguably the most facile and widely used ligands in coordination chemistry [4–7]. Of them, 1,2,4-triazole rings have been combined with pyridine rings to prepare bi and

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
S1	0.78422 (3)	−0.24333 (2)	0.01370 (2)	0.01791 (7)
N1	0.84375 (11)	0.02557 (7)	0.08601 (4)	0.01555 (15)
N2	0.75444 (10)	−0.00422 (7)	0.01292 (4)	0.01410 (14)
N3	0.64332 (11)	0.03174 (7)	−0.10979 (4)	0.01610 (15)
N4	0.66094 (11)	−0.08516 (7)	−0.09595 (4)	0.01603 (15)
H4	0.6293	−0.1362	−0.1311	0.019*
N5	0.88942 (12)	−0.10133 (7)	0.27665 (5)	0.01809 (15)
C1	0.65262 (18)	0.40477 (9)	−0.08053 (7)	0.0287 (2)
H1A	0.5513	0.4144	−0.0486	0.043*
H1B	0.6294	0.4481	−0.1284	0.043*
H1C	0.7642	0.4318	−0.0521	0.043*
C2	0.67312 (14)	0.27784 (9)	−0.10033 (5)	0.01984 (18)
H2A	0.7740	0.2684	−0.1334	0.024*
H2B	0.5609	0.2510	−0.1297	0.024*
C3	0.71077 (12)	0.20526 (8)	−0.02574 (5)	0.01575 (16)
H3A	0.8318	0.2244	−0.0006	0.019*
H3B	0.6201	0.2238	0.0108	0.019*
C4	0.70318 (11)	0.07964 (8)	−0.04300 (5)	0.01406 (15)
C5	0.73196 (12)	−0.11172 (8)	−0.02254 (5)	0.01472 (15)
C6	0.81785 (12)	−0.04320 (8)	0.14315 (5)	0.01588 (16)
H6	0.7380	−0.1054	0.1355	0.019*
C7	0.91716 (12)	−0.02152 (8)	0.22116 (5)	0.01482 (16)
C8	1.03382 (13)	0.07302 (8)	0.23567 (5)	0.01854 (17)
H8	1.0475	0.1273	0.1965	0.022*
C9	1.12899 (15)	0.08451 (9)	0.30973 (6)	0.02275 (19)
H9	1.2075	0.1466	0.3211	0.027*
C10	1.10449 (14)	0.00133 (10)	0.36662 (5)	0.0230 (2)
H10	1.1682	0.0061	0.4164	0.028*
C11	0.98324 (15)	−0.08899 (9)	0.34785 (5)	0.02172 (19)
H11	0.9662	−0.1436	0.3864	0.026*
O1	0.41055 (10)	0.24531 (6)	0.21332 (4)	0.01865 (14)
H1	0.3233	0.2881	0.2193	0.028*
C12	0.56624 (15)	0.31240 (10)	0.19461 (6)	0.0249 (2)
H12A	0.5914	0.3706	0.2341	0.037*
H12B	0.5394	0.3481	0.1441	0.037*
H12C	0.6721	0.2634	0.1932	0.037*

tridentate ligands [8, 9]. The simplest addition of a 1,2,4-triazole unit at a pyridine ring is the formation of the Schiff base compound. A number of N-heterocyclic Schiff base compounds have received much attention not only in the crystal engineering of coordination polymers [10], but also in the other fields [11–13]. In the course of our previous work, some 1,2,4-triazole derivative Schiff base ligands and their metal complexes have been development [6, 7, 14, 15]. As a part of our continuing investigation on complexes derived from different N-donor Schiff base ligands, herein we reported the structure of a new Schiff base. The title compound was characterized by Fourier transform IR spectroscopy, UV–vis spectroscopy, elemental analysis and X-ray diffraction.

The asymmetric unit of the title complex contains one target molecule and one methanol molecules. The title molecule is shown in the figure. All bond lengths and angles are in the expected ranges. Of them, the bond length of the C=S (1.682 Å) is close to the typical C=S double bond (1.646 Å) [16], which indicates that the thione form exists in the compound. In the molecule, the dihedral angle between the triazole and pyridine moiety is 34.43°. This twist is different from the reported 1,2,4-triazole derivative compound (4-[(5-bromo-2-hydroxybenzylidene) amino]-3-propyl-1*H*-1,2,4-triazole-5(4*H*)-thione and 4-[(5-chloro-2-hydroxybenzylidene) amino]-3-propyl-1*H*-1,2,4-triazole-5(4*H*)-thione) [6, 14]. In addition, there are intermolecular hydrogen bonds and weak π–π interactions in the structure. The title compound and methanol molecules were linked together through N4–H4···O1^{i} (*i* = 1–*x*, −1/2–*y*, 1/2–*z*) and O1^{i}–H1^{i}···N5^{ii} (*ii* = *x*, −1/2–*y*, 1/2+*z*) to form a chain structure. The geometry analysis shows a face-to-face alignment of the aromatic-aromatic rings, and the centroid distances of Cg1 ring (N2–C4–N3–N4–C5) and Cg1^{ii} (*ii* = 1–*x*, −*y*, −*z*) ring is 3.5413(9) Å.

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

Research funding: This work was supported by the Natural Science Foundation of Shanxi Province (201901D111014), Research Project Supported by Shanxi Scholarship Council of China (2020-001) and Project 2021019342 supported by Shanxi University's Training Program of Innovation and Entrepreneurship for Undergraduates.

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

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