Wen-Jun Lan, Chun-Yi Liu, Yan-Bi Luo, Qiang Fei* and Wen-Neng Wu

Crystal structure of 3-((2,4-dichlorobenzyl)thio)-5-methyl-7-(trifluoromethyl)-[1,2,4]triazolo [4,3-c]pyrimidine, $C_{14}H_9Cl_2F_3N_4S$

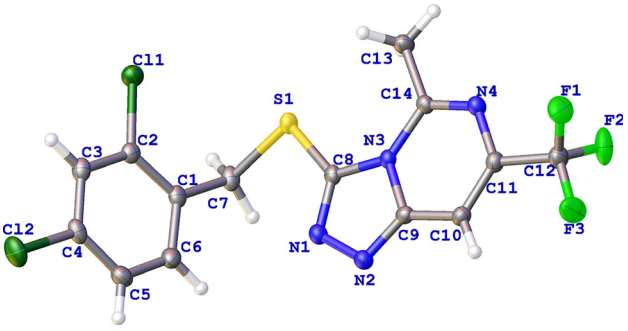

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.15 × 0.12 × 0.10 mm
Wavelength:	Cu *K*α radiation (1.54184 Å)
μ:	5.42 mm^{-1}
Diffractometer, scan mode:	XtaLAB AFC12 (RINC), ω
θ_{max}, completeness:	71.5°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	6825, 2942, 0.029
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2732
$N(param)_{refined}$:	218
Programs:	Olex2 [1], SHELX [2, 3]

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Abstract

$C_{14}H_9Cl_2F_3N_4S$, monoclinic, $P2_1/c$ (no. 14), a = 11.6567(3) Å, b = 4.80480(10) Å, c = 28.1051(7) Å, β = 101.336(2)°, V = 1543.40(6) Å^3, Z = 4, $R_{gt}(F)$ = 0.0358, $wR_{ref}(F^2)$ = 0.1014, T = 250 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.

Source of material

The mixture of 2-methyl-4-chloro-6-trifluoromethylpyrimidine (20 mmol) and hydrazine hydrate (30 mmol) was stirred in an ice bath in absolute ethanol (50 mL). Until the reaction completed, water was added to the system to obtain a yellow solid. Column chromatography was used to purify the residue to get a pale yellow solid. Then a mixture of the pale yellow solid (50 mmol), carbon disulfide (75 mmol) and triethylamine (75 mmol) were refluxed in anhydrous ethanol for 3 h. Then the system was cooled to room temperature, and 2,4-dichlorobenzyl chloride was added. After completion, the solvent was removed to get the residue, which was purified by column chromatography. Needle colorless crystals were obtained through cooling the ethanol solution of the target compound.

Experimental details

A suitable crystal was selected and tested on a XtaLAB AFC12 (RINC): Kappa single diffractometer. Using Olex2 [1], the structure was solved with the SHELXT [2] structure solution program using Intrinsic Phasing and refined with the SHELXL [3] refinement package using Least Squares minimisation.

Comment

Fused pyrimidine containing systems possess extensive biological activities [4–9] and were widely applied in medicine [10] and as pesticides [11]. This study aims to develop pyrimidine derivatives with new structures and good bioactivities. The target compound is shown in the

***Corresponding author: Qiang Fei**, Food and Pharmaceutical Engineering Institute, Guiyang University, Guiyang, Guizhou, China, E-mail: fqorganic@163.com. https://orcid.org/0000-0002-9825-0790
Wen-Jun Lan, Chun-Yi Liu, Yan-Bi Luo and Wen-Neng Wu, Food and Pharmaceutical Engineering Institute, Guiyang University, Guiyang, Guizhou, China. https://orcid.org/0000-0001-8704-226X (W.-N. Wu)

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.61526 (4)	0.91205 (11)	0.25194 (2)	0.04251 (15)
Cl2	0.96208 (5)	0.16952 (11)	0.29198 (2)	0.05383 (18)
S1	0.47856 (4)	0.86987 (9)	0.34993 (2)	0.03316 (14)
F1	0.06162 (11)	−0.0779 (3)	0.43520 (5)	0.0580 (3)
F2	0.07412 (13)	0.2490 (3)	0.48675 (6)	0.0679 (4)
F3	0.17998 (12)	−0.1150 (4)	0.50342 (6)	0.0727 (5)
N1	0.57288 (12)	0.5267 (3)	0.42441 (5)	0.0345 (3)
N2	0.53931 (12)	0.3321 (3)	0.45531 (5)	0.0347 (3)
N3	0.38007 (11)	0.4969 (3)	0.40633 (5)	0.0261 (3)
N4	0.18782 (12)	0.3698 (3)	0.40823 (5)	0.0329 (3)
C1	0.71388 (14)	0.7660 (4)	0.34453 (6)	0.0308 (4)
C2	0.71210 (14)	0.7250 (4)	0.29528 (6)	0.0309 (4)
C3	0.78759 (15)	0.5421 (4)	0.27899 (7)	0.0350 (4)
H3	0.783762	0.515206	0.245948	0.042*
C4	0.86860 (15)	0.4006 (4)	0.31271 (7)	0.0364 (4)
C5	0.87542 (16)	0.4398 (4)	0.36197 (7)	0.0405 (4)
H5	0.931283	0.345984	0.384463	0.049*
C6	0.79785 (16)	0.6205 (4)	0.37710 (7)	0.0382 (4)
H6	0.801876	0.645854	0.410192	0.046*
C7	0.63305 (15)	0.9672 (4)	0.36217 (7)	0.0357 (4)
H7A	0.640508	1.146722	0.347290	0.043*
H7B	0.658683	0.989547	0.396935	0.043*
C8	0.47907 (14)	0.6222 (3)	0.39548 (6)	0.0281 (3)
C9	0.42479 (15)	0.3169 (4)	0.44444 (6)	0.0288 (3)
C10	0.34531 (15)	0.1605 (4)	0.46578 (6)	0.0324 (4)
H10	0.370576	0.037512	0.491236	0.039*
C11	0.23138 (15)	0.1999 (4)	0.44718 (6)	0.0322 (4)
C12	0.13620 (17)	0.0626 (4)	0.46843 (7)	0.0409 (4)
C13	0.21438 (16)	0.6926 (4)	0.34560 (7)	0.0384 (4)
H13A	0.235632	0.882540	0.353452	0.058*
H13B	0.130731	0.677257	0.337164	0.058*
H13C	0.247868	0.634035	0.318649	0.058*
C14	0.25922 (13)	0.5126 (4)	0.38831 (6)	0.0286 (3)

figure. The N–N bond distance is 1.384(2) Å. The C–S bond lengths are 1.8266(18) and 1.7468(17) Å respectively. The C–Cl bond lengths are 1.7394(18) and 1.7347(18) Å. In addition, the characteristic C–S–C bond angle is 100.09(8)°. All geometric parameters are in expected ranges [12].

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

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