Xu Ning, Yun-Lian Shi, Xu-Gen Shi, Wen-Li Yang* and Da-Yong Peng*

Crystal structure of *N*-(methyl(oxo)(1-(6-(trifluoromethyl)pyridin-3-yl)ethyl)- λ^6 -sulfanylidene)cyanamide, $C_{10}H_{10}F_3N_3OS$

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Abstract

$[C_{10}H_{10}F_3N_3OS]$, monoclinic, $P2_1/c$ (no. 14), $a = 18.555(10)$ Å, $b = 6.785(4)$ Å, $c = 10.139(5)$ Å, $\beta = 105.86^\circ$, $V = 1227.9(11)$ Å³, $Z = 4$, $R_g(F) = 0.0571$, $wR_{ref}(F^2) = 0.1785$, $T = 296(2)$ 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.

Source of material

The synthesis process of the title compound was carried out using (*Z*)-*N*-(methyl(1-(6-(trifluoromethyl)pyridin-3-yl)ethyl)- λ^4 -sulfanylidene)cyanamide for start material, using an oxidation reaction [3–6]. A solution of (*Z*)-*N*-(methyl(1-(6-(trifluoromethyl)pyridin-3-yl)ethyl)-sulfanylidene)cyanamide (5.75 g, 22 mmol) in acetonitrile (50 mL) was cooled in an ice bath at 5 °C. To the well stirred solution

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.17 × 0.15 × 0.13 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.29 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max} , completeness:	25.5°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	8810, 2281, 0.031
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 1978
$N(param)_{refined}$:	165
Programs:	Bruker [1], SHELX [2]

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

Atom	x	y	z	U_{iso}^*/U_{eq}
C1	0.36996(17)	1.0306(5)	1.0228(3)	0.0536(8)
C2	0.37549(19)	0.8294(5)	1.0327(4)	0.0626(9)
H2	0.4160	0.7705	1.0952	0.075*
C3	0.32012(18)	0.7159(5)	0.9486(4)	0.0556(8)
H3	0.3225	0.5791	0.9532	0.067*
C4	0.26132(16)	0.8091(4)	0.8577(3)	0.0420(7)
C5	0.26111(18)	1.0135(5)	0.8571(3)	0.0547(8)
H5	0.2212	1.0768	0.7961	0.066*
C6	0.19847(16)	0.7000(4)	0.7587(3)	0.0410(6)
H6	0.1792	0.7873	0.6800	0.049*
C7	0.22099(19)	0.5094(5)	0.7032(4)	0.0531(8)
H7A	0.2569	0.5368	0.6531	0.080*
H7B	0.1775	0.4485	0.6433	0.080*
H7C	0.2428	0.4220	0.7779	0.080*
C8	0.4264(2)	1.1649(7)	1.1121(5)	0.0740(11)
C9	0.10955(16)	0.4215(4)	1.0105(3)	0.0443(7)
C10	0.04478(17)	0.6009(5)	0.7066(3)	0.0500(7)
H10A	0.0023	0.5885	0.7427	0.075*
H10B	0.0537	0.4775	0.6675	0.075*
H10C	0.0351	0.7011	0.6372	0.075*
N1	0.31390(16)	1.1252(4)	0.9374(3)	0.0572(7)
N2	0.15227(15)	0.4864(4)	0.9348(3)	0.0554(7)
N3	0.07752(19)	0.3587(5)	1.0833(3)	0.0650(8)
O1	0.10801(12)	0.8458(3)	0.9005(2)	0.0521(6)
S1	0.12346(4)	0.66558(10)	0.83841(7)	0.0377(3)
F1	0.4836(2)	1.0772(6)	1.1837(6)	0.202(3)
F2	0.4536(2)	1.2948(6)	1.0421(4)	0.1454(15)
F3	0.3980(2)	1.2778(8)	1.1862(5)	0.167(2)

*Corresponding authors: Wen-Li Yang, College of Water Conservancy and Ecological Engineering, Nanchang Institute of Technology, Nanchang 330000, P.R. China, e-mail: szyangwl@163.com; and Da-Yong Peng, College of Sciences, Jiangxi Agricultural University, Nanchang 330045, P.R. China, e-mail: dayongpeng@163.com. <https://orcid.org/0000-0003-2527-8714>

Xu Ning: College of Land Resource and Environment, Jiangxi Agricultural University, Nanchang 330045, P.R. China

Yun-Lian Shi and Xu-Gen Shi: College of Agronomy, Jiangxi Agricultural University, Nanchang 330045, P.R. China

was added (8.0 g, 22 mmol) of a 40% aqueous solution of NaMnO₄ over 0.5 h. During the addition the reaction temperature increased to about 20 °C. The resulting brown reaction slurry was allowed to stir for about 0.5 h and then cooled to about 5 °C. A 30% aqueous solution of sodium metabisulfite (29.8 g, 47 mmol) was added to the vigorously stirred reaction mixture in one portion. The temperature increased by 15 to 20 °C during the course of the addition, and then the reaction mixture slurry thickened. Additional acetonitrile (15 mL) and water (15 mL) were added to facilitate mixing. The collected grey solids were rinsed with acetonitrile (15 mL). The combined filtrate and wash was transferred to a separatory funnel. The lower aqueous phase was removed. The organic phase was concentrated in vacuo; the residue was purified by column chromatography (ethyl acetate/hexane = 1/1) to give the title compound (5.2 g), Yield 83.38%. The product was recrystallized from isopropyl alcohol. m.p. 139 °C–141 °C. Elemental Anal. Calcd. (%) for C₁₀H₁₀F₃N₃OS: C, 43.32; H, 3.64; N, 15.16; Found(%): C, 41.96; H, 3.87; N, 14.68.

Experimental details

All H atoms were included in calculated positions and refined as riding atoms, with C–H = 0.93 Å with $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{C})$ for methyl H atoms and $1.2 U_{\text{eq}}(\text{C})$ for all other H atoms.

Comment

Sulfoxaflor a new insecticide from Dow Agrosiences was sold for the first time in 2012, and the first product from this new class (the sulfoximines) of insect control agents, exhibits broad-spectrum efficacy against many sap-feeding insect pests, including aphids, white flies, hoppers, and lygus, with levels of activity that are comparable to those of other classes of insecticides [7–10]. Herein we report the crystal structure of Sulfoxaflor. The crystal structure may lead to a better understanding of mechanisms, which makes this kind of compound better understood and paves the way for the development of more novel sulfoximine compounds.

In the molecule of the title compound, bond lengths and angles are very similar to those given in the literature [11–13]. The molecular conformation is characterized by the C5–C4–C6–C7, C5–C4–C6–S1, C4–C6–S1–C8, and C4–C6–S1–N2 torsion angles of –144.8°, 88.2°, –164.6°, and 79.5°, respectively.

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