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Crystal structure of *N*-[[*(4,6*-dimethoxy-2-pyrimidinyl)amino]carbonyl]-3-(2,2,2-trifluoroethoxy)-2-pyridinesulfonamide, C₁₄H₁₄F₃N₅O₆S

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Abstract

[C₁₄H₁₄F₃N₅O₆S], monoclinic, *P*₂₁/*c* (No. 14), *a* = 12.512(4) Å, *b* = 12.572(4) Å, *c* = 12.511(4) Å, β = 114.39(1)°, *V* = 1792.5(11) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0366, *wR*_{ref}(*F*²) = 0.0849, *T* = 296(2) K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.20 × 0.16 × 0.14 mm
Wavelength:	Mo Kα radiation (0.71073 Å)
μ:	0.26 mm ^{−1}
Diffraction, scan mode:	Bruker APEX-II, φ and ω-scans
θ _{max} , completeness:	25.5°, >99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	12628, 3318, 0.031
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 2472
<i>N</i> (<i>param</i>) _{refined} :	165
Programs:	Bruker programs [1], SHELX [2]

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
S1	0.63850(5)	0.06938(5)	0.29627(5)	0.04598(17)
F1	0.82668(16)	−0.09147(14)	0.06422(15)	0.0877(5)
F2	0.90865(15)	0.05876(16)	0.09350(15)	0.0873(5)
F3	0.84952(16)	−0.01203(15)	−0.07686(14)	0.0881(6)
N1	0.47961(17)	0.20186(17)	0.16504(18)	0.0580(6)
N2	0.77815(15)	0.07704(14)	0.32940(15)	0.0419(4)
H2	0.8162	0.0187	0.3356	0.050*
N3	0.95512(16)	0.15901(14)	0.36863(17)	0.0482(5)
H3A	0.9950	0.2171	0.3871	0.058*
N4	0.96765(15)	−0.02456(14)	0.35129(15)	0.0390(4)
N5	1.12523(15)	0.09126(14)	0.37409(16)	0.0454(5)
O1	0.68198(15)	0.06548(14)	0.07600(13)	0.0564(4)
O2	0.61374(15)	0.11962(16)	0.38453(14)	0.0667(5)
O3	0.61253(14)	−0.04055(13)	0.27214(15)	0.0598(5)
O4	0.79615(15)	0.25675(13)	0.34548(15)	0.0605(5)
O5	1.29080(13)	0.03385(13)	0.37040(15)	0.0571(4)
O6	0.98551(14)	−0.20371(12)	0.32789(15)	0.0575(5)
C1	0.56546(18)	0.14157(18)	0.16281(19)	0.0422(5)
C2	0.5895(2)	0.12824(18)	0.06454(19)	0.0440(5)
C3	0.5177(2)	0.18091(19)	−0.0376(2)	0.0553(6)
H3	0.5299	0.1742	−0.1057	0.066*
C4	0.4291(2)	0.2426(2)	−0.0358(2)	0.0636(7)
H4	0.3797	0.2782	−0.1033	0.076*
C5	0.4125(2)	0.2523(2)	0.0654(3)	0.0666(7)
H5	0.3522	0.2957	0.0649	0.080*
C6	0.7080(2)	0.0563(2)	−0.0241(2)	0.0557(6)
H6A	0.7113	0.1261	−0.0555	0.067*
H6B	0.6478	0.0149	−0.0847	0.067*
C7	0.8228(2)	0.0027(2)	0.0145(2)	0.0591(7)
C8	0.8388(2)	0.17008(18)	0.34791(19)	0.0438(5)
C9	1.01885(18)	0.07003(17)	0.36470(18)	0.0395(5)
C10	1.18574(19)	0.00661(19)	0.36493(18)	0.0433(5)
C11	1.14309(19)	−0.09525(18)	0.35005(19)	0.0454(5)
H11	1.1869	−0.1532	0.3447	0.054*
C12	1.03118(19)	−0.10610(17)	0.34360(18)	0.0411(5)
C13	1.3644(2)	−0.0484(2)	0.3587(2)	0.0581(7)
H13A	1.3859	−0.0965	0.4239	0.087*
H13B	1.4340	−0.0173	0.3574	0.087*
H13C	1.3228	−0.0865	0.2869	0.087*
C14	0.8695(2)	−0.2166(2)	0.3216(2)	0.0609(7)
H14A	0.8666	−0.1901	0.3923	0.091*
H14B	0.8490	−0.2907	0.3128	0.091*
H14C	0.8150	−0.1778	0.2555	0.091*

Source of material

Trifloxysulfuron (0.0874 g, 0.2 mmol) was dissolved in ethanol (10 mL). CuCl₂ (0.0172 g, 0.1 mmol) in ethanol

(10 mL) was added slowly under stirring at room temperature. The mixture stirred well for 3 h at room temperature and some red powder generated. The resulting solution was filtered and washed with ethanol. The filtrate was left undisturbed at room temperature, and the colorless block crystal were obtained by slow evaporation of ethanol two weeks later, yield 70% (based on Trifloxysulfuron). Elemental Anal. Calcd. (%) for C₁₄H₁₄F₃N₅O₆S (437.36): C, 38.45; H, 3.23; N, 16.01. Found (%): C, 37.67; H, 3.39; N, 15.69.

Experimental details

All H atoms were included in calculated positions and refined as riding atoms, with C–H = 0.96–0.97 Å, N–H = 0.82–0.84 Å and O–H = 0.81–0.82 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.

Discussion

Herbicide, also commonly known as weedkillers, are chemical substances used to control unwanted plants [3, 4]. Trifloxysulfuron-sodium is a new type herbicide of sulfonylurea developed by syngenta company in 2001. Trifloxysulfuron, *N*-[[[(4,6-dimethoxy-2-pyrimidinyl)amino]carbonyl]-3-(2,2,2-trifluoroethoxy)-2-pyridinesulfonamide is mainly used to prevent the weeds of broad-leaved weeds and aromatic plants in cotton and sugarcane [5–9]. Because of its strong activities and its wide applications, the synthesis of trifloxysulfuron, its intermediates and its derivatives have attracted much attention [10, 11]. In order to find the new herbicide complex with high efficiency and low toxicity, and further study the structural changes of trifloxysulfuron may occurred in the application process, we have been engaged in synthesizing new metal complex. But we obtained single crystals of the title compound as a by-product.

There is one crystallographic independent molecule in the asymmetric unit. In the molecule of the title compound, bond lengths and angles range within similar deviations [12]. The molecular conformation is characterized by the C8–N3–C9–N4 and N2–S1–C1–N1 torsion angles of 170.8° and 136.9°, respectively. The pyridine C1/C2/C3/C4/C5/N1 ring and pyrimidine C9/N5/C10/C11/C12/N4 ring are each planar. The dihedral angle of two rings is 81.0°.

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References

1. Bruker. APEX2, SAINT and SADABS, Bruker AXS Inc., Madison, WI, USA (2009).
2. Sheldrick, G. M.: A short history of SHELX. *Acta Crystallogr. A* **64** (2008) 112–122.
3. Jian, Q.; Zheng, Z. T.; Song, W. C.: Degradation dynamics of MCPA-Ametryn-diuron residues in sugarcane and soil. *Acta Agric. Univ. Jiangxiensis*. **37** (2015) 825–831.
4. Liang, Y.; Jia, H. J.; Dong, X.; Li, J. J.; Liu, Y. L.; Cao, Z. J.; Wang, C. J.; Yang, D. S.: The effects of four soil applied herbicides on control of solanum nigrum L- and safety svaluation. *Acta Agric. Univ. Jiangxiensis*. **36** (2014) 106–108.
5. Tang, K. J.; Zhou, Z. E.; Yu, R. L.; Chen, S. W.: Separation of five sulfonylurea herbicides and application in soil sample. *Acta Agric. Univ. Jiangxiensis*. **36** (2014) 326–331.
6. Diao, J. X.; Wang, Z. Z.; Guo, W. L.; Zhang, L. L.; Li, W.; Zhao, Z. Y.; Wang, J. X.: Weed control efficacy and safety evaluation to cotton of combinations of trifloxysulfuron and quizalofop-p-ethyl. *Chine J. Pestic. Sci.* **16** (2014) 706–712.
7. Dodds, D. M.; Kirkpatrick, M. T.; Barber, L. T.; Reynolds, D. B.: Evaluation of trifloxysulfuron plus prometryn for weed control in cotton. *J. Cotton Sci.* **12** (2008) 311–317.
8. Anderson, R. D. O.; Silvério, D. P. F.; Ismael, L. J. F.: Control of cyperus rotundus L. with trifloxysulfuron-sodium in sugar-cane. *Revista Ceres*. **57**(2010) 736–741.
9. Md, K. U.; Abdul, S. J.; Mohd, R. I.; Mahfuza, B.; Radzah, O.; Anuar, A. R.: Efficacy of sea water in combination with trifloxy-sulfuron and quinclorac to control weeds. *Aust. J. Crop. Sci.* **5** (2011) 1305–1310.
10. Zhang, J. Z.; Xu, D. F.; Wu, J. P.: Synthesis of 3-(2,2,2-Trifluoroethoxy)-2-pyridinesulfonamide. *Asian J. Chem.* **22** (2010) 826–828.
11. Parikh, K. S.; Vyas, S. P.: Synthesis and spectral studies of some novel schiff base derived with pyrimidines. *J. Chem. Pharma. Res.* **4** (2012) 2109–2111.
12. Xia, J. Y.; Li, F. S.; Yin, L. H.; Yu, D. S.; Wu, D. Y.: Methyl 2-[[3-(4,6-dimethoxypyrimidin-2-yl)ureido]sulfonylmethyl]benzoate. *Acta Cryst.* **E64** (2008) o632.