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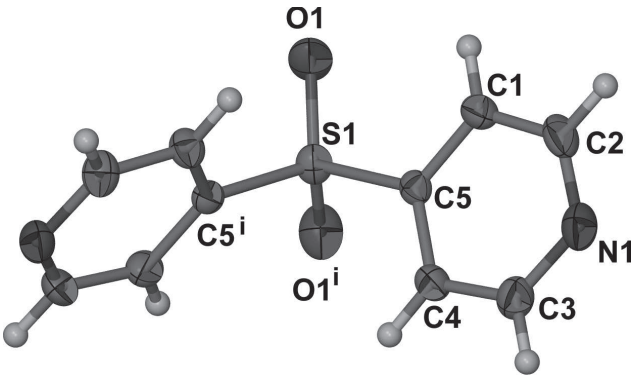
Hui Chen, Rui-Heng Liu and Hui Guo*

Crystal structure of 4,4′-sulfonyldipyridine, $C_{10}H_8N_2O_2S$



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Abstract

$C_{10}H_8N_2O_2S$, monoclinic, $C2/c$ (no. 15), $a = 7.29640(10)$ Å, $b = 13.1899(2)$ Å, $c = 10.6087(2)$ Å, $\beta = 94.763(1)°$, $V = 1017.44(3)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0344$, $wR_{ref}(F^2) = 0.1025$, $T = 296$ K.

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The crystal structure is shown in the figure. Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colorless, prism, size 0.12×0.28×0.44 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.97 cm^{-1}
Diffractometer, scan mode:	Bruker APEXII, ω scans
$2\theta_{max}$:	54.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7127, 1175
$N(param)_{refined}$:	70
Programs:	SHELX [7], checkCIF [8], Diamond [9]

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

Atom	Site	x	y	z	U_{iso}
H(1A)	8*f*	−0.0543	0.2859	0.4850	0.071
H(2A)	8*f*	0.0914	0.3878	0.3478	0.087
H(3A)	8*f*	0.4025	0.5066	0.6204	0.078
H(4A)	8*f*	0.2735	0.4079	0.7678	0.066

Source of material

All reagents and solvents from commercial sources were used without further purification. Di-4-pyridylsulfide was purchased form the J&K Chemical Industry Co. Ltd and used as starting materials. Di-4-pyridylsulfide (1 g, 5.3 mmol) was dissolved in glacial acetic acid (20 mL) that contains 30% w/w hydrogen peroxide (2 mL). The mixture was kept at room temperature for two weeks without stirring. The solution was then treated with water (25 mL). A white crystalline precipitate deposited, which was filtered and further purified through chromatography over silica-gel by using ethyl acetate/CH_2Cl_2 (1:1) as eluent. Sulfonyldipyridine was obtained as white powdery product 408 mg (Yield: 35%). The title compound was dissolved in methanol, and was left to stand in air. The solvent slowly evaporated, and colourless crystals of the title compound were deposited after one week.

Experimental details

All hydrogen atoms were identified in difference Fourier syntheses. The U_{iso} values of the hydrogen atoms of methyl groups were set to $1.5U_{eq}$(C) and the U_{iso} values of all other hydrogen atons were set to $1.2U_{eq}$(C, N).

Discussion

Di-pyridyl based organic molecule containing a CH_2, O, S or SO (sulfinyl) hinge were widely used as versatile angular-shaped linkers in the syntheses of a large number of transition metal complexes [1]. In contrast, the analogous ones bearing a SO_2 (sulfonyl) hinge group are rarely reported. There are several examples reported in the literature, such as sulfonyldiacetylide [2], sulfonyldiyne [1], sulfonyldicarboxylate [3, 4] and bis[3-(2-pyridylmethyleneamino)-phenyl]

***Corresponding author: Hui Guo,** Chemistry Department, Capital Normal University, Beijing 100048, P. R. China, e-mail: wanchongqq@163.com
Hui Chen and Rui-Heng Liu: Chemistry Department, Capital Normal University, Beijing 100048, P. R. China

Table 3: Fractional coordinates and atomic displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S(1)	4*e*	0	0.25375(4)	0.7500	0.0624(4)	0.0424(3)	0.0564(4)	0.0	0.0111(2)	0.0
O(1)	8*f*	0.1474(2)	0.19940(9)	0.8172(1)	0.0924(9)	0.0659(8)	0.0819(9)	0.0312(6)	0.0182(7)	0.0216(6)
N(1)	8*f*	0.2596(2)	0.4567(1)	0.4702(1)	0.0704(9)	0.079(1)	0.0593(8)	0.0075(7)	0.0195(7)	0.0126(7)
C(1)	8*f*	0.0393(2)	0.3297(1)	0.5148(2)	0.0514(8)	0.078(1)	0.0483(8)	0.0008(7)	−0.0013(6)	−0.0078(7)
C(2)	8*f*	0.1276(3)	0.3919(2)	0.4338(2)	0.069(1)	0.109(2)	0.0396(8)	0.017(1)	0.0031(7)	0.0059(9)
C(3)	8*f*	0.3101(2)	0.4612(1)	0.5930(2)	0.069(1)	0.0627(9)	0.066(1)	−0.0101(8)	0.0184(8)	−0.0035(8)
C(4)	8*f*	0.2338(2)	0.4026(1)	0.6825(1)	0.0608(9)	0.0603(9)	0.0452(7)	−0.0072(7)	0.0055(6)	−0.0056(6)
C(5)	8*f*	0.0966(2)	0.3357(1)	0.6412(1)	0.0447(7)	0.0458(7)	0.0429(7)	0.0064(5)	0.0058(5)	−0.0033(5)

sulfone [5]. In the title compound, the S1 = O1 bond length equals 1.4320(13) Å, and the C5—S1—C5^i angle is 104.68(8)°. The dihedral angle between the pair of the pyridyl rings is 70.12(9)°. Thus the molecule looks like a butterfly bearing pyridine wings (Figure). The S = O bond is comparable to that 1.422(2) Å – 1.434(2) Å found in sulfonyldipyridine reported [6].

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