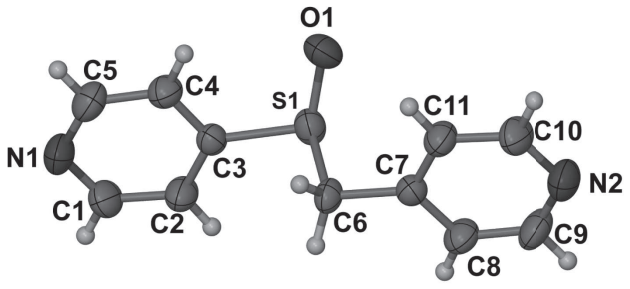
Open Access

Hao-Jie Yan*, Zhi-Peng Wang, Qi-Qi Zhang and Wen-Hua Chen

Crystal structure of 4-((pyridin-4-ylmethyl) sulfinyl)pyridine, $C_{11}H_{10}N_2OS$



DOI 10.1515/ncrs-2014-9102
Received June 1, 2015; accepted January 11, 2016; available online February 8, 2016

Abstract

$C_{11}H_{10}N_2OS$, monoclinic, *Pc* (no. 7), $a = 12.050(3)$ Å, $b = 5.5218(13)$ Å, $c = 8.1813(19)$ Å, $\beta = 107.934(6)°$, $V = 517.9(2)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0483$, $wR_{ref}(F^2) = 0.1247$, $T = 296$ K.

CCDC no.: 1446485

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, Block, size 0.30×0.40×0.50 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.84 cm^{-1}
Diffractometer, scan mode:	Bruker Smart ApexII, ω scans
$2\theta_{max}$:	55.71°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5839, 2375
$N(param)_{refined}$:	136
Programs:	Platon [8], Diamond [9], SHELX [10]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1A)	2*a*	0.3345	−0.1107	0.8208	0.064
H(2A)	2*a*	0.5233	−0.0461	0.8248	0.057
H(4A)	2*a*	0.5187	0.5801	1.0702	0.053
H(5A)	2*a*	0.3309	0.4912	1.0649	0.066
H(6A)	2*a*	0.7439	0.2228	1.2338	0.046
H(6B)	2*a*	0.7483	−0.0038	1.1186	0.046
H(8A)	2*a*	0.9238	−0.0767	1.0309	0.057
H(9A)	2*a*	1.1164	0.0028	1.0695	0.070
H(10A)	2*a*	1.1051	0.5865	1.3263	0.063
H(11A)	2*a*	0.9128	0.5251	1.2990	0.055

Source of material

All reagents and solvents obtained from commercial sources were used without further purification. The starting 4-((pyridin-4-yl)methylthio)pyridine was synthesized according to a modified procedure in literature [1]. A solution of 3-chlorobenzoperoxoic acid (72° m-CPMA, 750 mg, 3.22 mmol) in 20 mL of CH_2Cl_2 was added to a 10 mL CH_2Cl_2 solution of 4-((pyridin-4-yl)methylthio)pyridine (606 mg, 3 mmol) and cooled to 0°C. After stirring for 2 h at 0°C, the organic phase was treated with 30 mL of saturated $Na_2S_2O_3$ solution, saturated $NaHCO_3$ solution (30 mL), and brine (20 mL) and dried over $MgSO_4$. The combined organic extract was concentrated to give a white solid. The product was further purified by column chromatography on silica gel using ethyl acetate/CH_2Cl_2 (1:1) as eluent. 4-(Pyridin-4-ylmethylsulfinyl)pyridine was obtained as a white powder (401 mg). Yield: 63°, m.p. 100–103°C. Colorless crystals of the title compound were obtained by slow evaporation from dichloromethane/ethanol 2:1 (v/v) at room temperature after a week.

Experimental details

The Flack x parameter of 0.42(6) suggests the existence of an inversion twin.

Discussion

Interest in sulfoxide stemmed from stereochemical consequences arising from the configuration of the R-S-(O)-R-group [2]. Many such studies focused on their biological activity

***Corresponding author: Hao-Jie Yan,** Department of Chemistry, Capital Normal University, Beijing 100048, P. R. China, e-mail: wanchqq@163.com
Zhi-Peng Wang, Qi-Qi Zhang and Wen-Hua Chen: Department of Chemistry, Capital Normal University, Beijing 100048, P. R. China

Table 3: Atomic displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S(1)	2*a*	0.6851(1)	0.3411(2)	0.9401(1)	0.0365(5)	0.0476(5)	0.0470(5)	−0.0035(5)	0.0134(4)	0.0035(5)
O(1)	2*a*	0.7049(3)	0.6036(5)	0.9788(5)	0.053(2)	0.043(2)	0.083(3)	−0.007(2)	0.019(2)	0.011(2)
N(1)	2*a*	0.3143(3)	0.1820(7)	0.9437(6)	0.033(2)	0.061(2)	0.067(3)	0.003(2)	0.015(2)	−0.000(2)
N(2)	2*a*	1.1312(3)	0.3012(8)	1.2017(6)	0.035(2)	0.064(3)	0.070(3)	0.004(2)	0.014(2)	−0.001(2)
C(1)	2*a*	0.3725(4)	0.0288(9)	0.8731(7)	0.038(2)	0.048(2)	0.070(3)	−0.007(2)	0.011(2)	−0.003(2)
C(2)	2*a*	0.4857(4)	0.0664(8)	0.8739(6)	0.037(2)	0.042(2)	0.062(3)	−0.001(2)	0.014(2)	−0.009(2)
C(3)	2*a*	0.5418(3)	0.2741(7)	0.9488(5)	0.031(2)	0.037(2)	0.038(2)	0.001(2)	0.008(2)	0.004(2)
C(4)	2*a*	0.4832(4)	0.4372(8)	1.0202(6)	0.042(2)	0.039(2)	0.051(2)	0.002(2)	0.013(2)	−0.004(2)
C(5)	2*a*	0.3701(4)	0.3824(9)	1.0154(7)	0.044(2)	0.059(3)	0.064(3)	0.011(2)	0.021(2)	−0.001(2)
C(6)	2*a*	0.7654(3)	0.1678(7)	1.1350(5)	0.026(2)	0.043(2)	0.043(2)	0.000(2)	0.007(2)	−0.006(2)
C(7)	2*a*	0.8946(3)	0.2156(7)	1.1610(5)	0.034(2)	0.036(2)	0.039(2)	0.001(2)	0.011(2)	0.003(2)
C(8)	2*a*	0.9586(4)	0.0597(8)	1.0923(6)	0.044(2)	0.044(2)	0.056(3)	0.005(2)	0.018(2)	−0.008(2)
C(9)	2*a*	1.0747(4)	0.1098(9)	1.1162(7)	0.040(2)	0.061(3)	0.073(3)	0.015(2)	0.018(2)	−0.006(2)
C(10)	2*a*	1.0683(4)	0.4506(9)	1.2666(7)	0.036(2)	0.051(3)	0.065(3)	−0.004(2)	0.007(2)	−0.009(2)
C(11)	2*a*	0.9522(4)	0.4156(8)	1.2504(6)	0.039(2)	0.044(2)	0.056(3)	0.004(2)	0.016(2)	−0.006(2)

[3], synthetic applications in asymmetric synthesis [4] and their behavior as intermediates in homogeneous catalytic processes [5]. Furthermore, sulfoxides are versatile precursors in inorganic synthesis [6]. Herein, we report one new pyridyl-based sulfoxide containing compound (see the figure). Single crystal x-ray diffraction revealed that the value of the C3—S1—C6—C7 torsion angle is 176.0(3)°. The S1 = O1 bond length is 1.487(3) Å in the title molecule which is in good agreement with the average value of 1.49 Å in analogous sulfoxide complexes [7]. The molecule adapts a zigzag configuration with the pair of 4-pyridyl rings of each molecule exhibit a dihedral angle of 5.734(3)°.

Acknowledgements: I acknowledge support from Scientific Research Base Development Program of the Beijing Municipal Commission of Education.

References

1. Grassetti, D. R.; Brokke, M. E.; Murray Jr, J. F.: The effect of some sulfur-containing pyridine derivatives on the carbohydrate metabolism of ehrlich ascites tumor. J. Med. Chem. **8** (1965) 753–756.
2. Kovacs, J. A.: Synthetic analogues of cysteinate-ligated non-heme iron and non-corrinoid cobalt enzymes. Chem. Rev. **104** (2004) 825–848.
3. Bentley, R.: Role of sulfur chirality in the chemical processes of biology. Chem. Soc. Rev. **34** (2005) 609–624.
4. Posner, G. H.: Asymmetric synthesis of carbon-carbon bonds using sulfinyl cycloalkenones, alkenolides, and pyrones. Acc. Chem. Res. **20** (1987) 72–78.
5. Mariz, R.; Poater, A.; Gatti, M.; Drinkel, E.; Bürgi, J. J.; Luan, X.-J.; Blumentritt, S.; Linden, A.; Cavallo, L.; Dorta, R.: C2-symmetric chiral disulfoxide ligands in rhodium-catalyzed 1,4-addition: From ligand synthesis to the enantioselection pathway. Chem. Eur. J. **16** (2010) 14335–14347.
6. Alessio, E.: Synthesis and reactivity of Ru-, Os-, Rh-, and Ir-Halide- sulfoxide complexes. Chem. Rev. **104** (2004) 4203–4242.
7. Calligaris, M.: Structure and bonding in metal sulfoxide complexes: an update. Coord. Chem. Rev. **248** (2004) 351.
8. Spek, A. L.: Structure validation in chemical crystallography. Acta Crystallogr. **D65** (2009) 148–155.
9. Brandenburg, K.: DIAMOND. Visual crystal structure information system. Version 3.2i. Crystal Impact, Bonn, Germany 2012.
10. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112—122.