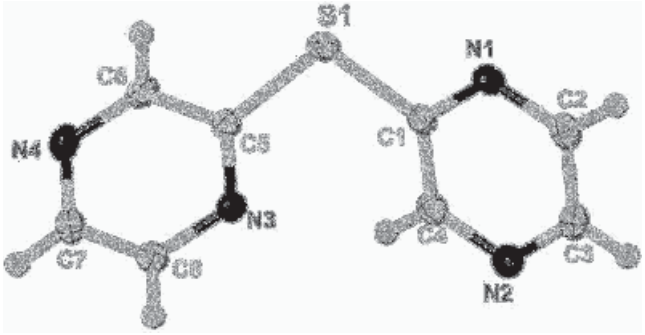
Crystal structure of di(pyrazin-2-yl)sulfane, $C_8H_6N_4S$

Xiao Chun Jia*, Chun Chen, Xiang Li, Bin Zhang and Yan Yan Yu

Tianjin Entry-Exit Inspection and Quarantine Bureau, Tianjin 30021, P.R. China

Received January 05, 2013, accepted April 09, 2013, available online June 28, 2013, CCDC no. 916933



Abstract

$C_8H_6N_4S$, monoclinic, $P2_1/n$ (no. 14), a = 3.9096(7) Å, b = 20.518(4) Å, c = 10.628(2) Å, β = 98.402(3)°, V = 843.4 Å^3, Z = 4, $R_{gt}(F)$ = 0.0395, $wR_{ref}(F^2)$ = 0.1094, T = 293 K.

Table 1. Data collection and handling.

Crystal:	yellow blocks, size 0.20×0.30×0.34 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	3.35 cm^{-1}
Diffractometer, scan mode:	Bruker APEXII CCD area detector, ω
$2\theta_{max}$:	56.56°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5803, 2095
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1586
$N(param)_{refined}$:	118
Programs:	SHELX [8], PLATON [9]

Source of material

Di(pyrazin-2-yl)sulfane $C_8H_6N_4S$ was obtained by using the same synthetic method as reported in the literature [1]. The product was purified by column chromatography on silica gel (eluent: dichloromethane/methanol = 95:5, *v*/*v*), and the title compound was obtained in 40.1% yield. The orange crystals with average dimensions of 0.34´0.30×0.20 mm were obtained by slow evaporation from methanol / acetonitrile 1/1 (*v*/*v*).

Experimental details

The hydrogen atoms were placed in idealized positions and allowed to ride on the relevant carbon atoms. U_{iso}(H) = 1.2 U_{eq}(C)$_{aryl}$.

Discussion

Chalcogenobispyridines and derivates display a wide range of biological and industrial applications, as well as of building blocks for supramolecular assembly [2-6]. However, nitrogen rich diheterocyclic sulfides have been rarely reported [7]. As shown in the figure, the S atom bears two pyrazinyl rings with a dihedral angle of 48.37(5)°. The S–C bond lengths are 1.7665(16) Å and 1.7683(16) Å, and comparable with the avarage value of 1.780 Å in dipyrimidinyl sulfane [7]. Along the *a* axis, the molecules are stacked with the distance between the pyrazinyl ring centroids of 3.910(3) Å. By those intermolecular $\pi\cdots\pi$ interactions, a clomun motif is formed with the molecules.

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(8A)	4*e*	–0.1186	0.0194	0.3861	0.058
H(6A)	4*e*	0.4009	0.2111	0.3326	0.057
H(7A)	4*e*	–0.0334	0.0522	0.1893	0.064
H(3A)	4*e*	0.4282	0.0652	1.0159	0.066
H(4A)	4*e*	0.6134	0.0759	0.6669	0.054
H(2A)	4*e*	0.2533	0.1709	1.0064	0.064

* Correspondence author (e-mail: xiaochuanjia2012@163.com)

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S	4*e*	0.3714(2)	0.20576(2)	0.58991(4)	0.0863(4)	0.0363(2)	0.0444(3)	–0.0137(2)	0.0135(2)	–0.0014(2)
N(3)	4*e*	0.0853(4)	0.09515(7)	0.4816(1)	0.0570(9)	0.0410(7)	0.0380(7)	–0.0088(6)	0.0086(7)	0.0030(6)
N(1)	4*e*	0.2887(4)	0.18634(7)	0.8268(1)	0.062(1)	0.0401(7)	0.0443(7)	0.0012(7)	0.0145(7)	–0.0078(6)
N(2)	4*e*	0.5418(5)	0.05881(7)	0.8418(1)	0.063(1)	0.0483(8)	0.0467(8)	0.0105(7)	–0.0023(8)	–0.0018(7)
N(4)	4*e*	0.1909(5)	0.13582(8)	0.2393(1)	0.076(1)	0.061(1)	0.0366(7)	0.0007(9)	0.0114(8)	0.0062(7)
C(5)	4*e*	0.2389(4)	0.15151(7)	0.4631(1)	0.0428(9)	0.0360(8)	0.0366(8)	0.0009(7)	0.0090(7)	0.0026(6)
C(1)	4*e*	0.3940(4)	0.15668(7)	0.7276(1)	0.0416(9)	0.0372(8)	0.0369(7)	–0.0059(7)	0.0068(7)	–0.0060(6)
C(8)	4*e*	–0.0119(5)	0.05930(9)	0.3775(2)	0.058(1)	0.0416(9)	0.0444(9)	–0.0069(8)	0.0010(8)	0.0005(7)
C(6)	4*e*	0.2916(5)	0.17148(9)	0.3417(2)	0.058(1)	0.0426(9)	0.0430(9)	0.0012(8)	0.0143(8)	0.0091(7)
C(7)	4*e*	0.0393(5)	0.0790(1)	0.2586(2)	0.065(1)	0.055(1)	0.0386(9)	0.0028(9)	0.0003(9)	–0.0038(8)
C(3)	4*e*	0.4290(6)	0.0883(1)	0.9406(2)	0.069(1)	0.057(1)	0.0382(9)	0.0016(9)	0.0014(9)	0.0012(8)
C(4)	4*e*	0.5293(5)	0.09381(8)	0.7367(2)	0.047(1)	0.0457(9)	0.0416(8)	0.0055(7)	0.0060(8)	–0.0090(7)
C(2)	4*e*	0.3148(6)	0.1516(1)	0.9338(2)	0.069(1)	0.056(1)	0.0383(8)	0.0016(9)	0.0128(9)	–0.0084(8)

Acknowledgments. General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China

References

1. Cheeseman, G. W. H.: The synthesis and tautomerism of some 2-substituted pyrazines. J. Chem. Soc. (1960) 242-245.
2. Dunne, S. J.; Summers, L. A.; von Nagy-Felsobuki, E. I.: Conformational and UV photoelectron spectroscopy analysis of the chalcogenobispyridines. Coord. Chem. Rev. **165** (1997) 1-92.
3. Baradello, L.; Schiavo, S. L., Nicolò, F.; Alibrandi, G.; Tresoldi, G. Lanza, S.: Restricted Phenyl Rotation in Pyridyl Thioether Ligands N,S-Chelated to Congested Diiminoruthenium Cores. Eur. J. Inorg. Chem. (2004) 3358-3369.
4. Bruno, G.; Nicolò, F.; Schiavo, S. L.; Sinicropi, M. S.; Tresoldi, G.: Synthesis and spectroscopic properties of di-2-pyridyl sulfide (dps) compounds. Crystal structure of [Ru(*dps*)$_2$Cl$_2$]. J. Chem. Soc., Dalton Trans. (1995) 17-24.
5. Jung, O.-S.; Kim, Y. J.; Lee, Y-A.; Chae, H. K.; Jang, H. G.; Hong, J.: Structures and Related Properties of AgX Bearing 3,3'-Thiobispyridine (X^-= NO_3^-, BF_4^-, ClO_4^-, and PF_6^-). Inorg. Chem. **40** (2001) 2105-2110.
6. Jung, O.-S.; Kim, Y. J.; Lee, Y.-A.; Park, K.-M.; Lee, S. S.: Subtle Role of Polyatomic Anions in Molecular Construction: Structures and Properties of AgX Bearing 2,4'-Thiobis(pyridine) (X^-= NO_3^-, BF_4^-, ClO_4^-, PF_6^-, $CF_3CO_2^-$, and $CF_3SO_3^-$). Inorg. Chem. **42** (2003) 844-850.
7. Wan, C.-Q.; Wang, Z.-J.; Wang, G.; Liu, H.; Deng, Y.-H.; Jin, Q.-H.: Conformation Restriction of Nonplanar Di-2-pyrimidyl Sulfide with Intramolecular N···C Interaction and Its Supramolecular Silver(I) Complexes. Cryst. Growth Des. **12** (2012) 376-386.
8. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112-122.
9. Spek, A. L.: Single-crystal structure validation with the program PLATON. J. Appl. Crystallogr. **36** (2003) 7-13.