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NOTE

Synthesis and Crystal Structure of 1,2-*bis*(Pyrazin-2-ylthio)ethane

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A new organic compound has been synthesized and characterized by elemental analysis and X-ray diffraction. The results show that the present compound 1,2-*bis*(pyrazin-2-ylthio)ethane is a double-thioether compound. At first, we tried to obtain a Co(II) complex containing pyrazine-2-thiol ligand. Unexpectedly, two new organic ligands of 1,2-*bis*(pyrazin-2-ylthio)ethane and 1-(2-(pyrazin-2-ylthio)ethyl)-pyrazine-2(1*H*)-thione which is isomer to each other were obtained from pyrazine-2-thiol.

Key Words: Thioether, Synthesis, Structure.

For a long time, thioether-type ligands have been a focus for researchers due to their good flexibilities and versatile coordination modes. Therefore, there were a lot of reports about the crystal structure related to thioether-type compounds recently[1,2]. As a part of our series of research about thioether-type compounds, we present herein the crystal structure of the 1,2-*bis*(pyrazin-2-ylthio)ethane. At first, we tried to obtain a Co(II) complex containing pyrazine-2-thiol ligand. Unexpectedly, two new organic ligands of 1,2-*bis*(pyrazin-2-ylthio)-ethane and 1-(2-(pyrazin-2-ylthio)-ethyl)pyrazine-2(1*H*)-thione which is isomer to each other were synthesized from pyrazine-2-thiol. The two compounds, one of which was previously reported last year[3], were separated according to their different solubility in water.

All solvents and chemicals obtained from commercial sources were of reagent grade and used without further purification. The elemental analyses for C, H, N and S were performed on a Perkin-Elmer 240 ºC elemental analyzer. Crystal data were collected at 298(2) K on a Bruker SMART CCD area detector diffractometer.

Synthesis of the title compound: The syntheses process of the title compound is according to the literature[3]. To a solution of pyraine-2-thiol (250 mg, 2 mmol) and 1,2-dibromo-ethane (0.09 mL, 1 mmol) in 15 mL sodium ethoxide ethanol solution, a aqua solution containing $CoCl_2 \cdot 6H_2O$ (47.9 mg, 0.2 mmol) was added with drop. The resulting solution was stirred at 352 K for 3 h and a portion of ethanol was removed by distillation under reduced pressure, then a yellow powder was obtained. The products were placed in distilled water and found that only a small part of the powder can dissolve in it. They were recrystallized with anhydrous ethanol to give two different colours of crystals suitable for X-ray diffraction. We found that the compound which is poorly soluble in water is the title compound. Anal. calcd. $C_{10}H_{10}N_4S_2$ for (%): C, 47.98; H, 4.02; N, 22.38; S, 25.62. Found (%): C, 48.12; H, 3.82; N, 22.56; S, 25.50.

X-ray crystallographic determination: A yellow-block single crystal of the title compound with dimensions of 0.40 mm × 0.38 mm × 0.19 mm was used for structure determination. Data were collected at 298(2) K on a Bruker SMART CCD area detector diffractometer equipped with a graphite-monochromatic MoK_α (λ = 0.71073 Å) radiation by using a ϕ-ω mode in the range of $3.73 \le \theta \le 25.01°$. The structure was solved by direct methods and refined by full-matrix least-squares on F^2 using SHELXL-97 program package[4]. All non-hydrogen atoms were assigned anisotropically. The hydrogen atoms were generated theoretically and treated by a mixture of independent and constrained refinement. The final cycle of refinement converged at R = 0.0419, wR = 0.0949. Selected bond lengths and bond angles of the title compound are listed in Table-1 and the hydrogen bond lengths and bond angles of the compound in Table-2.

Crystal structure description: The title compound, $C_{10}H_{10}N_4S_2$, is isostructural with 1-[2-(pyrazin-2-ylthio)ethyl]-pyrazine-2(1*H*)-thione reported last year by our group, whose structure has been described in detail[3].

TABLE-1
SELECTED BOND LENGTHS (Å) AN D BOND ANGLES (°) OF THE TITLE COMPOUND

Bond	Dist (Å)	Bond	Dist (Å)
N1–C1	1.332(4)	N1–C4	1.340(4)
S1–C1	1.758(3)	S1–C5	1.807
Angle	(°)	Angle	(°)
C1–N1–C4	115.6(3)	C2–N2–C3	115.5(3)
C1–S1–C5	102.62(15)		

TABLE-2
HYDROGEN BOND LENGTHS (Å) AND BOND ANGLES (°) OF THE COMPOUND

D–H···A	d (D–H)	d (H···A)	d (D···A)	∠ (DHA)
C5–H5B···S1	0.97	3.09	3.785 (37)	129
C2–H2···N1	0.93	2.68	3.482 (46)	145
C4–H4···N2	0.93	2.83	3.603 (53)	141
C5–H5A···N1	0.97	3.08	3.714 (13)	124

In the compound, the plane of the two pyrazine ring from the same molecule (mean deviation from plane = 0.0 Å) are parallel to each other and the distance of the two plane is 1.477 Å (Fig. 1). The distances of C-S bonds are 1.758(3) Å and 1.807 Å, which are longer than C=S double bonds[5] and match with the normal C-S range[1]. In the structure, there is short of any classical hydrogen bonds due to lack of relevant donors and acceptors for such bonds. However, there are many non-classical hydrogen bonds such as C-H···N (C_2-H_2···N_1, C_4-H_4···N_2, C_5-H_5A···N_1) and C-H···S (C_5-H_{5B}···S_1), which are beneficial to the stability of the crystal structure (Fig. 2).

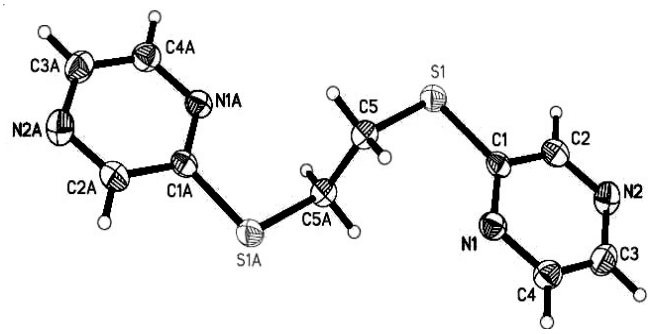

Fig. 1. Molecular view of the title compound showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30 % probability level. H atoms are represented as small spheres of arbitrary radii

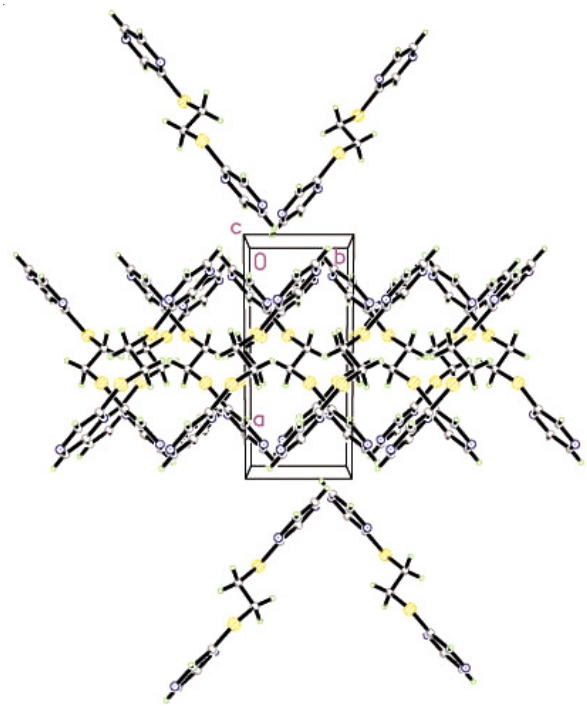

Fig. 2. Crystal packing of the title compound viewed along the c axis

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