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NOTE

Synthesis and Crystal Structure of 2-(Methylsulfonyl)-4-(pyridin-3-yl)pyrimidine

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A new compound with molecular formula as $C_{10}H_9N_3O_2S$, was derived from 2-(methylthio)-4-(pyridin-3-yl)pyrimidine. The crystal is monoclinic, space group C2/c with unit cell parameters: a = 5.4625 (8) Å, b = 10.8235 (16) Å, c = 17.520 (3) Å, $\alpha = 90°$, $\beta = 97.460$ (3)°, $\gamma = 90°$, V = 1027.1 (3) Å^3, Z = 4, Mr = 235.00, Dc = 1.521 g/cm^3, $\mu = 0.300$ mm^{-1}, F(000) = 488, R = 0.0443, wR = 0.1061 for 2413 reflections with $I > 2\sigma(I)$.

Key Words: 2-(Methylsulfonyl)-4-(pyridin-3-yl)pyrimidine, Hydrogen bonds, Sulfone.

Remarkable attention has been paid to the coordination polymers and metal-organic frameworks derived from heterocyclic thiolates or thioethers in recent years for their fascinating structural topologies and potential uses as functional materials[1-6].

In our previous paper, we reported some 3D supramolecular complexes derived from thioethers or heterocyclic sulfonate[3-8]. Herein reported is a new compound 2-(methylsulfonyl)-4-(pyridin-3-yl)pyrimidine.

All reagents for synthesis were commercially available and employed as received or purified by standard methods prior to use. 2-(Methylthio)-4-(pyridin-3-yl)pyrimidine was prepared by similar procedure reported in the literature[4]. Analyses for carbon, hydrogen and nitrogen were performed on a Perkin-Elmer 1400C analyzer. Infrared spectra (4000-400 cm^{-1}) were recorded with a Bruker Vector 22 FT-IR spectrophotometer on KBr disks.

Synthesis: A solution of H_2O_2 (30 mL, 30 %) was added dropwise into the suspension containing 2-(methylthio)-4-(pyridin-3-yl)pyrimidine (0.1 mol) and 20 mL of acetic acid. After addition, the mixture was stirred in the water bath for 0.5 h and then refluxed for 1 h. After cooling, yellow precipitates were filtered, washed with water and ethanol and dried in vacuum. Single crystals suitable for X-ray diffraction analysis were obtained by slow evaporation of the CH_2Cl_2 solution of 2-(methylsulfonyl)-4-(pyridin-3-yl)pyrimidine. Yield 71 %. Anal. calcd. for $C_{10}H_9N_3O_2S$: C, 51.05; H, 3.86; N, 17.86. Found: C, 51.11; H, 3.92; N, 17.81. Main IR (KBr, ν_{max}, cm^{-1}): 1585 s, 1541 m, 1492 w, 1458 m, 1372 w, 1255 s, 1213 s, 1034 s, 1019 m.

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Crystal structure determination: A single crystal of compound with dimensions of 0.3 mm × 0.2 mm × 0.2 mm was selected for crystallographic data collection at 291(2)K and structure determination on a Bruker SMART CCD-4K diffractometer employing graphite-monochromated MoKα radiation (λ = 0.71073 Å). A total of 6049 reflections were collected in the range of 2.2° ≤ q ≤ 28.3°, of which 2413 reflections were unique with R_{int} = 0.030. The data were collected using SMART and reduced by the program SAINT. All the structures were solved by direct methods and refined by full-matrix least squares method on F^2_{obs} by using SHELXTL-PC software package. Non-hydrogen atoms were placed in geometrically calculated positions. Hydrogen atoms were added according to theoretical model. The final full-matrix least-squares refinement including 146 variable parameters for 2413 reflections with $I > 2\sigma(I)$ and converged with unweighted and weighted agreement factors of

$$R_1 = \Sigma(||F_0| - |F_c||)/\Sigma|F_0| = 0.0443 \quad (1)$$

and

$$wR_2 = \{\Sigma[w(F_0^2 - F_C^2)^2]/\Sigma w(F_0^2)^2\}^{1/2} = 0.1061 \quad (2)$$

where $w = 1/[\sigma^2(F_0^2) + (0.0542P)^2]$ and $P = (F_0^2 + 2F_C^2)/3$. The maximum and minimum peaks on the final difference Fourier map are corresponding to 0.37 and -0.23 e/Å^3, respectively.

The selected bond lengths and bond angles are given in Table-1. Fig. 1 shows the molecular structure of the title compound. Fig. 2 shows the packing diagram of the title compound. The title complex crystallizes in C2/c space group. It is noted that the pyrimidinyl ring and the pyridyl ring of title complex is obviously not coplanar, the dihedral angle between the two heterocyclic rings is *ca*. 16.3°.

In the solid state, the adjacent molecules are stacked through C-H…O and C-H···N hydrogen bonding interactions to form 3D supramolecular framework.

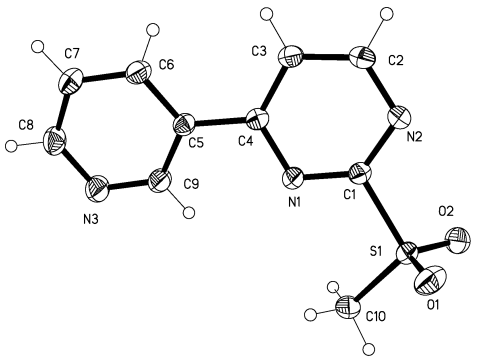

Fig. 1. Molecular structure of the present compound

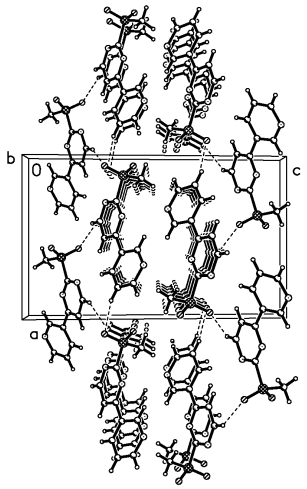

Fig. 2. View of a 3D supramolecular framework of present compound showing the intermolecular hydrogen bonding

TABLE-1
SELECTED BOND DISTANCES (Å) AND ANGLES (º)

S1-O1	1.4338(15)	S1-O2	1.4293 (17)	S1 -C1	1.8019(18)
S1-C10	1.745 (2)	N1-C1	1.319 (2)	N1-C4	1.347 (2)
N2-C1	1.324 (2)	N2-C2	1.337 (3)	N3-C8	1.337 (3)
N3-C9	1.340 (3)	C2-C3	1.372 (3)	C3-C4	1.385 (3)
C4-C5	1.479 (3)	O1-S1-O2	117.61 (9)	O1-S1 -C1	106.71 (8)
O1-S1-C10	109.00 (10)	O2-S1-C1	108.78 (8)	O2-S1 -C10	109.56 (10)
C1-S1-C10	104.34 (9)	C1-N1-C4	116.07 (15)	C1-N2-C2	113.24 (17)
C8-N3 -C9	116.38 (19)	S1-C1-N1	116.22 (13)	S1-C1-N2	114.07 (13)

TABLE-2
HYDROGEN BOND DISTANCES (Å) AND ANGLES (º)

Type (D-H…A)	d(D-H)	d(H…A)	∠(DHA)	d(D…A)	A
C2-H2…O1	0.9300	2.4800	141.00	3.255 (3)	-x, 1/2 + y, 1/2-z
C7-H7…O1	0.9300	2.5200	150.00	3.352 (2)	x, 1 + y, z
C9-H9…N1	0.9300	2.4800	101.00	2.815 (3)	–

Conclusion

Crystal structure of a new heterocyclic sulfone complex has been synthesized and characterized by IR, elemental analysis and X-ray diffraction analysis.

Supplementary material

Crystallographic data for the structure reported in this communication have been deposited with the Cambridge Crystallographic Data Center as supplementary publication No. CCDC 705138.

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