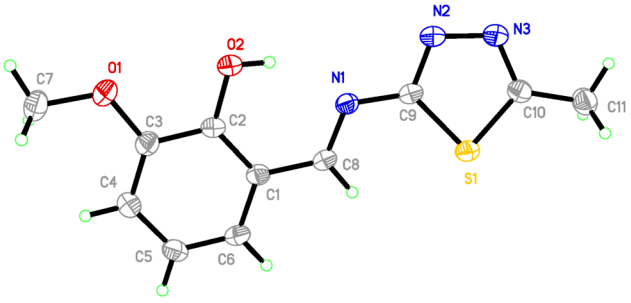
Qiuping Huang, Zhenfang Zeng and Qiuchan Huang*

The crystal structure of (*E*)-2-methoxy-6-(((5-methyl-1,3,4-thiadiazol-2-yl)imino)methyl) phenol, $C_{11}H_{11}N_3O_2S$



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Abstract

$C_{11}H_{11}N_3O_2S$, orthorhombic, $Pna2_1$ (no. 33), a = 22.6572(9) Å, b = 10.0205(4) Å, c = 4.9023(2) Å, V = 1113.01(8) Å^3, Z = 5, $R_{gt}(F)$ = 0.0341, w$R_{ref}(F^2)$ = 0.0857, T = 297 K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

1 Experimental details

The structure was solved with Shelxt program and refined with the Shelxt refinement package under Olex2 suite [1, 2]. All hydrogen atoms were placed in calculated positions and refined as riding atoms, with U_{iso}(H) = 1.5U_{eq}(C) for methyl H and hydroxyl H atoms. U_{iso}(H) was set to 1.2U_{eq}(C) for all other H atoms.

***Corresponding author: Qiuchan Huang**, College of Chemical and Biological Engineering, Guangxi Minzu Normal University, Chongzuo, Guangxi 532200, China, E-mail: huangqiuchan2021@163.com
Qiuping Huang and Zhenfang Zeng, College of Chemical and Biological Engineering, Guangxi Minzu Normal University, Chongzuo, Guangxi 532200, China, E-mail: huangqiuping@gxnun.edu.cn (Q. Huang), zengzhenfang@gxnun.edu.cn (Z. Zeng). https://orcid.org/0000-0002-2449-8195 (Q. Huang)

Table 1: Data collection and handling.

Crystal:	Colorless needle
Size:	0.21 × 0.18 × 0.14 mm
Wavelength:	MoKα radiation (0.71073 Å)
μ:	0.28 mm^{-1}
Diffractometer, scan mode:	ROD, Synergy Custom DW system
θ_{max}, completeness:	26.3°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	5529, 1998, 0.026
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 1768
$N(param)_{refined}$:	157
Programs:	Olex2 [1], Shelx [2]

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

Atom	x	y	z	U_{iso}*/U_{eq}
C1	0.12996 (13)	0.6004 (3)	0.7664 (7)	0.0379 (7)
C2	0.09815 (13)	0.7174 (3)	0.7188 (6)	0.0378 (7)
C3	0.05407 (13)	0.7189 (3)	0.5180 (7)	0.0429 (8)
C4	0.04305 (13)	0.6061 (3)	0.3677 (7)	0.0455 (8)
H4	0.0135	0.6068	0.2361	0.055*
C5	0.07605 (14)	0.4900 (3)	0.4114 (8)	0.0469 (7)
H5	0.0689	0.4145	0.3064	0.056*
C6	0.11856 (14)	0.4873 (3)	0.6071 (8)	0.0440 (8)
H6	0.1402	0.4098	0.6353	0.053*
C7	−0.01873 (16)	0.8466 (4)	0.2824 (8)	0.0627 (11)
H7A	−0.0006	0.8310	0.1084	0.094*
H7B	−0.0361	0.9340	0.2844	0.094*
H7C	−0.0488	0.7809	0.3134	0.094*
C8	0.17582 (13)	0.5952 (3)	0.9721 (7)	0.0393 (7)
H8	0.1951	0.5147	1.0024	0.047*
C9	0.23554 (13)	0.6852 (3)	1.3064 (7)	0.0402 (8)
C10	0.31278 (13)	0.6336 (3)	1.6205 (7)	0.0435 (7)
C11	0.35918 (15)	0.5776 (4)	1.7974 (8)	0.0584 (10)
H11A	0.3925	0.5521	1.6881	0.088*
H11B	0.3441	0.5006	1.8910	0.088*
H11C	0.3712	0.6434	1.9283	0.088*
N1	0.19088 (11)	0.6974 (2)	1.1146 (6)	0.0386 (6)
N2	0.25635 (13)	0.7905 (2)	1.4268 (7)	0.0537 (7)
N3	0.30113 (14)	0.7610 (3)	1.6066 (7)	0.0569 (8)
O1	0.02461 (11)	0.8376 (2)	0.4912 (5)	0.0587 (7)
O2	0.10777 (10)	0.8308 (2)	0.8615 (5)	0.0517 (7)
H2	0.1340	0.8180	0.9738	0.078*
S1	0.26903 (4)	0.53806 (7)	1.4100 (2)	0.0481 (3)

2 Source of material

All chemicals were commercially sourced and were not further purified. Weigh 0.1152 g (1.0 mmol) of 2-amino-5-methyl-1,3,4-thiadiazole and 0.1522 g (1.0 mmol) of orthovanillin into a three necked flask containing 8.0 mL of anhydrous ethanol stirring at room temperature. In addition, add 1 mL of acetic acid as the catalyst and reflux at 80 °C for 2 h, and the solution turns yellow. The solution was filtered and then put into a Teflon-lined autoclave, and heated for three days at 80 °C, cooled to room temperature slowly and colorless needle crystals formed.

3 Comment

Schiff bases are a class of compounds formed by double bonding carbon atoms with nitrogen atoms [3], and their excellent coordination structure is due to the presence of C=N in the structure. The different structures and functional groups of reactants give Schiff bases different properties. Therefore, Schiff base compounds have important applications in multiple fields [4]. Schiff bases can be divided into salicylaldehydes, amino acids, acetamides, etc. according to the types of reactants [5]. Among them, salicylaldehydes Schiff bases are the most and earliest studied. They are a type of Schiff base obtained by condensation reaction between salicylaldehydes and amines, which is easy to prepare and has good biological activity, and is an oxygen-containing ligand. It have various coordination modes with different metal ions, providing conditions for the formation of highly biologically active Schiff base compounds [6].

From the data of bond length and bond angle of the title Schiff base compound, it can be seen that the C=N double bond length N1–C8 in Schiff base is 1.286(4) Å, which is longer than the average bond length of the usual C=N double bond [7, 8]. The bond length between the phenolic hydroxyl oxygen O1 in Schiff base and the carbon atom C3 on the benzene ring is 1.370(4) Å, which is between the C–O single bond and the C=O double bond [9–11]. And the bond length of C1–C8 is equal to 1.449(4) Å, which is 0.091 Å shorter than the typical C–C single bond (1.54 Å). Schiff base has intramolecular hydrogen bonds, where the phenolic hydroxyl O atom on orthovanillin forms an O2–H1···N1 hydrogen bond with the N atom in the Schiff base C=N double bond, forming a stable six-membered ring with C2, C1, and C8.

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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