Li-Hua Wang and Xi-Shi Tai*

The crystal structure of (2-hydroxy-5-methyl-phenyl)-(1H-pyrazol-4-yl)-methanone hemihydrate, $C_{11}H_{10.5}N_2O_{2.5}$

Figure 1: The molecular structure of (2-hydroxy-5-methyl-phenyl)-(1H-pyrazol-4-yl)-methanone hemihydrate.

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Abstract

$C_{11}H_{10.5}N_2O_{2.5}$, monoclinic, $P2_1/c$ (no. 14), $a = 4.9681(4)$ Å, $b = 32.634(4)$ Å, $c = 6.2373(10)$ Å, $\beta = 96.244(10)^\circ$, $V = 1005.3(2)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0595$, $wR_{ref}(F^2) = 0.1112$, $T = 293(2)$ 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.

Source of material

The title compound was synthesized as following: 0.0941 g 3-formyl-6-methylchromone (0.5 mmol) and 0.0750 g

*Corresponding author: Xi-Shi Tai, College of Chemistry and Chemical Engineering, Weifang University, Weifang, Shandong 261061, P. R. China, E-mail: taixs@wfu.edu.cn

Li-Hua Wang, College of Chemistry and Chemical Engineering, Weifang University, Weifang, Shandong 261061, P. R. China

Table 1: Data collection and handling.

Crystal:	Colourless prism
Size:	$0.14 \times 0.11 \times 0.09$ mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.10 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
$\theta_{\max}$, completeness:	26.5° , 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	6368, 2068, 0.057
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1262
$N(\text{param})_{\text{refined}}$:	164
Programs:	Bruker [1], Olex2 [2], SHELX [3], Diamond [4]

phenylacetic acid hydrazide (0.5 mmol) were dissolved in 15 mL of a ethanol (95%)-water solution (v:v = 2:1) with stirring. Then 0.0549 g zinc acetate dihydrate (0.25 mmol) was added to the above solution. The mixture was stirred and refluxed for 1 h, and continually stirred for 8 h at 80°C , cooled to room temperature and filtered. The colourless crystals of (2-hydroxy-5-methyl-phenyl)-(1H-pyrazol-4-yl)-methanone hemihydrate were obtained from the filtrate after five weeks.

Experimental details

Most hydrogen atoms were positioned geometrically ($C-H = 0.93-0.96$ Å, $N-H = 0.88$ Å; see Table 2). Their U_{iso} values were set to $1.2U_{eq}$ or $1.5U_{eq}$ of the parent atoms. The water molecule is disordered over an inversion center.

Comment

Nitrogen heterocyclic compounds show excellent properties in many ways such as antiviral agent [5], exceptional impact and friction sensitivities [6], precursor for the preparation of divalent energetic materials [7]. In this paper, a new nitrogen heterocyclic compound is described. The title compound is made up of the organic target molecule and a disordered lattice water molecule (Figure 1). The bond distances of $C8-O2$ and $C2-O1$ are $1.246(3)$ and $1.353(3)$ Å, respectively, showing that the $C8-O2$ bond is double bond. The bond

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	<i>U</i> _{iso} ^a / <i>U</i> _{eq}
O1	1.2216 (3)	0.36061 (6)	0.9199 (3)	0.0360 (5)
H1	1.127239	0.379146	0.959739	0.054*
O2	0.8191 (3)	0.41007 (6)	0.8936 (3)	0.0357 (5)
H2	0.580 (5)	0.4209 (8)	0.225 (5)	0.044 (9)*
N1	0.2968 (4)	0.45836 (7)	0.3128 (4)	0.0358 (6)
N2	0.2171 (4)	0.46985 (7)	0.5065 (4)	0.0324 (6)
C1	0.8802 (4)	0.36404 (7)	0.6128 (5)	0.0252 (7)
C2	1.1108 (5)	0.34634 (8)	0.7264 (5)	0.0262 (6)
C3	1.2334 (5)	0.31289 (8)	0.6394 (5)	0.0325 (7)
H3	1.390893	0.301855	0.710989	0.039*
C4	1.1240 (5)	0.29620 (8)	0.4497 (5)	0.0321 (7)
H4	1.209410	0.273843	0.394069	0.038*
C5	0.8882 (5)	0.31146 (8)	0.3361 (5)	0.0285 (7)
H5	0.341 (5)	0.4545 (7)	0.804 (5)	0.026 (8)*
C6	0.7726 (5)	0.34535 (8)	0.4200 (5)	0.0289 (7)
H6	0.616612	0.356315	0.345783	0.035*
C7	0.7620 (6)	0.29137 (9)	0.1324 (5)	0.0415 (8)
H7A	0.590366	0.303959	0.087880	0.062*
H7B	0.735855	0.262724	0.158396	0.062*
H7C	0.879257	0.294605	0.020967	0.062*
H2N ^a	0.095 (9)	0.4889 (15)	0.518 (9)	0.031 (16)*
C8	0.7530 (5)	0.39999 (8)	0.7025 (5)	0.0268 (7)
C9	0.5496 (5)	0.42466 (8)	0.5741 (5)	0.0263 (7)
C10	0.4969 (5)	0.43106 (8)	0.3529 (6)	0.0328 (7)
C11	0.3684 (5)	0.45067 (8)	0.6621 (6)	0.0308 (7)
O3 ^a	−0.0781 (7)	0.49057 (13)	0.0006 (8)	0.0429 (12)
H3A	0.053 (17)	0.480 (3)	0.106 (14)	0.40 (6)*

^aOccupancy: 0.5.

distances of C10–N1 and C11–N2 are 1.338(3) and 1.319(4) Å, respectively. The bond distances of C–O and C–N are consistent with literature [8–10]. The dihedral angle between the ring 1 (C1–C2–C3–C4–C5–C6) and ring 2 (C9–C10–N1–N2–C11) is 33.25°, indicating that the whole molecule is not coplanar. The title compound forms dimers by bridging hydrogen bonds between water molecule and (2-hydroxy-5-methyl-phenyl)-(1*H*-pyrazol-4-yl)-methanone. Meanwhile, the title compound form a 3D network by weak π – π interactions, a O–H···O hydrogen bond (intramolecular) and O–H···N hydrogen bonds (intermolecular).

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

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