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Crystal structure of 4,4'-(hydrazonomethylene) diphenol dihydrate, $C_{13}H_{16}N_2O_4$

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.25 × 0.22 × 0.20 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.10 mm ⁻¹
Diffraction, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	26.4°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	8255, 2699, 0.025
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2159
$N(\text{param})_{\text{refined}}$:	192
Programs:	Bruker [1], SHELX [2]

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.70338(12)	0.60136(16)	0.51437(11)	0.0303(3)
C2	0.60446(12)	0.69725(16)	0.45168(11)	0.0305(3)
C3	0.57264(13)	0.74779(19)	0.33567(12)	0.0403(4)
H3	0.6153	0.7214	0.2959	0.048*
C4	0.47923(13)	0.83595(19)	0.27891(13)	0.0405(4)
H4	0.4595	0.8682	0.2015	0.049*
C5	0.41472(12)	0.87661(16)	0.33652(12)	0.0309(3)
C6	0.44638(12)	0.83136(18)	0.45264(12)	0.0351(3)
H6	0.4047	0.8606	0.4928	0.042*
C7	0.53972(12)	0.74292(18)	0.50870(12)	0.0347(3)
H7	0.5600	0.7130	0.5867	0.042*
C8	0.75334(12)	0.59600(16)	0.64717(11)	0.0294(3)
C9	0.82007(13)	0.71342(17)	0.71250(12)	0.0353(3)
H9	0.8320	0.7964	0.6734	0.042*
C10	0.86951(13)	0.70967(18)	0.83523(12)	0.0370(4)
H10	0.9152	0.7889	0.8780	0.044*
C11	0.85087(12)	0.58826(17)	0.89389(12)	0.0330(3)
C12	0.78317(15)	0.47190(18)	0.83024(13)	0.0466(4)
H12	0.7695	0.3906	0.8695	0.056*
C13	0.73523(15)	0.47574(18)	0.70737(13)	0.0459(4)
H13	0.6901	0.3959	0.6648	0.055*
N1	0.74422(10)	0.52826(14)	0.45293(10)	0.0333(3)
N2	0.84453(11)	0.44787(16)	0.51673(12)	0.0403(3)
H2A	0.8399(14)	0.388(2)	0.5705(15)	0.048*
H2B	0.8603(14)	0.3901(19)	0.4644(15)	0.048*
O1	0.32020(8)	0.96100(13)	0.28416(9)	0.0408(3)
H1	0.3091	0.9809	0.2158	0.061*
O2	0.89659(10)	0.57959(13)	1.01517(8)	0.0466(3)
H2	0.9360	0.6539	1.0437	0.070*
O3	0.98293(10)	0.17271(15)	0.89021(9)	0.0481(3)
H3A	0.9483(14)	0.169(2)	0.8149(8)	0.072*
H3B	1.0296(13)	0.1002(18)	0.9099(15)	0.072*
O4	0.84997(10)	0.14319(14)	0.64954(10)	0.0486(3)
H4A	0.7974(12)	0.102(2)	0.6605(17)	0.073*
H4B	0.8688(15)	0.0780(18)	0.6112(16)	0.073*

<https://doi.org/10.1515/ncrs-2019-0276>

Received April 16, 2019; accepted June 20, 2019; available online August 23, 2019

Abstract

$C_{13}H_{16}N_2O_4$, monoclinic, $P2_1/c$ (no. 14), $a = 13.312(2)$ Å, $b = 8.8222(13)$ Å, $c = 12.4784(18)$ Å, $\beta = 115.350(2)^\circ$, $V = 1324.4(3)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0372$, $wR_{\text{ref}}(F^2) = 0.0739$, $T = 293$ K.

CCDC no.: 1935384

The asymmetric unit of 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

All of starting materials are commercially available. The title compound was synthesized via bis(4-hydroxyphenyl)methanone with excess hydrazine hydrate in ethanol solution containing a few drops glacial acetic acid

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under refluxing. The solvent was removed to give the white product. Colourless crystals suitable for single X-ray diffraction study were obtained by recrystallization from anhydrous ethanol.

Experimental details

Hydrogen atoms were placed in calculated positions and were included in the refinement using the riding model approximation, with $U_{\text{iso}} = 1.2U_{\text{eq}}(\text{C})$. Nitrogen-bound and oxygen-bound H atoms were refined subject to distance restraints with $U_{\text{iso}} = 1.2U_{\text{eq}}(\text{N})$ and $U_{\text{iso}} = 1.5U_{\text{eq}}(\text{O})$, respectively.

Comment

Owing to their multiple coordination chemistry properties and biological activities, pharmaceutical properties, Schiff bases have received considerable attention [3–10]. In order to search for new Schiff bases with higher biological activities, the title compound was synthesized and its crystal structure is reported here.

The asymmetric unit of the title compound consists of one formula unit (*cf.* the figure). In the crystal structure of title compound, the short distance $d(\text{N1}–\text{C1}) = 1.2869(18)$ Å has a value of a typical C=N double bond. The C=N bond length is similar to the previous reported structure, (bis(4-methoxyphenyl)methylene)hydrazine of 1.2912(16) Å [11]. The two phenyl rings are almost vertical to each other with the dihedral angle of 89.19°. While in (bis(4-methoxyphenyl)methylene)hydrazine, this dihedral angle is 80.57°. The molecules are packed in the crystal structure via abundant classical O–H···O, O–H···N and N–H···O hydrogen bonds.

Acknowledgements: This work was financially supported by Nanyang Institute of Technology.

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