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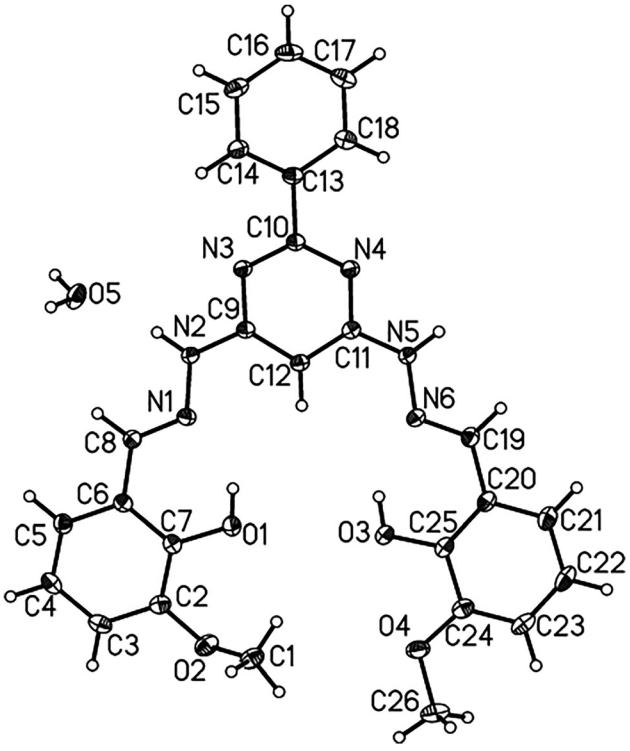
Crystal structure of 6,6′-((1*E*,1′*E*)-((2-phenylpyrimidine-4,6-diyl)bis(hydrazin-2-yl-1-ylidene))bis(methaneylylidene)) bis(2-methoxyphenol)monohydrate, $C_{26}H_{26}N_6O_5$



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Abstract

$C_{26}H_{26}N_6O_5$, monoclinic, $P2_1/c$ (no. 14), a = 10.9393(1) Å, b = 16.8724(2) Å, c = 13.5836(2) Å, β = 102.843(1)°, V = 2444.43(5) Å^3, Z = 4, $R_{gt}(F)$ = 0.0318, $wR_{ref}(F^2)$ = 0.0827, T = 173 K.

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Table 1: Data collection and handling.

Crystal:	Colourless octahedral
Size:	0.30 × 0.30 × 0.30 mm
Wavelength:	Cu *Kα* radiation (1.54184 Å)
μ:	0.80 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
θ_{max}, completeness:	65.0°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	15,390, 4107, 0.019
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 3837
$N(param)_{refined}$:	342
Programs:	CrysAlis^{PRO},[1] Olex2,[2] SHELX[3,4]

CCDC no.: 2335017

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 Source of materials

All reagents and starting materials were commercially available and used as received.

The title compound was synthesized by a two-step reaction. Firstly, the intermediate 4,6-dihydrazineyl-2-phenylpyrimidine was synthesized by nucleophilic substitution reaction based on the previous work of our research group.[5] Secondly, the title compound was synthesized by condensation reaction of the above intermediate and 2-hydroxy-3-methoxybenzaldehyde. 4,6-dihydrazineyl-2-phenylpyrimidine (2 mmol, 0.432 g) and 2-hydroxy-3-methoxybenzaldehyde (4.2 mmol, 0.638 g) were dissolved in ethanol. The mixture was stirred at room temperature for 2 h, and then refluxed at 78 °C. White solids were gradually precipitated during the reaction, and the reaction process was monitored by thin-layer chromatography. Finally, the precipitated solid was filtered, washed with ethanol, and then purified by column chromatography (petroleum ether: ethyl acetate = 1:1 v/v) to afford the title compound as white powder. Yield: ca. 63 %. Crystallization

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O1	0.53123 (9)	0.37172 (5)	0.40806 (6)	0.0339 (2)
H1	0.468743	0.401947	0.399565	0.051*
O2	0.72219 (9)	0.26102 (5)	0.46417 (7)	0.0366 (2)
O3	0.62598 (9)	0.48751 (6)	0.21450 (7)	0.0384 (2)
H3	0.554099	0.506214	0.211595	0.058*
O4	0.84353 (8)	0.43362 (6)	0.19820 (7)	0.0391 (2)
N1	0.33808 (9)	0.44322 (6)	0.45587 (7)	0.0264 (2)
N2	0.23314 (9)	0.48953 (6)	0.43701 (8)	0.0290 (2)
H2	0.182657	0.489377	0.479214	0.035*
N3	0.09995 (9)	0.57879 (6)	0.34376 (7)	0.0244 (2)
N4	0.13818 (9)	0.63718 (6)	0.19358 (7)	0.0258 (2)
N5	0.30995 (9)	0.60880 (6)	0.13371 (7)	0.0277 (2)
H5	0.277539	0.640256	0.082939	0.033*
N6	0.42555 (9)	0.57638 (6)	0.13882 (7)	0.0267 (2)
C1	0.79157 (13)	0.32184 (8)	0.42546 (11)	0.0368 (3)
H1A	0.733011	0.357195	0.380895	0.055*
H1B	0.847834	0.297194	0.387402	0.055*
H1C	0.840992	0.352419	0.481811	0.055*
C2	0.64772 (11)	0.28364 (7)	0.52923 (9)	0.0285 (3)
C3	0.66542 (12)	0.24466 (7)	0.62094 (10)	0.0331 (3)
H3A	0.733173	0.208539	0.640045	0.040*
C4	0.58495 (12)	0.25786 (8)	0.68541 (10)	0.0337 (3)
H4	0.596161	0.229895	0.747481	0.040*
C5	0.48917 (12)	0.31165 (7)	0.65859 (9)	0.0288 (3)
H5A	0.435334	0.321472	0.703317	0.035*
C6	0.46935 (11)	0.35232 (7)	0.56655 (9)	0.0251 (3)
C7	0.54908 (11)	0.33714 (7)	0.50038 (9)	0.0257 (3)
C8	0.36396 (11)	0.40640 (7)	0.54103 (9)	0.0267 (3)
H8	0.312876	0.414847	0.588211	0.032*
C9	0.20646 (10)	0.53601 (7)	0.35287 (9)	0.0235 (3)
C10	0.07261 (10)	0.62776 (7)	0.26433 (9)	0.0236 (3)
C11	0.24392 (11)	0.59361 (7)	0.20516 (9)	0.0241 (3)
C12	0.28173 (11)	0.54002 (7)	0.28348 (9)	0.0254 (3)
H12	0.354553	0.508147	0.289121	0.030*
C13	−0.03979 (11)	0.67907 (7)	0.25598 (9)	0.0264 (3)
C14	−0.08526 (12)	0.69779 (7)	0.34110 (10)	0.0312 (3)
H14	−0.046401	0.675809	0.404751	0.037*
C15	−0.18662 (12)	0.74818 (8)	0.33353 (11)	0.0381 (3)
H15	−0.216220	0.761225	0.392170	0.046*
C16	−0.24494 (13)	0.77956 (9)	0.24138 (12)	0.0442 (4)
H16	−0.314540	0.814172	0.236430	0.053*
C17	−0.20166 (13)	0.76044 (9)	0.15622 (12)	0.0436 (4)
H17	−0.242543	0.781323	0.092440	0.052*
C18	−0.09901 (12)	0.71101 (8)	0.16336 (10)	0.0344 (3)
H18	−0.068933	0.698877	0.104662	0.041*
C19	0.48262 (12)	0.59882 (7)	0.07041 (9)	0.0284 (3)
H19	0.442741	0.636161	0.021320	0.034*
C20	0.60527 (12)	0.56933 (7)	0.06543 (9)	0.0283 (3)
C21	0.65858 (13)	0.59466 (8)	−0.01408 (10)	0.0358 (3)
H21	0.614971	0.631659	−0.061979	0.043*
C22	0.77297 (14)	0.56649 (9)	−0.02320 (11)	0.0421 (4)
H22	0.808035	0.583926	−0.077459	0.051*
C23	0.83805 (13)	0.51251 (8)	0.04653 (11)	0.0389 (3)
H23	0.917388	0.493269	0.039768	0.047*
C24	0.78782 (12)	0.48684 (7)	0.12543 (10)	0.0313 (3)
C25	0.67080 (12)	0.51503 (7)	0.13550 (9)	0.0284 (3)
C26	0.96218 (14)	0.40182 (10)	0.19022 (13)	0.0508 (4)
H26A	0.953087	0.374199	0.125534	0.076*
H26B	0.991774	0.364470	0.245646	0.076*
H26C	1.022951	0.445013	0.194023	0.076*
O5	0.11035 (9)	0.49871 (6)	0.59788 (7)	0.0413 (3)
H5B	0.046578	0.474642	0.609192	0.062*
H5C	0.146930	0.517352	0.654939	0.062*

from acetonitrile at room temperature gave the title compound as white block crystals.

2 Experimental details

The structure was solved by Direct Methods and refined by using Olex2 and SHELXTL software. The H atoms were added using riding models. Their U_{iso} values were set to 1.2 U_{eq} of the parent atoms.

3 Comment

Pyrimidine derivatives have attracted considerable attention due to their wide range of pharmacological properties including anticancer,[6,7] anti-inflammatory[8] and antibacterial[9] activities. It was found that incorporating hydrazone functional group into the pyrimidine backbone helps to enhance the biological activity.[10] Therefore, there is growing interest in synthesizing and exploring the crystal structure of compounds containing pyrimidine and hydrazone moieties. As a continuation of our research,[5,11] a new pyrimidine dihydrazone derivative was synthesized and characterized by X-ray diffraction.

The title compound crystallizes in the monoclinic space group $P2_1/c$ with one molecule in the asymmetric unit (see the figure). The molecule structure consists of a phenylpyrimidine group and two 2-hydroxy-3-methoxy substituted phenyl groups linked by hydrazone groups. The crystal lattice also contains one water molecule. Bond lengths and angles are all in the normal range. The bond lengths of C8–N1 (1.2879 Å) and C19–N6 (1.2864 Å) indicate the existence of C=N double bond, which is typical for similar hydrazone compounds.[12–14] The molecule adopts *E* configuration about the C=N double bond. In addition, two hydrazone arms are slightly twisted out of the plane of phenylpyrimidine unit, which was also observed for the pyrimidine dihydrazone compound investigated earlier.[12] The dihedral angle between pyrimidine and substituted phenyl rings are 8.43° and

5.15°, respectively. Intramolecular O–H···N hydrogen bonds are found between phenolic oxygen atoms and imine nitrogen atoms [O1···N1: 2.6366 (14) Å, O3···N6: 2.6665 (13) Å].

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

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