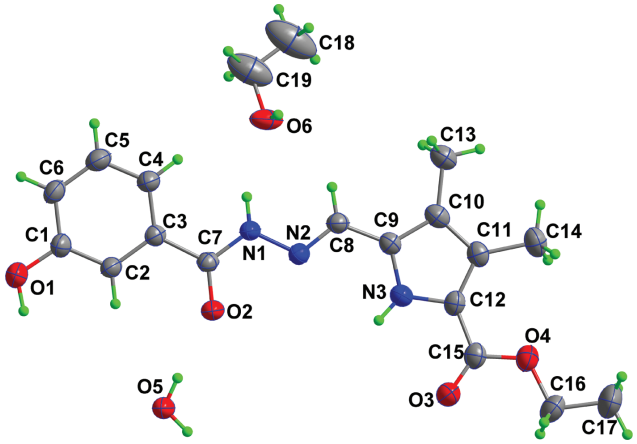
Liangjun Lv, Zhengli Hao, Long Lin* and Yuan Wang

Crystal structure of ethyl (*E*)-5-((2-(3-hydroxybenzoyl)hydrazono)methyl)-3,4-dimethyl-1*H*-pyrrole-2-carboxylate – water – ethanol (1/1/1), $C_{19}H_{27}N_3O_6$



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Abstract

$C_{19}H_{27}N_3O_6$, monoclinic, $P2_1/c$ (no. 14), $a = 8.132(7)$ Å, $b = 8.762(7)$ Å, $c = 30.22(3)$ Å, $\beta = 97.19(2)°$, $V = 2136(3)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0579$, $wR_{ref}(F^2) = 0.1892$, $T = 296(2)$ K.

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The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

The title compound was prepared according to the literature method [1]. Crystals of the title crystals were obtained by the recrystallization of the compound from ethanol solution.

***Corresponding author: Long Lin,** School of Materials Science and Engineering, Henan Polytechnic University, Jiaozuo, 454000, P. R. China, e-mail: linlong@hpu.edu.cn
Liangjun Lv and Zhengli Hao: Department of Foudamental Science, Yellow River Conservancy Technical Institute, Kaifeng, 475001, P.R. China
Yuan Wang: College of Chemistry and Chemical Engineering, Henan Polytechnic University, Jiaozuo, 454000, P.R. China

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.19 × 0.16 × 0.15 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.09 mm^{-1}
Diffractometer, scan mode:	Bruker SMART, φ and ω-scans
$2\theta_{max}$, completeness:	25°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	10899, 3767, 0.065
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1771
$N(param)_{refined}$:	263
Programs:	Bruker programs, SHELX [2, 3]

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

Atom	x	y	z	U_{iso}*/U_{eq}
C1	0.5981(5)	0.8544(4)	−0.18148(11)	0.0635(10)
C2	0.5596(4)	0.8496(4)	−0.13786(11)	0.0585(9)
H2	0.5822	0.9338	−0.1193	0.070*
C3	0.4878(4)	0.7207(4)	−0.12173(10)	0.0512(8)
C4	0.4535(5)	0.5971(4)	−0.14994(11)	0.0649(10)
H4	0.4057	0.5097	−0.1396	0.078*
C5	0.4902(5)	0.6031(4)	−0.19332(13)	0.0777(11)
H5	0.4658	0.5197	−0.2120	0.093*
C6	0.5624(5)	0.7310(4)	−0.20940(12)	0.0708(11)
H6	0.5866	0.7338	−0.2387	0.085*
C7	0.4481(4)	0.7248(4)	−0.07476(11)	0.0514(8)
C8	0.2483(4)	0.4881(4)	−0.00558(11)	0.0573(9)
H8	0.2235	0.4081	−0.0255	0.069*
C9	0.1968(4)	0.4803(4)	0.03805(11)	0.0526(8)
C10	0.1143(4)	0.3620(4)	0.05735(12)	0.0593(9)
C11	0.0916(4)	0.4093(4)	0.10099(12)	0.0590(9)
C12	0.1620(4)	0.5535(4)	0.10701(11)	0.0576(9)
C13	0.0613(5)	0.2121(4)	0.03588(13)	0.0762(11)
H13A	0.0073	0.1517	0.0563	0.114*
H13B	0.1568	0.1585	0.0283	0.114*
H13C	−0.0142	0.2304	0.0094	0.114*
C14	0.0115(5)	0.3159(4)	0.13413(13)	0.0787(12)
H14A	−0.0262	0.2208	0.1207	0.118*
H14B	−0.0811	0.3709	0.1430	0.118*
H14C	0.0907	0.2963	0.1598	0.118*
C15	0.1899(5)	0.6550(5)	0.14460(12)	0.0654(10)
C16	0.1547(6)	0.6922(5)	0.22089(13)	0.0976(14)
H16A	0.1026	0.7916	0.2166	0.117*
H16B	0.2728	0.7071	0.2290	0.117*
C17	0.0846(9)	0.6081(6)	0.25646(17)	0.170(3)
H17A	0.1035	0.6649	0.2838	0.255*
H17B	0.1371	0.5101	0.2605	0.255*
H17C	−0.0324	0.5947	0.2482	0.255*

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C18	0.1626(9)	0.0942(7)	−0.1169(3)	0.197(4)
H18A	0.0985	0.0505	−0.1427	0.296*
H18B	0.0895	0.1324	−0.0969	0.296*
H18C	0.2336	0.0176	−0.1021	0.296*
C19	0.2548(7)	0.2089(7)	−0.1297(2)	0.152(3)
H19A	0.1848	0.2770	−0.1491	0.182*
H19B	0.3385	0.1678	−0.1466	0.182*
O1	0.6687(4)	0.9779(4)	−0.19876(9)	0.0959(10)
O2	0.4790(3)	0.8375(3)	−0.05045(7)	0.0692(7)
O3	0.2662(4)	0.7751(3)	0.14453(8)	0.0854(9)
O4	0.1246(3)	0.6038(3)	0.18027(8)	0.0776(8)
O5	0.5850(4)	1.1381(3)	−0.04310(7)	0.0772(8)
H5A	0.5731	1.0418	−0.0446	0.116*
H5B	0.5791	1.1677	−0.0166	0.116*
O6	0.3347(4)	0.2944(3)	−0.09225(10)	0.0934(9)
H6A	0.4099	0.2433	−0.0790	0.140*
N1	0.3728(3)	0.6013(3)	−0.06004(8)	0.0555(7)
H1A	0.3528	0.5225	−0.0768	0.067*
N2	0.3281(3)	0.6040(3)	−0.01734(8)	0.0537(7)
N3	0.2244(3)	0.5938(3)	0.06838(9)	0.0580(8)
H3	0.2737	0.6784	0.0642	0.070*
H1	0.696(6)	1.055(6)	−0.1778(16)	0.136(19)*

Experimental details

The hydrogen atoms were placed at calculated positions and refined as riding atoms with isotropic displacement parameters.

Discussion

Acylhydrazones are an important class of ligands in coordination chemistry and have been found extensive application in different fields [4]. Our previous work shows that acylhydrazone ligands bearing pyrrole units and their complexes exhibit considerable antibacterial and antitumor activity [1, 5, 6]. In fact, the structure of DMF solvate of ethyl (*E*)-5-((2-(4-hydroxybenzoyl)hydrazono)methyl)-3,4-dimethyl-1*H*-pyrrole-2-carboxylate has been reported [1]. As its isomer, the title compound was synthesized and characterized by X-ray diffraction.

In the title crystal structure, the acylhydrazone molecule is in a ketone form and adopts an *E* configuration at the C = N double bond, in which the bond length (1.237(4) Å) is comparable to that of the 4-hydroxybenzohydrazone isomer (1.233(3) Å) [1]. The dihedral angle between the pyrrole ring (N3/C9—C12, r.m.s. deviation 0.0019 Å) and the mean plane of the hydroxyphenyl moiety (C1—C6, r.m.s. deviation 0.0031 Å) is 4.1°. The torsion angles of C9—C8—N2—N1 and C7—N1—N2—C8 are 179.9(3)° and 176.1(3)°, respectively. In the solid state, two arylhydrazone molecules are linked by neighboring water molecules through intermolecular O—H···O and N—H···O hydrogen bonds, forming a centrosymmetry dimer. Intermolecular N—H···O hydrogen bonds between the ethanol molecule and the dimeric units are also present. A further ladder-like structure is constructed *via* pairs of O—H···O hydrogen bonds between the ethanol and water molecules.

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