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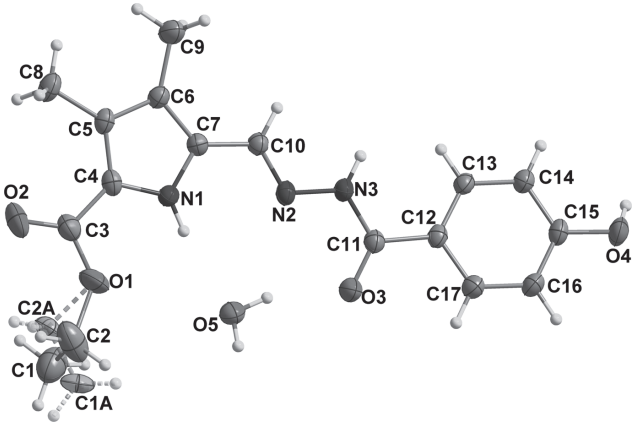
N′-(5-ethoxycarbonyl-3,4-dimethyl-pyrrol-2-yl-methylidene)-4-hydroxybenzohydrazide monohydrate, $C_{17}H_{21}N_3O_5$



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Abstract

$C_{17}H_{21}N_3O_5$, monoclinic, $P2_1/n$ (no. 14), $a = 9.2278(16)$ Å, $b = 15.093(3)$ Å, $c = 12.698(2)$ Å, $\beta = 105.195(12)°$, $V = 1706.7(5)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0553$, $wR_{ref}(F^2) = 0.1662$, $T = 296$ K.

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

Source of materials

N′-(5-ethoxycarbonyl-3,4-dimethyl-pyrrol-2-yl-methylidene)-4-hydroxybenzohydrazide was prepared according to the

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Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.21 × 0.19 × 0.15 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	1.0 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$2\theta_{max}$, completeness:	50°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	12944, 3000, 0.046
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2016
$N(param)_{refined}$:	253
Programs:	SHELXL [1], Bruker programs [2]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
N2	0.6348(2)	0.02933(14)	0.93676(16)	0.0445(5)
N1	0.8876(2)	−0.06095(13)	1.05922(16)	0.0426(5)
H1A	0.8379	−0.0924	1.0052	0.051*
N3	0.5149(2)	0.07672(14)	0.87624(16)	0.0463(6)
H3A	0.5003	0.1306	0.8929	0.056*
O3	0.4397(2)	−0.04019(14)	0.76629(16)	0.0633(6)
C12	0.2949(3)	0.09113(17)	0.72426(19)	0.0418(6)
O4	−0.0472(2)	0.23575(14)	0.51712(15)	0.0651(6)
H4A	−0.0879	0.2690	0.5517	0.098*
C10	0.7236(3)	0.06866(17)	1.0172(2)	0.0426(6)
H10A	0.7049	0.1269	1.0337	0.051*
C14	0.1387(3)	0.22183(18)	0.6892(2)	0.0472(7)
H14A	0.1123	0.2767	0.7120	0.057*
C16	0.1013(3)	0.1067(2)	0.5556(2)	0.0547(7)
H16A	0.0500	0.0840	0.4881	0.066*
C15	0.0637(3)	0.18871(19)	0.5880(2)	0.0465(6)
O5	0.6797(2)	−0.15668(13)	0.87010(16)	0.0598(6)
C11	0.4201(3)	0.03682(18)	0.7897(2)	0.0443(6)
C13	0.2526(3)	0.17317(17)	0.7559(2)	0.0442(6)
H13A	0.3025	0.1958	0.8239	0.053*
C7	0.8525(3)	0.02272(16)	1.08231(19)	0.0402(6)
C4	1.0142(3)	−0.08758(18)	1.1352(2)	0.0442(6)
O2	1.1826(3)	−0.20519(16)	1.2033(2)	0.0844(7)
C6	0.9603(3)	0.05104(17)	1.1745(2)	0.0435(6)
C17	0.2150(3)	0.05843(19)	0.6233(2)	0.0519(7)
H17A	0.2388	0.0028	0.6010	0.062*
C5	1.0615(3)	−0.01860(18)	1.2083(2)	0.0439(6)
C9	0.9659(4)	0.1397(2)	1.2290(3)	0.0688(9)

Table 2 (continued)

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
H9A	1.0513	0.1419	1.2913	0.103*
H9B	0.8758	0.1483	1.2520	0.103*
H9C	0.9743	0.1855	1.1784	0.103*
O1	1.0057(3)	−0.21960(15)	1.0479(2)	0.0994(9)
C3	1.0783(4)	−0.1743(2)	1.1351(3)	0.0621(8)
C8	1.1961(3)	−0.0185(2)	1.3039(2)	0.0631(8)
H8A	1.2039	0.0377	1.3403	0.095*
H8B	1.2847	−0.0285	1.2793	0.095*
H8C	1.1865	−0.0647	1.3536	0.095*
C1[a]	0.9520(14)	−0.3628(9)	1.0424(15)	0.110(5)
H1B[a]	0.9868	−0.3626	1.1206	0.165*
H1C[a]	0.9545	−0.4221	1.0158	0.165*
H1D[a]	0.8509	−0.3407	1.0207	0.165*
C1A[b]	0.9574(12)	−0.3590(6)	0.9697(12)	0.090(4)
H1A1[b]	0.8638	−0.3672	0.9881	0.135*
H1A2[b]	0.9984	−0.4157	0.9589	0.135*
H1A3[b]	0.9408	−0.3248	0.9038	0.135*
C2A[b]	1.0609(13)	−0.3130(5)	1.0569(11)	0.079(4)
H2A1[b]	1.1622	−0.3160	1.0483	0.095*
H2A2[b]	1.0602	−0.3381	1.1271	0.095*
C2[a]	1.050(2)	−0.3054(8)	0.9965(16)	0.118(7)
H2A[a]	1.0216	−0.3042	0.9173	0.141*
H2B[a]	1.1559	−0.3199	1.0236	0.141*
H5B	0.685(6)	−0.188(3)	0.815(3)	0.177*
H5A	0.626(5)	−0.111(2)	0.845(4)	0.177*

[a]Occupancy: 0.464(18); [b]Occupancy: 0.536(18).

literature method [3]. The title crystals were obtained by slow evaporating of a THF/H_2O (1:1, *v:v*) solution at room temperature.

Experimental details

All H atoms were situated into idealized positions with the carrier atom-H distances = 0.93 Å for aryl, 0.96 Å for the methyl and 0.86 Å for the secondary amine H atoms. The U_{iso} values were constrained to be $1.5U_{eq}$ of the carrier atom for the methyl H atoms and $1.2U_{eq}$ for the remaining H atoms. Difference Fourier maps indicate methyl H atoms disorder, which has not been modelled.

Comment

Acylhydrazones are an important class of ligands in coordination chemistry and have found extensive application in different fields [4]. Our previous work shows that acylhydrazone ligands bearing pyrrole units and their Cu(II) complexes exhibit considerable antibacterial and antitumor activity [3, 5, 6]. The structure of *N′*-(5-ethoxycarbonyl-3,4-dimethyl-pyrrol-2-yl-methylidene)-4-hydroxybenzohydrazide DMF adduct has been reported [3], while its monohydrate was characterized by X-ray diffraction in this work.

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 all the bond lengths are comparable with those observed in the DMF adduct [6]. The dihedral angle between the pyrrole (N1/C4—C7, r.m.s. deviation 0.0034 Å) and the phenyl rings (C12—C17, r.m.s. deviation 0.0070 Å) is 10.7°. The torsion angles of N3—N2—C10—C7 and C11—N3—N2—C10 are 179.3(2)° and −178.95(19)°, respectively. In the solid state, the acylhydrazone molecules are linked into a two-dimensional supermolecular network by the crystal water molecules *via* intermolecular N—H···O and O–H···O hydrogen bonds.

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