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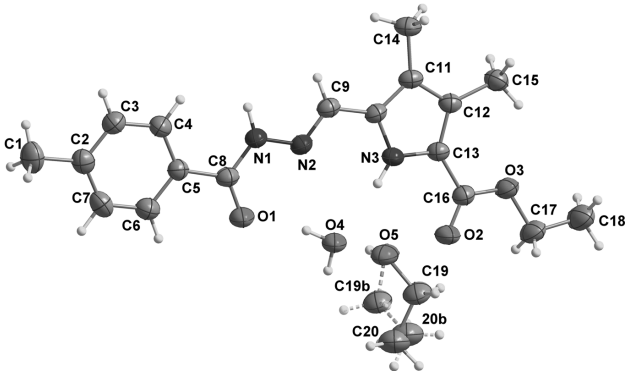
Crystal Structure of ethyl (*E*)-3,4-dimethyl-5-((2-(4-methylbenzoyl) hydrazono)methyl)-1*H*-pyrrole-2-carboxylate — water — ethanol (1/1/1), $C_{20}H_{29}N_3O_5$



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Abstract

$C_{20}H_{29}N_3O_5$, triclinic, $P\bar{1}$ (no. 2), $a = 9.1571(18)$ Å, $b = 10.505(2)$ Å, $c = 11.576(2)$ Å, $\alpha = 94.509(4)°$, $\beta = 92.570(4)°$, $\gamma = 102.312(3)°$, $V = 1082.4(4))$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0543$, $wR_{ref}(F^2) = 0.1704$, $T = 296(2)$ 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 materials

The title compound was prepared according to the literature method [3]. The crystals were obtained by the recrystallization of the title compound from ethanol solution.

Experimental details

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

The ethanol molecule is disordered (*cf.* the figure).

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Table 1: Data collection and handling.

Crystal:	Prism, colorless
Size:	0.18 × 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
θ_{max}, completeness:	25°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	5720, 3789, 0.021
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1996
$N(param)_{refined}$:	273
Programs:	Bruker programs [1], SHELX [2]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.6973(4)	−0.3007(4)	−0.1146(3)	0.1081(12)
H1A	0.8044	−0.2888	−0.1080	0.162*
H1B	0.6672	−0.2726	−0.1871	0.162*
H1C	0.6521	−0.3916	−0.1114	0.162*
C2	0.6476(3)	−0.2210(3)	−0.0160(3)	0.0759(9)
C3	0.5550(4)	−0.2782(3)	0.0644(3)	0.0799(9)
H3	0.5225	−0.3687	0.0580	0.096*
C4	0.5087(3)	−0.2052(3)	0.1545(3)	0.0731(8)
H4	0.4458	−0.2470	0.2073	0.088*
C5	0.5550(3)	−0.0704(3)	0.1668(2)	0.0611(7)
C6	0.6489(3)	−0.0125(3)	0.0867(3)	0.0788(9)
H6	0.6827	0.0780	0.0930	0.095*
C7	0.6928(4)	−0.0872(4)	−0.0021(3)	0.0864(10)
H7	0.7556	−0.0456	−0.0551	0.104*
C8	0.5087(3)	0.0163(3)	0.2594(2)	0.0635(7)
C9	0.2654(3)	−0.0104(3)	0.4852(2)	0.0635(8)
H9A	0.2278	−0.1004	0.4776	0.076*
C10	0.2160(3)	0.0708(2)	0.5739(2)	0.0573(7)
C11	0.1095(3)	0.0388(3)	0.6547(2)	0.0607(7)
C12	0.0996(3)	0.1554(3)	0.7186(2)	0.0587(7)
C13	0.2010(3)	0.2558(2)	0.6753(2)	0.0576(7)
C14	0.0272(3)	−0.0968(3)	0.6740(3)	0.0788(9)
H14A	0.0270	−0.1543	0.6052	0.118*
H14B	−0.0741	−0.0954	0.6910	0.118*
H14C	0.0760	−0.1275	0.7380	0.118*
C15	−0.0009(3)	0.1667(3)	0.8165(3)	0.0774(9)
H15A	0.0192	0.2556	0.8501	0.116*
H15B	0.0176	0.1107	0.8746	0.116*
H15C	−0.1036	0.1409	0.7872	0.116*
C16	0.2545(3)	0.3946(3)	0.7084(3)	0.0659(8)
C17	0.2332(4)	0.5827(3)	0.8263(3)	0.0974(11)
H17A	0.2193	0.6328	0.7614	0.117*
H17B	0.3390	0.6019	0.8506	0.117*

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C18	0.1455(5)	0.6178(4)	0.9236(4)	0.1216(14)
H18A	0.0412	0.5998	0.8983	0.182*
H18B	0.1788	0.7092	0.9484	0.182*
H18C	0.1594	0.5671	0.9871	0.182*
C19[a]	0.7716(8)	0.4277(6)	0.6831(6)	0.1037(18)
H19A[a]	0.8344	0.4125	0.7481	0.124*
H19B[a]	0.6936	0.4672	0.7145	0.124*
C19B[b]	0.8388(13)	0.3907(9)	0.6039(10)	0.1037(18)
H19C[b]	0.8604	0.3794	0.5229	0.124*
H19D[b]	0.9199	0.3706	0.6509	0.124*
C20[a]	0.865(4)	0.5226(11)	0.612(3)	0.133(4)
H20A[a]	0.9146	0.5987	0.6613	0.200*
H20B[a]	0.8012	0.5479	0.5536	0.200*
H20C[a]	0.9375	0.4825	0.5755	0.200*
C20B[b]	0.827(6)	0.5281(16)	0.635(4)	0.133(4)
H20D[b]	0.9019	0.5862	0.5970	0.200*
H20E[b]	0.8428	0.5480	0.7173	0.200*
H20F[b]	0.7296	0.5389	0.6097	0.200*
O1	0.5606(2)	0.1357(2)	0.26954(18)	0.0844(7)
O2	0.3604(3)	0.46048(19)	0.6665(2)	0.0937(8)
O3	0.1807(2)	0.44304(18)	0.79201(19)	0.0782(6)
O4	0.5070(2)	0.32191(18)	0.44231(18)	0.0882(7)
H4A	0.5395	0.3927	0.4095	0.132*
H4B	0.5095	0.2574	0.3903	0.132*
O5	0.7044(3)	0.3066(2)	0.6231(2)	0.0943(7)
H5	0.640(4)	0.310(4)	0.568(3)	0.141*
N1	0.4064(3)	−0.0386(2)	0.3319(2)	0.0636(6)
H1	0.367(3)	−0.128(3)	0.331(2)	0.076*
N2	0.3618(2)	0.0419(2)	0.4165(2)	0.0629(6)
N3	0.2698(2)	0.2020(2)	0.58752(19)	0.0605(6)
H3A	0.3369	0.2449	0.5471	0.073*

Occupancy: [a] = 0.609(5), [b] = 0.391(5).

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 [4–6]. As part of our continuous work, 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 of carbonyl group (1.236(3) Å) is comparable to that of the 4-hydroxybenzohydrazone isomer (1.233(3) Å) [4]. The dihedral angle between pyrrole moiety (N3/C10—C13, r.m.s. deviation 0.0015 Å) and phenyl moiety (C2—C7, r.m.s. deviation 0.0022 Å) is 7.1°. The torsion angles of C5—C8—N2—N1 and C8—N1—N2—C9 are −179.0(2) and −179.8(2)°, respectively. Bond lengths and angles are almost in the expected ranges [7].

In the solid state, two arylhydrazone molecules are linked by both neighboring crystal 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 ethanol and water molecules.

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