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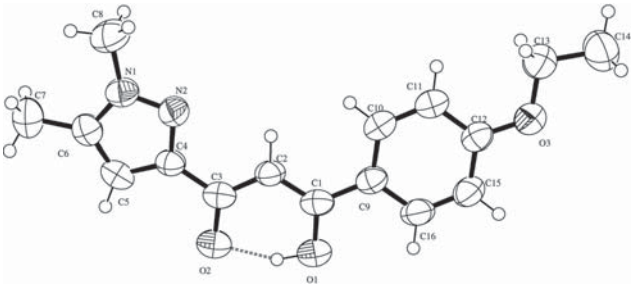
Crystal structure of (*Z*)-1-(1,5-dimethyl-1*H*-pyrazol-3-yl)-3-(4-ethoxyphenyl)-3-hydroxyprop-2-en-1-one, $C_{16}H_{18}N_2O_3$



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Abstract

$C_{16}H_{18}N_2O_3$, orthorhombic , $Pca2_1$ (no. 29), $a = 14.3074(1)$ Å, $b = 8.5321(1)$Å, $c = 12.2534(2)$Å, $V = 1495.8(4)$Å^3, $Z = 4$, $R_{gt}(F) = = 0.071$, $wR_{ref}(F^2) = 0.1266$, $T = 302$ 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.

Table 1: Data collection and handling.

Crystal:	Colourless plate
Size:	0.46 × 0.13 × 0.08 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.9 cm^{-1}
Diffractometer, scan mode:	Xcalibur Sapphire3, ω-scans
$2\theta_{max}$, completeness:	57°, >97%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	12864, 3442, 0.071
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1894
$N(param)_{refined}$:	201
Programs:	CrysAlis [4], SHELX [5], ORTEP [6]

Source of material

To a mixture of ethyl-1,5-dimethyl-1-*H*-pyrazole-3-carboxylate (2.02 g; 12.01 mmol) and metallic sodium (0.35 g; 15.21 mmol) in 50 mL of anhydrous toluene was added 1-(4-ethoxphenyl)ethanone (1.97 g; 12.01 mmol) at 0 °C. The resulting mixture was kept under stirring at room temperature for 7 days. The obtaining precipitate was filtered, washed, dissolved in water and neutralized with acetic acid. The organic layer extracted with CH_2Cl_2 was concentrated in vacuo and chromatographed on silica gel using CH_2Cl_2/MeOH (9.5/0.5) as eluant to give the desired product. Crystals of the title compound were obtained from hot ethanol by slow evaporation. yield: 47%; M.p. 159–160 °C.

Experimental details

All hydrogen atoms were inserted at their calculated positions and then refined using a riding model. The U_{iso} values of the hydrogen atoms of methyl groups were set to $1.5U_{eq}$(C) and the U_{iso} values of all other hydrogen atoms were set to $1.2U_{eq}$ of their parent atoms.

Discussion

Some β-keto-enols containing a pyrazolyl moiety are of interest for their use in synthetic procedures [1], their remarkable biological activities [2] and efficient coordination properties [3].

The crystal structure shows the formation of an intramolecular (O1—H—O2) interaction balancing the intraelectrostatic forces and van der Waals interactions. The two oxygen atoms O1, O2 are separated by a distance of 2.514(4) Å. Furthermore, the N1—N2 distance (1.337(5) Å) is in good agreement with the lengths of bonds reported for

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.1375(2)	0.6070(4)	0.4372(4)	0.0572(9)
C2	0.1730(3)	0.6918(4)	0.3505(3)	0.0545(9)
H2	0.2374	0.7011	0.3433	0.065*
C3	0.1158(3)	0.7636(4)	0.2737(4)	0.0563(9)
C4	0.1566(2)	0.8526(4)	0.1839(3)	0.0505(9)
C5	0.1138(3)	0.9228(4)	0.0939(4)	0.0619(10)
H5	0.0503	0.9234	0.0776	0.074*
C6	0.1831(3)	0.9893(4)	0.0355(3)	0.0581(10)
C7	0.1826(3)	1.0826(5)	−0.0674(4)	0.0822(13)
H7A	0.1193	1.1087	−0.0864	0.123*
H7B	0.2101	1.0220	−0.1251	0.123*
H7C	0.2179	1.1771	−0.0570	0.123*
C8	0.3579(3)	1.0026(5)	0.0614(4)	0.0834(15)
H8A	0.3896	1.0409	0.1250	0.125*
H8B	0.3560	1.0835	0.0069	0.125*
H8C	0.3905	0.9132	0.0330	0.125*
C9	0.1958(3)	0.5269(4)	0.5181(3)	0.0530(9)
C10	0.2918(3)	0.5273(4)	0.5109(3)	0.0610(11)
H10	0.3202	0.5796	0.4532	0.073*
C11	0.3467(3)	0.4529(4)	0.5865(4)	0.0618(11)
H11	0.4114	0.4554	0.5795	0.074*
C12	0.3065(3)	0.3744(4)	0.6731(3)	0.0568(9)
C13	0.4544(3)	0.2979(5)	0.7430(4)	0.0759(12)
H13A	0.4779	0.4045	0.7448	0.091*
H13B	0.4734	0.2504	0.6746	0.091*
C14	0.4929(3)	0.2071(6)	0.8361(5)	0.0966(15)
H14A	0.4779	0.2594	0.9032	0.145*
H14B	0.5595	0.1994	0.8288	0.145*
H14C	0.4661	0.1040	0.8364	0.145*
C15	0.2104(3)	0.3739(5)	0.6817(4)	0.0659(11)
H15	0.1822	0.3224	0.7400	0.079*
C16	0.1564(3)	0.4477(5)	0.6063(4)	0.0655(11)
H16	0.0917	0.4452	0.6136	0.079*
N1	0.2635(2)	0.9579(3)	0.0901(3)	0.0601(8)
N2	0.2486(2)	0.8744(3)	0.1809(3)	0.0588(8)
O1	0.04715(18)	0.5961(3)	0.4501(3)	0.0821(9)
H1	0.0220	0.6549	0.3911	0.123*
O2	0.02659(18)	0.7558(3)	0.2792(3)	0.0800(9)
O3	0.35492(19)	0.2981(3)	0.7513(2)	0.0700(8)

analogous compounds [1]. The torsion angle O1—C1—C9—C10 of 178.3(4)° indicates that the plane of phenyl ring undergoes a slight inclination relative to plane of the molecule. Also, the N1, N2, C4, C5, and C6 atoms are in the same plane, and the r.m.s. deviation from their mean plane is of 0.001 Å. The torsion angle O2—C3—C4—N2 of 174.3(3)° indicates that the pyrazole ring undergoes a greater inclination with respect to the core of the molecule (*cf*. the figure).

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