Crystal structure of (Z)-1-(1,5-dimethyl-1H-pyrazol-3-yl)-3-hydroxy-3-(4-methoxyphenyl)prop-2-en-1-one, C15H16N2O3

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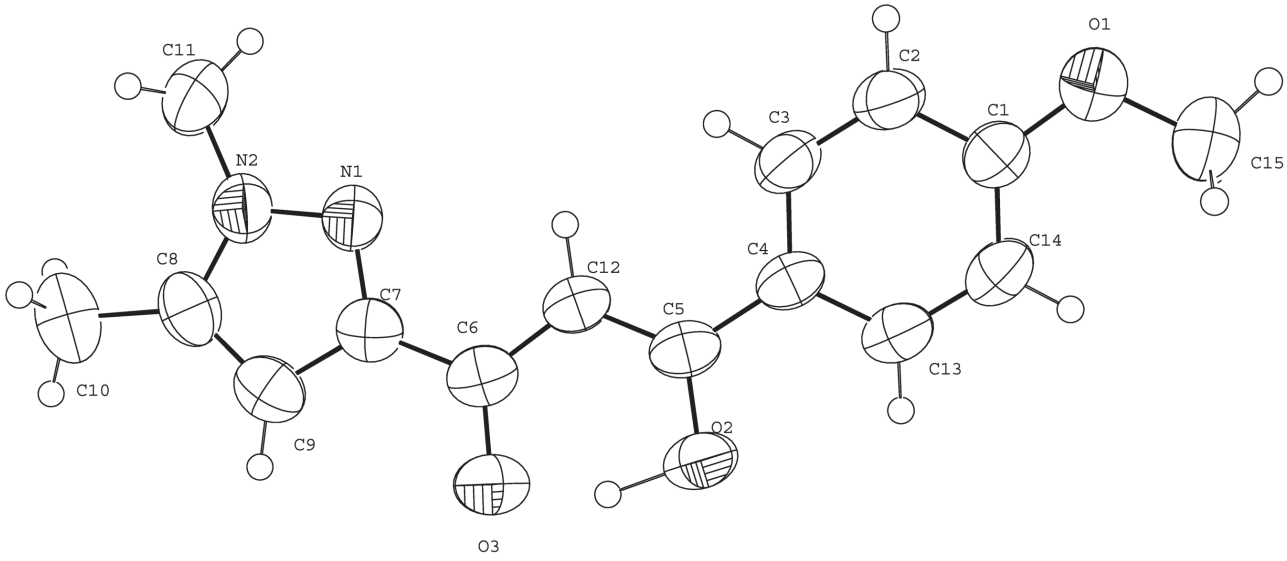

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Abstract

$C_{15}H_{16}N_2O_3$, monoclinic, $P2_1/c$ (no. 14), $a = 11.223(2)$ Å, $b = 11.9339(5)$ Å, $c = 11.945(2)$ Å, $\beta = 117.43(2)°$, $V = 1419.9(5)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0528$, $wR_{ref}(F^2) = 0.1346$, $T = 300(2)$ 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.

Table 1: Data collection and handling.

Crystal:	Colourless parallelepiped
Size:	0.42 × 0.26 × 0.25 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:	56°, >96%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	10796, 3274, 0.023
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2193
$N(param)_{refined}$:	192
Programs:	CrysAlis^{PRO} [5], SHELX [6], ORTEP [7]

Source of material

A solution of pyrazolic carboxylate (12.01 mmol) in 20 mL of THF was added to a suspension of sodium (15.21 mmol) in 15 mL of THF, then *p*-OMe-phenyl methyl ketone

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.33437(18)	0.58646(14)	0.41262(17)	0.0603(4)
C2	0.44193(19)	0.65956(15)	0.45537(18)	0.0689(5)
H13	0.4613	0.7043	0.5254	0.083*
C3	0.52015(18)	0.66644(14)	0.39523(17)	0.0638(5)
H12	0.5917	0.7164	0.4247	0.077*
C4	0.49428(16)	0.59970(13)	0.29052(15)	0.0545(4)
C5	0.57340(17)	0.60809(14)	0.22126(15)	0.0561(4)
C6	0.76077(17)	0.67033(13)	0.18904(16)	0.0569(4)
C7	0.88827(17)	0.72914(14)	0.23243(16)	0.0568(4)
C8	1.0758(2)	0.80374(15)	0.24927(19)	0.0667(5)
C9	0.9671(2)	0.74205(15)	0.17018(18)	0.0654(5)
H2	0.9490	0.7143	0.0912	0.079*
C10	1.1934(2)	0.84747(19)	0.2357(2)	0.0934(7)
H3	1.1851	0.9271	0.2234	0.140*
H4	1.1959	0.8127	0.1643	0.140*
H5	1.2748	0.8306	0.3106	0.140*
C11	1.1482(2)	0.8853(2)	0.4655(2)	0.0985(8)
H6	1.1072	0.8925	0.5201	0.148*
H7	1.1658	0.9584	0.4430	0.148*
H8	1.2310	0.8447	0.5083	0.148*
C12	0.69219(17)	0.66641(13)	0.26202(16)	0.0568(4)
H9	0.7274	0.7037	0.3390	0.068*
C13	0.38824(19)	0.52484(14)	0.25148(16)	0.0625(5)
H11	0.3704	0.4780	0.1834	0.075*
C14	0.30858(19)	0.51805(14)	0.31091(17)	0.0651(5)
H10	0.2376	0.4675	0.2826	0.078*
C15	0.1469(2)	0.5168(2)	0.4333(2)	0.0930(7)
H14	0.0877	0.5323	0.3462	0.140*
H15	0.1003	0.5289	0.4824	0.140*
H16	0.1762	0.4402	0.4420	0.140*
N1	0.94514(15)	0.78047(12)	0.34485(14)	0.0641(4)
N2	1.05841(15)	0.82513(12)	0.35234(14)	0.0670(4)
O1	0.26014(14)	0.58896(12)	0.47654(13)	0.0811(4)
O2	0.52254(13)	0.55531(11)	0.11275(11)	0.0745(4)
H1	0.6030	0.5720	0.0780	0.112*
O3	0.71546(13)	0.62190(10)	0.08166(11)	0.0703(4)

(12.01 mmol) in 7 mL of THF was added at 0 °C. The mixture was stirred for 2 days at room temperature. The resulting precipitate was filtered, washed, dissolved in water and neutralized to pH 5 using acetic acid. The extracted and dried organic layer was concentrated in vacuo. The obtained residue was purified by silica using CH_2Cl_2/MeOH as eluant. Crystals were obtained by slow evaporation of methanol from the mixture; 31%; M.p. 122–124 °C.

Experimental details

All hydrogen atoms were inserted at 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 heterocyclic compounds bearing β-keto-enol functionality have recently attracted a great attention due to their applications in organic and inorganic chemistry [1–4]. In this work we coupled ethyl pyrazole carboxylate with *p*-OMe-phenyl methyl ketone to produce the title compound in acceptable yield. The whole molecule is almost planar, with an r.m.s. deviation of 0.008 Å from the plane through all non-hydrogen atoms. In the title crystal structure an intramolecular (OH$\cdots$O=C) interaction balances the electrostatic positive and negative charges respectively of O1—H1 proton and oxygen (O2). The title molecule may act as a tridentate and bidentate ligand, respectively. However, the orientation is fixed by means of an intramolecular hydrogen bond of 2.489(1) Å.

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