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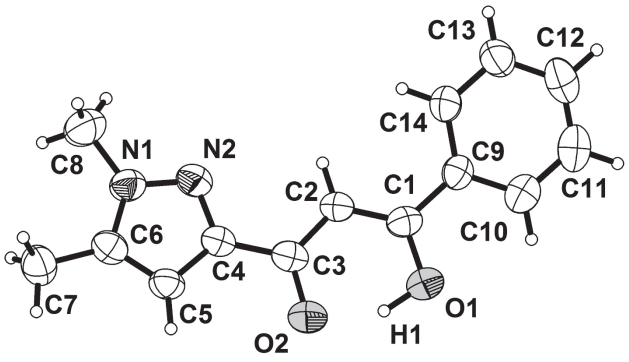
Radi Smaail*, Said Tighadouini, Driss Eddike, Monique Tillard and Yahia N. Mabkhot

Crystal structure of (*Z*)-1-(1,5-dimethyl-1*H*-pyrazol-3-yl)-3-hydroxy-3-phenylprop-2-en-1-one, $C_{14}H_{14}N_2O_2$



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Abstract

$C_{14}H_{14}N_2O_2$, orthorhombic, $P2_12_12_1$ (no. 19), $a = 5.6736(2)$ Å, $b = 7.7803(4)$ Å, $c = 28.7821(13)$ Å, $V = 1270.5(1)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0454$, $wR_{ref}(F^2) = 0.0879$, $T = 298$ K.

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Table 1: Data collection and handling.

Crystal:	Colourless plate
Size:	0.40 × 0.15 × 0.07 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:	55.4°, >89% (99.4% up to 50.5°)
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	8426, 2596, 0.035
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1646
$N(param)_{refined}$:	173
Programs:	CrysAlis [11], SHELX [12], ORTEP [13]

Source of material

Ethyl-1,5-dimethyl-1*H*-pyrazole-3-carboxylate (2.02 g; 12.01 mmol) was slowly added to a solution of metallic sodium (0.35 g; 15.21 mmol) in 50 mL of anhydrous toluene. Then, acetophenone (1.44 g; 12.01 mmol) in 10 mL of toluene was added at 0 °C. The resulting mixture was stirred at room temperature for 7 days. The precipitate formed was filtered, washed with toluene, dissolved in water and neutralized with acetic acid. The product extracted with CH_2Cl_2 was concentrated in vacuo and precipitated in hexane. Crystals of the title compound were obtained from hot ethanol by slow evaporation. Yield: 32%; M.p. 108–110 °C.

Experimental details

All hydrogen atoms were inserted at calculated positions and 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

Many heterocycles branched β-keto-enol are useful synthetic intermediates in a wide variety of applications including anti-tumor [1–3], anti-inflammatory [4] and anti-HIV [5, 6] activities. Recently, we have reported some new heterocyclic keto-enol compounds that have shown remarkable biological activities [7] and efficient coordination properties [8, 9]. Herein, we report a β-keto-enol compound: (*Z*)-1-(1,5-dimethyl-1*H*-pyrazol-3-yl)-3-hydroxy-3-phenylprop-2-en-1-one, prepared in acceptable yield. The crystal structure shows the formation of an intramolecular (O1—H—O2) interaction balancing the intra-electrostatic forces. The two oxygen atoms

***Corresponding author: Radi Smaail,** LCAE, Department of Chemistry, Faculty of Sciences, Mohamed Premier University, Oujda 60000, Morocco, e-mail: radi_smaail@yahoo.fr
Said Tighadouini: LCAE, Department of Chemistry, Faculty of Sciences, Mohamed Premier University, Oujda 60000, Morocco
Driss Eddike: LCSMA, Faculte des Sciences, Universitè Mohamed I, 60 000 Oujda, Morocco
Monique Tillard: Institut Charles Gerhardt, AIME, UMR CNRS 5253 CC15, Université de Montpellier II, Sciences et Techniques du Languedoc, 2 Place Eugène Bataillon, 34095 Montpellier Cédex 5, France
Yahia N. Mabkhot: Department of Chemistry, College of Science, King Saud University, P.O. Box 2455, Riyadh 1451, Saudi Arabia

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.4206(5)	0.7032(4)	0.15932(9)	0.0458(8)
C2	0.6125(5)	0.6156(4)	0.14197(9)	0.0476(8)
H2	0.6893	0.5360	0.1607	0.057*
C3	0.6944(5)	0.6438(4)	0.09665(9)	0.0480(8)
C4	0.8970(4)	0.5512(4)	0.07777(9)	0.0446(7)
C5	1.0080(5)	0.5761(4)	0.03494(9)	0.0476(8)
H5	0.9669	0.6555	0.0122	0.057*
C6	1.1873(5)	0.4616(4)	0.03326(9)	0.0479(8)
C7	1.3676(5)	0.4268(5)	−0.00323(9)	0.0627(9)
H7A	1.5219	0.4490	0.0091	0.094*
H7B	1.3398	0.5002	−0.0295	0.094*
H7C	1.3572	0.3088	−0.0128	0.094*
C8	1.3290(5)	0.2316(5)	0.08903(11)	0.0763(11)
H8A	1.4696	0.2776	0.1028	0.114*
H8B	1.3704	0.1617	0.0628	0.114*
H8C	1.2466	0.1630	0.1115	0.114*
C9	0.3333(4)	0.6900(4)	0.20725(9)	0.0426(7)
C10	0.1240(5)	0.7691(4)	0.21946(10)	0.0565(9)
H10	0.0343	0.8236	0.1968	0.068*
C11	0.0467(5)	0.7681(5)	0.26485(13)	0.0671(10)
H11	−0.0941	0.8222	0.2726	0.080*
C12	0.1768(6)	0.6876(5)	0.29873(11)	0.0646(9)
H12	0.1254	0.6886	0.3294	0.078*
C13	0.3833(5)	0.6055(4)	0.28713(10)	0.0606(9)
H13	0.4705	0.5492	0.3098	0.073*
C14	0.4608(5)	0.6071(4)	0.24151(10)	0.0513(8)
H14	0.6007	0.5516	0.2338	0.062*
N1	1.1782(4)	0.3720(3)	0.07386(8)	0.0519(6)
N2	1.0014(4)	0.4251(3)	0.10172(7)	0.0538(7)
O1	0.3059(3)	0.8116(3)	0.13283(6)	0.0647(6)
H1	0.3959	0.8169	0.1024	0.097*
O2	0.5935(3)	0.7515(3)	0.06995(7)	0.0665(7)

O1, O2 are separated by a distance of 2.480(3) Å. Furthermore, the N1—N2 distance (1.349(3) Å) is in good agreement with the lengths of bonds reported for analogous compounds [10]. The N1, N2, C4, C5, and C6 atoms are in the same plane. The r.m.s. deviation is 0.002 Å. The torsion angle O2—C3—C4—N2 of -174.1(3) indicates that the pyrazole ring undergoes slight inclination with respect to the binding C3—C4. Also the phenyl atoms are in the same plane (0.005 Å), while the torsion angle O1—C1—C9—C14 of -167.6(3)° indicates that the plane of phenyl ring undergoes a inclination relative to plane of the rest of the molecule.

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