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1-[5-(4-Hydroxy-3-methoxyphenyl)-3-methyl-4,5-dihydro-1*H*-pyrazol-1-yl]ethanone

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.049
wR factor = 0.141
Data-to-parameter ratio = 17.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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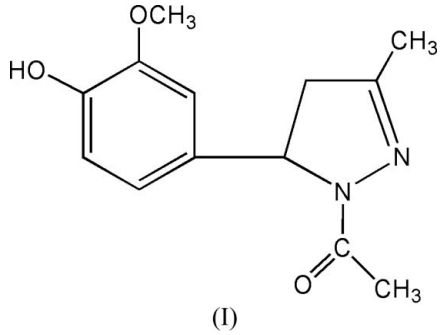
The title compound, $C_{13}H_{16}N_2O_3$, was synthesized from vanillin, acetone and hydrazine monohydrate. An intermolecular O—H···O hydrogen bond connects the molecules into infinite one-dimensional chains.

Comment

Research in the field of pyrazole derivatives has been significantly developed in recent years as a result of their potent bioactivities against hypertension, heart breakdown and anxiety neurosis (Camacho *et al.*, 2004; Bekhit & Abdel, 2004). Thus, we are interested in the synthesis, bioactivities and structural studies of pyrazole derivatives. In this context, we have synthesized the title compound, (I).



(I)

All the bond lengths and angles are within normal ranges (Allen *et al.*, 1987) and the structural data confirm that the benzene ring plane is perpendicular to the plane of the pyrazole ring [dihedral angle 85.3 (1)°]. An intermolecular O1—H1···O3 hydrogen bond links adjacent molecules to form infinite one-dimensional chains (Table 1).

Experimental

Vanillin (1.52 g, 10 mmol) was dissolved in acetone (10 ml), and dilute aqueous NaOH solution (10%, 6 ml) was added to the acetone solution with stirring. The mixture was allowed to stand overnight at room temperature, and the mixture was then acidified with dilute aqueous HCl to give 4-(4-hydroxy-3-methoxyphenyl)but-3-en-2-one as a yellow solid (yield 53%), a precursor for the synthesis of the title compound. To a solution of 4-(4-hydroxy-3-methoxyphenyl)but-3-en-2-one (0.96 g, 5 mmol) in acetic acid (10 ml) was added hydrazine monohydrate (1.25 ml, 25 mmol) and the reaction mixture was refluxed for 5 h. The solvent was evaporated and cold water (50 ml) was added to the yellow residue. The resulting precipitate was filtered off, washed with water and recrystallized from acetone, standing in air over a period of three days. After about three-quarters of the original solvent had evaporated, the title compound, (I), was obtained as colourless plate-like crystals (yield 68%).

Crystal data

$C_{13}H_{16}N_2O_3$
M_r = 248.28
Monoclinic, $P2_1/n$
a = 7.5862 (6) Å
b = 10.5414 (9) Å
c = 15.6452 (13) Å
β = 102.913 (1)°
V = 1219.49 (17) Å^3
Z = 4
D_x = 1.352 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4666 reflections
θ = 2.4–27.9°
μ = 0.10 mm^{-1}
T = 293 (2) K
Plate, colourless
0.22 × 0.12 × 0.04 mm

Data collection

Bruker SMART CCD area detector diffractometer
φ and ω scans
Absorption correction: none
10433 measured reflections
2893 independent reflections
2271 reflections with $I > 2\sigma(I)$
R_{int} = 0.061
θ_{max} = 27.9°
$h = -9 \rightarrow 9$
$k = -13 \rightarrow 13$
$l = -20 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.141
S = 1.05
2893 reflections
167 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.081P)^2 + 0.0609P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O3^{i}	0.82	1.93	2.752 (2)	176

Symmetry code: (i) $-x+\frac{5}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$.

H atoms were positioned geometrically [C—H = 0.93 (Csp^2—H), 0.96 (methyl), 0.97 (methylene) and 0.98 (methine) Å, and O—H = 0.82 Å]. U_{iso}(H) values were set equal to xU_{eq}(carrier atom), where x = 1.2 for CH and CH_2, and 1.5 for hydroxy and methyl H atoms.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 2000); software used to prepare material for publication: *SHELXTL*.

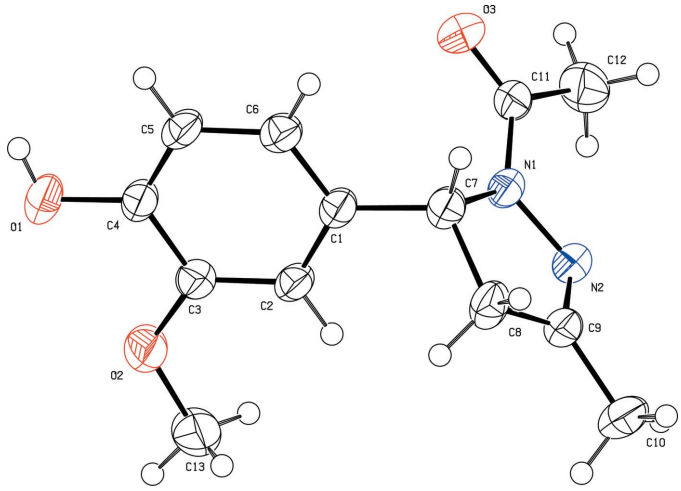


Figure 1
The structure of the title compound, (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

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