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(5-Hydroxy-3-methyl-5-phenyl-4,5-dihydro-1*H*-pyrazol-1-yl)(pyridin-4-yl)-methanone monohydrate

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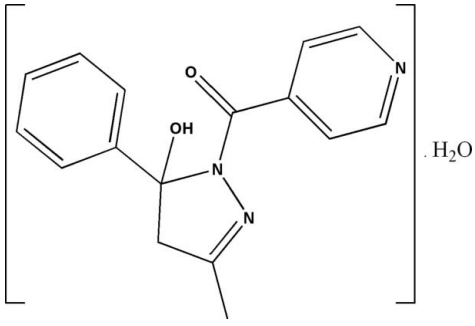
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.048; wR factor = 0.121; data-to-parameter ratio = 15.3.

In the title compound, $C_{16}H_{15}N_3O_2\cdot H_2O$, the mean plane of the approximately planar pyrazole ring [maximum deviation = 0.0474 (18) Å] makes dihedral angles of 86.32 (11) and 45.04 (10)° with the phenyl and pyridine rings, respectively. The dihedral angle between the phenyl and pyridine rings is 69.62 (11)°. In the crystal, intermolecular O—H···O and O—H···N hydrogen bonds connect the components into chains along [010]. The crystal structure is further stabilized by π–π stacking interactions with centroid–centroid distances of 3.7730 (12) Å.

Related literature

For standard values of bond lengths, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{16}H_{15}N_3O_2\cdot H_2O$
M_r = 299.33
Monoclinic, $P2_1/c$
a = 16.9676 (10) Å
b = 7.0266 (5) Å
c = 12.6135 (6) Å
β = 93.004 (3)°
V = 1501.77 (16) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 296 K
0.32 × 0.24 × 0.18 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.971, T_{max} = 0.983
8525 measured reflections
3059 independent reflections
1916 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.121
S = 1.03
3059 reflections
200 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O1W^{i}	0.92	1.84	2.7490 (19)	173
O1W—H1W1···O2^{ii}	0.92	1.89	2.8003 (19)	169
O1W—H2W1···N3^{iii}	0.85	2.13	2.976 (2)	173

Symmetry codes: (i) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (ii) $x, -y+\frac{3}{2}, z+\frac{1}{2}$; (iii) $-x, y-\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL* and *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXTL* and *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5187).

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supplementary materials

Acta Cryst. (2011). E**67**, o209 [**doi:10.1107/S160053681005275X**]

(5-Hydroxy-3-methyl-5-phenyl-4,5-dihydro-1*H*-pyrazol-1-yl)(pyridin-4-yl)methanone monohydrate

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Comment

The asymmetric unit of the title compound, Fig. 1, comprises a substituted pyrazole molecule and a solvent water molecule. The bond lengths (Allen *et al.,* 1987) and angles are within the normal ranges. The dihedral angle the mean plane of the pyrazole ring makes with the phenyl and pyridine rings are 86.32 (11) and 45.04 (10)°, respectively. The dihedral angle between the phenyl ring and the pyridine ring is 69.62 (11)Å.

In the crystal structure, intermolecular O—H···O and O—H···N hydrogen bonds connect the components of the structure to form one-dimensional chains along [0 1 0]. The crystal structure is further stabilized by intermolecular π–π stacking interactions [$Cg1\cdots Cg1^{iv}$ = 3.7730 (11)Å, (iv) -*x*, 1 - *y*, -*z*, *Cg*1 is the centroid of the C12-C16/N3 ring].

Experimental

The title compound was synthesized by adding isoniazide (2 mmol) to a solution of benzoylacetone (2 mmol) in ethanol (20 ml). The mixture was refluxed with stirring for half an hour. The resultant solution was filtered and the white single crystals suitable for *X*-ray structure determination were recrystallized from ethanol by slow evaporation of the solvents at room temperature over several days.

Refinement

The H atoms of the water and hydroxy groups were located in a difference Fourier map and constraied to refine on the parent atom with U_{iso}(H) = 1.5 U_{eq}(O), see Table 1. The remaining H atoms were positioned geometrically with C—H = 0.93-0.97 Å and included in a riding-model approximation with U_{iso}(H) = 1.2 or 1.5 U_{eq}(C). A rotating group model was used for the methyl group.

Figures

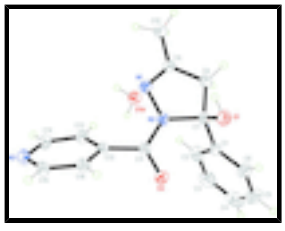

Fig. 1. The asymmetric unit of the title compound, showing 30% probability displacement ellipsoids and the atomic numbering.

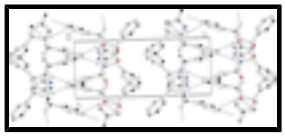

Fig. 2. The packing of the compound viewed along the *c*-axis showing 1-D extended chains along the *b*-axis through hydrogen bonds. All H atoms removed except those involved in the hydrogen bonds. Hydrogen bonds are shown as dashed lines.

(5-Hydroxy-3-methyl-5-phenyl-4,5-dihydro-1*H*-pyrazol-1-yl)(pyridin- 4-yl)methanone monohydrate

Crystal data

$C_{16}H_{15}N_3O_2 \cdot H_2O$	$F(000) = 632$
$M_r = 299.33$	$D_x = 1.324$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 2540 reflections
$a = 16.9676$ (10) Å	$\theta = 2.5$–$27.5°$
$b = 7.0266$ (5) Å	$\mu = 0.09$ mm^{-1}
$c = 12.6135$ (6) Å	$T = 296$ K
$\beta = 93.004$ (3)°	Prism, white
$V = 1501.77$ (16) Å^3	0.32 × 0.24 × 0.18 mm
$Z = 4$	

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	3059 independent reflections
Radiation source: fine-focus sealed tube	1916 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.038$
φ and ω scans	$\theta_{max} = 26.5°$, $\theta_{min} = 2.4°$
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)	$h = -18 \rightarrow 21$
$T_{min} = 0.971$, $T_{max} = 0.983$	$k = -7 \rightarrow 8$
8525 measured reflections	$l = -15 \rightarrow 14$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.048$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.121$	H-atom parameters constrained
$S = 1.03$	$w = 1/[\sigma^2(F_o^2) + (0.0554P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
3059 reflections	$(\Delta/\sigma)_{max} < 0.001$
200 parameters	$\Delta\rho_{max} = 0.15$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.18$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds

in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.32045 (8)	0.3424 (2)	−0.01490 (10)	0.0461 (4)
H1	0.2849	0.2478	−0.0322	0.069*
O2	0.22503 (8)	0.70361 (19)	0.00147 (10)	0.0455 (4)
O1W	0.21323 (8)	0.4293 (2)	0.41563 (10)	0.0522 (4)
H1W1	0.2123	0.5449	0.4498	0.078*
H2W1	0.1656	0.3911	0.4143	0.078*
N1	0.17899 (8)	0.3038 (2)	0.15669 (11)	0.0339 (4)
N2	0.22186 (8)	0.4400 (2)	0.10150 (11)	0.0347 (4)
N3	−0.05005 (9)	0.7733 (2)	0.10396 (13)	0.0431 (4)
C1	0.36077 (10)	0.5444 (3)	0.12956 (14)	0.0370 (5)
C2	0.35148 (13)	0.6223 (3)	0.22873 (16)	0.0544 (6)
H2	0.3115	0.5788	0.2703	0.065*
C3	0.40176 (17)	0.7653 (4)	0.2661 (2)	0.0726 (8)
H3A	0.3947	0.8188	0.3323	0.087*
C4	0.46131 (16)	0.8286 (4)	0.2071 (3)	0.0770 (8)
H4	0.4951	0.9238	0.2333	0.092*
C5	0.47118 (14)	0.7520 (4)	0.1099 (2)	0.0726 (8)
H5	0.5119	0.7947	0.0694	0.087*
C6	0.42080 (12)	0.6104 (3)	0.07061 (17)	0.0521 (6)
H6	0.4277	0.5596	0.0036	0.062*
C7	0.30659 (10)	0.3854 (3)	0.09099 (13)	0.0359 (5)
C8	0.31075 (11)	0.2087 (3)	0.16286 (16)	0.0449 (5)
H8A	0.3310	0.1000	0.1255	0.054*
H8B	0.3443	0.2320	0.2262	0.054*
C9	0.22789 (11)	0.1762 (3)	0.19037 (13)	0.0339 (4)
C10	0.20331 (12)	0.0106 (3)	0.25333 (15)	0.0469 (5)
H10A	0.1476	0.0176	0.2628	0.070*
H10B	0.2314	0.0116	0.3214	0.070*
H10C	0.2150	−0.1048	0.2166	0.070*
C11	0.18790 (11)	0.5957 (3)	0.05705 (13)	0.0334 (4)
C12	0.10340 (10)	0.6415 (2)	0.07655 (13)	0.0297 (4)
C13	0.07060 (11)	0.6357 (3)	0.17484 (14)	0.0375 (5)
H13	0.0990	0.5858	0.2335	0.045*
C14	−0.00432 (12)	0.7045 (3)	0.18447 (15)	0.0430 (5)
H14	−0.0247	0.7031	0.2515	0.052*
C15	−0.01844 (11)	0.7738 (3)	0.00927 (15)	0.0407 (5)
H15	−0.0493	0.8176	−0.0488	0.049*
C16	0.05681 (11)	0.7135 (3)	−0.00760 (14)	0.0361 (5)

H16	0.0764	0.7210	−0.0750	0.043*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0399 (8)	0.0500 (9)	0.0490 (8)	−0.0025 (7)	0.0074 (6)	−0.0062 (7)
O2	0.0370 (8)	0.0445 (9)	0.0556 (8)	0.0018 (7)	0.0082 (6)	0.0166 (7)
O1W	0.0428 (8)	0.0447 (9)	0.0690 (9)	−0.0001 (7)	0.0023 (6)	−0.0063 (7)
N1	0.0297 (9)	0.0353 (9)	0.0368 (8)	−0.0024 (8)	0.0008 (7)	0.0024 (7)
N2	0.0240 (8)	0.0362 (9)	0.0438 (8)	0.0016 (7)	0.0022 (6)	0.0106 (7)
N3	0.0319 (9)	0.0421 (10)	0.0553 (11)	0.0033 (8)	0.0027 (8)	−0.0013 (8)
C1	0.0274 (10)	0.0373 (11)	0.0457 (11)	0.0028 (9)	−0.0031 (8)	0.0043 (9)
C2	0.0458 (13)	0.0606 (16)	0.0564 (13)	0.0085 (12)	−0.0017 (10)	−0.0066 (11)
C3	0.0676 (18)	0.0689 (19)	0.0786 (17)	0.0129 (16)	−0.0232 (14)	−0.0247 (14)
C4	0.0537 (17)	0.0506 (17)	0.123 (2)	−0.0011 (14)	−0.0303 (16)	−0.0094 (16)
C5	0.0484 (15)	0.0663 (19)	0.102 (2)	−0.0183 (14)	−0.0027 (14)	0.0144 (16)
C6	0.0413 (12)	0.0551 (15)	0.0598 (13)	−0.0080 (11)	0.0028 (10)	0.0046 (11)
C7	0.0253 (10)	0.0393 (12)	0.0434 (11)	0.0041 (9)	0.0048 (8)	0.0036 (9)
C8	0.0352 (11)	0.0387 (12)	0.0605 (12)	0.0049 (10)	0.0006 (9)	0.0118 (10)
C9	0.0355 (11)	0.0314 (11)	0.0345 (9)	0.0005 (9)	0.0000 (8)	−0.0005 (8)
C10	0.0472 (12)	0.0370 (12)	0.0567 (12)	0.0009 (11)	0.0055 (9)	0.0069 (10)
C11	0.0294 (10)	0.0355 (11)	0.0349 (10)	0.0017 (9)	−0.0009 (8)	0.0008 (9)
C12	0.0289 (10)	0.0249 (10)	0.0351 (10)	−0.0014 (8)	0.0004 (8)	−0.0009 (8)
C13	0.0348 (11)	0.0420 (12)	0.0355 (10)	0.0022 (10)	−0.0015 (8)	0.0012 (9)
C14	0.0381 (12)	0.0480 (13)	0.0431 (11)	−0.0005 (10)	0.0053 (9)	−0.0035 (9)
C15	0.0343 (11)	0.0386 (12)	0.0480 (12)	0.0020 (10)	−0.0095 (9)	0.0048 (9)
C16	0.0363 (11)	0.0358 (11)	0.0360 (10)	0.0017 (9)	−0.0013 (8)	0.0013 (8)

Geometric parameters (Å, °)

O1—C7	1.401 (2)	C5—C6	1.386 (3)
O1—H1	0.9166	C5—H5	0.9300
O2—C11	1.229 (2)	C6—H6	0.9300
O1W—H1W1	0.9200	C7—C8	1.536 (3)
O1W—H2W1	0.8517	C8—C9	1.483 (3)
N1—C9	1.279 (2)	C8—H8A	0.9700
N1—N2	1.408 (2)	C8—H8B	0.9700
N2—C11	1.345 (2)	C9—C10	1.481 (3)
N2—C7	1.501 (2)	C10—H10A	0.9600
N3—C15	1.334 (2)	C10—H10B	0.9600
N3—C14	1.336 (2)	C10—H10C	0.9600
C1—C6	1.373 (3)	C11—C12	1.502 (2)
C1—C2	1.382 (3)	C12—C16	1.386 (2)
C1—C7	1.511 (3)	C12—C13	1.386 (2)
C2—C3	1.384 (3)	C13—C14	1.371 (3)
C2—H2	0.9300	C13—H13	0.9300
C3—C4	1.360 (4)	C14—H14	0.9300
C3—H3A	0.9300	C15—C16	1.372 (3)
C4—C5	1.358 (4)	C15—H15	0.9300

C4—H4	0.9300	C16—H16	0.9300
C7—O1—H1	104.0	C9—C8—H8A	110.9
H1W1—O1W—H2W1	104.4	C7—C8—H8A	110.9
C9—N1—N2	107.33 (14)	C9—C8—H8B	110.9
C11—N2—N1	122.54 (15)	C7—C8—H8B	110.9
C11—N2—C7	124.25 (15)	H8A—C8—H8B	108.9
N1—N2—C7	113.06 (13)	N1—C9—C10	122.21 (17)
C15—N3—C14	115.90 (17)	N1—C9—C8	114.87 (16)
C6—C1—C2	118.6 (2)	C10—C9—C8	122.91 (17)
C6—C1—C7	122.12 (17)	C9—C10—H10A	109.5
C2—C1—C7	119.22 (17)	C9—C10—H10B	109.5
C1—C2—C3	119.9 (2)	H10A—C10—H10B	109.5
C1—C2—H2	120.1	C9—C10—H10C	109.5
C3—C2—H2	120.1	H10A—C10—H10C	109.5
C4—C3—C2	120.9 (2)	H10B—C10—H10C	109.5
C4—C3—H3A	119.5	O2—C11—N2	121.24 (17)
C2—C3—H3A	119.5	O2—C11—C12	118.90 (16)
C5—C4—C3	119.6 (2)	N2—C11—C12	119.85 (16)
C5—C4—H4	120.2	C16—C12—C13	117.15 (17)
C3—C4—H4	120.2	C16—C12—C11	117.65 (15)
C4—C5—C6	120.3 (2)	C13—C12—C11	124.86 (16)
C4—C5—H5	119.9	C14—C13—C12	119.10 (17)
C6—C5—H5	119.9	C14—C13—H13	120.4
C1—C6—C5	120.7 (2)	C12—C13—H13	120.4
C1—C6—H6	119.7	N3—C14—C13	124.36 (18)
C5—C6—H6	119.7	N3—C14—H14	117.8
O1—C7—N2	110.42 (13)	C13—C14—H14	117.8
O1—C7—C1	109.67 (14)	N3—C15—C16	123.97 (17)
N2—C7—C1	110.60 (15)	N3—C15—H15	118.0
O1—C7—C8	112.56 (16)	C16—C15—H15	118.0
N2—C7—C8	99.74 (14)	C15—C16—C12	119.47 (17)
C1—C7—C8	113.51 (15)	C15—C16—H16	120.3
C9—C8—C7	104.34 (15)	C12—C16—H16	120.3
C9—N1—N2—C11	−179.02 (16)	N2—C7—C8—C9	7.23 (18)
C9—N1—N2—C7	5.31 (18)	C1—C7—C8—C9	124.87 (16)
C6—C1—C2—C3	0.6 (3)	N2—N1—C9—C10	179.31 (16)
C7—C1—C2—C3	178.63 (19)	N2—N1—C9—C8	0.2 (2)
C1—C2—C3—C4	−1.1 (4)	C7—C8—C9—N1	−5.2 (2)
C2—C3—C4—C5	0.7 (4)	C7—C8—C9—C10	175.69 (17)
C3—C4—C5—C6	0.2 (4)	N1—N2—C11—O2	−173.53 (15)
C2—C1—C6—C5	0.2 (3)	C7—N2—C11—O2	1.7 (3)
C7—C1—C6—C5	−177.7 (2)	N1—N2—C11—C12	7.4 (2)
C4—C5—C6—C1	−0.6 (4)	C7—N2—C11—C12	−177.38 (15)
C11—N2—C7—O1	−64.9 (2)	O2—C11—C12—C16	39.6 (2)
N1—N2—C7—O1	110.71 (15)	N2—C11—C12—C16	−141.39 (18)
C11—N2—C7—C1	56.7 (2)	O2—C11—C12—C13	−133.48 (19)
N1—N2—C7—C1	−127.72 (15)	N2—C11—C12—C13	45.6 (3)
C11—N2—C7—C8	176.49 (17)	C16—C12—C13—C14	−1.5 (3)

N1—N2—C7—C8	−7.92 (18)	C11—C12—C13—C14	171.52 (18)
C6—C1—C7—O1	−8.6 (2)	C15—N3—C14—C13	−0.4 (3)
C2—C1—C7—O1	173.52 (17)	C12—C13—C14—N3	2.0 (3)
C6—C1—C7—N2	−130.57 (19)	C14—N3—C15—C16	−1.7 (3)
C2—C1—C7—N2	51.5 (2)	N3—C15—C16—C12	2.1 (3)
C6—C1—C7—C8	118.3 (2)	C13—C12—C16—C15	−0.4 (3)
C2—C1—C7—C8	−59.6 (2)	C11—C12—C16—C15	−173.97 (17)
O1—C7—C8—C9	−109.82 (17)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O1W^{i}	0.92	1.84	2.7490 (19)	173.
O1W—H1W1···O2^{ii}	0.92	1.89	2.8003 (19)	169.
O1W—H2W1···N3^{iii}	0.85	2.13	2.976 (2)	173.

Symmetry codes: (i) *x*, −*y*+1/2, *z*−1/2; (ii) *x*, −*y*+3/2, *z*+1/2; (iii) −*x*, *y*−1/2, −*z*+1/2.

Fig. 1

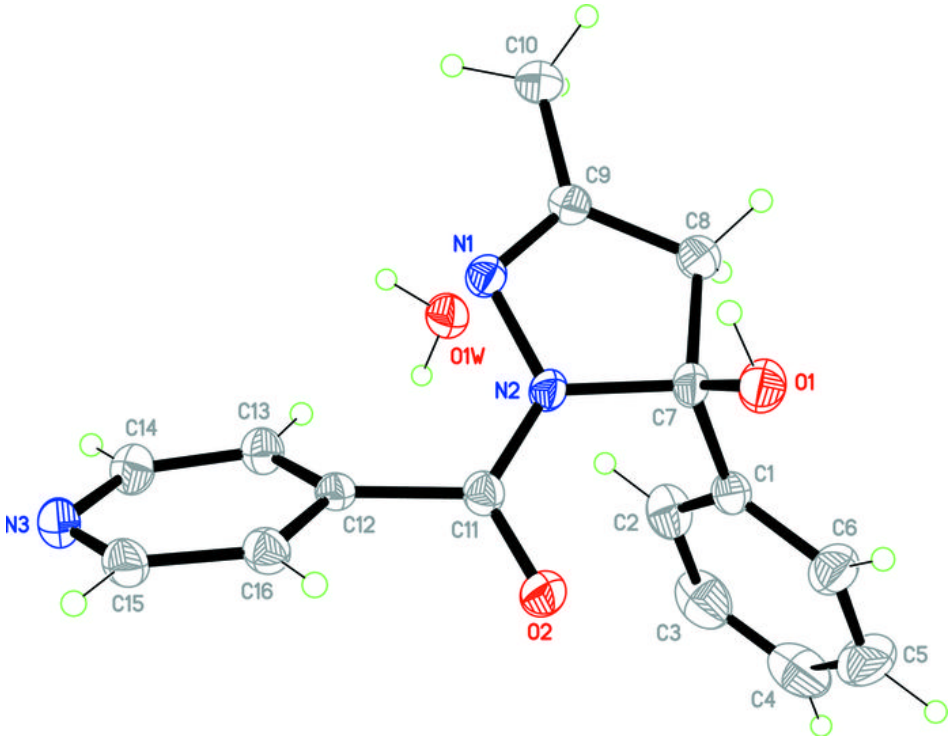
C10
C9
C8
N1
O1W
O1
N2
C7
C1
C14
C13
N3
C12
C11
C2
C6
C16
C15
O2
C3
C5
C4

Fig. 2