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5-(2-Hydroxybenzoyl)-1-methyl-3-nitropyridin-2(1*H*)-one

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Keywords: crystal structure; pyridine derivative; hydrogen bonding.

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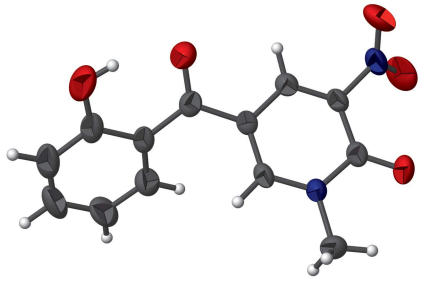
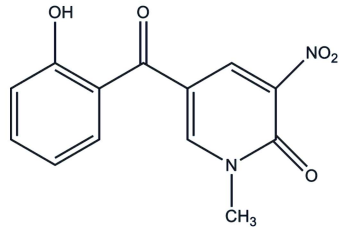
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{13}H_{10}N_2O_5$, the dihedral angle between the pyridine and phenyl ring is 50.47 (2)°. The hydroxyl H and ketone O atoms form an intramolecular O—H···O hydrogen bond with the hydroxyl group almost coplanar with the phenyl ring. In the crystal, molecules are linked by two C—H···O hydrogen bonds, forming dimers. The dimers are linked by further C—H···O hydrogen bonds, forming a three-dimensional architecture.

3D view



Chemical scheme



Structure description

The title compound is an important nitropyridine compound which is widely used in organic synthesis, especially in the synthesis of heterocyclic drugs and cytokine inhibitors (Hu *et al.*, 2011). Studies of pyridine and pyrimidine derivatives related to the title compound are also of interest owing to their putative fluorescence properties (Kawai *et al.* 2001; Abdullah, 2005). For related crystal structures, see: Aznan *et al.*, (2011).

In the title compound the dihedral angle between the pyridine and phenyl ring is 50.47 (2)°. The hydroxyl H and ketone O atoms form an intramolecular O—H···O hydrogen bond with the hydroxyl group almost coplanar with the phenyl ring (Fig. 1). In the crystal, molecules are linked into dimers *via* two C—H···O hydrogen bonds (Table 1), resulting in an $R_2^2(10)$ graph-set motif (Fig. 2). The dimers are linked by further C—H···O hydrogen bonds, forming a three-dimensional architecture.

Synthesis and crystallization

A mixture of 3-formylchromone (1 mmol), (*Z*)-*N*-methyl-1-(methyl-thio)-2-nitro-ethenamine (1 mmol), and indium trifluoromethanesulfonate (0.020 mmol) in ethanol (3 ml) were charged in a 25 ml round-bottomed flask and the mixture was heated at reflux. The resulting solution was stirred for 1.5 h. The consumption of the starting

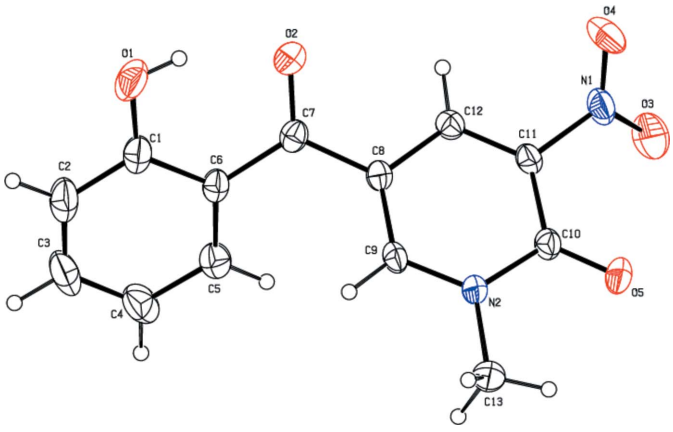


Figure 1
The molecular structure of the title compound, with the atomic numbering scheme and displacement ellipsoids drawn at 30% probability level.

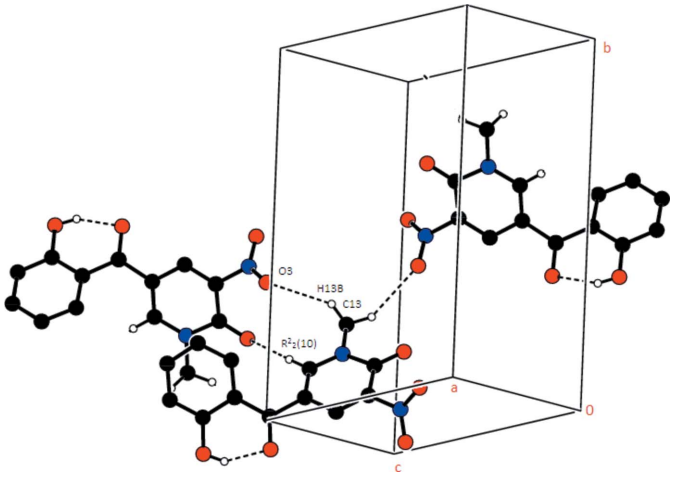


Figure 2
A partial view of the crystal packing of the title compound is viewed along the *b* axis, showing intramolecular O—H···O hydrogen bonds and molecules linked by C—H···O intermolecular interactions (see Table 1).

material was monitored by TLC. After completion of the reaction, the compound was purified by column chromatography to obtain pure product. The purified compound was recrystallized from ethanol and DMSO-D6 by slow evaporation. The yield of the isolated product was 88%, giving block-like crystals suitable for X-ray diffraction.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

The authors thank Dr Babu Varghese, SAIF, IIT, Chennai, India, for the data collection.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2	0.82	1.90	2.603 (3)	142
C4—H4···O1^{i}	0.93	2.58	3.498 (5)	169
C9—H9···O5^{ii}	0.93	2.41	3.311 (3)	164
C13—H13*A*···O4^{iii}	0.96	2.53	3.388 (4)	148
C13—H13*B*···O3^{ii}	0.96	2.59	3.431 (5)	146

Symmetry codes: (i) $-x+\frac{5}{2}, y+\frac{1}{2}, -z+\frac{5}{2}$; (ii) $x+\frac{1}{2}, -y+\frac{1}{2}, z+\frac{1}{2}$; (iii) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{13}H_{10}N_2O_5$
M_r	274.23
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	293
a, *b*, *c* (Å)	7.4998 (8), 13.8350 (14), 12.2324 (12)
β (°)	107.474 (4)
V (Å^3)	1210.7 (2)
Z	4
Radiation type	Mo *K*α
μ (mm^{-1})	0.12
Crystal size (mm)	0.22 × 0.20 × 0.18
Data collection	
Diffractometer	Bruker Kappa APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2004)
T_{min}, T_{max}	0.974, 0.979
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	18309, 2126, 1288
R_{int}	0.057
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.056, 0.176, 1.01
No. of reflections	2091
No. of parameters	181
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.42, −0.30

Computer programs: *APEX2*, *SAINT* and *XPREP* (Bruker, 2004), *SHELXS97* and *SHELXL97* (Sheldrick, 2008), *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009).

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full crystallographic data

IUCrData (2016). **1**, x160527 [doi:10.1107/S2414314616005277]

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Crystal data

$C_{13}H_{10}N_2O_5$
M_r = 274.23
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 7.4998 (8) Å
b = 13.8350 (14) Å
c = 12.2324 (12) Å
β = 107.474 (4)°
V = 1210.7 (2) Å^3
Z = 4
$F(000)$ = 568
D_x = 1.505 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1288 reflections
θ = 2.3–25.0°
μ = 0.12 mm^{-1}
T = 293 K
Block, colourless
0.22 × 0.20 × 0.18 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scan
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.974, T_{max} = 0.979
18309 measured reflections
2126 independent reflections
1288 reflections with $I > 2\sigma(I)$
R_{int} = 0.057
θ_{max} = 25.0°, θ_{min} = 2.3°
h = −8→8
k = −16→16
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.176
S = 1.01
2091 reflections
181 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.083P)^2 + 0.9837P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O2	0.8511 (3)	−0.05625 (17)	1.07410 (18)	0.0501 (6)
N2	0.4933 (3)	0.21890 (17)	0.95459 (19)	0.0352 (6)
O5	0.2617 (3)	0.22615 (17)	0.78498 (18)	0.0523 (7)
O1	1.0686 (4)	−0.0637 (2)	1.2845 (2)	0.0662 (8)
H1	0.9808	−0.0823	1.2307	0.099*
C8	0.7065 (4)	0.0878 (2)	1.0007 (2)	0.0338 (7)
C12	0.6121 (4)	0.0460 (2)	0.8948 (2)	0.0361 (7)
H12	0.6469	−0.0148	0.8759	0.043*
N1	0.3812 (4)	0.0517 (2)	0.7088 (2)	0.0511 (8)
C9	0.6406 (4)	0.1738 (2)	1.0272 (2)	0.0357 (7)
H9	0.6993	0.2022	1.0978	0.043*
C11	0.4714 (4)	0.0938 (2)	0.8206 (2)	0.0372 (8)
C6	1.0100 (4)	0.0801 (2)	1.1684 (2)	0.0378 (8)
O3	0.3589 (5)	0.1021 (2)	0.6255 (2)	0.0864 (10)
C7	0.8572 (4)	0.0327 (2)	1.0816 (2)	0.0369 (7)
C10	0.3981 (4)	0.1837 (2)	0.8458 (2)	0.0373 (8)
O4	0.3364 (4)	−0.0328 (2)	0.7060 (2)	0.0750 (9)
C5	1.0684 (4)	0.1731 (3)	1.1538 (3)	0.0493 (9)
H5	1.0048	0.2080	1.0888	0.059*
C1	1.1098 (4)	0.0286 (3)	1.2664 (3)	0.0477 (9)
C13	0.4218 (5)	0.3087 (2)	0.9891 (3)	0.0538 (10)
H13A	0.3173	0.3312	0.9277	0.081*
H13B	0.5185	0.3568	1.0063	0.081*
H13C	0.3827	0.2970	1.0558	0.081*
C2	1.2540 (5)	0.0719 (4)	1.3476 (3)	0.0640 (11)
H2	1.3150	0.0388	1.4145	0.077*
C4	1.2182 (5)	0.2144 (3)	1.2333 (3)	0.0646 (11)
H4	1.2582	0.2761	1.2216	0.078*
C3	1.3088 (5)	0.1631 (4)	1.3310 (4)	0.0726 (13)
H3	1.4086	0.1913	1.3863	0.087*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.0543 (14)	0.0430 (15)	0.0478 (14)	0.0061 (11)	0.0074 (11)	0.0055 (11)
N2	0.0360 (14)	0.0363 (15)	0.0288 (13)	0.0027 (11)	0.0028 (11)	−0.0011 (11)
O5	0.0506 (14)	0.0564 (15)	0.0388 (13)	0.0125 (11)	−0.0036 (11)	0.0050 (11)
O1	0.0713 (17)	0.0667 (19)	0.0514 (15)	0.0189 (14)	0.0047 (13)	0.0210 (13)
C8	0.0338 (16)	0.0388 (18)	0.0273 (16)	−0.0004 (13)	0.0070 (13)	0.0012 (13)
C12	0.0378 (17)	0.0374 (18)	0.0327 (17)	−0.0006 (13)	0.0099 (14)	−0.0022 (13)

N1	0.0454 (16)	0.065 (2)	0.0345 (17)	0.0050 (15)	−0.0004 (13)	−0.0135 (16)
C9	0.0355 (16)	0.0421 (19)	0.0247 (15)	−0.0037 (14)	0.0018 (13)	−0.0010 (13)
C11	0.0394 (17)	0.0409 (19)	0.0275 (16)	−0.0027 (14)	0.0042 (14)	−0.0044 (13)
C6	0.0337 (16)	0.049 (2)	0.0291 (17)	0.0064 (14)	0.0073 (13)	0.0012 (14)
O3	0.121 (3)	0.096 (2)	0.0275 (14)	0.0062 (19)	0.0009 (15)	−0.0046 (15)
C7	0.0381 (17)	0.044 (2)	0.0299 (16)	0.0054 (14)	0.0117 (13)	0.0053 (14)
C10	0.0391 (17)	0.0402 (18)	0.0285 (16)	−0.0015 (14)	0.0042 (14)	0.0036 (14)
O4	0.0760 (19)	0.067 (2)	0.0690 (19)	−0.0128 (15)	0.0019 (14)	−0.0323 (15)
C5	0.0418 (19)	0.056 (2)	0.048 (2)	−0.0010 (16)	0.0095 (16)	−0.0015 (16)
C1	0.0411 (19)	0.065 (2)	0.0346 (18)	0.0132 (17)	0.0078 (15)	−0.0002 (17)
C13	0.060 (2)	0.048 (2)	0.047 (2)	0.0135 (17)	0.0068 (17)	−0.0123 (16)
C2	0.052 (2)	0.096 (3)	0.036 (2)	0.020 (2)	0.0003 (18)	−0.008 (2)
C4	0.045 (2)	0.072 (3)	0.072 (3)	−0.0133 (19)	0.010 (2)	−0.018 (2)
C3	0.043 (2)	0.105 (4)	0.058 (3)	0.001 (2)	−0.0036 (19)	−0.035 (3)

Geometric parameters (Å, º)

O2—C7	1.234 (4)	C11—C10	1.431 (4)
N2—C9	1.345 (4)	C6—C5	1.388 (5)
N2—C10	1.395 (4)	C6—C1	1.402 (4)
N2—C13	1.465 (4)	C6—C7	1.463 (4)
O5—C10	1.219 (3)	C5—C4	1.370 (5)
O1—C1	1.348 (4)	C5—H5	0.9300
O1—H1	0.8200	C1—C2	1.367 (5)
C8—C9	1.364 (4)	C13—H13A	0.9600
C8—C12	1.402 (4)	C13—H13B	0.9600
C8—C7	1.472 (4)	C13—H13C	0.9600
C12—C11	1.342 (4)	C2—C3	1.361 (6)
C12—H12	0.9300	C2—H2	0.9300
N1—O3	1.205 (4)	C4—C3	1.381 (6)
N1—O4	1.214 (4)	C4—H4	0.9300
N1—C11	1.453 (4)	C3—H3	0.9300
C9—H9	0.9300		
C9—N2—C10	123.5 (3)	O5—C10—N2	120.6 (3)
C9—N2—C13	120.2 (2)	O5—C10—C11	126.4 (3)
C10—N2—C13	116.3 (2)	N2—C10—C11	112.9 (2)
C1—O1—H1	109.5	C4—C5—C6	121.2 (3)
C9—C8—C12	117.5 (3)	C4—C5—H5	119.4
C9—C8—C7	123.5 (3)	C6—C5—H5	119.4
C12—C8—C7	118.7 (3)	O1—C1—C2	117.8 (3)
C11—C12—C8	119.8 (3)	O1—C1—C6	122.2 (3)
C11—C12—H12	120.1	C2—C1—C6	120.0 (4)
C8—C12—H12	120.1	N2—C13—H13A	109.5
O3—N1—O4	124.5 (3)	N2—C13—H13B	109.5
O3—N1—C11	118.2 (3)	H13A—C13—H13B	109.5
O4—N1—C11	117.3 (3)	N2—C13—H13C	109.5
N2—C9—C8	122.1 (3)	H13A—C13—H13C	109.5

N2—C9—H9	118.9	H13B—C13—H13C	109.5
C8—C9—H9	118.9	C3—C2—C1	120.5 (4)
C12—C11—C10	123.9 (3)	C3—C2—H2	119.8
C12—C11—N1	119.2 (3)	C1—C2—H2	119.8
C10—C11—N1	116.8 (3)	C5—C4—C3	119.1 (4)
C5—C6—C1	118.3 (3)	C5—C4—H4	120.5
C5—C6—C7	122.0 (3)	C3—C4—H4	120.5
C1—C6—C7	119.6 (3)	C2—C3—C4	120.8 (3)
O2—C7—C6	120.3 (3)	C2—C3—H3	119.6
O2—C7—C8	117.6 (3)	C4—C3—H3	119.6
C6—C7—C8	122.1 (3)		

C9—C8—C12—C11	4.7 (4)	C9—N2—C10—O5	177.7 (3)
C7—C8—C12—C11	178.5 (3)	C13—N2—C10—O5	−0.9 (4)
C10—N2—C9—C8	−1.5 (5)	C9—N2—C10—C11	1.1 (4)
C13—N2—C9—C8	177.0 (3)	C13—N2—C10—C11	−177.5 (3)
C12—C8—C9—N2	−1.4 (4)	C12—C11—C10—O5	−173.9 (3)
C7—C8—C9—N2	−174.9 (3)	N1—C11—C10—O5	3.9 (5)
C8—C12—C11—C10	−5.4 (5)	C12—C11—C10—N2	2.4 (4)
C8—C12—C11—N1	176.9 (3)	N1—C11—C10—N2	−179.8 (3)
O3—N1—C11—C12	−131.4 (3)	C1—C6—C5—C4	−0.8 (5)
O4—N1—C11—C12	47.3 (4)	C7—C6—C5—C4	−176.5 (3)
O3—N1—C11—C10	50.7 (4)	C5—C6—C1—O1	−176.5 (3)
O4—N1—C11—C10	−130.6 (3)	C7—C6—C1—O1	−0.7 (5)
C5—C6—C7—O2	153.2 (3)	C5—C6—C1—C2	3.5 (5)
C1—C6—C7—O2	−22.5 (4)	C7—C6—C1—C2	179.4 (3)
C5—C6—C7—C8	−26.8 (4)	O1—C1—C2—C3	176.3 (3)
C1—C6—C7—C8	157.5 (3)	C6—C1—C2—C3	−3.7 (5)
C9—C8—C7—O2	147.5 (3)	C6—C5—C4—C3	−1.7 (6)
C12—C8—C7—O2	−25.8 (4)	C1—C2—C3—C4	1.2 (6)
C9—C8—C7—C6	−32.4 (4)	C5—C4—C3—C2	1.6 (6)
C12—C8—C7—C6	154.2 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2	0.82	1.90	2.603 (3)	142
C4—H4···O1^{i}	0.93	2.58	3.498 (5)	169
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