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4-Oxo-*N*-phenyl-1,4-dihydropyridine-3-carboxamide

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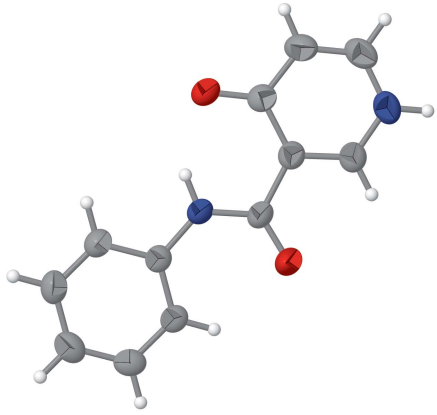
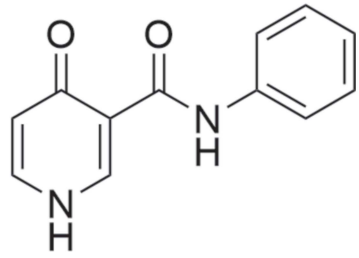
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{12}H_{10}N_2O_2$, shows a nearly planar conformation. The crystal structure is sustained by hydrogen bonds between the NH and the carbonyl O function of the 4-oxo-1,4-dihydropyridine ring of the molecules, forming infinite chains along the *b*-axis direction.

3D view



Chemical scheme



Structure description

The title compound (Fig. 1) is a derivative of *N*-phenylnicotinamide, which is an efficient molluscicide (Dunlop *et al.*, 1980). In addition, the compound can be used as a raw material for many chemical reactions. We were interested in its solid-state behavior since it is a structural isomer of *N*-phenyl-2-hydroxynicotinanilide, which has interesting structural properties (Zhoujin *et al.*, 2021). In our study, the compound was synthesized by an amide condensation reaction (Narajan *et al.*, 2016), and single crystals were obtained by slow evaporation of an acetone solution of the compound. The compound has a nearly planar conformation as evidenced by the dihedral angle between the 4-oxo-1,4-dihydropyridine and benzene rings of 6.80 (8)°. An intramolecular hydrogen bond is formed between the NH of the amide and the carbonyl O atom on the 4-oxo-1,4-dihydropyridine ring. In the crystal, the molecules form chains along the *b*-axis direction through hydrogen bonds between the NH group and the carbonyl O atom of the 4-oxo-1,4-dihydropyridine ring (Fig. 2, Table 1).

Synthesis and crystallization

4-Hydroxynicotinic acid (0.51 g, 3.67 mmol), 1-ethyl-3-(3-dimethylaminopropyl)carbodiimide hydrochloride (EDC, 1.06 g, 5.51 mmol) and hydroxybenzotriazole (HOBT, 0.60 g, 4.40 mmol) were dissolved in 6 ml of DMF and stirred at 0°C for 1 h. Then diisopropylethylamine (DIPEA, 0.95 g, 7.34 mmol) and aniline (0.28 ml, 3.67 mmol) were

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.91 (2)	1.84 (3)	2.744 (2)	171 (2)
C8—H8···O2	0.93	2.27	2.861 (2)	121
N2—H2···O1	0.88 (3)	1.91 (2)	2.6731 (18)	144 (2)

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

added and the reaction was completed under continuous stirring at 50°C for 12 h. Then 20 ml of deionized water were added to the reaction mixture, which was placed into a refrigerator at 5°C overnight. The resulting precipitate was collected by filtration and washed with deionized water to obtain 0.306 g (39% based on 4-hydroxynicotinic acid) of the title compound (Fig. 3). The obtained compound was fully

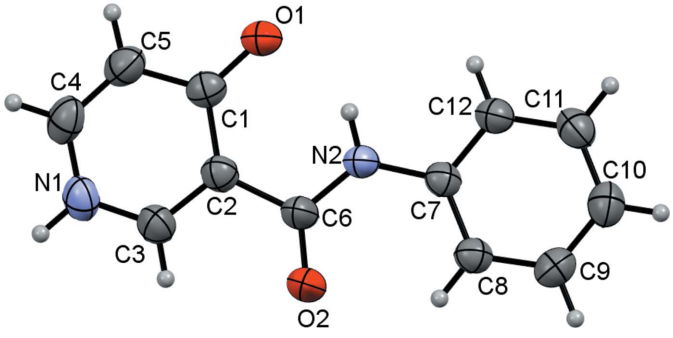


Figure 1
Molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level.

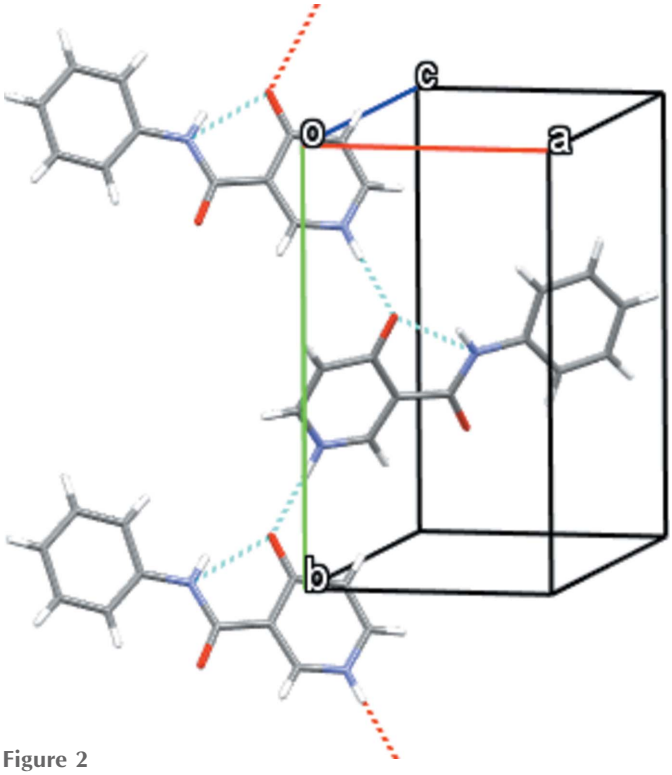


Figure 2
Packing of the molecules in the title compound viewed along the *b* axis.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{12}H_{10}N_2O_2$
M_r	214.22
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	276
a, b, c (Å)	6.68228 (10), 11.79159 (18), 13.1717 (2)
V (Å^3)	1037.86 (3)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.79
Crystal size (mm)	0.1 × 0.07 × 0.05
Data collection	
Diffractometer	Rigaku Oxford Diffraction, Synergy Custom system, HyPix
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2021)
T_{min}, T_{max}	0.864, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	6101, 2075, 2003
R_{int}	0.019
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.632
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.030, 0.083, 1.06
No. of reflections	2075
No. of parameters	154
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.21, −0.14
Absolute structure	Flack x determined using 788 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	0.11 (8)

Computer programs: *CrysAlis PRO* (Rigaku OD, 2021), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2018/3* (Sheldrick, 2015*b*), *OLEX2* (Dolomanov *et al.*, 2009) and *Mercury* (Macrae *et al.*, 2020).

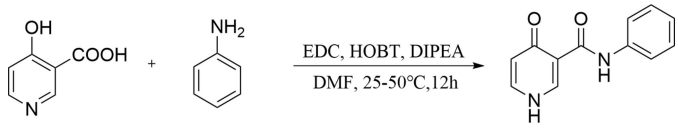


Figure 3
Reaction scheme.

Figure 4
A representative crystal of the title compound.

dissolved in acetone under ultrasound until a clear solution was obtained, which was then filtered into a transparent glass bottle. The bottle was placed in a fume hood for slow evaporation of the solvent. Colorless rod-shaped crystals (Fig. 4) were obtained in a few days.

Refinement

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

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full crystallographic data

IUCrData (2023). **8**, x230601 [https://doi.org/10.1107/S2414314623006016]

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Crystal data

$C_{12}H_{10}N_2O_2$
M_r = 214.22
Orthorhombic, $P2_12_12_1$
a = 6.68228 (10) Å
b = 11.79159 (18) Å
c = 13.1717 (2) Å
V = 1037.86 (3) Å^3
Z = 4
$F(000)$ = 448
D_x = 1.371 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 5537 reflections
θ = 3.3–77.1°
μ = 0.79 mm^{-1}
T = 276 K
Block, clear light colourless
0.1 × 0.07 × 0.05 mm

Data collection

Rigaku Oxford Diffraction, Synergy Custom system, HyPix diffractometer
Radiation source: Rotating-anode X-ray tube, Rigaku (Cu) X-ray Source
Mirror monochromator
Detector resolution: 10.0000 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2021)
T_{min} = 0.864, T_{max} = 1.000
6101 measured reflections
2075 independent reflections
2003 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 77.2°, θ_{min} = 5.0°
h = −8→8
k = −13→14
l = −14→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.083
S = 1.06
2075 reflections
154 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0478P)^2 + 0.0929P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}
Extinction correction: SHELXL-2018/3 (Sheldrick 2015b), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.027 (3)
Absolute structure: Flack x determined using 788 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.11 (8)

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. The positions of H atoms at N1 and N2 were obtained from the difference Fourier map and were refined freely. Other H atoms were positioned geometrically with C—H = 0.93 Å and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2 U_{eq}(C) (Table 2).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.25139 (19)	0.41568 (10)	0.25242 (11)	0.0504 (4)
O2	0.4568 (3)	0.68655 (14)	0.4334 (2)	0.0985 (8)
N1	−0.0559 (2)	0.71242 (14)	0.26837 (14)	0.0522 (4)
H1	−0.122 (4)	0.780 (2)	0.2688 (18)	0.060 (6)*
N2	0.5233 (2)	0.50907 (12)	0.37556 (12)	0.0418 (3)
C1	0.1596 (3)	0.50951 (14)	0.25749 (13)	0.0415 (4)
C2	0.2281 (3)	0.60428 (14)	0.31705 (14)	0.0417 (4)
C3	0.1157 (3)	0.70093 (15)	0.32024 (16)	0.0497 (4)
H3	0.159842	0.761165	0.359932	0.060*
C4	−0.1229 (3)	0.62714 (18)	0.20986 (16)	0.0553 (5)
H4	−0.240686	0.636483	0.173182	0.066*
C5	−0.0226 (3)	0.52847 (17)	0.20341 (15)	0.0534 (5)
H5	−0.073310	0.470945	0.162547	0.064*
C6	0.4136 (3)	0.60500 (15)	0.38116 (17)	0.0485 (5)
C7	0.7012 (2)	0.48310 (14)	0.42740 (12)	0.0372 (4)
C8	0.8102 (3)	0.56149 (14)	0.48339 (13)	0.0414 (4)
H8	0.766564	0.636194	0.488177	0.050*
C9	0.9846 (3)	0.52788 (17)	0.53218 (15)	0.0489 (4)
H9	1.056878	0.580418	0.569958	0.059*
C10	1.0519 (3)	0.41749 (18)	0.52534 (16)	0.0534 (5)
H10	1.168367	0.395585	0.558657	0.064*
C11	0.9447 (3)	0.33961 (16)	0.46848 (15)	0.0512 (5)
H11	0.989888	0.265248	0.463118	0.061*
C12	0.7708 (3)	0.37210 (15)	0.41965 (14)	0.0447 (4)
H12	0.699818	0.319469	0.381348	0.054*
H2	0.471 (3)	0.455 (2)	0.3385 (19)	0.061 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0506 (7)	0.0403 (6)	0.0602 (8)	−0.0014 (5)	−0.0053 (6)	−0.0110 (5)
O2	0.0752 (11)	0.0582 (10)	0.162 (2)	0.0273 (8)	−0.0592 (12)	−0.0567 (11)
N1	0.0498 (8)	0.0454 (8)	0.0614 (10)	0.0053 (7)	−0.0058 (7)	0.0124 (7)
N2	0.0383 (7)	0.0354 (7)	0.0518 (8)	−0.0002 (6)	−0.0030 (6)	−0.0095 (6)
C1	0.0434 (8)	0.0399 (8)	0.0411 (8)	−0.0060 (7)	0.0023 (7)	0.0022 (7)
C2	0.0404 (8)	0.0369 (8)	0.0478 (9)	−0.0013 (6)	0.0012 (7)	0.0006 (7)

C3	0.0482 (9)	0.0395 (9)	0.0613 (11)	0.0018 (7)	−0.0056 (8)	0.0019 (8)
C4	0.0516 (10)	0.0588 (11)	0.0555 (11)	−0.0042 (9)	−0.0138 (9)	0.0172 (9)
C5	0.0586 (11)	0.0510 (10)	0.0506 (10)	−0.0091 (9)	−0.0146 (9)	0.0035 (8)
C6	0.0429 (9)	0.0349 (8)	0.0677 (12)	0.0013 (7)	−0.0071 (8)	−0.0117 (8)
C7	0.0363 (7)	0.0371 (8)	0.0383 (7)	−0.0004 (6)	0.0048 (6)	−0.0016 (6)
C8	0.0422 (8)	0.0374 (8)	0.0445 (9)	−0.0024 (6)	0.0020 (7)	−0.0028 (7)
C9	0.0469 (9)	0.0532 (10)	0.0465 (9)	−0.0069 (8)	−0.0044 (8)	−0.0033 (8)
C10	0.0470 (9)	0.0621 (11)	0.0510 (10)	0.0061 (8)	−0.0060 (8)	0.0042 (9)
C11	0.0550 (10)	0.0451 (9)	0.0536 (11)	0.0116 (8)	0.0016 (9)	−0.0004 (8)
C12	0.0483 (9)	0.0376 (8)	0.0482 (9)	0.0002 (7)	−0.0003 (8)	−0.0051 (7)

Geometric parameters (Å, º)

O1—C1	1.267 (2)	C4—C5	1.346 (3)
O2—C6	1.217 (2)	C5—H5	0.9300
N1—H1	0.91 (2)	C7—C8	1.389 (2)
N1—C3	1.341 (3)	C7—C12	1.393 (2)
N1—C4	1.344 (3)	C8—H8	0.9300
N2—C6	1.350 (2)	C8—C9	1.388 (2)
N2—C7	1.405 (2)	C9—H9	0.9300
N2—H2	0.88 (3)	C9—C10	1.380 (3)
C1—C2	1.440 (2)	C10—H10	0.9300
C1—C5	1.428 (2)	C10—C11	1.384 (3)
C2—C3	1.365 (2)	C11—H11	0.9300
C2—C6	1.500 (2)	C11—C12	1.382 (3)
C3—H3	0.9300	C12—H12	0.9300
C4—H4	0.9300		
C3—N1—H1	120.0 (15)	O2—C6—N2	124.36 (17)
C3—N1—C4	120.09 (17)	O2—C6—C2	121.23 (16)
C4—N1—H1	119.8 (15)	N2—C6—C2	114.41 (15)
C6—N2—C7	128.00 (15)	C8—C7—N2	123.83 (15)
C6—N2—H2	115.0 (16)	C8—C7—C12	119.29 (15)
C7—N2—H2	116.8 (16)	C12—C7—N2	116.87 (15)
O1—C1—C2	123.54 (16)	C7—C8—H8	120.1
O1—C1—C5	121.55 (16)	C9—C8—C7	119.73 (16)
C5—C1—C2	114.91 (16)	C9—C8—H8	120.1
C1—C2—C6	125.01 (15)	C8—C9—H9	119.6
C3—C2—C1	119.31 (16)	C10—C9—C8	120.84 (17)
C3—C2—C6	115.63 (15)	C10—C9—H9	119.6
N1—C3—C2	122.60 (17)	C9—C10—H10	120.3
N1—C3—H3	118.7	C9—C10—C11	119.48 (17)
C2—C3—H3	118.7	C11—C10—H10	120.3
N1—C4—H4	119.4	C10—C11—H11	119.9
N1—C4—C5	121.13 (17)	C12—C11—C10	120.18 (18)
C5—C4—H4	119.4	C12—C11—H11	119.9
C1—C5—H5	119.0	C7—C12—H12	119.8
C4—C5—C1	121.92 (17)	C11—C12—C7	120.47 (17)

C4—C5—H5	119.0	C11—C12—H12	119.8
O1—C1—C2—C3	−178.02 (16)	C5—C1—C2—C3	1.9 (3)
O1—C1—C2—C6	−0.6 (3)	C5—C1—C2—C6	179.37 (17)
O1—C1—C5—C4	178.76 (18)	C6—N2—C7—C8	−9.9 (3)
N1—C4—C5—C1	−0.4 (3)	C6—N2—C7—C12	171.15 (18)
N2—C7—C8—C9	179.95 (16)	C6—C2—C3—N1	−178.90 (18)
N2—C7—C12—C11	−179.93 (16)	C7—N2—C6—O2	0.9 (4)
C1—C2—C3—N1	−1.2 (3)	C7—N2—C6—C2	−178.75 (16)
C1—C2—C6—O2	−176.4 (2)	C7—C8—C9—C10	0.4 (3)
C1—C2—C6—N2	3.3 (3)	C8—C7—C12—C11	1.1 (3)
C2—C1—C5—C4	−1.2 (3)	C8—C9—C10—C11	0.4 (3)
C3—N1—C4—C5	1.2 (3)	C9—C10—C11—C12	−0.5 (3)
C3—C2—C6—O2	1.2 (3)	C10—C11—C12—C7	−0.3 (3)
C3—C2—C6—N2	−179.15 (17)	C12—C7—C8—C9	−1.1 (2)
C4—N1—C3—C2	−0.4 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.91 (2)	1.84 (3)	2.744 (2)	171 (2)
C8—H8···O2	0.93	2.27	2.861 (2)	121
N2—H2···O1	0.88 (3)	1.91 (2)	2.6731 (18)	144 (2)

Symmetry code: (i) −*x*, *y*+1/2, −*z*+1/2.