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ISSN 2414-3146

N-(3-Chloro-2-methylphenyl)-6-oxo-1,6-dihydropyridine-3-carboxamide

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Received 23 May 2023
Accepted 7 July 2023

Edited by L. Van Meervelt, Katholieke Universiteit Leuven, Belgium

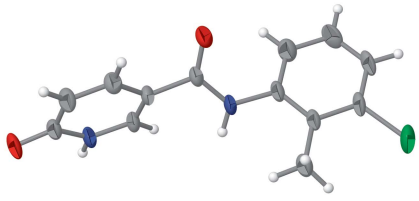
Keywords: crystal structure; 6-oxo-1,6-dihydropyridine; N—H···O hydrogen bonds.

CCDC reference: 2280200

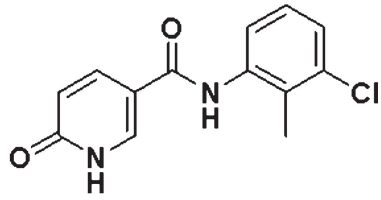
Structural data: full structural data are available from iucrdata.iucr.org

In the crystal structure of the title compound, $C_{13}H_{11}ClN_2O_2$, the molecules form a three-dimensional network based on two types of hydrogen bonds between NH groups and the carbonyl oxygen atoms and amides. The molecule is highly twisted, as evidenced by the dihedral angle between the 6-oxo-1,6-dihydropyridine and benzene rings [88.1 (2)°].

3D view



Chemical scheme



Structure description

The title compound (Fig. 1) is a structural isomer of *N*-phenyl-2-hydroxynicotinanilide, which has interesting structural properties (Liu *et al.*, 2020; Zhoujin *et al.*, 2021). We wondered if isomerization would lead to completely different synthons in the crystal structure. In our study, crystals were obtained by slowly evaporating a pyridine solution of the title compound. The molecule is highly twisted, as evidenced by the dihedral angle between the 6-oxo-1,6-dihydropyridine and benzene rings [88.1 (2)°]. In the crystal, the molecules form chains running in the *a*-axis direction through hydrogen bonds between NH groups and the carbonyl oxygen atoms of the amides (Fig. 2, Table 1). The 6-oxo-1,6-dihydropyridine rings form dimers through additional N—H···O hydrogen bonds.

Synthesis and crystallization

The title compound was synthesized with 6-oxo-1,6-dihydropyridine-3-carboxylic acid and 3-chloro-2-methylaniline as starting materials (Fig. 3). The pure sample was dissolved in bulk pyridine at 323 K, and the resulting solution was left in a refrigerator. Colorless block-shaped crystals (Fig. 4) were harvested after several days.

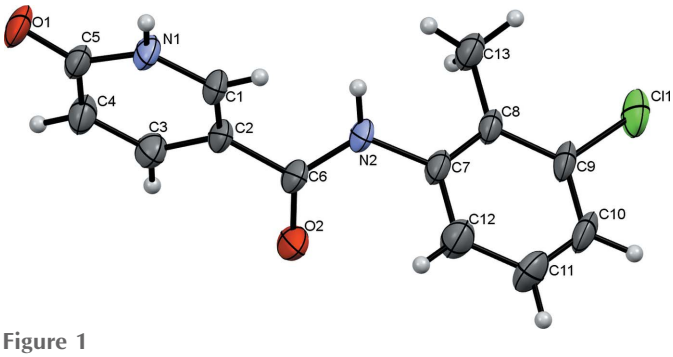


Figure 1
Molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level.

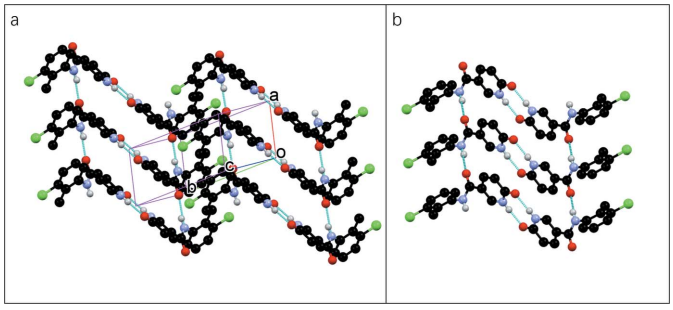


Figure 2
(*a*) Packing of the molecules in the title compound; (*b*) chain and dimer formation supported by intermolecular N–H···O═C hydrogen bonds (indicated by blue dashed lines).

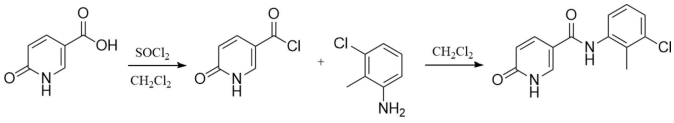


Figure 3
Synthesis of the title compound.

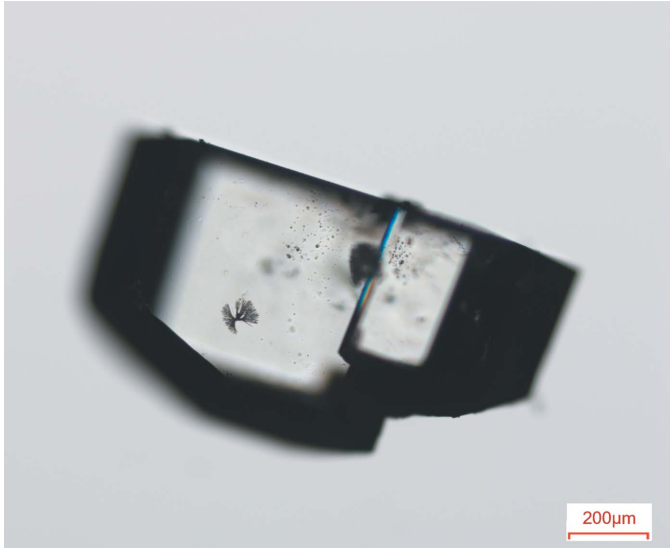


Figure 4
A representative crystal of the title compound.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.86	1.93	2.793 (3)	177
N2—H2···O2^{ii}	0.86	2.08	2.926 (3)	166

Symmetry codes: (i) $-x, -y, -z$; (ii) $x-1, y, z$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{13}H_{11}ClN_2O_2$
M_r	262.69
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	268
a, b, c (Å)	4.91237 (15), 10.3037 (3), 12.5876 (3)
α, β, γ (°)	105.890 (2), 96.422 (2), 99.361 (2)
V (Å^3)	596.35 (3)
Z	2
Radiation type	Cu $K\alpha$
μ (mm^{-1})	2.81
Crystal size (mm)	0.11 × 0.05 × 0.04
Data collection	
Diffractometer	Rigaku Oxford Diffraction, Synergy Custom system, HyPix
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2021)
T_{min}, T_{max}	0.482, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	5870, 2362, 2023
R_{int}	0.059
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.633
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.060, 0.184, 1.09
No. of reflections	2362
No. of parameters	164
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.34, −0.47

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

Refinement

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

References

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full crystallographic data

IUCrData (2023). **8**, x230602 [https://doi.org/10.1107/S2414314623006028]

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Crystal data

$C_{13}H_{11}ClN_2O_2$
$M_r = 262.69$
Triclinic, $P\overline{1}$
$a = 4.91237$ (15) Å
$b = 10.3037$ (3) Å
$c = 12.5876$ (3) Å
$\alpha = 105.890$ (2)°
$\beta = 96.422$ (2)°
$\gamma = 99.361$ (2)°
$V = 596.35$ (3) Å^3
$Z = 2$
$F(000) = 272$
$D_x = 1.463$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 4316 reflections
$\theta = 3.7$–$77.4°$
$\mu = 2.81$ mm^{-1}
$T = 268$ K
Block, clear light colourless
0.11 × 0.05 × 0.04 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)
$T_{min} = 0.482$, $T_{max} = 1.000$
5870 measured reflections
2362 independent reflections
2023 reflections with $I > 2\sigma(I)$
$R_{int} = 0.059$
$\theta_{max} = 77.6°$, $\theta_{min} = 3.7°$
$h = -6\rightarrow6$
$k = -12\rightarrow12$
$l = -15\rightarrow11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.060$
$wR(F^2) = 0.184$
$S = 1.09$
2362 reflections
164 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1286P)^2 + 0.0079P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.34$ e Å^{-3}
$\Delta\rho_{min} = -0.47$ 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. positions of H atoms at N1 and N2 were obtained from a difference Fourier map. Other H atoms were positioned geometrically with C—H = 0.93 Å (aromatic H) or 0.96 Å (methyl H), and constrained to ride on their parent atoms, with U_{iso}(H) = xU_{eq}(C,*O*), where *x*=1.5 for methyl H, and *x*=1.2 for all other H atoms.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.70169 (15)	0.48597 (7)	0.88602 (4)	0.0568 (3)
O1	0.2394 (4)	0.10879 (18)	−0.04783 (12)	0.0467 (4)
O2	1.0545 (3)	0.2472 (2)	0.40507 (14)	0.0554 (5)
N1	0.2702 (4)	0.07879 (18)	0.12455 (14)	0.0351 (4)
H1	0.113762	0.020388	0.103199	0.042*
N2	0.6373 (4)	0.23089 (19)	0.46700 (13)	0.0367 (4)
H2	0.459644	0.221023	0.448300	0.044*
C1	0.3982 (4)	0.1067 (2)	0.23115 (16)	0.0344 (5)
H1A	0.313595	0.064270	0.278844	0.041*
C2	0.6488 (4)	0.1959 (2)	0.27013 (16)	0.0327 (4)
C3	0.7723 (5)	0.2563 (2)	0.19387 (18)	0.0387 (5)
H3	0.946826	0.314972	0.217320	0.046*
C4	0.6407 (5)	0.2301 (2)	0.08749 (18)	0.0403 (5)
H4	0.724547	0.272452	0.039606	0.048*
C5	0.3741 (4)	0.1379 (2)	0.04762 (16)	0.0338 (5)
C6	0.7985 (4)	0.2265 (2)	0.38631 (17)	0.0357 (5)
C7	0.7463 (4)	0.2510 (2)	0.58157 (16)	0.0330 (5)
C8	0.6758 (4)	0.3534 (2)	0.66538 (16)	0.0312 (4)
C9	0.7869 (5)	0.3634 (2)	0.77585 (17)	0.0369 (5)
C10	0.9636 (5)	0.2817 (3)	0.80184 (19)	0.0436 (5)
H10	1.034339	0.292535	0.876128	0.052*
C11	1.0340 (6)	0.1845 (3)	0.7173 (2)	0.0498 (6)
H11	1.156189	0.129966	0.733910	0.060*
C12	0.9234 (6)	0.1669 (2)	0.6069 (2)	0.0465 (6)
H12	0.967423	0.098984	0.549550	0.056*
C13	0.4974 (5)	0.4497 (2)	0.64070 (18)	0.0413 (5)
H13A	0.614874	0.532952	0.638879	0.062*
H13B	0.384069	0.471175	0.697991	0.062*
H13C	0.378964	0.406824	0.569413	0.062*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0737 (5)	0.0721 (5)	0.0188 (4)	0.0169 (3)	0.0033 (3)	0.0046 (3)
O1	0.0527 (10)	0.0628 (10)	0.0219 (8)	0.0063 (7)	−0.0090 (7)	0.0172 (7)
O2	0.0327 (9)	0.1011 (14)	0.0257 (9)	0.0141 (8)	−0.0021 (6)	0.0103 (9)
N1	0.0348 (9)	0.0473 (9)	0.0185 (9)	0.0040 (7)	−0.0040 (6)	0.0080 (7)
N2	0.0337 (9)	0.0570 (10)	0.0151 (9)	0.0098 (7)	−0.0028 (6)	0.0060 (7)
C1	0.0374 (11)	0.0466 (10)	0.0172 (10)	0.0085 (8)	0.0000 (8)	0.0078 (8)
C2	0.0349 (10)	0.0440 (10)	0.0158 (9)	0.0105 (8)	−0.0012 (7)	0.0037 (8)
C3	0.0354 (11)	0.0483 (11)	0.0284 (11)	0.0051 (8)	0.0008 (8)	0.0087 (9)

C4	0.0455 (12)	0.0502 (11)	0.0258 (11)	0.0050 (9)	0.0024 (9)	0.0162 (9)
C5	0.0400 (11)	0.0406 (10)	0.0211 (10)	0.0116 (8)	−0.0009 (8)	0.0101 (8)
C6	0.0344 (11)	0.0491 (11)	0.0186 (10)	0.0098 (8)	−0.0036 (8)	0.0045 (8)
C7	0.0355 (10)	0.0430 (10)	0.0184 (10)	0.0052 (8)	−0.0026 (7)	0.0102 (8)
C8	0.0344 (10)	0.0409 (10)	0.0181 (10)	0.0031 (7)	0.0002 (7)	0.0125 (8)
C9	0.0455 (12)	0.0473 (11)	0.0155 (10)	0.0026 (8)	−0.0009 (8)	0.0116 (8)
C10	0.0500 (13)	0.0597 (13)	0.0229 (11)	0.0053 (10)	−0.0048 (9)	0.0227 (10)
C11	0.0577 (15)	0.0569 (13)	0.0415 (14)	0.0171 (11)	−0.0031 (11)	0.0269 (12)
C12	0.0582 (15)	0.0492 (12)	0.0333 (13)	0.0196 (10)	0.0010 (10)	0.0117 (10)
C13	0.0504 (13)	0.0490 (11)	0.0247 (11)	0.0157 (9)	−0.0013 (9)	0.0114 (9)

Geometric parameters (Å, º)

Cl1—C9	1.743 (2)	C3—C4	1.355 (3)
O1—C5	1.238 (2)	C4—C5	1.434 (3)
O2—C6	1.225 (3)	C7—C8	1.391 (3)
N1—C1	1.349 (3)	C7—C12	1.394 (3)
N1—C5	1.379 (3)	C8—C9	1.406 (3)
N2—C6	1.351 (3)	C8—C13	1.497 (3)
N2—C7	1.427 (2)	C9—C10	1.376 (3)
C1—C2	1.360 (3)	C10—C11	1.366 (4)
C2—C3	1.418 (3)	C11—C12	1.387 (3)
C2—C6	1.487 (3)		
C1—N1—C5	124.19 (18)	N2—C6—C2	116.39 (17)
C6—N2—C7	123.50 (17)	C8—C7—N2	119.96 (17)
N1—C1—C2	121.18 (19)	C8—C7—C12	121.34 (18)
C1—C2—C3	117.44 (18)	C12—C7—N2	118.70 (19)
C1—C2—C6	122.53 (18)	C7—C8—C9	116.00 (18)
C3—C2—C6	119.98 (18)	C7—C8—C13	122.62 (17)
C4—C3—C2	121.2 (2)	C9—C8—C13	121.36 (19)
C3—C4—C5	120.99 (19)	C8—C9—Cl1	118.95 (17)
O1—C5—N1	119.92 (19)	C10—C9—Cl1	117.88 (16)
O1—C5—C4	125.14 (19)	C10—C9—C8	123.2 (2)
N1—C5—C4	114.93 (17)	C11—C10—C9	119.3 (2)
O2—C6—N2	123.42 (19)	C10—C11—C12	120.1 (2)
O2—C6—C2	120.18 (19)	C11—C12—C7	120.1 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.86	1.93	2.793 (3)	177
N2—H2···O2^{ii}	0.86	2.08	2.926 (3)	166

Symmetry codes: (i) −*x*, −*y*, −*z*; (ii) *x*−1, *y*, *z*.