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

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Key indicators

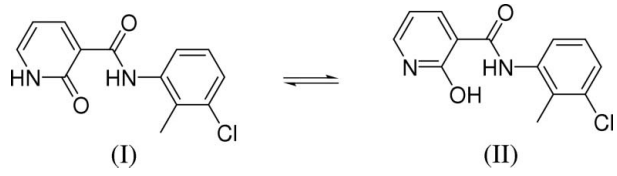
Single-crystal X-ray study
T = 90 K
Mean σ(C–C) = 0.003 Å
R factor = 0.045
wR factor = 0.112
Data-to-parameter ratio = 15.6

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

The title compound, $C_{13}H_{11}ClN_2O_2$, contains two aromatic rings linked by an amide group, and adopts a near-planar conformation. There are intra- and intermolecular N—H···O hydrogen bonds, forming centrosymmetric dimers.

Comment

The title compound, (I), was first synthesized by Alvarez *et al.* (1979) in an attempt to prepare substituted 2-hydroxynicotinamide derivatives. We obtained (I) as a by-product during efforts to make 2-(3-chloro-2-methylanilino)nicotinic acid *via* a procedure modified from Ting *et al.* (1990). It was thought to be a hydroxy–pyridine tautomer, (II), but the crystal structure analysis revealed that it is the keto–amine tautomer, (I) (Fig. 1 and Table 1). The molecule contains two aromatic rings linked by an amide group. Since it can form a π-conjugation system throughout the whole molecule *via* the amide group, the molecule has a near-planar conformation. There are intra- and intermolecular N—H···O hydrogen bonds (Table 2), forming centrosymmetric dimers.



Experimental

2-Chloronicotinic acid (18.2 g, 0.11 mol) and 3-chloro-2-methylaniline (16.35 g, 0.11 mol) were dissolved in pyridine (10 ml, 0.12 mol), followed by addition of *p*-toluenesulfonic acid (3.0 g, 0.018 mol) in 40 ml of water. The resulting solution was refluxed

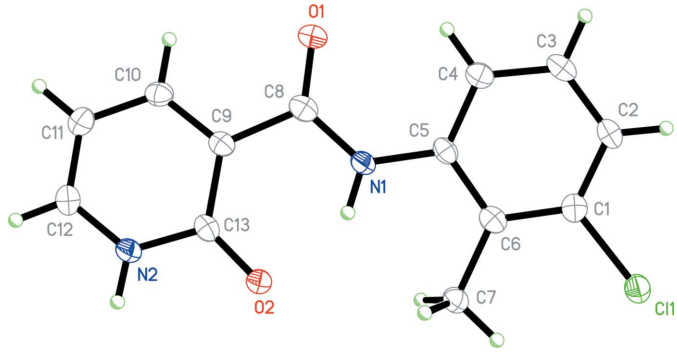


Figure 1
The molecular structure of (I), showing displacement ellipsoids at the 50% probability level.

overnight. Upon cooling of the solution, a colorless solid precipitated out and it was characterized by NMR to be the compound (I). Crystals were grown by slow evaporation of a methanol solution (yield 32%, m.p. 583–587 K).

Crystal data

$C_{13}H_{11}ClN_2O_2$
$M_r = 262.69$
Triclinic, $P\overline{1}$
$a = 7.1514$ (2) Å
$b = 7.5805$ (2) Å
$c = 10.3268$ (3) Å
$\alpha = 87.8186$ (14)°
$\beta = 89.5131$ (14)°
$\gamma = 89.2959$ (13)°
$V = 559.36$ (3) Å^3
$Z = 2$
$D_x = 1.560$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.34$ mm^{-1}
$T = 90.0$ (2) K
Irregular block, colorless
0.20 × 0.10 × 0.03 mm

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
$T_{min} = 0.936$, $T_{max} = 0.990$
4917 measured reflections
2560 independent reflections
1811 reflections with $I > 2\sigma(I)$
$R_{int} = 0.048$
$\theta_{max} = 27.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.112$
$S = 1.04$
2560 reflections
164 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0508P)^2 + 0.1363P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.31$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C9—C10	1.374 (3)	C12—N2	1.350 (3)
C9—C13	1.442 (3)	C13—O2	1.261 (2)
C10—C11	1.404 (3)	C13—N2	1.370 (3)
C11—C12	1.351 (3)		
N1—C8—C9—C13	4.8 (3)	C4—C5—N1—C8	4.1 (3)
C9—C8—N1—C5	179.65 (19)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2	0.88	1.89	2.655 (2)	144
N2—H2A···O2^{i}	0.88	1.92	2.790 (2)	170

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

H atoms were located in difference Fourier maps and subsequently placed in idealized positions, with constrained C—H distances of 0.95 (atomatic), 0.98 (methyl) and 0.88 Å (NH). U_{iso}(H) values were set at $1.2U_{eq}$(C,N) or $1.5U_{eq}$(methyl C).

Data collection: *COLLECT* (Nonius, 2002); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL/PC* (Sheldrick, 1995); software used to prepare material for publication: *SHELXL97* and local procedures.

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