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2-Amino-*N*-(2-chloropyridin-3yl)benzamide

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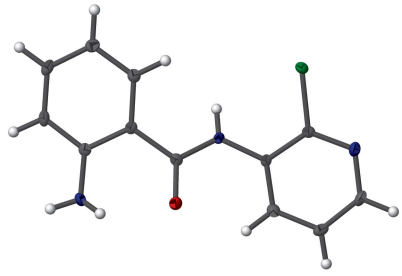
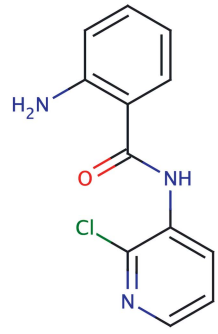
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{12}H_{10}ClN_3O$, is a condensation product of 3-amino-2-chloropyridine and ethyl 2-aminobenzoate in which the aromatic rings are almost coplanar [dihedral angle = 2.28 (9)°] and an intramolecular N—H···O hydrogen bond occurs. In the crystal, N—H···O and N—H···N hydrogen bonds link the molecules into (100) sheets.

3D view



Chemical scheme



Structure description

The title compound results from our research on subtype-selective ligands for muscarinic receptors (Mohr *et al.*, 2004, 2010). It was isolated in 23% yield as a ring-opened side product in a condensation reaction between 3-amino-2-chloropyridine and ethyl 2-aminobenzoate (Holzgrabe & Heller, 2003; Riad *et al.*, 2015).

The molecular structure is shown in Fig. 1. As expected, the central amide group adopts an almost planar orientation (O=C—N—H torsion angle = 174°). The C12—C7—C6=O1 torsion angle is 145.9 (2)° and an intramolecular N—H···O hydrogen bond (Table 1) closes an *S*(6) ring. The aromatic rings are essentially coplanar [dihedral angle = 2.28 (9)°].

In the crystal, molecules are linked by N—H···O and N—H···N hydrogen bonds to generate (100) sheets (Table 1, Fig. 2).

Synthesis and crystallization

The title compound was synthesized according to our previously reported procedure (Riad *et al.*, 2017). Colourless blocks were obtained by recrystallization from a solvent mixture of methanol and toluene.

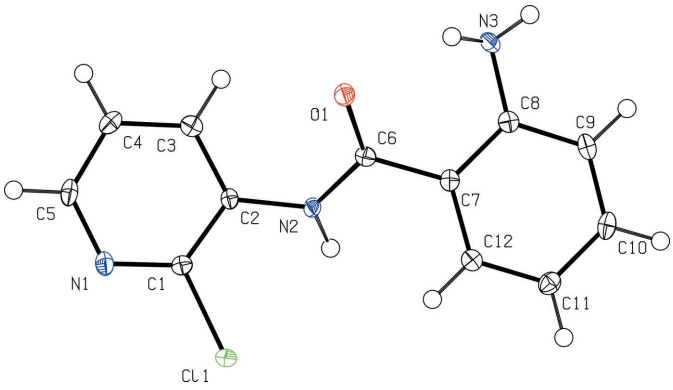


Figure 1
The molecular structure, with displacement ellipsoids drawn at the 50% probability level.

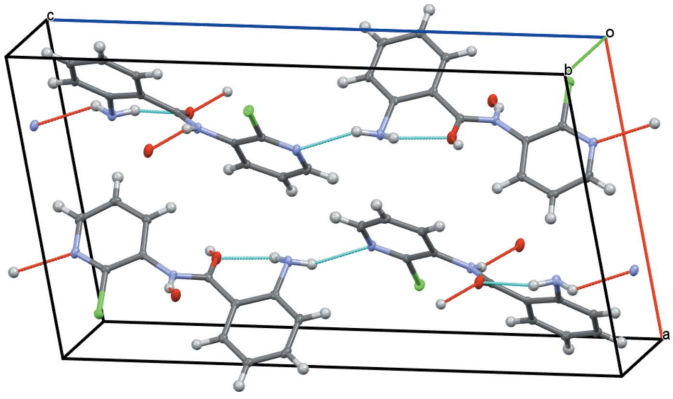


Figure 2
A view along the *c* axis of the packing. Hydrogen bonds (see Table 1) are shown as dashed lines.

Refinement

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

Acknowledgements

The authors thank Andreas Lorbach and Todd B. Marder (Institute of Inorganic Chemistry, Wuerzburg University) for the data collection and structure solution.

Funding information

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References

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3A···O1	0.86	2.10	2.7734 (19)	135
N2—H2···O1^{i}	0.88	2.07	2.882 (2)	152
N3—H3B···N1^{ii}	0.86	2.42	3.087 (2)	134

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{12}H_{10}ClN_3O$
M_r	247.68
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	100
a, b, c (Å)	11.0965 (9), 4.7669 (4), 20.6624 (17)
β (°)	97.556 (3)
V (Å^3)	1083.47 (15)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.34
Crystal size (mm)	0.57 × 0.39 × 0.21
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2013)
T_{min}, T_{max}	0.862, 0.957
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	11807, 2283, 1963
R_{int}	0.044
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.633
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.037, 0.091, 1.07
No. of reflections	2283
No. of parameters	155
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.35, −0.44

Computer programs: *APEX2* and *SAINT* (Bruker, 2013), *olex2.solve* (Bourhis *et al.*, 2015), *SHELXL* (Sheldrick, 2008) and *OLEX2* (Dolomanov *et al.*, 2009).

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full crystallographic data

IUCrData (2017). **2**, x171536 [https://doi.org/10.1107/S241431461701536X]

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Crystal data

$C_{12}H_{10}ClN_3O$
M_r = 247.68
Monoclinic, $P2_1/c$
a = 11.0965 (9) Å
b = 4.7669 (4) Å
c = 20.6624 (17) Å
β = 97.556 (3)°
V = 1083.47 (15) Å^3
Z = 4
$F(000)$ = 512
D_x = 1.518 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2859 reflections
θ = 2.5–26.6°
μ = 0.34 mm^{-1}
T = 100 K
Block, colourless
0.57 × 0.39 × 0.21 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2013)
T_{min} = 0.862, T_{max} = 0.957
11807 measured reflections
2283 independent reflections
1963 reflections with $I > 2\sigma(I)$
R_{int} = 0.044
θ_{max} = 26.8°, θ_{min} = 1.9°
h = −13→14
k = −6→5
l = −25→26

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.091
S = 1.07
2283 reflections
155 parameters
0 restraints
Primary atom site location: iterative
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0341P)^2 + 0.9297P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.44 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.

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

	x	y	z	U_{iso}*/U_{eq}
Cl1	0.81489 (4)	0.23796 (9)	0.39806 (2)	0.01471 (13)

O1	0.70743 (12)	0.9552 (3)	0.22843 (6)	0.0161 (3)
N3	0.70224 (13)	0.8909 (3)	0.09469 (7)	0.0137 (3)
H3A	0.7063	1.0033	0.1275	0.016*
H3B	0.7206	0.9803	0.0611	0.016*
N2	0.71512 (13)	0.5248 (3)	0.27552 (7)	0.0113 (3)
H2	0.7345	0.3474	0.2712	0.014*
N1	0.65077 (14)	0.5388 (3)	0.44509 (7)	0.0139 (3)
C8	0.78996 (15)	0.6915 (4)	0.11373 (9)	0.0112 (4)
C9	0.85353 (16)	0.5653 (4)	0.06707 (9)	0.0141 (4)
H9	0.8375	0.6234	0.0228	0.017*
C6	0.74137 (15)	0.7094 (4)	0.22874 (8)	0.0109 (4)
C2	0.65890 (15)	0.6017 (4)	0.33025 (8)	0.0099 (3)
C7	0.81176 (15)	0.5917 (4)	0.17868 (8)	0.0110 (4)
C5	0.56237 (17)	0.7324 (4)	0.44136 (9)	0.0159 (4)
H5	0.5291	0.7798	0.4800	0.019*
C11	0.96468 (16)	0.2701 (4)	0.14867 (9)	0.0146 (4)
H11	1.0256	0.1328	0.1606	0.018*
C12	0.90038 (15)	0.3850 (4)	0.19498 (9)	0.0126 (4)
H12	0.9166	0.3225	0.2389	0.015*
C3	0.56528 (15)	0.7972 (4)	0.32742 (9)	0.0131 (4)
H3	0.5346	0.8827	0.2871	0.016*
C4	0.51739 (16)	0.8661 (4)	0.38375 (9)	0.0147 (4)
H4	0.4548	1.0026	0.3830	0.018*
C1	0.69709 (15)	0.4811 (4)	0.39114 (9)	0.0113 (4)
C10	0.93868 (16)	0.3588 (4)	0.08413 (9)	0.0153 (4)
H10	0.9801	0.2760	0.0515	0.018*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0156 (2)	0.0151 (2)	0.0135 (2)	0.00489 (17)	0.00259 (16)	0.00396 (16)
O1	0.0258 (7)	0.0090 (6)	0.0148 (7)	0.0026 (5)	0.0075 (5)	0.0002 (5)
N3	0.0171 (8)	0.0138 (8)	0.0102 (8)	0.0010 (6)	0.0021 (6)	0.0025 (6)
N2	0.0156 (7)	0.0084 (7)	0.0105 (7)	0.0015 (6)	0.0042 (6)	−0.0009 (6)
N1	0.0171 (8)	0.0144 (8)	0.0111 (8)	−0.0010 (6)	0.0046 (6)	−0.0012 (6)
C8	0.0105 (8)	0.0097 (8)	0.0133 (9)	−0.0038 (7)	0.0017 (7)	−0.0004 (7)
C9	0.0159 (9)	0.0169 (9)	0.0101 (9)	−0.0045 (7)	0.0035 (7)	−0.0009 (7)
C6	0.0117 (8)	0.0101 (8)	0.0102 (8)	−0.0015 (7)	−0.0011 (7)	−0.0015 (6)
C2	0.0121 (8)	0.0082 (8)	0.0096 (8)	−0.0022 (7)	0.0025 (6)	−0.0017 (6)
C7	0.0124 (8)	0.0087 (8)	0.0121 (9)	−0.0029 (7)	0.0027 (7)	−0.0016 (7)
C5	0.0181 (9)	0.0170 (9)	0.0142 (9)	−0.0006 (8)	0.0078 (7)	−0.0040 (7)
C11	0.0123 (8)	0.0140 (9)	0.0179 (10)	−0.0005 (7)	0.0034 (7)	−0.0005 (7)
C12	0.0124 (8)	0.0124 (8)	0.0127 (9)	−0.0018 (7)	0.0012 (7)	−0.0001 (7)
C3	0.0121 (8)	0.0133 (9)	0.0135 (9)	−0.0017 (7)	0.0002 (7)	0.0005 (7)
C4	0.0116 (8)	0.0128 (9)	0.0203 (10)	0.0007 (7)	0.0041 (7)	−0.0022 (7)
C1	0.0115 (8)	0.0097 (8)	0.0125 (9)	−0.0006 (7)	0.0005 (7)	−0.0010 (7)
C10	0.0145 (9)	0.0170 (9)	0.0159 (10)	−0.0033 (7)	0.0070 (7)	−0.0042 (7)

Geometric parameters (Å, º)

Cl1—C1	1.7387 (17)	C6—C7	1.486 (2)
O1—C6	1.230 (2)	C2—C3	1.391 (2)
N3—H3A	0.8605	C2—C1	1.398 (2)
N3—H3B	0.8612	C7—C12	1.402 (2)
N3—C8	1.380 (2)	C5—H5	0.9500
N2—H2	0.8800	C5—C4	1.384 (3)
N2—C6	1.367 (2)	C11—H11	0.9500
N2—C2	1.410 (2)	C11—C12	1.380 (3)
N1—C5	1.342 (2)	C11—C10	1.393 (3)
N1—C1	1.316 (2)	C12—H12	0.9500
C8—C9	1.403 (2)	C3—H3	0.9500
C8—C7	1.414 (2)	C3—C4	1.381 (3)
C9—H9	0.9500	C4—H4	0.9500
C9—C10	1.378 (3)	C10—H10	0.9500
H3A—N3—H3B	109.4	N1—C5—H5	118.5
C8—N3—H3A	104.0	N1—C5—C4	122.98 (17)
C8—N3—H3B	109.8	C4—C5—H5	118.5
C6—N2—H2	118.0	C12—C11—H11	120.6
C6—N2—C2	123.91 (15)	C12—C11—C10	118.85 (17)
C2—N2—H2	118.0	C10—C11—H11	120.6
C1—N1—C5	117.33 (15)	C7—C12—H12	119.2
N3—C8—C9	119.93 (16)	C11—C12—C7	121.53 (17)
N3—C8—C7	121.89 (16)	C11—C12—H12	119.2
C9—C8—C7	117.98 (16)	C2—C3—H3	120.3
C8—C9—H9	119.3	C4—C3—C2	119.46 (16)
C10—C9—C8	121.36 (17)	C4—C3—H3	120.3
C10—C9—H9	119.3	C5—C4—H4	120.7
O1—C6—N2	121.56 (16)	C3—C4—C5	118.68 (17)
O1—C6—C7	122.99 (16)	C3—C4—H4	120.7
N2—C6—C7	115.44 (15)	N1—C1—Cl1	116.28 (13)
C3—C2—N2	123.17 (15)	N1—C1—C2	124.78 (16)
C3—C2—C1	116.73 (16)	C2—C1—Cl1	118.94 (13)
C1—C2—N2	120.10 (15)	C9—C10—C11	120.71 (17)
C8—C7—C6	119.58 (15)	C9—C10—H10	119.6
C12—C7—C8	119.44 (16)	C11—C10—H10	119.6
C12—C7—C6	120.98 (15)		
O1—C6—C7—C8	−33.5 (2)	C6—N2—C2—C3	39.1 (2)
O1—C6—C7—C12	145.85 (17)	C6—N2—C2—C1	−140.53 (17)
N3—C8—C9—C10	−177.76 (16)	C6—C7—C12—C11	178.71 (16)
N3—C8—C7—C6	−1.9 (2)	C2—N2—C6—O1	−6.5 (3)
N3—C8—C7—C12	178.73 (15)	C2—N2—C6—C7	174.11 (14)
N2—C6—C7—C8	145.84 (16)	C2—C3—C4—C5	−1.6 (3)
N2—C6—C7—C12	−34.8 (2)	C7—C8—C9—C10	−2.8 (3)
N2—C2—C3—C4	−178.27 (16)	C5—N1—C1—Cl1	177.95 (13)

N2—C2—C1—Cl1	0.4 (2)	C5—N1—C1—C2	−1.6 (3)
N2—C2—C1—N1	179.89 (16)	C12—C11—C10—C9	2.4 (3)
N1—C5—C4—C3	0.2 (3)	C3—C2—C1—Cl1	−179.30 (13)
C8—C9—C10—C11	−0.4 (3)	C3—C2—C1—N1	0.2 (3)
C8—C7—C12—C11	−1.9 (3)	C1—N1—C5—C4	1.4 (3)
C9—C8—C7—C6	−176.72 (15)	C1—C2—C3—C4	1.4 (2)
C9—C8—C7—C12	3.9 (2)	C10—C11—C12—C7	−1.3 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*A*···O1	0.86	2.10	2.7734 (19)	135
N2—H2···O1^{i}	0.88	2.07	2.882 (2)	152
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