

3,6-Dichloro-*N*-phenylpicolinamideXian-Hong Yin,<sup>a\*</sup> Kai Zhao,<sup>b</sup> Yu Feng<sup>a</sup> and Jie Zhu<sup>b</sup>

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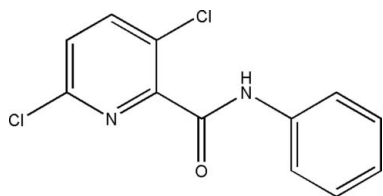
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Key indicators: single-crystal X-ray study;  $T = 298$  K; mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å;  $R$  factor = 0.037;  $wR$  factor = 0.100; data-to-parameter ratio = 13.3.

The title compound,  $\text{C}_{12}\text{H}_8\text{Cl}_2\text{N}_2\text{O}$ , is found in the keto form. The molecules are approximately planar (r.m.s. deviation = 0.058 Å for 17 non-H atoms) and lie in sheets. Intermolecular  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds are not observed. Instead, the  $\text{N}-\text{H}$  group of the amide forms an intramolecular  $\text{N}-\text{H}\cdots\text{N}$  contact to the pyridyl ring and the  $\text{C}=\text{O}$  group is involved in an intermolecular  $\text{C}-\text{H}\cdots\text{O}$  contact.

## Related literature

For similar structures with an unsubstituted pyridyl group and a Cl or Br atom in the *para* position of the phenyl ring, see: Zhang *et al.* (2006); Qi *et al.* (2003).



## Experimental

## Crystal data

 $\text{C}_{12}\text{H}_8\text{Cl}_2\text{N}_2\text{O}$  $M_r = 267.10$ Monoclinic,  $P2_1/c$  $a = 6.8404$  (11) Å $b = 12.2627$  (19) Å $c = 14.156$  (2) Å $\beta = 101.044$  (2)° $V = 1165.4$  (3) Å<sup>3</sup> $Z = 4$ Mo  $K\alpha$  radiation $\mu = 0.54$  mm<sup>-1</sup> $T = 298$  (2) K

0.42 × 0.37 × 0.35 mm

## Data collection

Bruker SMART CCD diffractometer  
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)  
 $T_{\min} = 0.805$ ,  $T_{\max} = 0.834$

5729 measured reflections  
2055 independent reflections  
1339 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.023$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.037$   
 $wR(F^2) = 0.100$   
 $S = 1.08$   
2055 reflections

154 parameters  
H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.25$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.18$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N2}-\text{H2}\cdots\text{N1}$	0.86	2.14	2.606 (3)	113
$\text{C4}-\text{H4}\cdots\text{O1}^i$	0.93	2.48	3.349 (3)	156

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: B12247).

## References

- Bruker (2000). *SMART*, *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.  
Qi, J. Y., Yang, Q. Y., Lam, K. H., Zhou, Z. Y. & Chan, A. S. C. (2003). *Acta Cryst.* **E59**, o374–o375.  
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.  
Zhang, Q., Zhang, S.-P. & Shao, S.-C. (2006). *Acta Cryst.* **E62**, o4695–o4696.

**supplementary materials**

*Acta Cryst.* (2007). E63, o4617 [ doi:10.1107/S1600536807055936 ]

### 3,6-Dichloro-*N*-phenylpicolinamide

X.-H. Yin, K. Zhao, Y. Feng and J. Zhu

#### Comment

Amide compounds play an important role in the development of coordination chemistry related to catalysis, magnetism and molecular architectures. As part of our studies on the synthesis and characterization of these compounds, we report here the synthesis and crystal structure of 3,6-dichloro-*N*-phenylpicolinamide.

The bond length of 1.345 (3) Å between atoms C7 and N2 is between the values expected for a double and single bond, because of conjugation effects in the molecule. The C=O bond length of 1.212 (3) Å indicates that the molecule is in the keto form.

In the crystal structure, the molecules are stabilized by intramolecular N—H···N hydrogen bonds and intermolecular C—H···O contacts (Table 1 and Fig. 2).

#### Experimental

A solution of 3,6-dichloropicolinoyl chloride (10 mmol) in 50 ml toluene was added to a solution of aniline (10 mmol) in 10 ml toluene. The reaction mixture was refluxed for 1 h with stirring then the resulting white precipitate was obtained by filtration, washed several times with ethanol and dried *in vacuo* (yield 90%). Elemental analysis calculated: C 53.96, H 3.02, Cl 26.55, N 10.49, O 5.99; found: C 53.90, H 3.08, Cl 26.50, N 10.45, O 6.08. Crystals were obtained by slow evaporation of a solution in methanol.

#### Refinement

H atoms were placed geometrically and refined using a riding model, with C—H = 0.93 Å, N—H = 0.86 Å and  $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C/N})$ .

#### Figures

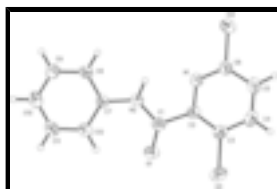


Fig. 1. The molecular structure of the title compound showing 50% probability displacement ellipsoids for non-H atoms.

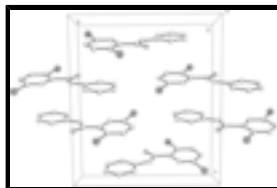


Fig. 2. Packing diagram (H atoms omitted).

## 3,6-Dichloro-*N*-phenylpicolinamide

### Crystal data

$C_{12}H_8Cl_2N_2O$

$M_r = 267.10$

Monoclinic,  $P2_1/c$

Hall symbol: -P 2ybc

$a = 6.8404$  (11) Å

$b = 12.2627$  (19) Å

$c = 14.156$  (2) Å

$\beta = 101.044$  (2)°

$V = 1165.4$  (3) Å<sup>3</sup>

$Z = 4$

$F_{000} = 544$

$D_x = 1.522$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation

$\lambda = 0.71073$  Å

Cell parameters from 1704 reflections

$\theta = 2.2$ – $24.2$ °

$\mu = 0.54$  mm<sup>-1</sup>

$T = 298$  (2) K

Block, colourless

$0.42 \times 0.37 \times 0.35$  mm

### Data collection

Bruker SMART CCD  
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298$ (2) K

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan  
(SADABS; Sheldrick, 1996)

$T_{\min} = 0.805$ ,  $T_{\max} = 0.834$

5729 measured reflections

2055 independent reflections

1339 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.023$

$\theta_{\text{max}} = 25.0$ °

$\theta_{\text{min}} = 2.2$ °

$h = -8$ → $8$

$k = -14$ → $14$

$l = -16$ → $11$

### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.037$

$wR(F^2) = 0.100$

$S = 1.08$

2055 reflections

154 parameters

Primary atom site location: structure-invariant direct  
methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring  
sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0324P)^2 + 0.4782P]$$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} < 0.001$

$\Delta\rho_{\text{max}} = 0.25$  e Å<sup>-3</sup>

$\Delta\rho_{\text{min}} = -0.18$  e Å<sup>-3</sup>

Extinction correction: none

*Special details*

**Geometry.** All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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  $F^2 > 2\sigma(F^2)$  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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	1.07918 (11)	0.66623 (7)	0.22741 (7)	0.0821 (3)
C12	0.29924 (11)	0.90851 (6)	0.07090 (7)	0.0838 (3)
N1	0.5113 (3)	0.73478 (17)	0.11835 (14)	0.0476 (5)
N2	0.4635 (3)	0.52408 (17)	0.12230 (15)	0.0534 (6)
H2	0.3779	0.5760	0.1103	0.064*
O1	0.7923 (3)	0.49406 (17)	0.17281 (19)	0.0927 (8)
C1	0.6531 (4)	0.5561 (2)	0.15056 (19)	0.0515 (7)
C2	0.6792 (4)	0.6782 (2)	0.15207 (17)	0.0460 (6)
C3	0.8567 (4)	0.7321 (2)	0.18511 (18)	0.0529 (7)
C4	0.8595 (4)	0.8446 (2)	0.1857 (2)	0.0618 (8)
H4	0.9772	0.8817	0.2095	0.074*
C5	0.6894 (4)	0.9011 (2)	0.1514 (2)	0.0610 (8)
H5	0.6877	0.9769	0.1506	0.073*
C6	0.5198 (4)	0.8412 (2)	0.11777 (19)	0.0536 (7)
C7	0.3836 (4)	0.41788 (19)	0.10930 (17)	0.0458 (6)
C8	0.1789 (4)	0.4101 (2)	0.07842 (18)	0.0529 (7)
H8	0.1019	0.4731	0.0685	0.063*
C9	0.0898 (4)	0.3096 (2)	0.0624 (2)	0.0617 (8)
H9	-0.0472	0.3050	0.0409	0.074*
C10	0.2011 (5)	0.2165 (2)	0.0778 (2)	0.0627 (8)
H10	0.1408	0.1485	0.0664	0.075*
C11	0.4025 (5)	0.2244 (2)	0.1103 (2)	0.0631 (8)
H11	0.4779	0.1611	0.1222	0.076*
C12	0.4958 (4)	0.3240 (2)	0.1258 (2)	0.0568 (7)
H12	0.6329	0.3280	0.1471	0.068*

*Atomic displacement parameters ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C11	0.0448 (4)	0.0895 (6)	0.1053 (7)	0.0040 (4)	-0.0022 (4)	-0.0149 (5)
C12	0.0701 (5)	0.0529 (5)	0.1224 (8)	0.0124 (4)	0.0033 (5)	0.0079 (5)
N1	0.0483 (13)	0.0439 (13)	0.0500 (13)	0.0013 (10)	0.0080 (10)	-0.0008 (10)

## supplementary materials

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N2	0.0413 (12)	0.0434 (12)	0.0712 (15)	0.0087 (10)	-0.0001 (11)	0.0008 (11)
O1	0.0484 (12)	0.0600 (13)	0.162 (2)	0.0099 (11)	0.0000 (13)	0.0147 (14)
C1	0.0448 (15)	0.0530 (16)	0.0572 (17)	0.0061 (13)	0.0109 (13)	0.0036 (13)
C2	0.0414 (14)	0.0534 (16)	0.0434 (15)	0.0021 (13)	0.0090 (12)	-0.0019 (12)
C3	0.0457 (15)	0.0620 (18)	0.0507 (17)	-0.0003 (13)	0.0083 (13)	-0.0087 (14)
C4	0.0560 (18)	0.068 (2)	0.0617 (19)	-0.0163 (15)	0.0114 (15)	-0.0153 (15)
C5	0.071 (2)	0.0477 (16)	0.0650 (19)	-0.0078 (15)	0.0140 (16)	-0.0102 (14)
C6	0.0572 (17)	0.0469 (17)	0.0560 (18)	0.0006 (13)	0.0088 (14)	-0.0033 (13)
C7	0.0485 (15)	0.0419 (15)	0.0453 (15)	0.0039 (12)	0.0051 (12)	0.0019 (12)
C8	0.0507 (16)	0.0467 (16)	0.0560 (17)	0.0094 (13)	-0.0029 (13)	0.0013 (13)
C9	0.0559 (17)	0.0592 (19)	0.066 (2)	-0.0046 (15)	0.0020 (14)	-0.0045 (15)
C10	0.079 (2)	0.0469 (17)	0.0629 (19)	-0.0053 (16)	0.0149 (16)	-0.0047 (14)
C11	0.071 (2)	0.0438 (16)	0.075 (2)	0.0118 (15)	0.0164 (16)	0.0028 (14)
C12	0.0534 (16)	0.0497 (17)	0.0666 (19)	0.0106 (14)	0.0095 (14)	0.0026 (14)

### *Geometric parameters (Å, °)*

C11—C3	1.725 (3)	C5—C6	1.378 (4)
C12—C6	1.736 (3)	C5—H5	0.930
N1—C6	1.307 (3)	C7—C12	1.378 (3)
N1—C2	1.349 (3)	C7—C8	1.388 (3)
N2—C1	1.341 (3)	C8—C9	1.374 (3)
N2—C7	1.411 (3)	C8—H8	0.930
N2—H2	0.860	C9—C10	1.367 (4)
O1—C1	1.212 (3)	C9—H9	0.930
C1—C2	1.508 (4)	C10—C11	1.370 (4)
C2—C3	1.382 (3)	C10—H10	0.930
C3—C4	1.380 (4)	C11—C12	1.376 (4)
C4—C5	1.362 (4)	C11—H11	0.930
C4—H4	0.930	C12—H12	0.930
C6—N1—C2	118.7 (2)	N1—C6—C12	116.1 (2)
C1—N2—C7	129.6 (2)	C5—C6—C12	119.4 (2)
C1—N2—H2	115.2	C12—C7—C8	119.5 (2)
C7—N2—H2	115.2	C12—C7—N2	124.0 (2)
O1—C1—N2	124.1 (3)	C8—C7—N2	116.5 (2)
O1—C1—C2	122.3 (2)	C9—C8—C7	120.1 (2)
N2—C1—C2	113.6 (2)	C9—C8—H8	119.9
N1—C2—C3	120.5 (2)	C7—C8—H8	119.9
N1—C2—C1	114.5 (2)	C10—C9—C8	120.5 (3)
C3—C2—C1	125.0 (2)	C10—C9—H9	119.8
C4—C3—C2	119.3 (2)	C8—C9—H9	119.8
C4—C3—C11	117.1 (2)	C9—C10—C11	119.2 (3)
C2—C3—C11	123.6 (2)	C9—C10—H10	120.4
C5—C4—C3	119.8 (3)	C11—C10—H10	120.4
C5—C4—H4	120.1	C10—C11—C12	121.5 (3)
C3—C4—H4	120.1	C10—C11—H11	119.3
C4—C5—C6	117.2 (3)	C12—C11—H11	119.3
C4—C5—H5	121.4	C11—C12—C7	119.2 (3)
C6—C5—H5	121.4	C11—C12—H12	120.4

N1—C6—C5	124.5 (3)	C7—C12—H12	120.4
C7—N2—C1—O1	-2.0 (5)	C2—N1—C6—C5	-1.4 (4)
C7—N2—C1—C2	177.8 (2)	C2—N1—C6—C12	178.61 (18)
C6—N1—C2—C3	-0.1 (4)	C4—C5—C6—N1	1.3 (4)
C6—N1—C2—C1	179.4 (2)	C4—C5—C6—C12	-178.8 (2)
O1—C1—C2—N1	175.3 (3)	C1—N2—C7—C12	0.8 (4)
N2—C1—C2—N1	-4.6 (3)	C1—N2—C7—C8	-179.6 (2)
O1—C1—C2—C3	-5.2 (4)	C12—C7—C8—C9	-1.5 (4)
N2—C1—C2—C3	175.0 (2)	N2—C7—C8—C9	178.9 (2)
N1—C2—C3—C4	1.7 (4)	C7—C8—C9—C10	0.8 (4)
C1—C2—C3—C4	-177.8 (2)	C8—C9—C10—C11	0.6 (4)
N1—C2—C3—C11	-178.81 (19)	C9—C10—C11—C12	-1.4 (4)
C1—C2—C3—C11	1.7 (4)	C10—C11—C12—C7	0.8 (4)
C2—C3—C4—C5	-1.9 (4)	C8—C7—C12—C11	0.6 (4)
C11—C3—C4—C5	178.7 (2)	N2—C7—C12—C11	-179.7 (3)
C3—C4—C5—C6	0.4 (4)		

*Hydrogen-bond geometry* ( $\text{\AA}$ ,  $^\circ$ )

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
N2—H2 $\cdots$ N1	0.86	2.14	2.606 (3)	113
C4—H4 $\cdots$ O1 <sup>i</sup>	0.93	2.48	3.349 (3)	156

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Fig. 1

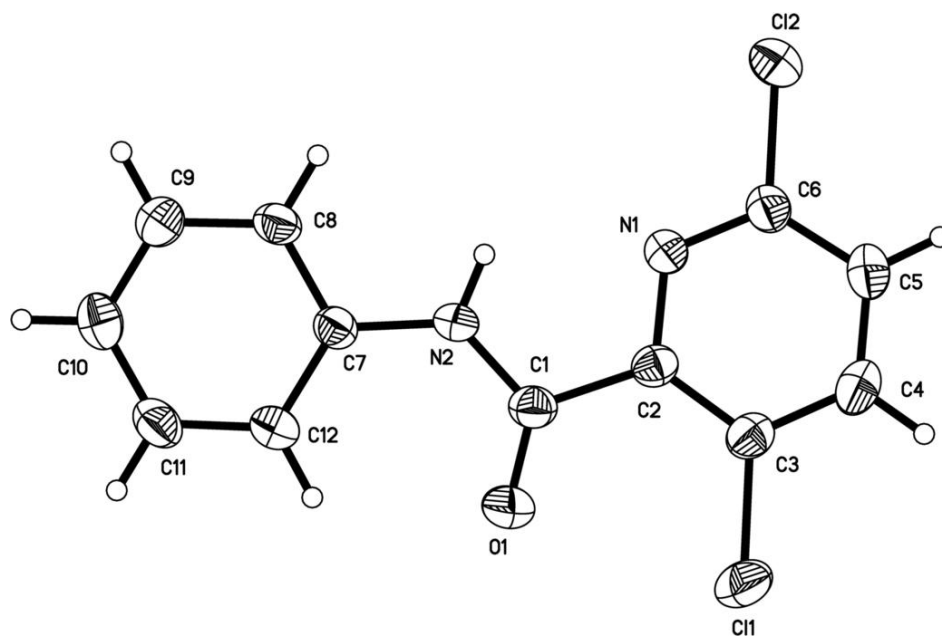




Fig. 2

