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2-(4-Chlorophenyl)acetamide

Dong-Sheng Ma,* Pei-Jiang Liu, Shuai Zhang and
Guang-Feng HouCollege of Chemistry and Materials Science, Heilongjiang University, Harbin
150080, People's Republic of China

Correspondence e-mail: hgf1000@163.com

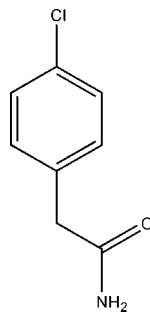
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å;
 R factor = 0.037; wR factor = 0.083; data-to-parameter ratio = 16.7.

In the title compound, $\text{C}_8\text{H}_8\text{ClNO}$, the acetamide group is twisted out the benzene plane with a dihedral angle of $83.08(1)^\circ$. In the crystal, molecules are linked by $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds, forming layers parallel to the ab plane.

Related literature

For details of the nitrile hydrolysis of the same substrate (4-chlorobenzonitrile) by another method, see: Moorthy & Singhal (2005).



Experimental

Crystal data

 $\text{C}_8\text{H}_8\text{ClNO}$ $M_r = 169.60$ Orthorhombic, $P2_12_12_1$ $a = 4.917(2)$ Å $b = 6.033(4)$ Å $c = 26.680(12)$ Å $V = 791.5(7)$ Å³ $Z = 4$ Mo $K\alpha$ radiation $\mu = 0.42$ mm⁻¹ $T = 293$ K $0.29 \times 0.22 \times 0.07$ mm

Data collection

Rigaku R-Axis RAPID
diffractometerAbsorption correction: multi-scan
(*ABSCOR*; Higashi, 1995) $T_{\min} = 0.887$, $T_{\max} = 0.970$

7733 measured reflections

1807 independent reflections

1451 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.041$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.037$ $wR(F^2) = 0.083$ $S = 1.05$

1807 reflections

108 parameters

2 restraints

H atoms treated by a mixture of
independent and constrained
refinement $\Delta\rho_{\max} = 0.17$ e Å⁻³ $\Delta\rho_{\min} = -0.17$ e Å⁻³Absolute structure: Flack (1983),
704 Friedel pairsFlack parameter: $-0.12(8)$

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H11}\cdots\text{O1}^i$	0.88 (1)	2.05 (1)	2.911 (2)	165 (2)
$\text{N1}-\text{H12}\cdots\text{O1}^{ii}$	0.89 (1)	2.22 (1)	3.064 (3)	157 (2)

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

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalClear* (Rigaku/MS, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

The authors thank Heilongjiang University for supporting this work.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV5191).

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supplementary materials

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2-(4-Chlorophenyl)acetamide

D.-S. Ma, P.-J. Liu, S. Zhang and G.-F. Hou

Comment

The title compound is formed by hydrolysis of appropriate nitriles (Moorthy *et al.*, 2005), while the final product of hydrolysis of nitriles should be carboxylic acid. In this paper, we report the synthesis and the crystal structure of the title compound prepared from 4-cyanobenzylchloride under solvothermal condition.

In the title molecule (Fig.1), the acetamide group is twisted out the benzene plane with a dihedral angle of 83.08 (1) °. In the crystal packing, the molecules are linked by N—H···O hydrogen bonds to form layers parallel to *ab* plane (Fig. 2, Table 1).

Experimental

A mixture of NaN₃ (0.39 g, 6 mmol), CuCl₂·2H₂O (0.684 g, 4 mmol), and 4-cyanobenzylchloride (0.606 g, 4 mmol) was sealed in a 15 ml teflon-lined reactor and heated in an oven at 150 ° C for 72 hrs and slowly cooled to room temperature. The resulting mixture was washed with water, and pale yellow blocklike crystals were collected (yeild 31%).

Refinement

N-bound H atoms were located in a differece Fourier map and refined with restraint of N—H = 0.89 (1) Å. C-bound H atoms were placed in calculated positions and treated as riding on their parent atoms, with C—H = 0.93 Å (aromatic); C—H = 0.97 Å (methylene), and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Figures

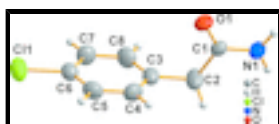


Fig. 1. The molecular structure of the title compound, showing displacement ellipsoids at the 50% probability level.

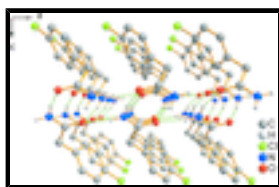


Fig. 2. A portion of the crystal packing, showing a two-dimensional structure formed by N—H···O hydrogen bonds (dashed lines).

2-(4-Chlorophenyl)acetamide

Crystal data

C₈H₈ClNO

$F(000) = 352$

supplementary materials

$$M_r = 169.60$$

Orthorhombic, $P2_12_12_1$

Hall symbol: P 2ac 2ab

$$a = 4.917 (2) \text{ \AA}$$

$$b = 6.033 (4) \text{ \AA}$$

$$c = 26.680 (12) \text{ \AA}$$

$$V = 791.5 (7) \text{ \AA}^3$$

$$Z = 4$$

$$D_x = 1.423 \text{ Mg m}^{-3}$$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 5994 reflections

$$\theta = 3.1\text{--}27.4^\circ$$

$$\mu = 0.42 \text{ mm}^{-1}$$

$$T = 293 \text{ K}$$

Block, colorless

$$0.29 \times 0.22 \times 0.07 \text{ mm}$$

Data collection

Rigaku R-Axis RAPID
diffractometer

Radiation source: fine-focus sealed tube

graphite

ω scan

Absorption correction: multi-scan
(*ABSCOR*; Higashi, 1995)

$$T_{\min} = 0.887, T_{\max} = 0.970$$

7733 measured reflections

1807 independent reflections

1451 reflections with $I > 2\sigma(I)$

$$R_{\text{int}} = 0.041$$

$$\theta_{\max} = 27.4^\circ, \theta_{\min} = 3.1^\circ$$

$$h = -6 \rightarrow 6$$

$$k = -7 \rightarrow 7$$

$$l = -34 \rightarrow 33$$

Refinement

Refinement on F^2

Least-squares matrix: full

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

$$wR(F^2) = 0.083$$

$$S = 1.05$$

1807 reflections

108 parameters

2 restraints

Primary atom site location: structure-invariant direct
methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring
sites

H atoms treated by a mixture of independent and
constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.036P)^2 + 0.1017P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

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

$$\Delta\rho_{\max} = 0.17 \text{ e \AA}^{-3}$$

$$\Delta\rho_{\min} = -0.17 \text{ e \AA}^{-3}$$

Absolute structure: Flack (1983), 704 Friedel pairs

$$\text{Flack parameter: } -0.12 (8)$$

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. 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.3632 (4)	0.5073 (3)	0.79843 (7)	0.0335 (4)
C2	0.2527 (4)	0.3677 (5)	0.84087 (10)	0.0603 (7)
H2A	0.1560	0.4638	0.8639	0.072*
H2B	0.1222	0.2633	0.8272	0.072*
C3	0.4640 (4)	0.2408 (4)	0.86973 (8)	0.0429 (5)
C4	0.5575 (5)	0.0374 (4)	0.85279 (8)	0.0487 (6)
H4	0.4907	-0.0195	0.8228	0.058*
C5	0.7475 (5)	-0.0822 (3)	0.87944 (8)	0.0451 (5)
H5	0.8079	-0.2185	0.8676	0.054*
C6	0.8470 (4)	0.0017 (4)	0.92375 (8)	0.0410 (5)
C7	0.7620 (5)	0.2049 (4)	0.94146 (8)	0.0472 (6)
H7	0.8324	0.2622	0.9711	0.057*
C8	0.5697 (5)	0.3221 (4)	0.91430 (8)	0.0487 (5)
H8	0.5100	0.4586	0.9262	0.058*
C11	1.08553 (13)	-0.14906 (11)	0.95804 (2)	0.0605 (2)
N1	0.1788 (3)	0.6234 (4)	0.77353 (7)	0.0441 (4)
H11	0.004 (2)	0.613 (4)	0.7811 (8)	0.046 (6)*
H12	0.225 (5)	0.714 (4)	0.7485 (7)	0.064 (8)*
O1	0.6073 (3)	0.5149 (3)	0.78808 (5)	0.0435 (4)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0263 (9)	0.0376 (11)	0.0367 (10)	-0.0005 (9)	0.0007 (8)	-0.0027 (8)
C2	0.0291 (11)	0.0819 (19)	0.0699 (15)	0.0050 (12)	0.0050 (11)	0.0347 (15)
C3	0.0310 (11)	0.0528 (13)	0.0449 (11)	0.0000 (9)	0.0035 (9)	0.0145 (9)
C4	0.0493 (13)	0.0554 (14)	0.0416 (11)	-0.0093 (12)	-0.0077 (11)	-0.0001 (10)
C5	0.0511 (13)	0.0368 (12)	0.0475 (12)	0.0016 (9)	0.0072 (11)	-0.0041 (9)
C6	0.0381 (11)	0.0444 (12)	0.0406 (10)	0.0018 (10)	0.0046 (9)	0.0071 (9)
C7	0.0514 (13)	0.0514 (14)	0.0387 (11)	0.0009 (10)	-0.0027 (10)	-0.0056 (10)
C8	0.0503 (13)	0.0433 (13)	0.0525 (12)	0.0118 (12)	0.0069 (12)	-0.0003 (10)
C11	0.0520 (3)	0.0695 (4)	0.0598 (3)	0.0147 (3)	-0.0015 (3)	0.0194 (3)
N1	0.0243 (8)	0.0587 (12)	0.0494 (10)	-0.0006 (8)	0.0013 (8)	0.0149 (10)
O1	0.0237 (6)	0.0529 (9)	0.0538 (8)	-0.0038 (7)	0.0051 (7)	0.0046 (7)

Geometric parameters (\AA , $^\circ$)

C1—O1	1.232 (2)	C5—C6	1.376 (3)
C1—N1	1.324 (3)	C5—H5	0.9300
C1—C2	1.512 (3)	C6—C7	1.379 (3)
C2—C3	1.503 (3)	C6—C11	1.744 (2)
C2—H2A	0.9700	C7—C8	1.386 (3)
C2—H2B	0.9700	C7—H7	0.9300
C3—C4	1.386 (3)	C8—H8	0.9300
C3—C8	1.387 (3)	N1—H11	0.883 (10)

supplementary materials

C4—C5	1.378 (3)	N1—H12	0.892 (10)
C4—H4	0.9300		
O1—C1—N1	122.34 (19)	C6—C5—C4	119.5 (2)
O1—C1—C2	122.57 (18)	C6—C5—H5	120.3
N1—C1—C2	115.08 (17)	C4—C5—H5	120.3
C3—C2—C1	114.78 (17)	C5—C6—C7	120.9 (2)
C3—C2—H2A	108.6	C5—C6—C11	119.88 (18)
C1—C2—H2A	108.6	C7—C6—C11	119.21 (17)
C3—C2—H2B	108.6	C6—C7—C8	118.8 (2)
C1—C2—H2B	108.6	C6—C7—H7	120.6
H2A—C2—H2B	107.5	C8—C7—H7	120.6
C4—C3—C8	117.9 (2)	C7—C8—C3	121.6 (2)
C4—C3—C2	120.9 (2)	C7—C8—H8	119.2
C8—C3—C2	121.2 (2)	C3—C8—H8	119.2
C5—C4—C3	121.3 (2)	C1—N1—H11	120.9 (16)
C5—C4—H4	119.3	C1—N1—H12	121.7 (18)
C3—C4—H4	119.3	H11—N1—H12	117 (2)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H11 \cdots O1 ⁱ	0.88 (1)	2.05 (1)	2.911 (2)	165 (2)
N1—H12 \cdots O1 ⁱⁱ	0.89 (1)	2.22 (1)	3.064 (3)	157 (2)

Symmetry codes: (i) $x-1, y, z$; (ii) $-x+1, y+1/2, -z+3/2$.

Fig. 1

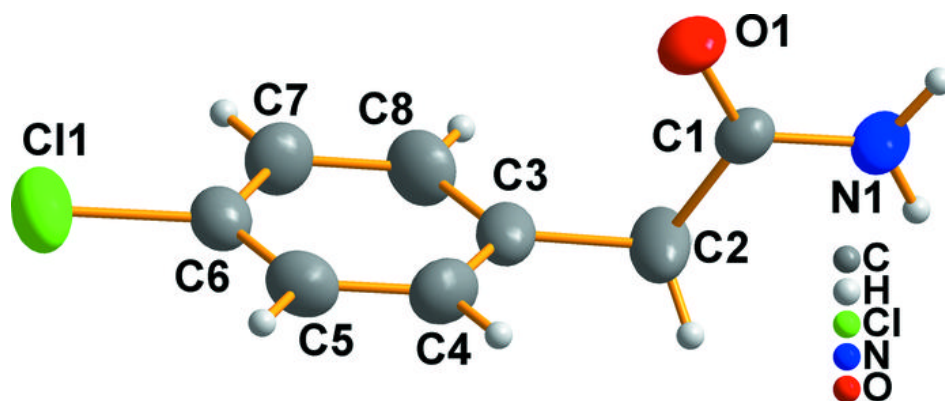


Fig. 2

