Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-Chloro-*N*-methyl-*N*-[2-(methylamino)-phenyl]acetamide

Yu-Bin Bai, Xia-Hui Chen, Ya-Tuan Ma, An-Ling Zhang and Jin-Ming Gao*

College of Science, Northwest A&F University, Yangling Shaanxi 712100, People's Republic of China
Correspondence e-mail: jinminggao@nwsuaf.edu.cn

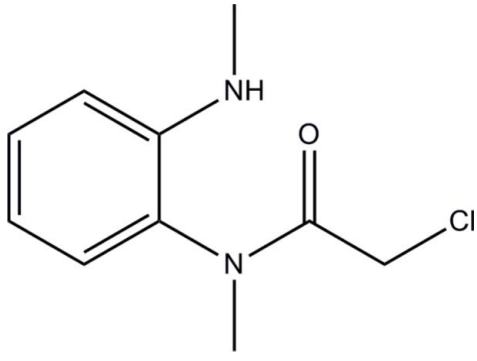
Received 17 December 2012; accepted 6 January 2013

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.045; wR factor = 0.140; data-to-parameter ratio = 15.6.

The title compound, $C_{10}H_{13}ClN_2O$, was obtained as a by-product in the reaction of 2-chloromethyl-1*H*-benzimidazole, dimethyl sulfate and toluene to synthesise 2-chloromethyl-1-methylbenzimidazole. The dihedral angle between the benzene ring and the acetamide group is 89.72 (6)° while that between the aromatic ring and the chloracetyl group is 84.40 (4)°. In the crystal, adjacent molecules are linked by pairs of N—H···O hydrogen bonds into inversion dimers.

Related literature

For the synthesis of similar compounds, see: Turner & Wood (1965); Bai *et al.* (2008).



Experimental

Crystal data

$C_{10}H_{13}ClN_2O$
M_r = 212.67
Monoclinic, $P2_1/n$
a = 9.2483 (18) Å
b = 6.6630 (13) Å
c = 17.622 (3) Å
β = 94.377 (2)°
V = 1082.8 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.32 mm^{-1}
T = 296 K
0.50 × 0.35 × 0.21 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.855, T_{max} = 0.935
7714 measured reflections
2011 independent reflections
1487 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.140
S = 1.01
2011 reflections
129 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.86	2.23	2.926 (2)	138

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; 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: *SHELXTL*.

This work was supported financially by grants from the National Natural Science Foundation of China (No. 30971882), the Program of Natural Science Basis Research in Shaanxi (No. 2009JM3010) and Shaanxi Province Science and Technology (No. 2011k02–07).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NC2302).

References

Bai, Y., Li, C., Sun, W., Zhao, G. & Shi, Z. (2008). *Hua Xue Shiji*, **30**, 409–411.
Bruker (2004). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconin, USA.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Turner, A. B. & Wood, H. C. S. (1965). *J. Chem. Soc.* pp. 5270–5275.

supporting information

Acta Cryst. (2013). E**69**, o229 [doi:10.1107/S1600536813000494]

2-Chloro-*N*-methyl-*N*-[2-(methylamino)phenyl]acetamide

Yu-Bin Bai, Xia-Hui Chen, Ya-Tuan Ma, An-Ling Zhang and Jin-Ming Gao

S1. Experimental

2-chloromethyl-1*H*-benzimidazole (1.01 g, 6.07 mmol), toluene (10 ml),dimethyl sulfate (0.63 ml, 6.67 mmol) were refluxed for 3 h and the reaction was followed by TLC monitoring). After cooling 10 mL of water and an excess of ammonia were added. After filtration, the solution was extracted with chloroform (3 *x* 20 ml). The organic layer was dried over anhydrous sodium sulfate, concentrated and purified by columnchromatography on silica gel eluting with 4:1–3:1 petroleum ether-acetone. Crystals of the title compound were grown by slow evaporation of the solvent.

S2. Refinement

All H atoms were positioned with idealized geometry and refined isotropic with U_{iso}(H) = 1.2 U_{eq}(C,N) using a riding model.

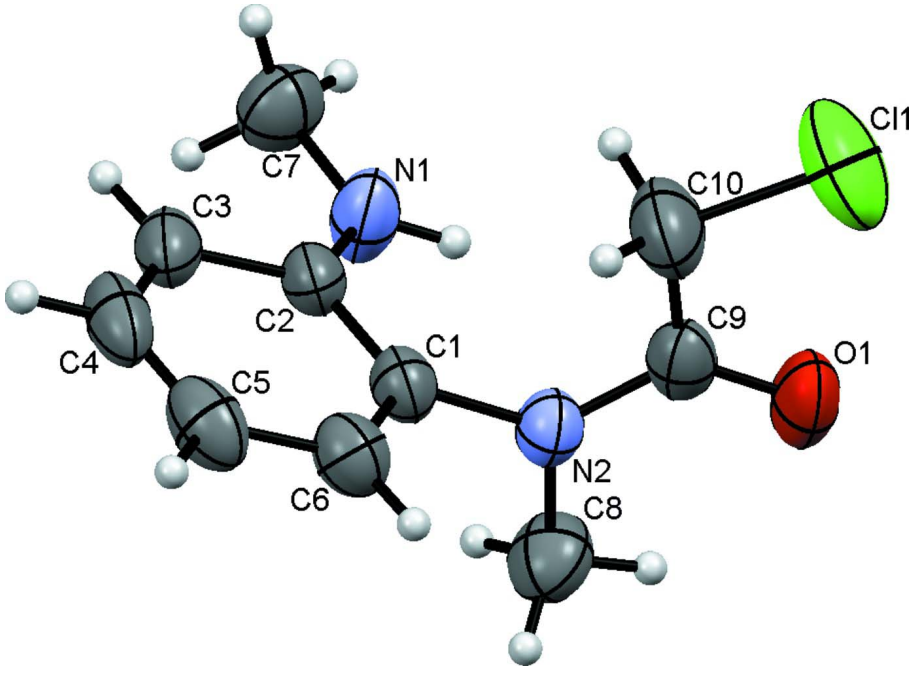


Figure 1

The molecular structure of the title compound with labeling and isplacement ellipsoids drawn at the 30% probability level.

2-Chloro-*N*-methyl-*N*-[2-(methylamino)phenyl]acetamide

Crystal data

$C_{10}H_{13}ClN_2O$
$M_r = 212.67$
Monoclinic, $P2_1/n$
$a = 9.2483$ (18) Å
$b = 6.6630$ (13) Å
$c = 17.622$ (3) Å
$\beta = 94.377$ (2)°
$V = 1082.8$ (4) Å^3
$Z = 4$
$F(000) = 448$
$D_x = 1.305$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2090 reflections
$\theta = 2.6$–25.0°
$\mu = 0.32$ mm^{-1}
$T = 296$ K
Block, colourless
0.50 × 0.35 × 0.21 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.855$, $T_{max} = 0.935$
7714 measured reflections
2011 independent reflections
1487 reflections with $I > 2\sigma(I)$
$R_{int} = 0.023$
$\theta_{max} = 25.5°$, $\theta_{min} = 2.3°$
$h = -11 \rightarrow 11$
$k = -8 \rightarrow 8$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.140$
$S = 1.01$
2011 reflections
129 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0697P)^2 + 0.4335P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	1.0326 (2)	0.5640 (3)	0.20070 (11)	0.0443 (5)

C2	0.9122 (2)	0.6930 (3)	0.19791 (11)	0.0443 (5)
C3	0.8956 (3)	0.8109 (3)	0.26283 (13)	0.0526 (6)
H3	0.8181	0.8998	0.2633	0.063*
C4	0.9928 (3)	0.7961 (4)	0.32578 (13)	0.0594 (7)
H4	0.9793	0.8755	0.3681	0.071*
C5	1.1092 (3)	0.6675 (4)	0.32793 (13)	0.0594 (7)
H5	1.1733	0.6584	0.3711	0.071*
C6	1.1285 (2)	0.5519 (4)	0.26424 (12)	0.0526 (6)
H6	1.2072	0.4650	0.2644	0.063*
C7	0.6897 (3)	0.8227 (4)	0.12831 (16)	0.0648 (7)
H7A	0.6262	0.7888	0.1670	0.097*
H7B	0.6398	0.8034	0.0791	0.097*
H7C	0.7188	0.9605	0.1340	0.097*
C8	0.9806 (3)	0.2351 (4)	0.14078 (16)	0.0655 (7)
H8A	0.9882	0.1661	0.0934	0.098*
H8B	0.8802	0.2518	0.1497	0.098*
H8C	1.0281	0.1580	0.1814	0.098*
C9	1.1273 (2)	0.4748 (4)	0.07858 (11)	0.0487 (5)
C10	1.1969 (3)	0.6811 (4)	0.08199 (16)	0.0740 (8)
H10A	1.2469	0.7003	0.1319	0.089*
H10B	1.1217	0.7823	0.0753	0.089*
Cl1	1.31970 (9)	0.71410 (17)	0.01279 (4)	0.0981 (4)
N1	0.8153 (2)	0.6964 (3)	0.13566 (11)	0.0612 (6)
H1	0.8305	0.6177	0.0984	0.073*
N2	1.04930 (19)	0.4308 (3)	0.13748 (9)	0.0453 (4)
O1	1.14232 (19)	0.3589 (3)	0.02587 (9)	0.0643 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0489 (11)	0.0492 (12)	0.0357 (10)	−0.0029 (10)	0.0084 (9)	−0.0016 (9)
C2	0.0519 (12)	0.0455 (12)	0.0369 (11)	0.0002 (9)	0.0128 (9)	−0.0001 (9)
C3	0.0641 (14)	0.0470 (13)	0.0496 (13)	−0.0030 (10)	0.0228 (11)	−0.0044 (10)
C4	0.0806 (17)	0.0599 (15)	0.0400 (12)	−0.0238 (13)	0.0205 (12)	−0.0133 (11)
C5	0.0654 (15)	0.0733 (17)	0.0390 (12)	−0.0196 (13)	0.0006 (10)	−0.0026 (11)
C6	0.0528 (13)	0.0604 (14)	0.0444 (12)	−0.0039 (11)	0.0028 (10)	0.0001 (11)
C7	0.0593 (15)	0.0652 (16)	0.0700 (16)	0.0143 (12)	0.0053 (12)	−0.0009 (13)
C8	0.0724 (17)	0.0552 (15)	0.0696 (17)	−0.0062 (12)	0.0108 (13)	−0.0129 (12)
C9	0.0470 (12)	0.0606 (14)	0.0380 (11)	0.0130 (10)	0.0011 (9)	−0.0007 (10)
C10	0.0861 (19)	0.0806 (19)	0.0593 (16)	−0.0093 (15)	0.0314 (14)	−0.0046 (13)
Cl1	0.0836 (6)	0.1459 (9)	0.0684 (5)	−0.0247 (5)	0.0304 (4)	0.0038 (5)
N1	0.0624 (12)	0.0763 (14)	0.0449 (11)	0.0249 (10)	0.0041 (9)	−0.0101 (10)
N2	0.0484 (10)	0.0480 (10)	0.0398 (9)	0.0030 (8)	0.0054 (7)	−0.0055 (8)
O1	0.0742 (11)	0.0775 (12)	0.0417 (9)	0.0169 (9)	0.0064 (8)	−0.0138 (8)

Geometric parameters (Å, º)

C1—C6	1.377 (3)	C7—H7B	0.9600
C1—C2	1.405 (3)	C7—H7C	0.9600
C1—N2	1.442 (3)	C8—N2	1.454 (3)
C2—N1	1.363 (3)	C8—H8A	0.9600
C2—C3	1.406 (3)	C8—H8B	0.9600
C3—C4	1.377 (4)	C8—H8C	0.9600
C3—H3	0.9300	C9—O1	1.224 (3)
C4—C5	1.374 (4)	C9—N2	1.341 (3)
C4—H4	0.9300	C9—C10	1.517 (4)
C5—C6	1.384 (3)	C10—Cl1	1.742 (3)
C5—H5	0.9300	C10—H10A	0.9700
C6—H6	0.9300	C10—H10B	0.9700
C7—N1	1.432 (3)	N1—H1	0.8600
C7—H7A	0.9600		
C6—C1—C2	121.60 (19)	H7B—C7—H7C	109.5
C6—C1—N2	119.48 (19)	N2—C8—H8A	109.5
C2—C1—N2	118.76 (18)	N2—C8—H8B	109.5
N1—C2—C3	122.7 (2)	H8A—C8—H8B	109.5
N1—C2—C1	120.60 (19)	N2—C8—H8C	109.5
C3—C2—C1	116.7 (2)	H8A—C8—H8C	109.5
C4—C3—C2	120.7 (2)	H8B—C8—H8C	109.5
C4—C3—H3	119.6	O1—C9—N2	123.3 (2)
C2—C3—H3	119.6	O1—C9—C10	122.0 (2)
C5—C4—C3	121.9 (2)	N2—C9—C10	114.78 (19)
C5—C4—H4	119.0	C9—C10—Cl1	112.58 (19)
C3—C4—H4	119.0	C9—C10—H10A	109.1
C4—C5—C6	118.3 (2)	Cl1—C10—H10A	109.1
C4—C5—H5	120.8	C9—C10—H10B	109.1
C6—C5—H5	120.8	Cl1—C10—H10B	109.1
C1—C6—C5	120.8 (2)	H10A—C10—H10B	107.8
C1—C6—H6	119.6	C2—N1—C7	124.2 (2)
C5—C6—H6	119.6	C2—N1—H1	117.9
N1—C7—H7A	109.5	C7—N1—H1	117.9
N1—C7—H7B	109.5	C9—N2—C1	124.03 (19)
H7A—C7—H7B	109.5	C9—N2—C8	119.30 (19)
N1—C7—H7C	109.5	C1—N2—C8	116.63 (18)
H7A—C7—H7C	109.5		
C6—C1—C2—N1	−177.2 (2)	N2—C9—C10—Cl1	170.27 (17)
N2—C1—C2—N1	−1.9 (3)	C3—C2—N1—C7	1.9 (4)
C6—C1—C2—C3	0.8 (3)	C1—C2—N1—C7	179.8 (2)
N2—C1—C2—C3	176.12 (18)	O1—C9—N2—C1	178.84 (19)
N1—C2—C3—C4	177.1 (2)	C10—C9—N2—C1	−1.0 (3)
C1—C2—C3—C4	−0.8 (3)	O1—C9—N2—C8	1.3 (3)
C2—C3—C4—C5	0.2 (3)	C10—C9—N2—C8	−178.6 (2)

C3—C4—C5—C6	0.6 (4)	C6—C1—N2—C9	−91.2 (3)
C2—C1—C6—C5	0.0 (3)	C2—C1—N2—C9	93.3 (3)
N2—C1—C6—C5	−175.32 (19)	C6—C1—N2—C8	86.4 (3)
C4—C5—C6—C1	−0.7 (3)	C2—C1—N2—C8	−89.1 (2)
O1—C9—C10—Cl1	−9.6 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.86	2.23	2.926 (2)	138

Symmetry code: (i) −*x*+2, −*y*+1, −*z*.