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## Structure Reports

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## 2-Chloro-8-methylquinoline-3-carbaldehyde

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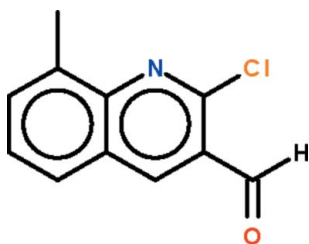
Received 6 October 2009; accepted 6 October 2009

Key indicators: single-crystal X-ray study;  $T = 290$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.048;  $wR$  factor = 0.136; data-to-parameter ratio = 16.9.

The quinoline fused-ring system of the title compound,  $\text{C}_{11}\text{H}_8\text{ClNO}$ , is planar (r.m.s. deviation =  $0.005$  Å); the formyl group is slightly bent out of the plane [ $\text{C}-\text{C}-\text{C}-\text{O}$ 1 torsion angles =  $8.8$  (7) and  $-172.8$  (4)°].

## Related literature

For a review of the synthesis of quinolines by the Vilsmeier–Haack reaction, see: Meth-Cohn (1993).



## Experimental

## Crystal data

 $\text{C}_{11}\text{H}_8\text{ClNO}$  $M_r = 205.63$ 

Orthorhombic,  $P2_12_12_1$   
 $a = 6.8576$  (5) Å  
 $b = 7.4936$  (6) Å  
 $c = 18.5003$  (14) Å  
 $V = 950.70$  (13) Å<sup>3</sup>

$Z = 4$   
Mo  $K\alpha$  radiation  
 $\mu = 0.36$  mm<sup>-1</sup>  
 $T = 290$  K  
 $0.26 \times 0.22 \times 0.17$  mm

## Data collection

Bruker SMART area-detector diffractometer  
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.912$ ,  $T_{\max} = 0.941$

8224 measured reflections  
2174 independent reflections  
1734 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.043$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.048$   
 $wR(F^2) = 0.136$   
 $S = 1.00$   
2174 reflections  
129 parameters  
H-atom parameters constrained

$\Delta\rho_{\max} = 0.25$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.33$  e Å<sup>-3</sup>  
Absolute structure: Flack (1983),  
838 Friedel pairs  
Flack parameter: 0.2 (2)

Data collection: SMART (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: XSEED (Barbour, 2001); software used to prepare material for publication: publCIF (Westrip, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5085).

## References

- Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.  
Bruker (2004). SAINT and SMART. Bruker AXS Inc., Madison, Wisconsin, USA.  
Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.  
Meth-Cohn, O. (1993). *Heterocycles*, **35**, 539–557.  
Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.  
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.  
Westrip, S. P. (2009). publCIF. In preparation.

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## supporting information

*Acta Cryst.* (2009). E65, o2722 [https://doi.org/10.1107/S1600536809040859]

### 2-Chloro-8-methylquinoline-3-carbaldehyde

**F. Nawaz Khan, R. Subashini, Atul Kumar Kushwaha, Venkatesha R. Hathwar and Seik Weng Ng**

#### S1. Experimental

The Vilsmeier-Haack reagent prepared from phosphorus oxytrichloride (6.5 ml, 70 mmol) and *N,N*-dimethylformamide (2.3 ml, 30 mmol) at 273 K was added *N*-(2-tolyl)acetamide (1.49 g, 10 mmol). The mixture was heated at 353 K for 15 h. The mixture was poured onto ice; the white product was collected and dried. The compound was purified by recrystallization from a petroleum ether/ethyl acetate mixture.

#### S2. Refinement

Carbon-bound H-atoms were placed in calculated positions (C–H 0.93–0.96 Å) and were included in the refinement in the riding model approximation, with  $U(\text{H})$  set to  $1.2\text{--}1.5U(\text{C})$ .

The crystal had two domains related by a translation of  $(1/2, 0, 0)$  so that all reflections with  $h = 2n$  are affected. A scale factor was added for all reflections with  $h = 2n$ . The *hkl* file had a scale factor of 1 for  $h = 2n + 1$  and a scale factor of 2 for the  $h = 2n$  reflections.

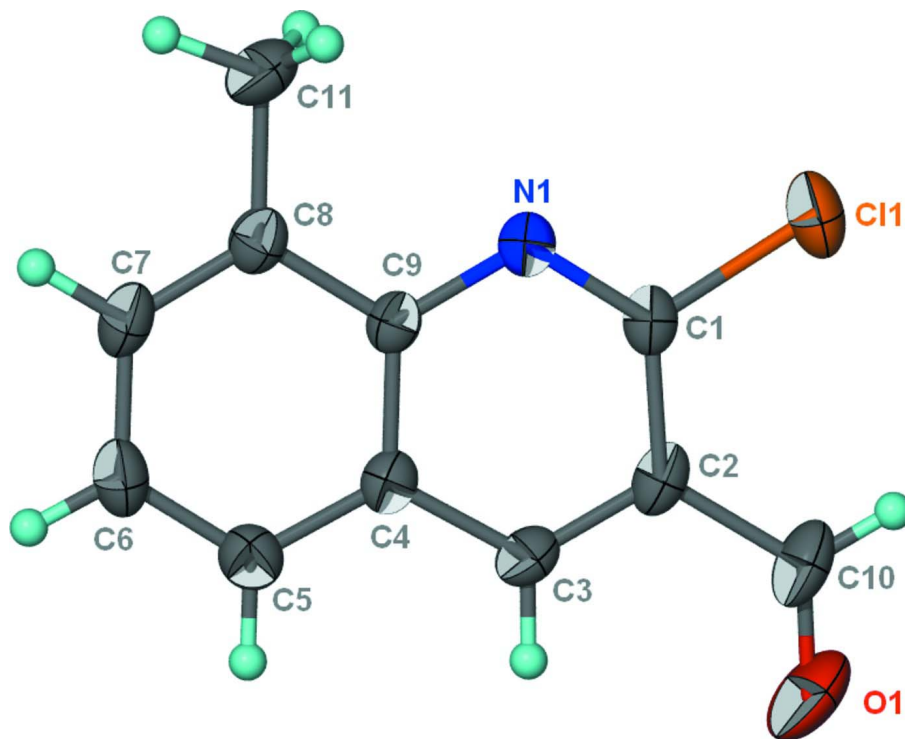


Figure 1

Thermal ellipsoid plot (Barbour, 2001) of  $C_{11}H_8ClNO$  at the 50% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

### 2-Chloro-8-methylquinoline-3-carbaldehyde

#### Crystal data

$C_{11}H_8ClNO$

$M_r = 205.63$

Orthorhombic,  $P2_12_12_1$

Hall symbol:  $P\ 2ac\ 2ab$

$a = 6.8576\ (5)\ \text{\AA}$

$b = 7.4936\ (6)\ \text{\AA}$

$c = 18.5003\ (14)\ \text{\AA}$

$V = 950.70\ (13)\ \text{\AA}^3$

$Z = 4$

$F(000) = 424$

$D_x = 1.437\ \text{Mg m}^{-3}$

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

Cell parameters from 867 reflections

$\theta = 2.0\text{--}24.4^\circ$

$\mu = 0.36\ \text{mm}^{-1}$

$T = 290\ \text{K}$

Block, colorless

$0.26 \times 0.22 \times 0.17\ \text{mm}$

#### Data collection

Bruker SMART area-detector  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\varphi$  and  $\omega$  scans

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

$T_{\min} = 0.912$ ,  $T_{\max} = 0.941$

8224 measured reflections

2174 independent reflections

1734 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.043$

$\theta_{\max} = 27.5^\circ$ ,  $\theta_{\min} = 2.2^\circ$

$h = -8 \rightarrow 8$

$k = -9 \rightarrow 9$

$l = -22 \rightarrow 24$

*Refinement*Refinement on  $F^2$ 

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.048$  $wR(F^2) = 0.136$  $S = 1.00$ 

2174 reflections

129 parameters

0 restraints

Primary atom site location: structure-invariant  
direct methodsSecondary atom site location: difference Fourier  
mapHydrogen site location: inferred from  
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0861P)^2 + 0.0263P]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\max} = 0.001$  $\Delta\rho_{\max} = 0.25 \text{ e } \text{\AA}^{-3}$  $\Delta\rho_{\min} = -0.33 \text{ e } \text{\AA}^{-3}$ Absolute structure: Flack (1983), 838 Friedel  
pairs

Absolute structure parameter: 0.2 (2)

*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	0.87010 (13)	1.14505 (9)	0.37890 (4)	0.0579 (3)
O1	0.8672 (5)	0.6106 (3)	0.29577 (10)	0.0763 (6)
N1	0.8717 (4)	0.9871 (2)	0.50346 (10)	0.0366 (4)
C1	0.8734 (5)	0.9563 (3)	0.43459 (13)	0.0368 (5)
C2	0.8754 (5)	0.7858 (3)	0.40176 (11)	0.0398 (5)
C3	0.8734 (5)	0.6423 (4)	0.44734 (12)	0.0389 (5)
H3	0.8741	0.5276	0.4281	0.047*
C4	0.8705 (4)	0.6651 (3)	0.52293 (10)	0.0344 (5)
C5	0.8691 (5)	0.5212 (3)	0.57186 (14)	0.0443 (6)
H5	0.8707	0.4045	0.5547	0.053*
C6	0.8654 (6)	0.5531 (3)	0.64410 (13)	0.0468 (6)
H6	0.8633	0.4585	0.6766	0.056*
C7	0.8648 (5)	0.7301 (4)	0.66955 (13)	0.0443 (6)
H7	0.8614	0.7491	0.7192	0.053*
C8	0.8690 (4)	0.8754 (3)	0.62492 (11)	0.0372 (5)
C9	0.8702 (4)	0.8426 (3)	0.54918 (11)	0.0327 (4)
C10	0.8840 (6)	0.7545 (5)	0.32246 (14)	0.0551 (7)
H10	0.9034	0.8521	0.2922	0.066*
C11	0.8698 (7)	1.0624 (3)	0.65349 (14)	0.0547 (7)
H11A	0.8779	1.0598	0.7053	0.082*
H11B	0.7519	1.1218	0.6392	0.082*
H11C	0.9801	1.1255	0.6343	0.082*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C11	0.0687 (4)	0.0591 (4)	0.0459 (4)	0.0008 (5)	0.0006 (4)	0.0218 (3)
O1	0.0887 (16)	0.1007 (17)	0.0394 (10)	-0.008 (2)	-0.0031 (13)	-0.0254 (11)
N1	0.0371 (10)	0.0403 (10)	0.0324 (10)	-0.0007 (12)	0.0005 (12)	0.0028 (7)
C1	0.0327 (11)	0.0459 (13)	0.0318 (12)	0.0001 (17)	-0.0007 (16)	0.0062 (10)
C2	0.0347 (11)	0.0576 (14)	0.0272 (10)	-0.0003 (15)	-0.0008 (12)	-0.0019 (10)
C3	0.0393 (11)	0.0427 (11)	0.0348 (12)	-0.002 (2)	-0.0005 (14)	-0.0084 (10)

C4	0.0323 (10)	0.0410 (11)	0.0300 (10)	-0.0007 (15)	0.0005 (11)	-0.0004 (8)
C5	0.0509 (14)	0.0398 (12)	0.0422 (14)	0.0026 (19)	0.0008 (19)	0.0040 (10)
C6	0.0509 (14)	0.0519 (14)	0.0375 (12)	0.0021 (17)	0.0007 (16)	0.0120 (10)
C7	0.0457 (14)	0.0596 (15)	0.0276 (11)	0.0043 (19)	-0.0022 (15)	0.0020 (11)
C8	0.0344 (10)	0.0461 (12)	0.0310 (11)	0.0029 (12)	-0.0009 (13)	-0.0014 (9)
C9	0.0292 (10)	0.0407 (11)	0.0280 (10)	-0.0001 (16)	0.0004 (12)	0.0002 (9)
C10	0.0538 (17)	0.084 (2)	0.0278 (12)	0.000 (2)	-0.0010 (17)	-0.0032 (13)
C11	0.0716 (17)	0.0543 (15)	0.0384 (13)	0.003 (2)	-0.0005 (19)	-0.0128 (11)

*Geometric parameters (Å, °)*

C11—C1	1.750 (2)	C5—H5	0.9300
O1—C10	1.191 (4)	C6—C7	1.407 (4)
N1—C1	1.295 (3)	C6—H6	0.9300
N1—C9	1.374 (3)	C7—C8	1.367 (3)
C1—C2	1.415 (3)	C7—H7	0.9300
C2—C3	1.367 (3)	C8—C9	1.423 (3)
C2—C10	1.487 (3)	C8—C11	1.498 (3)
C3—C4	1.409 (3)	C10—H10	0.9300
C3—H3	0.9300	C11—H11A	0.9600
C4—C5	1.408 (3)	C11—H11B	0.9600
C4—C9	1.416 (3)	C11—H11C	0.9600
C5—C6	1.358 (4)		
C1—N1—C9	117.73 (18)	C8—C7—C6	123.3 (2)
N1—C1—C2	125.68 (19)	C8—C7—H7	118.4
N1—C1—C11	115.80 (18)	C6—C7—H7	118.4
C2—C1—C11	118.52 (18)	C7—C8—C9	117.2 (2)
C3—C2—C1	116.47 (19)	C7—C8—C11	122.2 (2)
C3—C2—C10	119.0 (2)	C9—C8—C11	120.6 (2)
C1—C2—C10	124.5 (2)	N1—C9—C4	121.95 (19)
C2—C3—C4	121.1 (2)	N1—C9—C8	118.06 (19)
C2—C3—H3	119.4	C4—C9—C8	119.99 (19)
C4—C3—H3	119.4	O1—C10—C2	123.2 (3)
C5—C4—C3	123.0 (2)	O1—C10—H10	118.4
C5—C4—C9	119.9 (2)	C2—C10—H10	118.4
C3—C4—C9	117.0 (2)	C8—C11—H11A	109.5
C6—C5—C4	119.9 (2)	C8—C11—H11B	109.5
C6—C5—H5	120.1	H11A—C11—H11B	109.5
C4—C5—H5	120.1	C8—C11—H11C	109.5
C5—C6—C7	119.7 (2)	H11A—C11—H11C	109.5
C5—C6—H6	120.2	H11B—C11—H11C	109.5
C7—C6—H6	120.2		
C9—N1—C1—C2	-0.5 (5)	C6—C7—C8—C9	1.3 (5)
C9—N1—C1—C11	178.7 (2)	C6—C7—C8—C11	-179.5 (4)
N1—C1—C2—C3	0.6 (5)	C1—N1—C9—C4	0.0 (4)
C11—C1—C2—C3	-178.6 (3)	C1—N1—C9—C8	179.8 (3)

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N1—C1—C2—C10	-177.9 (3)	C5—C4—C9—N1	179.9 (3)
C11—C1—C2—C10	2.9 (5)	C3—C4—C9—N1	0.3 (5)
C1—C2—C3—C4	-0.3 (5)	C5—C4—C9—C8	0.2 (4)
C10—C2—C3—C4	178.3 (3)	C3—C4—C9—C8	-179.4 (2)
C2—C3—C4—C5	-179.8 (3)	C7—C8—C9—N1	179.1 (3)
C2—C3—C4—C9	-0.1 (5)	C11—C8—C9—N1	-0.1 (5)
C3—C4—C5—C6	-179.7 (4)	C7—C8—C9—C4	-1.1 (4)
C9—C4—C5—C6	0.7 (5)	C11—C8—C9—C4	179.6 (3)
C4—C5—C6—C7	-0.6 (6)	C3—C2—C10—O1	8.8 (7)
C5—C6—C7—C8	-0.4 (6)	C1—C2—C10—O1	-172.8 (4)

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