

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

2-Chloro-8-methoxyquinoline-3-carbaldehyde

R. Subashini,^a F. Nawaz Khan,^a Machhindra Gund,^a
Venkatesha R. Hathwar^b and Seik Weng Ng^{c*}^aChemistry Division, School of Science and Humanities, VIT University, Vellore 632 014, Tamil Nadu, India, ^bSolid State and Structural Chemistry Unit, Indian Institute of Science, Bangalore 560 012, Karnataka, India, and ^cDepartment of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
Correspondence e-mail: seikweng@um.edu.my

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 = 0.037; wR factor = 0.149; data-to-parameter ratio = 16.1.

In the title compound, $\text{C}_{11}\text{H}_8\text{ClNO}_2$, the quinoline fused-ring system is almost planar (r.m.s. deviation = 0.020 Å). The formyl group is slightly bent out of the quinoline plane [deviation of the O atom = 0.371 (2) Å].

Related literature

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



Experimental

Crystal data

 $\text{C}_{11}\text{H}_8\text{ClNO}_2$
 $M_r = 221.63$
Monoclinic, $P2_1/n$
 $a = 14.4763$ (8) Å
 $b = 3.9246$ (2) Å
 $c = 17.6295$ (9) Å
 $\beta = 104.802$ (3)° $V = 968.36$ (9) Å³
 $Z = 4$
Mo $K\alpha$ radiation
 $\mu = 0.37$ mm⁻¹
 $T = 290$ K
 $0.30 \times 0.18 \times 0.11$ mm

Data collection

Bruker SMART area-detector diffractometer
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.897$, $T_{\max} = 0.961$ 8150 measured reflections
2212 independent reflections
1769 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.026$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.037$
 $wR(F^2) = 0.149$
 $S = 1.18$
2212 reflections137 parameters
H-atom parameters constrained
 $\Delta\rho_{\max} = 0.30$ e Å⁻³
 $\Delta\rho_{\min} = -0.38$ e Å⁻³

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: X-SEED (Barbour, 2001); software used to prepare material for publication: publCIF (Westrip, 2009).

We thank the Department of Science and Technology, India, for use of the diffraction facility at IISc under the IRHPA–DST program; FNK thanks the DST for Fast Track Proposal funding. We also thank VIT University and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5131).

References

- Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Bruker (2004). SAINT and SMART. Bruker AXS Inc., Madison, Wisconsin, USA.
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.

supporting information

Acta Cryst. (2009). E65, o2720 [https://doi.org/10.1107/S1600536809040835]

2-Chloro-8-methoxyquinoline-3-carbaldehyde

R. Subashini, F. Nawaz Khan, Machhindra Gund, Venkatesha R. Hathwar and Seik Weng Ng

S1. Experimental

A Vilsmeier-Haack adduct 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-anisyl)acetamide (1.65 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 to yield colourless blocks of (I).

S2. Refinement

The 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 1.2–1.5 $U(\text{C})$.

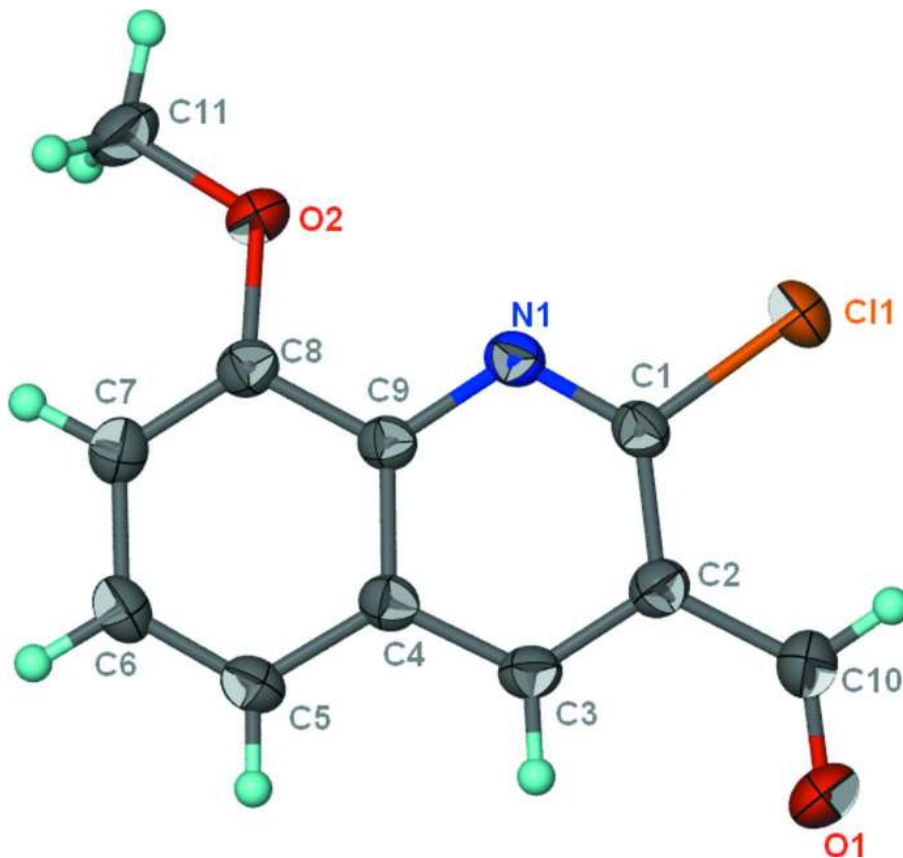


Figure 1

The molecular structure of (I) at the 50% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

2-Chloro-8-methoxyquinoline-3-carbaldehyde

Crystal data

C₁₁H₈ClNO₂ $M_r = 221.63$ Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

 $a = 14.4763$ (8) Å $b = 3.9246$ (2) Å $c = 17.6295$ (9) Å $\beta = 104.802$ (3)° $V = 968.36$ (9) Å³ $Z = 4$ $F(000) = 456$ $D_x = 1.520$ Mg m⁻³Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 943 reflections

 $\theta = 1.9$ – 24.6 ° $\mu = 0.37$ mm⁻¹ $T = 290$ K

Block, colourless

 $0.30 \times 0.18 \times 0.11$ mm

Data collection

Bruker SMART area-detector

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 φ and ω scans

Absorption correction: multi-scan

(SADABS; Sheldrick, 1996)

 $T_{\min} = 0.897$, $T_{\max} = 0.961$

8150 measured reflections

2212 independent reflections

1769 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.026$ $\theta_{\text{max}} = 27.5$ °, $\theta_{\text{min}} = 2.1$ ° $h = -18 \rightarrow 18$ $k = -4 \rightarrow 5$ $l = -22 \rightarrow 22$

Refinement

Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.037$ $wR(F^2) = 0.149$ $S = 1.18$

2212 reflections

137 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

 $w = 1/[\sigma^2(F_o^2) + (0.0883P)^2 + 0.0632P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\text{max}} = 0.001$ $\Delta\rho_{\text{max}} = 0.30$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.38$ e Å⁻³Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.80902 (4)	0.23452 (14)	0.68475 (3)	0.0499 (2)
N1	0.71583 (11)	0.3286 (4)	0.54057 (8)	0.0338 (4)
O1	1.02603 (11)	0.8263 (5)	0.61921 (10)	0.0627 (5)
O2	0.54659 (9)	0.2331 (4)	0.44173 (8)	0.0450 (4)
C1	0.79779 (12)	0.3856 (5)	0.58921 (9)	0.0333 (4)
C2	0.87625 (12)	0.5588 (5)	0.57179 (10)	0.0347 (4)
C3	0.86350 (13)	0.6661 (5)	0.49563 (10)	0.0344 (4)
H3	0.9129	0.7785	0.4811	0.041*
C4	0.77669 (12)	0.6081 (5)	0.43926 (10)	0.0320 (4)
C5	0.75979 (14)	0.7149 (5)	0.36018 (11)	0.0377 (4)
H5	0.8080	0.8205	0.3426	0.045*
C6	0.67289 (14)	0.6619 (5)	0.31039 (10)	0.0395 (4)
H6	0.6620	0.7325	0.2585	0.047*

C7	0.59819 (13)	0.5015 (5)	0.33542 (10)	0.0381 (4)
H7	0.5390	0.4710	0.3001	0.046*
C8	0.61230 (12)	0.3907 (5)	0.41126 (10)	0.0339 (4)
C9	0.70294 (11)	0.4414 (4)	0.46533 (9)	0.0304 (4)
C10	0.96654 (14)	0.6382 (6)	0.63092 (11)	0.0451 (5)
H10	0.9774	0.5355	0.6799	0.054*
C11	0.45218 (13)	0.1914 (6)	0.39243 (13)	0.0477 (5)
H11A	0.4134	0.0738	0.4208	0.072*
H11B	0.4548	0.0613	0.3469	0.072*
H11C	0.4249	0.4110	0.3764	0.072*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0550 (4)	0.0636 (4)	0.0312 (3)	-0.0071 (2)	0.0116 (2)	0.00723 (19)
N1	0.0347 (8)	0.0368 (8)	0.0325 (7)	-0.0019 (6)	0.0132 (6)	-0.0004 (6)
O1	0.0409 (8)	0.0860 (13)	0.0560 (10)	-0.0244 (8)	0.0028 (7)	0.0043 (9)
O2	0.0321 (7)	0.0598 (10)	0.0421 (8)	-0.0124 (6)	0.0078 (6)	0.0026 (6)
C1	0.0373 (9)	0.0355 (9)	0.0288 (8)	-0.0012 (7)	0.0115 (6)	-0.0005 (7)
C2	0.0315 (9)	0.0369 (10)	0.0360 (9)	-0.0019 (7)	0.0088 (7)	-0.0028 (7)
C3	0.0311 (9)	0.0372 (9)	0.0377 (9)	-0.0035 (7)	0.0138 (7)	-0.0011 (7)
C4	0.0338 (9)	0.0318 (9)	0.0326 (8)	0.0006 (7)	0.0127 (7)	-0.0016 (7)
C5	0.0399 (10)	0.0411 (11)	0.0353 (10)	-0.0009 (8)	0.0157 (8)	0.0031 (7)
C6	0.0451 (10)	0.0443 (11)	0.0297 (9)	0.0047 (8)	0.0110 (7)	0.0032 (7)
C7	0.0359 (9)	0.0411 (10)	0.0358 (9)	0.0021 (8)	0.0062 (7)	-0.0036 (7)
C8	0.0312 (9)	0.0344 (10)	0.0372 (9)	-0.0005 (7)	0.0109 (7)	-0.0034 (7)
C9	0.0310 (8)	0.0310 (9)	0.0315 (8)	-0.0002 (7)	0.0121 (6)	-0.0024 (6)
C10	0.0403 (11)	0.0549 (12)	0.0376 (10)	-0.0038 (9)	0.0053 (8)	0.0011 (9)
C11	0.0304 (10)	0.0542 (13)	0.0553 (13)	-0.0076 (8)	0.0053 (8)	-0.0010 (9)

Geometric parameters (Å, °)

C11—C1	1.7536 (17)	C4—C9	1.425 (2)
N1—C1	1.294 (2)	C5—C6	1.354 (3)
N1—C9	1.365 (2)	C5—H5	0.9300
O1—C10	1.192 (3)	C6—C7	1.416 (3)
O2—C8	1.355 (2)	C6—H6	0.9300
O2—C11	1.429 (2)	C7—C8	1.371 (2)
C1—C2	1.423 (3)	C7—H7	0.9300
C2—C3	1.374 (2)	C8—C9	1.425 (2)
C2—C10	1.482 (2)	C10—H10	0.9300
C3—C4	1.407 (2)	C11—H11A	0.9600
C3—H3	0.9300	C11—H11B	0.9600
C4—C5	1.416 (2)	C11—H11C	0.9600
C1—N1—C9	117.55 (15)	C7—C6—H6	119.2
C8—O2—C11	118.05 (15)	C8—C7—C6	120.64 (16)
N1—C1—C2	125.95 (16)	C8—C7—H7	119.7

N1—C1—C11	114.99 (13)	C6—C7—H7	119.7
C2—C1—C11	119.05 (13)	O2—C8—C7	125.91 (16)
C3—C2—C1	116.23 (15)	O2—C8—C9	114.72 (15)
C3—C2—C10	119.81 (16)	C7—C8—C9	119.37 (16)
C1—C2—C10	123.91 (16)	N1—C9—C8	118.58 (15)
C2—C3—C4	120.89 (16)	N1—C9—C4	122.33 (15)
C2—C3—H3	119.6	C8—C9—C4	119.09 (16)
C4—C3—H3	119.6	O1—C10—C2	123.71 (19)
C3—C4—C5	123.09 (16)	O1—C10—H10	118.1
C3—C4—C9	117.00 (16)	C2—C10—H10	118.1
C5—C4—C9	119.89 (16)	O2—C11—H11A	109.5
C6—C5—C4	119.43 (17)	O2—C11—H11B	109.5
C6—C5—H5	120.3	H11A—C11—H11B	109.5
C4—C5—H5	120.3	O2—C11—H11C	109.5
C5—C6—C7	121.57 (17)	H11A—C11—H11C	109.5
C5—C6—H6	119.2	H11B—C11—H11C	109.5
C9—N1—C1—C2	0.5 (3)	C11—O2—C8—C9	176.32 (16)
C9—N1—C1—C11	179.85 (12)	C6—C7—C8—O2	-179.69 (18)
N1—C1—C2—C3	-1.7 (3)	C6—C7—C8—C9	0.6 (3)
C11—C1—C2—C3	179.00 (14)	C1—N1—C9—C8	-178.13 (16)
N1—C1—C2—C10	175.74 (18)	C1—N1—C9—C4	1.7 (3)
C11—C1—C2—C10	-3.6 (3)	O2—C8—C9—N1	0.5 (2)
C1—C2—C3—C4	0.6 (3)	C7—C8—C9—N1	-179.81 (16)
C10—C2—C3—C4	-176.92 (17)	O2—C8—C9—C4	-179.36 (16)
C2—C3—C4—C5	179.70 (17)	C7—C8—C9—C4	0.3 (3)
C2—C3—C4—C9	1.3 (3)	C3—C4—C9—N1	-2.6 (3)
C3—C4—C5—C6	-177.25 (17)	C5—C4—C9—N1	178.97 (16)
C9—C4—C5—C6	1.1 (3)	C3—C4—C9—C8	177.22 (16)
C4—C5—C6—C7	-0.1 (3)	C5—C4—C9—C8	-1.2 (3)
C5—C6—C7—C8	-0.8 (3)	C3—C2—C10—O1	9.2 (3)
C11—O2—C8—C7	-3.4 (3)	C1—C2—C10—O1	-168.2 (2)
