

## 8-Quinolyguanidinium chloride

**Chang-Mei Wei**

Department of Chemistry of Huaiyin Teachers College, Jiangsu Key Laboratory for the Chemistry of Low-Dimensional Materials, Huaian 223300, People's Republic of China

Correspondence e-mail: weichangmei@sina.com

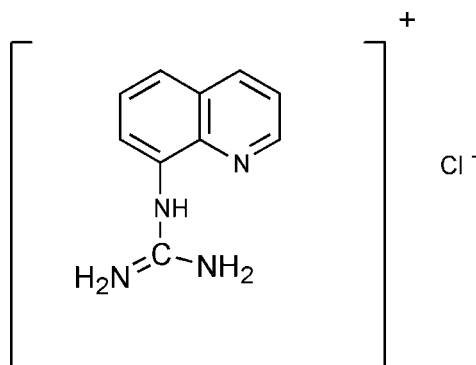
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 Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å;  $R$  factor = 0.062;  $wR$  factor = 0.108; data-to-parameter ratio = 17.6.

The title compound,  $\text{C}_{10}\text{H}_{11}\text{N}_4^+\cdot\text{Cl}^-$ , has been synthesized by the reaction of 8-aminoquinoline and cyanamide. The dihedral angle between the plane of the guanidine group and the quinoline ring system is  $68.64(13)^\circ$ . The crystal structure is stabilized by intermolecular  $\text{N}-\text{H}\cdots\text{Cl}$  hydrogen bonds.

### Related literature

For related literature, see: Hughes & Liu (1976); Juyal & Anand (2003); Knhla *et al.* (1986); Orner & Hamilton (2001).



### Experimental

#### Crystal data

 $\text{C}_{10}\text{H}_{11}\text{N}_4^+\cdot\text{Cl}^-$ 
 $M_r = 222.68$ 

 Orthorhombic,  $P2_12_12_1$ 
 $a = 8.7410(17)$  Å

 $b = 9.0230(18)$  Å

 $c = 13.942(3)$  Å

 $V = 1099.6(4)$  Å<sup>3</sup>
 $Z = 4$ 

 Mo  $K\alpha$  radiation

 $\mu = 0.32$  mm<sup>-1</sup>
 $T = 293(2)$  K

 $0.20 \times 0.20 \times 0.20$  mm

#### Data collection

Siemens P4 diffractometer  
Absorption correction: multi-scan  
(*XPRED* in *SHELXTL*;  
Sheldrick, 2008)  
 $T_{\min} = 0.939$ ,  $T_{\max} = 0.969$   
3398 measured reflections

2398 independent reflections  
2340 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.0301$   
3 standard reflections  
every 97 reflections  
intensity decay: 2.1%

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.062$   
 $wR(F^2) = 0.108$   
 $S = 0.99$   
2398 reflections  
136 parameters  
H-atom parameters constrained

$\Delta\rho_{\max} = 0.17$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.28$  e Å<sup>-3</sup>  
Absolute structure: Flack (1983),  
500 Friedel pairs  
Flack parameter: 0.02 (10)

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1A}\cdots\text{Cl1}^{\text{i}}$	0.86	2.34	3.171 (3)	162
$\text{N2}-\text{H2A}\cdots\text{Cl1}^{\text{i}}$	0.86	2.65	3.401 (3)	146
$\text{N2}-\text{H2B}\cdots\text{Cl1}^{\text{ii}}$	0.86	2.64	3.405 (3)	149
$\text{N3}-\text{H3A}\cdots\text{Cl1}^{\text{ii}}$	0.86	2.39	3.198 (3)	158
$\text{N3}-\text{H3B}\cdots\text{Cl1}$	0.86	2.46	3.269 (3)	156

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

Data collection: *XSCANS* (Bruker, 2000); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Sheldrick, 2008); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2213).

### References

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**supplementary materials**

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## 8-Quinolylguanidinium chloride

C.-M. Wei

### Comment

Guanidine is used in variety of supramolecular recognition processes across the spectrum of organic, biological and medicinal chemistry (Orner & Hamilton, 2001). Guanidine compounds containing a quinolyl ring are used as decongestive agents (Hughes & Liu, 1976) and in the treatment of gastrointestinal motility disorders (Knhla *et al.*, 1986). Guanidine derivatives are also employed as inhibitors of the reactions responsible for sedimentation in fuels as they efficiently disperse the gum and sediments formed (Juyal & Anand, 2003). These important compounds are therefore of interest from a structural viewpoint. In this paper, we report the crystal structure of the title compound, (I), which, to our knowledge, represents the first structure containing the 8-quinolylguanidinium cation. A perspective view of (I) is shown in Fig.1. In (I), bond lengths and angles within the 8-quinolylguanidinium cation (Table 1) indicate a partial conjugation between the guanidine group and the quinoline ring. The dihedral angle formed by the plane of the guanidine group and the quinoline ring is 68.64 (13)°. In the crystal packing, The chloride anion interacts with the cations though N—H···Cl hydrogen bonds forming a three dimensions network (Fig. 2, Table 2).

### Experimental

The title compound was synthesized as following. A mixture of 8-aminoquinoline (68.06 mmol), cyanamide (83.3 mmol) and ethanol (50 ml) was heated under reflux for 3 h with stirring. The reaction mixture was evaporated to give a residue. Single crystals suitable for X-ray analysis were obtained by slow evaporation of an aqueous solution.

### Refinement

All H atoms were placed in calculated positions with C—H = 0.93 Å, N—H = 0.86 Å, and refined as riding with  $U_{iso}(H) = 1.2 U_{eq}(C, N)$ .

### Figures

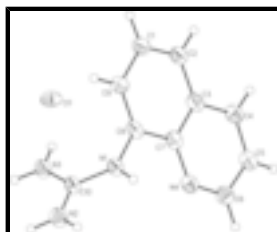


Fig. 1. The molecular structure drawing for (I) showing 50% probability of displacement ellipsoids and the atom-numbering scheme.

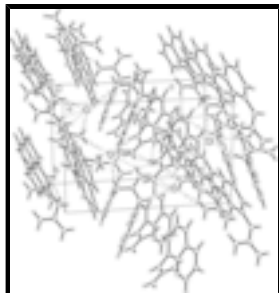


Fig. 2. The molecular packing diagram in the crystal for (I).

**'8-quinolyguanidine monohydrochloride'**

*Crystal data*

$C_{10}H_{11}N_4^+ \cdot Cl^-$   
 $M_r = 222.68$

Orthorhombic,  $P2(1)2(1)2(1)$

$a = 8.7410(17) \text{ \AA}$

$b = 9.0230(18) \text{ \AA}$

$c = 13.942(3) \text{ \AA}$

$V = 1099.6(4) \text{ \AA}^3$

$Z = 4$

$F_{000} = 464$

$D_x = 1.345 \text{ Mg m}^{-3}$

Melting point = 533–534 K

Mo  $K\alpha$  radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 25 reflections

$\theta = 2.1\text{--}25.6^\circ$

$\mu = 0.32 \text{ mm}^{-1}$

$T = 293(2) \text{ K}$

Block, yellow

$0.20 \times 0.20 \times 0.20 \text{ mm}$

*Data collection*

Siemens P4  
 diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293(2) \text{ K}$

$2\theta/\omega$  scans

Absorption correction: multi-scan  
 (XPREP in SHELXTL; Sheldrick, 2008)

$T_{\min} = 0.939$ ,  $T_{\max} = 0.969$

3398 measured reflections

2398 independent reflections

2340 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.030$

$\theta_{\max} = 27.0^\circ$

$\theta_{\min} = 2.7^\circ$

$h = -11 \rightarrow 11$

$k = -11 \rightarrow 11$

$l = -17 \rightarrow 17$

3 standard reflections

every 97 reflections

intensity decay: 2.1%

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.062$

$wR(F^2) = 0.108$

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0513P)^2 + 0.585P]$$

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

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

$S = 0.99$   $\Delta\rho_{\max} = 0.18 \text{ e } \text{\AA}^{-3}$   
 2398 reflections  $\Delta\rho_{\min} = -0.28 \text{ e } \text{\AA}^{-3}$   
 136 parameters Extinction correction: none  
 Primary atom site location: structure-invariant direct methods Absolute structure: Flack (1983), 500 Friedel pairs  
 Secondary atom site location: difference Fourier map Flack parameter: 0.02 (10)

*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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	0.87040 (9)	0.86783 (8)	0.66733 (5)	0.04274 (19)
N1	0.7592 (3)	0.9935 (3)	0.97479 (18)	0.0385 (5)
H1A	0.7204	1.0096	1.0305	0.046*
N2	0.8958 (3)	1.1997 (3)	0.99486 (19)	0.0470 (6)
H2A	0.8569	1.2079	1.0513	0.056*
H2B	0.9600	1.2646	0.9745	0.056*
N3	0.9244 (3)	1.0767 (3)	0.85594 (17)	0.0424 (6)
H3A	0.9918	1.1397	0.8371	0.051*
H3B	0.8995	1.0037	0.8195	0.051*
N4	0.4631 (3)	0.9657 (3)	0.90750 (19)	0.0424 (6)
C1	0.7557 (4)	0.6150 (4)	0.8683 (2)	0.0451 (7)
H1	0.8235	0.5370	0.8586	0.054*
C2	0.6087 (3)	0.6031 (3)	0.8407 (2)	0.0435 (7)
H2	0.5741	0.5145	0.8144	0.052*
C3	0.5062 (4)	0.7226 (3)	0.8511 (2)	0.0437 (7)
C4	0.3507 (4)	0.7159 (3)	0.8217 (2)	0.0455 (7)
H4A	0.3118	0.6310	0.7929	0.055*
C5	0.2599 (4)	0.8362 (3)	0.8366 (2)	0.0479 (7)
H5	0.1583	0.8351	0.8169	0.057*
C6	0.3210 (4)	0.9621 (4)	0.8819 (2)	0.0463 (7)
H6	0.2585	1.0436	0.8936	0.056*
C7	0.5564 (4)	0.8547 (4)	0.8952 (2)	0.0426 (7)
C8	0.7119 (3)	0.8643 (4)	0.92611 (19)	0.0398 (6)
C9	0.8061 (4)	0.7491 (3)	0.9126 (2)	0.0434 (7)
H9	0.9072	0.7566	0.9327	0.052*
C10	0.8584 (3)	1.0921 (3)	0.9418 (2)	0.0392 (6)

## supplementary materials

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C11	0.0583 (4)	0.0381 (3)	0.0318 (3)	0.0130 (3)	-0.0029 (3)	0.0014 (3)
N1	0.0418 (13)	0.0343 (12)	0.0395 (12)	-0.0066 (10)	-0.0012 (10)	-0.0012 (10)
N2	0.0490 (15)	0.0475 (15)	0.0445 (14)	-0.0093 (13)	0.0009 (12)	-0.0055 (12)
N3	0.0438 (14)	0.0413 (13)	0.0420 (14)	-0.0112 (11)	0.0029 (10)	-0.0047 (10)
N4	0.0424 (14)	0.0408 (14)	0.0440 (14)	-0.0057 (11)	-0.0028 (11)	0.0004 (11)
C1	0.0506 (17)	0.0380 (17)	0.0468 (15)	-0.0019 (15)	0.0020 (13)	-0.0019 (13)
C2	0.0483 (17)	0.0376 (15)	0.0446 (15)	-0.0059 (12)	0.0004 (14)	-0.0005 (13)
C3	0.0496 (17)	0.0398 (16)	0.0418 (17)	-0.0083 (13)	0.0030 (13)	-0.0009 (13)
C4	0.0478 (17)	0.0423 (15)	0.0464 (16)	-0.0071 (13)	-0.0045 (15)	0.0004 (13)
C5	0.0498 (17)	0.0454 (17)	0.0485 (16)	-0.0079 (14)	-0.0006 (16)	0.0026 (15)
C6	0.0493 (17)	0.0444 (17)	0.0452 (17)	-0.0024 (14)	0.0000 (14)	-0.0008 (13)
C7	0.0456 (16)	0.0396 (16)	0.0426 (15)	-0.0060 (14)	0.0010 (12)	0.0002 (14)
C8	0.0455 (15)	0.0357 (14)	0.0382 (14)	-0.0090 (14)	-0.0028 (12)	0.0011 (13)
C9	0.0460 (16)	0.0379 (15)	0.0462 (16)	-0.0013 (14)	0.0020 (13)	-0.0006 (13)
C10	0.0387 (15)	0.0367 (14)	0.0423 (14)	-0.0079 (12)	-0.0001 (13)	-0.0009 (11)

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

N1—C10	1.324 (4)	C1—H1	0.9300
N1—C8	1.411 (4)	C2—C3	1.410 (4)
N1—H1A	0.8600	C2—H2	0.9300
N2—C10	1.263 (4)	C3—C7	1.411 (4)
N2—H2A	0.8600	C3—C4	1.420 (4)
N2—H2B	0.8600	C4—C5	1.361 (4)
N3—C10	1.337 (4)	C4—H4A	0.9300
N3—H3A	0.8600	C5—C6	1.406 (4)
N3—H3B	0.8600	C5—H5	0.9300
N4—C6	1.293 (4)	C6—H6	0.9300
N4—C7	1.303 (4)	C7—C8	1.429 (4)
C1—C2	1.345 (5)	C8—C9	1.339 (5)
C1—C9	1.429 (4)	C9—H9	0.9300
C10—N1—C8	125.4 (3)	C5—C4—H4A	120.6
C10—N1—H1A	117.3	C3—C4—H4A	120.6
C8—N1—H1A	117.3	C4—C5—C6	119.5 (3)
C10—N2—H2A	120.0	C4—C5—H5	120.3
C10—N2—H2B	120.0	C6—C5—H5	120.3
H2A—N2—H2B	120.0	N4—C6—C5	120.6 (3)
C10—N3—H3A	120.0	N4—C6—H6	119.7
C10—N3—H3B	120.0	C5—C6—H6	119.7
H3A—N3—H3B	120.0	N4—C7—C3	120.8 (3)
C6—N4—C7	123.1 (3)	N4—C7—C8	120.6 (3)
C2—C1—C9	119.0 (3)	C3—C7—C8	118.6 (3)
C2—C1—H1	120.5	C9—C8—N1	121.9 (3)
C9—C1—H1	120.5	C9—C8—C7	119.7 (3)

C1—C2—C3	121.1 (3)	N1—C8—C7	118.3 (3)
C1—C2—H2	119.4	C8—C9—C1	122.0 (3)
C3—C2—H2	119.4	C8—C9—H9	119.0
C2—C3—C7	119.5 (3)	C1—C9—H9	119.0
C2—C3—C4	123.1 (3)	N2—C10—N1	118.8 (3)
C7—C3—C4	117.3 (3)	N2—C10—N3	119.5 (3)
C5—C4—C3	118.7 (3)	N1—C10—N3	121.6 (3)

*Hydrogen-bond geometry (Å, °)*

<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>
N1—H1A $\cdots$ C11 <sup>i</sup>	0.86	2.34	3.171 (3)	162
N2—H2A $\cdots$ C11 <sup>i</sup>	0.86	2.65	3.401 (3)	146
N2—H2B $\cdots$ C11 <sup>ii</sup>	0.86	2.64	3.405 (3)	149
N3—H3A $\cdots$ C11 <sup>ii</sup>	0.86	2.39	3.198 (3)	158
N3—H3B $\cdots$ C11	0.86	2.46	3.269 (3)	156

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

Fig. 1

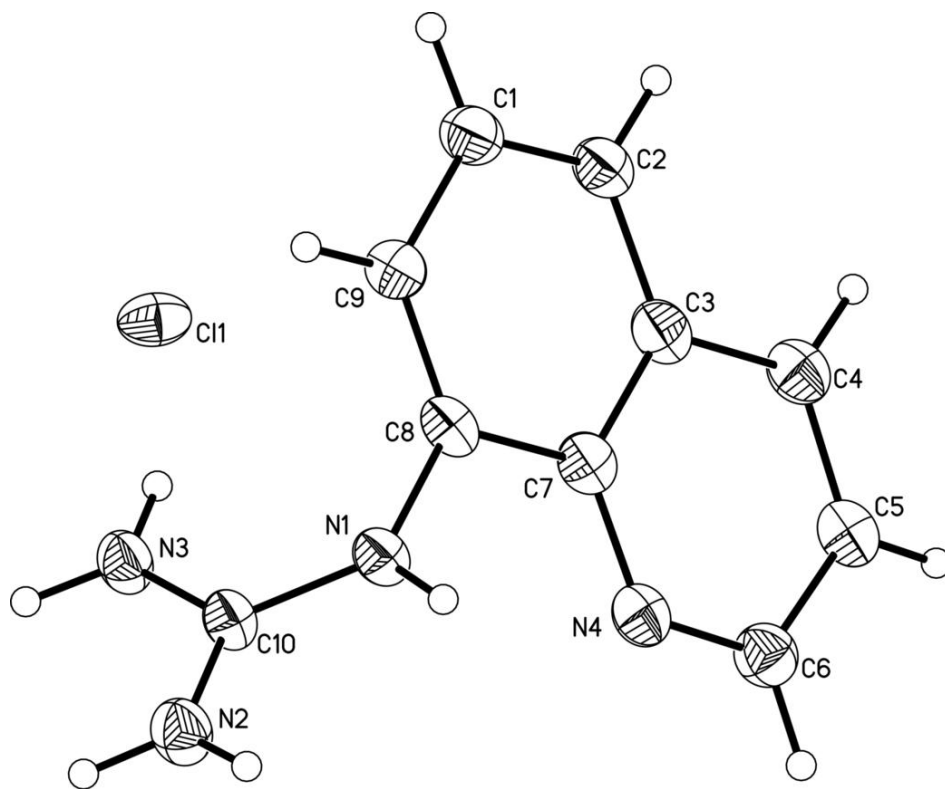




Fig. 2

