Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

8-Quinolylguanidinium chloride

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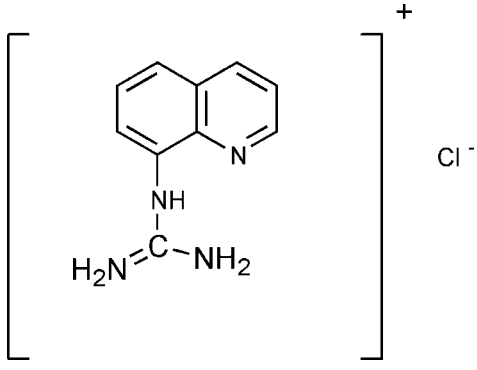
Received 20 April 2008; accepted 7 May 2008

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.062; wR factor = 0.108; data-to-parameter ratio = 17.6.

The title compound, $C_{10}H_{11}N_4^+\cdot Cl^-$, 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)°. The crystal structure is stabilized by intermolecular N—H···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

$C_{10}H_{11}N_4^+\cdot Cl^-$
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^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.32 mm^{-1}
T = 293 (2) K
0.20 × 0.20 × 0.20 mm

Data collection

Siemens P4 diffractometer
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_{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^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}
Absolute structure: Flack (1983), 500 Friedel pairs
Flack parameter: 0.02 (10)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···Cl1^{i}	0.86	2.34	3.171 (3)	162
N2—H2A···Cl1^{i}	0.86	2.65	3.401 (3)	146
N2—H2B···Cl1^{ii}	0.86	2.64	3.405 (3)	149
N3—H3A···Cl1^{ii}	0.86	2.39	3.198 (3)	158
N3—H3B···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*.

The author is grateful to the Science Foundation of Jiangsu Education Bureau (05KJD 150039), the Professor Foundation of Huaiyin Teachers College (05 HSJS018) and the Science Foundation of Jangsu Key Laboratory for the Chemistry of Low-Dimensional Materials (JSKC 06028) for financial support.

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

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supporting information

Acta Cryst. (2008). E**64**, o1244 [doi:10.1107/S1600536808013640]

8-Quinolylguanidinium chloride

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S1. 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-quinolylguanidium cation. A perspective view of (I) is shown in Fig.1. In (I), bond lengths and angles within the 8-quinolylguanidium 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).

S2. 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. Singles crystals suitable for X-ray analysis were obtained by slow evaporation of an aqueous solution.

S3. 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).

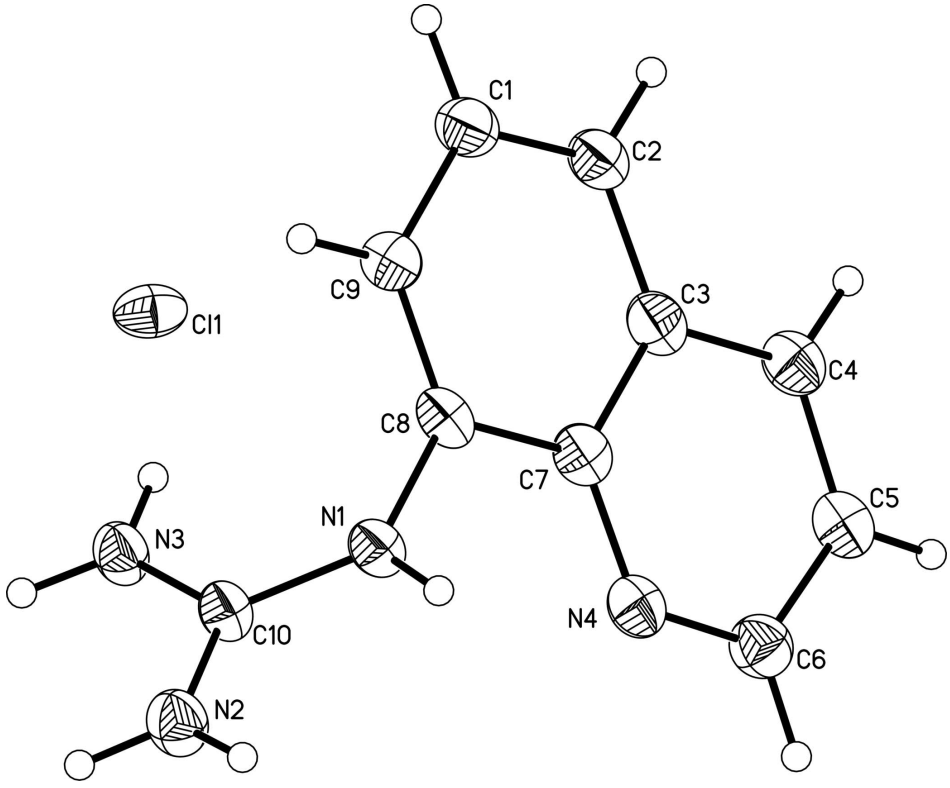

Figure 1

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

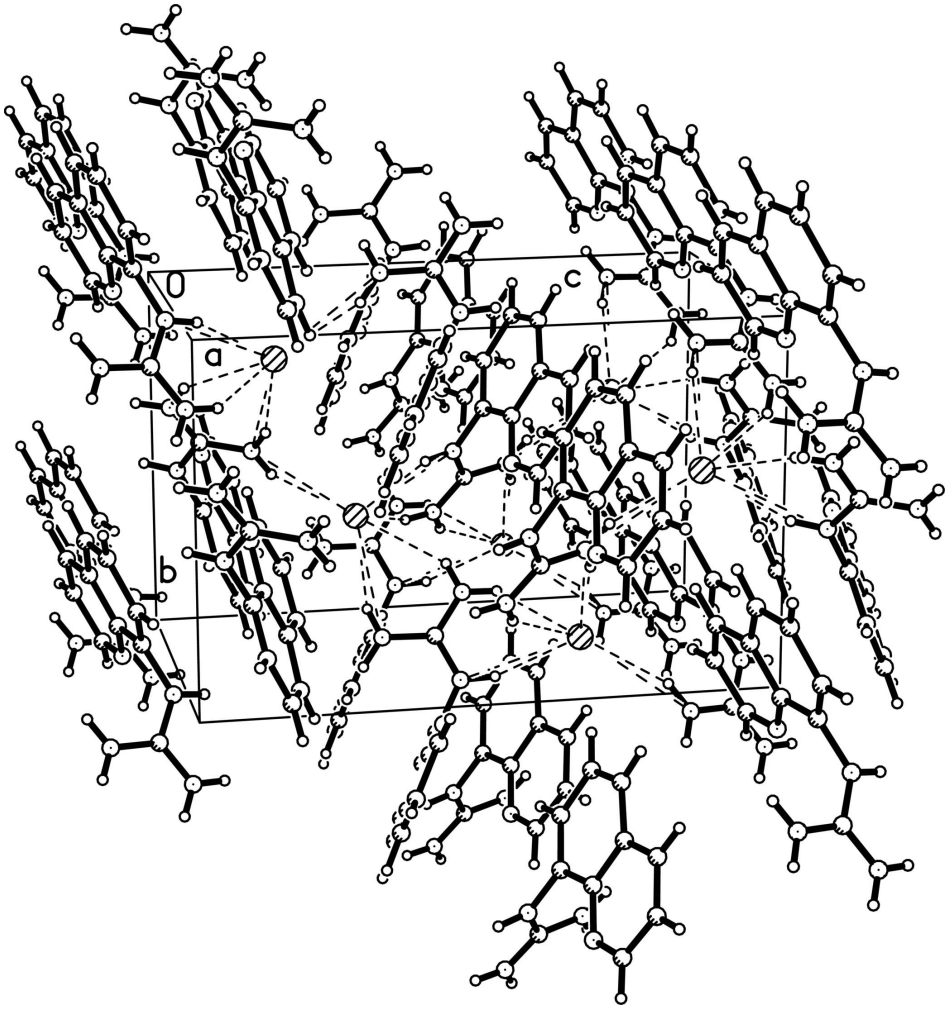

Figure 2

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

′8-quinolylguanidine monohydrochloride′

Crystal data

$C_{10}H_{11}N_4^+ \cdot Cl^-$
$M_r = 222.68$
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^3
$Z = 4$
$F(000) = 464$
$D_x = 1.345$ Mg m^{-3}
Melting point = 533–534 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 2.1$–$25.6°$
$\mu = 0.32$ mm^{-1}
$T = 293$ K
Block, yellow
0.20 × 0.20 × 0.20 mm

Data collection

Siemens P4
diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$2\theta/\omega$ 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 $I > 2\sigma(I)$
$R_{int} = 0.030$
$\theta_{max} = 27.0°$, $\theta_{min} = 2.7°$
$h = -11 \rightarrow 11$
$k = -11 \rightarrow 11$
$l = -17 \rightarrow 17$
3 standard reflections every 97 reflections
intensity decay: 2.1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.062$
$wR(F^2) = 0.108$
$S = 0.99$
2398 reflections
136 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
$w = 1/[\sigma^2(F_o^2) + (0.0513P)^2 + 0.585P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}
Absolute structure: Flack (1983), 500 Friedel pairs
Absolute structure 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	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)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	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 (Å, º)

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, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···Cl1^{i}	0.86	2.34	3.171 (3)	162
N2—H2*A*···Cl1^{i}	0.86	2.65	3.401 (3)	146
N2—H2*B*···Cl1^{ii}	0.86	2.64	3.405 (3)	149
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N3—H3*B*···Cl1	0.86	2.46	3.269 (3)	156

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