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## Structure Reports

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## 3-Carbamoylquinoxalin-1-ium chloride

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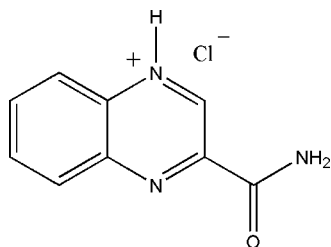
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Key indicators: single-crystal X-ray study;  $T = 150$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.034;  $wR$  factor = 0.085; data-to-parameter ratio = 13.4.

The title compound,  $\text{C}_9\text{H}_8\text{N}_3\text{O}^+\cdot\text{Cl}^-$ , was isolated from a liquid culture of *streptomyces* sp. In the cation, the ring system makes a dihedral angle of  $0.2(2)^\circ$  with the amide group. The protonation creating the cation occurs at one of the N atoms in the quinoxaline ring system. In the crystal, the ions are linked through  $\text{N}-\text{H}\cdots\text{O}$  and  $\text{N}-\text{H}\cdots\text{Cl}$  hydrogen bonds, forming a two-dimensional network parallel to  $(10\bar{3})$ .

## Related literature

For a description of the bioactivity and mode of action of compounds containing the quinoxaline moiety, see: Bailly *et al.* (1999); May *et al.* (2004); Mollegaard *et al.* (2000); Waring (1993). For crystal structures of the molecules triostin A, echinomycin and their derivatives, which all contain two quinoxalines, see: Hossain *et al.* (1982); Sheldrick *et al.* (1984, 1995); Viswamitra *et al.* (1981); Wang *et al.* (1984); Ughetto *et al.* (1985). For a description of the Streptomyces producing the title compound, see: Castillo *et al.* (2003).



## Experimental

## Crystal data

 $\text{C}_9\text{H}_8\text{N}_3\text{O}^+\cdot\text{Cl}^-$ 
 $M_r = 209.63$ 

 Monoclinic,  $P2_1/n$ 
 $a = 5.6476(2)$  Å

 $b = 15.1045(9)$  Å

 $c = 11.2556(6)$  Å

 $\beta = 99.993(3)^\circ$ 
 $V = 945.58(8)$  Å<sup>3</sup>
 $Z = 4$ 

 Mo  $K\alpha$  radiation

 $\mu = 0.37$  mm<sup>-1</sup>
 $T = 150$  K

 $0.25 \times 0.20 \times 0.08$  mm

## Data collection

Nonius KappaCCD diffractometer

Absorption correction: multi-scan

(DENZO-SMN; Otwinowski &amp;

Minor, 1997)

 $T_{\min} = 0.913$ ,  $T_{\max} = 0.971$ 

3671 measured reflections

2147 independent reflections

 1798 reflections with  $I > 2\sigma(I)$ 
 $R_{\text{int}} = 0.018$ 

## Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.034$ 
 $wR(F^2) = 0.085$ 
 $S = 1.05$ 

2147 reflections

160 parameters

All H-atom parameters refined

 $\Delta\rho_{\max} = 0.25$  e Å<sup>-3</sup>
 $\Delta\rho_{\min} = -0.24$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1A}\cdots\text{O1}^{\text{i}}$	0.86 (2)	2.04 (2)	2.9008 (17)	173.5 (17)
$\text{N1}-\text{H1B}\cdots\text{Cl1}$	0.90 (2)	2.44 (2)	3.2590 (13)	152.0 (17)
$\text{N3}-\text{H3N}\cdots\text{Cl1}^{\text{ii}}$	0.94 (2)	2.02 (2)	2.9501 (13)	169.8 (15)

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

Data collection: COLLECT (Nonius, 1998); cell refinement: DENZO-SMN (Otwinowski & Minor, 1997); data reduction: DENZO-SMN; program(s) used to solve structure: SIR97 (Altomare *et al.*, 1999); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: WinGX (Farrugia, 1999) and ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: SHELXL97.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5381).

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**supplementary materials**

*Acta Cryst.* (2012). E68, o79-o80 [ doi:10.1107/S1600536811052457 ]

### 3-Carbamoylquinoxalin-1-ium chloride

J. K. Harper, G. Strobel and A. M. Arif

#### Comment

The quinoxaline ring is an essential component of the DNA intercalators echinomycin and triostin A. The two quinoxaline rings present in each of these compounds bind the minor groove of double stranded DNA and thereby inhibit RNA synthesis (Bailey *et al.*, 1999; May *et al.*, 2004; Mollegaard *et al.*, 2000; Waring, 1993). Presently, the quinoxaline ring has been characterized crystallographically only as part of a significantly larger molecular assembly (Hossain *et al.*, 1982; Sheldrick *et al.*, 1984; Sheldrick *et al.*, 1995; Viswamitra *et al.*, 1981; Wang *et al.*, 1984; Ughetto *et al.*, 1985). Accordingly, the resolution of the quinoxaline moieties currently established is relatively low. Here, characterization of a simpler quinoxaline ring system provides a higher resolution dataset for a compound having a substitution pattern identical to that found in the quinoxaline antibiotics. The conformation about the C1—C2 bond in the title compound is shown in Figure 1 and matches that reported for triostin A and echinomycin. Molecules in the crystal are linked through N1—H···O1<sup>1</sup> (see Table 1 for symmetry codes) hydrogen bonds as well as N1—H···Cl···H—N3 interaction. The structure viewed along the *a* axis is shown in figure 2.

#### Experimental

The title compound was obtained by liquid-liquid extraction (CH<sub>2</sub>Cl<sub>2</sub>/H<sub>2</sub>O) of a culture of an endophytic *Streptomyces* sp. described elsewhere (Castillo *et al.*, 2003). A crystal was grown by slow evaporation of a 1:1 mix of CHCl<sub>3</sub>:MeOH

#### Refinement

All H atoms were refined independently with isotropic displacement parameters.

#### Figures

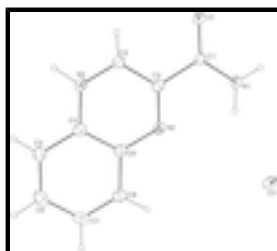


Fig. 1. Molecular structure of the title compound. Displacement ellipsoids are shown at the 50% probability level on non-hydrogen atoms.

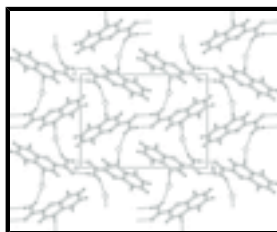


Fig. 2. Part of the crystal structure viewed along the *a* axis. The dashed lines indicate N—H···O and N—H···Cl hydrogen bonds.

## 3-Carbamoylquinoxalin-1-ium chloride

### Crystal data

$C_9H_8N_3O^+ \cdot Cl^-$

$M_r = 209.63$

Monoclinic,  $P2_1/n$

Hall symbol:  $-P\ 2yn$

$a = 5.6476\ (2)\ \text{\AA}$

$b = 15.1045\ (9)\ \text{\AA}$

$c = 11.2556\ (6)\ \text{\AA}$

$\beta = 99.993\ (3)^\circ$

$V = 945.58\ (8)\ \text{\AA}^3$

$Z = 4$

$F(000) = 432$

$D_x = 1.473\ \text{Mg m}^{-3}$

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

Cell parameters from 1998 reflections

$\theta = 1.0\text{--}27.5^\circ$

$\mu = 0.37\ \text{mm}^{-1}$

$T = 150\ \text{K}$

Plate, pale yellow

$0.25 \times 0.20 \times 0.08\ \text{mm}$

### Data collection

Nonius KappaCCD  
diffractometer

Radiation source: fine-focus sealed tube  
graphite

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan  
(*DENZO-SMN*; Otwinowski & Minor, 1997)

$T_{\min} = 0.913$ ,  $T_{\max} = 0.971$

3671 measured reflections

2147 independent reflections

1798 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.018$

$\theta_{\max} = 27.5^\circ$ ,  $\theta_{\min} = 3.9^\circ$

$h = -7 \rightarrow 7$

$k = -18 \rightarrow 19$

$l = -14 \rightarrow 14$

### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.034$

$wR(F^2) = 0.085$

$S = 1.05$

2147 reflections

160 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

All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0397P)^2 + 0.2499P]$

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

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

$\Delta\rho_{\max} = 0.25\ \text{e \AA}^{-3}$

$\Delta\rho_{\min} = -0.24\ \text{e \AA}^{-3}$

Extinction correction: *SHELXL97* (Sheldrick, 2008),

$F_c^* = kF_c[1 + 0.001 \times F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.012 (4)

*Special details*

**Experimental.** The program *DENZO-SMN* (Otwinowski & Minor, 1997) uses a scaling algorithm that effectively corrects for absorption effects. High redundancy data were used in the scaling program hence the 'multi-scan' code word was used. No transmission coefficients are available from the program (only scale factors for each frame). The scale factors in the experimental table are calculated from the 'size' command in the *SHELXL97* input file.

**Geometry.** All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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	0.08949 (6)	0.30272 (3)	0.19526 (3)	0.03255 (15)
O1	0.76925 (19)	0.43611 (7)	0.55896 (10)	0.0336 (3)
N1	0.4423 (2)	0.39023 (9)	0.42509 (12)	0.0269 (3)
N2	0.6771 (2)	0.23505 (8)	0.39555 (10)	0.0234 (3)
N3	1.1403 (2)	0.21019 (8)	0.51784 (11)	0.0248 (3)
C1	0.6625 (3)	0.38080 (9)	0.48828 (13)	0.0247 (3)
C2	0.7884 (2)	0.29539 (9)	0.46906 (12)	0.0234 (3)
C3	1.0240 (3)	0.28321 (10)	0.53318 (13)	0.0254 (3)
C4	1.0391 (2)	0.14589 (9)	0.43990 (12)	0.0237 (3)
C5	1.1680 (3)	0.06898 (10)	0.42012 (14)	0.0289 (3)
C6	1.0562 (3)	0.00651 (11)	0.34196 (14)	0.0338 (4)
C7	0.8153 (3)	0.01774 (11)	0.28407 (14)	0.0331 (4)
C8	0.6884 (3)	0.09223 (10)	0.30207 (13)	0.0276 (3)
C9	0.7997 (2)	0.15942 (9)	0.37975 (12)	0.0229 (3)
H1A	0.370 (3)	0.4397 (13)	0.4318 (16)	0.036 (5)*
H1B	0.381 (4)	0.3498 (15)	0.3699 (19)	0.051 (6)*
H3	1.102 (3)	0.3227 (12)	0.5857 (17)	0.035 (5)*
H3N	1.290 (3)	0.2011 (11)	0.5682 (17)	0.034 (5)*
H5	1.324 (3)	0.0634 (12)	0.4615 (16)	0.035 (5)*
H6	1.139 (3)	-0.0467 (12)	0.3273 (15)	0.032 (4)*
H7	0.738 (3)	-0.0276 (13)	0.2325 (17)	0.041 (5)*
H8	0.523 (3)	0.1023 (10)	0.2602 (15)	0.027 (4)*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C11	0.0270 (2)	0.0419 (2)	0.0258 (2)	-0.00693 (15)	-0.00344 (14)	-0.00243 (15)
O1	0.0323 (6)	0.0264 (5)	0.0365 (6)	0.0033 (4)	-0.0097 (5)	-0.0054 (5)

## supplementary materials

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N1	0.0258 (6)	0.0235 (6)	0.0284 (7)	0.0029 (5)	-0.0037 (5)	-0.0018 (5)
N2	0.0240 (6)	0.0249 (6)	0.0205 (6)	-0.0011 (5)	0.0015 (5)	0.0019 (5)
N3	0.0217 (6)	0.0290 (6)	0.0225 (6)	0.0013 (5)	0.0000 (5)	0.0016 (5)
C1	0.0263 (7)	0.0231 (7)	0.0228 (7)	-0.0001 (6)	-0.0011 (5)	0.0023 (6)
C2	0.0235 (7)	0.0251 (7)	0.0212 (7)	-0.0014 (5)	0.0030 (5)	0.0016 (5)
C3	0.0244 (7)	0.0268 (7)	0.0232 (7)	-0.0009 (6)	-0.0007 (6)	-0.0007 (6)
C4	0.0253 (7)	0.0258 (7)	0.0204 (7)	-0.0011 (5)	0.0051 (5)	0.0029 (5)
C5	0.0286 (8)	0.0315 (8)	0.0274 (8)	0.0053 (6)	0.0075 (6)	0.0033 (6)
C6	0.0433 (9)	0.0288 (8)	0.0321 (8)	0.0056 (7)	0.0144 (7)	-0.0001 (7)
C7	0.0428 (9)	0.0303 (8)	0.0279 (8)	-0.0050 (7)	0.0102 (7)	-0.0071 (7)
C8	0.0296 (8)	0.0314 (8)	0.0220 (7)	-0.0046 (6)	0.0052 (6)	-0.0018 (6)
C9	0.0257 (7)	0.0246 (7)	0.0189 (7)	-0.0008 (6)	0.0050 (5)	0.0022 (5)

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

O1—C1	1.2361 (17)	C3—H3	0.900 (19)
N1—C1	1.3285 (18)	C4—C5	1.409 (2)
N1—H1A	0.86 (2)	C4—C9	1.4178 (19)
N1—H1B	0.90 (2)	C5—C6	1.368 (2)
N2—C2	1.3154 (18)	C5—H5	0.928 (17)
N2—C9	1.3635 (18)	C6—C7	1.413 (2)
N3—C3	1.3104 (19)	C6—H6	0.957 (18)
N3—C4	1.3660 (19)	C7—C8	1.368 (2)
N3—H3N	0.94 (2)	C7—H7	0.95 (2)
C1—C2	1.5066 (19)	C8—C9	1.414 (2)
C2—C3	1.411 (2)	C8—H8	0.980 (16)
C1—N1—H1A	117.3 (12)	N3—C4—C9	117.53 (13)
C1—N1—H1B	120.9 (13)	C5—C4—C9	121.29 (13)
H1A—N1—H1B	121.2 (18)	C6—C5—C4	118.49 (15)
C2—N2—C9	117.67 (12)	C6—C5—H5	123.5 (11)
C3—N3—C4	121.30 (13)	C4—C5—H5	118.0 (11)
C3—N3—H3N	117.5 (10)	C5—C6—C7	120.93 (15)
C4—N3—H3N	120.9 (10)	C5—C6—H6	120.4 (10)
O1—C1—N1	125.36 (13)	C7—C6—H6	118.6 (10)
O1—C1—C2	118.78 (12)	C8—C7—C6	121.19 (15)
N1—C1—C2	115.85 (12)	C8—C7—H7	118.8 (11)
N2—C2—C3	122.45 (13)	C6—C7—H7	120.0 (11)
N2—C2—C1	119.85 (12)	C7—C8—C9	119.58 (14)
C3—C2—C1	117.69 (12)	C7—C8—H8	122.4 (9)
N3—C3—C2	119.48 (13)	C9—C8—H8	118.0 (9)
N3—C3—H3	116.4 (12)	N2—C9—C8	120.04 (13)
C2—C3—H3	124.2 (12)	N2—C9—C4	121.50 (13)
N3—C4—C5	121.17 (13)	C8—C9—C4	118.46 (13)
C9—N2—C2—C3	-1.7 (2)	C9—C4—C5—C6	-0.6 (2)
C9—N2—C2—C1	179.05 (12)	C4—C5—C6—C7	-1.3 (2)
O1—C1—C2—N2	179.06 (13)	C5—C6—C7—C8	1.6 (2)
N1—C1—C2—N2	-1.63 (19)	C6—C7—C8—C9	0.1 (2)
O1—C1—C2—C3	-0.2 (2)	C2—N2—C9—C8	179.78 (13)
N1—C1—C2—C3	179.12 (13)	C2—N2—C9—C4	-0.14 (19)

C4—N3—C3—C2	0.7 (2)	C7—C8—C9—N2	178.09 (13)
N2—C2—C3—N3	1.5 (2)	C7—C8—C9—C4	-2.0 (2)
C1—C2—C3—N3	-179.26 (12)	N3—C4—C9—N2	2.22 (19)
C3—N3—C4—C5	177.53 (13)	C5—C4—C9—N2	-177.80 (12)
C3—N3—C4—C9	-2.5 (2)	N3—C4—C9—C8	-177.70 (12)
N3—C4—C5—C6	179.33 (13)	C5—C4—C9—C8	2.3 (2)

*Hydrogen-bond geometry* (Å, °)

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N1—H1A···O1 <sup>i</sup>	0.86 (2)	2.04 (2)	2.9008 (17)	173.5 (17)
N1—H1B···C11	0.90 (2)	2.44 (2)	3.2590 (13)	152.0 (17)
N3—H3N···C11 <sup>ii</sup>	0.94 (2)	2.02 (2)	2.9501 (13)	169.8 (15)

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

Fig. 1

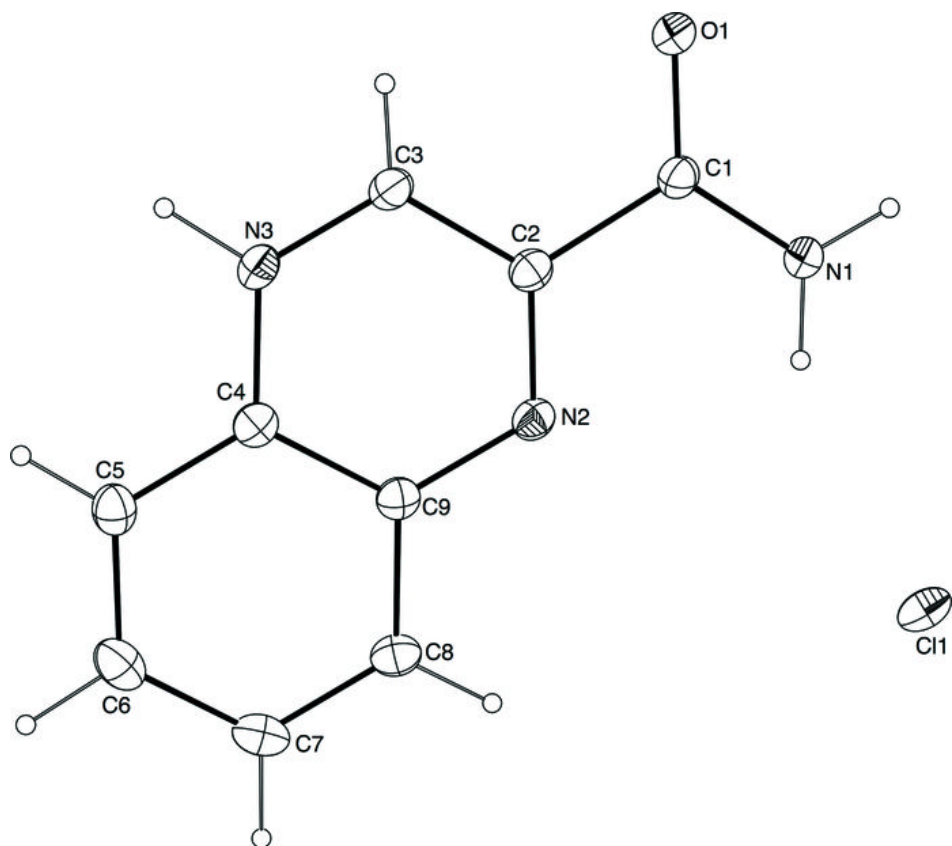


Fig. 2

