Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-Carbamoylquinoxalin-1-ium chloride

James K. Harper,[a]* Gary Strobel[b] and Atta M. Arif[c]

[a]University of Central Florida, Department of Chemistry, 4000 Central Florida Blvd., Orlando, FL 32816, USA, [b]Montana State University, Department of Plant Sciences and Plant Patology, Bozeman, MT 59717, USA, and [c]University of Utah, Department of Chemistry, 315 S. 1400 E. Rm. 2020, Salt Lake City, UT 84112, USA
Correspondence e-mail: james.harper@ucf.edu

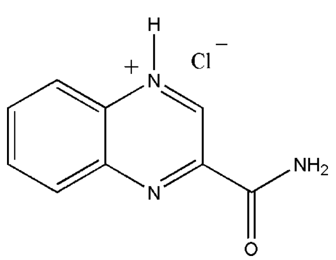
Received 15 November 2011; accepted 5 December 2011

Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.002 Å; R factor = 0.034; wR factor = 0.085; data-to-parameter ratio = 13.4.

The title compound, $C_9H_8N_3O^+\cdot Cl^-$, was isolated from a liquid culture of *streptomyces* sp. In the cation, the ring system makes a dihedral angle of 0.2 (2)° with the amide group. The protonation creating the cation occurs at ome of the N atoms in the quinoxaline ring system. In the crystal, the ions are linked through N—H···O and N—H···Cl hydrogen bonds, forming a two-dimensional network parallel to $(10\overline{3})$.

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 Streptomycete producing the title compound, see: Castillo *et al.* (2003).



Experimental

Crystal data

$C_9H_8N_3O^+\cdot Cl^-$
M_r = 209.63
Monoclinic, $P2_1/n$
a = 5.6476 (2) Å
b = 15.1045 (9) Å
c = 11.2556 (6) Å
β = 99.993 (3)°
V = 945.58 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.37 mm^{-1}
T = 150 K
0.25 × 0.20 × 0.08 mm

Data collection

Nonius KappaCCD diffractometer
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_{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^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^i	0.86 (2)	2.04 (2)	2.9008 (17)	173.5 (17)
N1—H1B···Cl1	0.90 (2)	2.44 (2)	3.2590 (13)	152.0 (17)
N3—H3N···Cl1^{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). E**68**, o79-o80 [**doi:10.1107/S1600536811052457**]

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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 (Bailly *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^{i} (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_2Cl_2/H_2O) 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 $CHCl_3$:MeOH

Refinement

All H atoms were refined independently with isotropic displacement parameters.

Figures

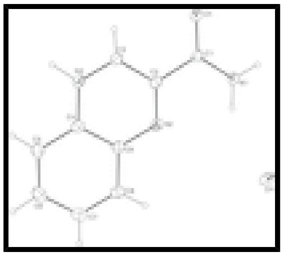

Fig. 1. Molecular structure of the title compound. Displacement ellipsoids are shown at the 50% probability level on non-hydrogen atoms.

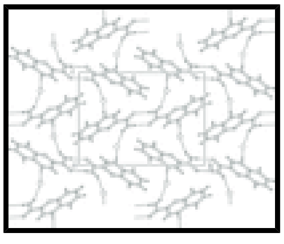

Fig. 2. Part of the crystal structure viewed along the *a* axis. The dashed lines indicate N—H···O and N—H···Cl hydrogen bonds.

3-Carbamoylquinoxalin-1-ium chloride

Crystal data

$C_9H_8N_3O^+\cdot Cl^-$	$F(000) = 432$
$M_r = 209.63$	$D_x = 1.473$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 1998 reflections
$a = 5.6476$ (2) Å	$\theta = 1.0$–27.5°
$b = 15.1045$ (9) Å	$\mu = 0.37$ mm^{-1}
$c = 11.2556$ (6) Å	$T = 150$ K
$\beta = 99.993$ (3)°	Plate, pale yellow
$V = 945.58$ (8) Å^3	0.25 × 0.20 × 0.08 mm
$Z = 4$	

Data collection

Nonius KappaCCD diffractometer	2147 independent reflections
Radiation source: fine-focus sealed tube	1798 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.018$
φ and ω scans	$\theta_{max} = 27.5°$, $\theta_{min} = 3.9°$
Absorption correction: multi-scan (*DENZO-SMN*; Otwinowski & Minor, 1997)	$h = -7 \rightarrow 7$
$T_{min} = 0.913$, $T_{max} = 0.971$	$k = -18 \rightarrow 19$
3671 measured reflections	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.034$	All H-atom parameters refined
$wR(F^2) = 0.085$	$w = 1/[\sigma^2(F_o^2) + (0.0397P)^2 + 0.2499P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{max} < 0.001$
2147 reflections	$\Delta\rho_{max} = 0.25$ e Å^{-3}
160 parameters	$\Delta\rho_{min} = -0.24$ e Å^{-3}
0 restraints	Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	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 (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	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)

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 (Å, °)

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, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···O1^{i}	0.86 (2)	2.04 (2)	2.9008 (17)	173.5 (17)
N1—H1B···Cl1	0.90 (2)	2.44 (2)	3.2590 (13)	152.0 (17)
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Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) *x*+3/2, −*y*+1/2, *z*+1/2.

Fig. 1

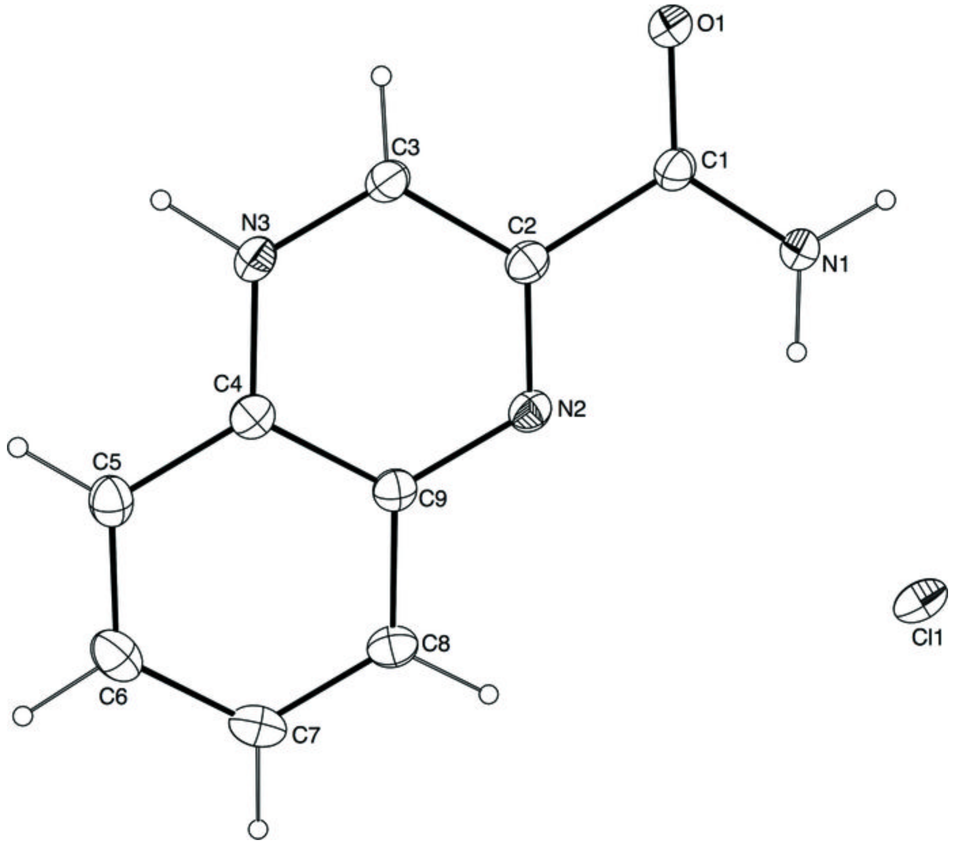

Fig. 2