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5-Chloro-8-hydroxyquinolinium nitrate

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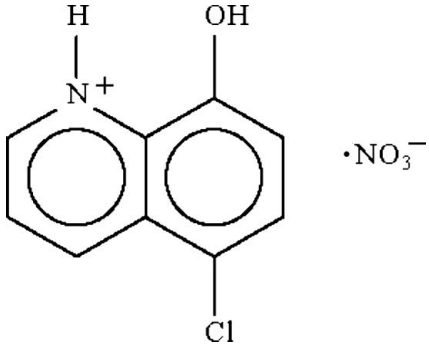
Key indicators: single-crystal X-ray study; T = 123 K; mean σ(C–C) = 0.005 Å; R factor = 0.062; wR factor = 0.192; data-to-parameter ratio = 14.4.

The 5-chloro-8-hydroxyquinolinium cation in the the title ion pair, $C_9H_7ClNO^+\cdot NO_3^-$, is approximately coplanar with the nitrate anion [dihedral angle = 16.1 (1)°]. Two ion pairs are hydrogen bonded (2 × O—H···O and 2 × N—H···O) about a center of inversion, generating an $R_4^4(14)$ ring.

Related literature

The 8-hydroxyquinolinium cation has been isolated as a number of salts; for the 8-hydroxyquinolinium chloride hydrate, see: Skakle *et al.* (2006). For the crystal structure of 5-chloro-8-hydroxyquinoline, see: Banerjee & Saha (1986).



Experimental

Crystal data

$C_9H_7ClNO^+\cdot NO_3^-$ M_r = 242.62
Monoclinic, $P2_1/n$
a = 7.4379 (3) Å
b = 11.5518 (6) Å
c = 11.2288 (5) Å
β = 95.831 (3)°
V = 959.80 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.40 mm^{-1}
T = 123 K
0.20 × 0.05 × 0.05 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.925, T_{max} = 0.980
6472 measured reflections
2196 independent reflections
1574 reflections with $I > 2\sigma(I)$
R_{int} = 0.049

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.192
S = 1.07
2196 reflections
153 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 1.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1o···O2^{i}	0.84 (1)	1.87 (1)	2.695 (3)	169 (4)
N1—H1n···O2	0.88 (1)	1.95 (1)	2.816 (3)	167 (4)

Symmetry code: (i) $-x+1, -y+1, -z+1$.

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); 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).

I thank the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2465).

References

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supplementary materials

Acta Cryst. (2009). E**65**, o1450 [**doi:10.1107/S160053680901993X**]

5-Chloro-8-hydroxyquinolinium nitrate

S. W. Ng

Comment

(type here to add)

Experimental

Zinc acetate (0.19 g, 1 mmol) and 5-chloro-8-hydroxyquinoline (0.36 g, 2 mmol) were loaded into a convection tube; the tube was filled with dry methanol and kept at 333 K. Yellow crystals were collected from the side-arm after several days.

Refinement

Carbon-bound H-atoms were placed in calculated positions (C–H 0.95 Å) and were included in the refinement in the riding model approximation with *U*(H) fixed at 1.2*U*(C). The ammonium and hydroxy H-atoms were located in a difference Fourier map, and were refined with distance restraints of N–H = 0.88±0.01 Å and O–H = 0.84±0.01 Å; their isotropic temperature factors were refined.

The final difference Fourier map had a large peak at about 1 Å from the Cl1 atom.

Figures

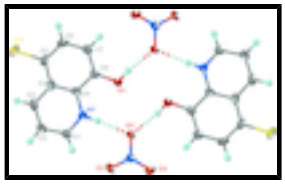

Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of a pair of hydrogen-bonded $[C_9H_7ClNO][NO_3]$ ion pairs drawn at the 70% probability level. Hydrogen atoms are drawn spheres of arbitrary radius and dashed lines denote hydrogen bonds.

5-Chloro-8-hydroxyquinolinium nitrate

Crystal data

$C_9H_7ClNO^+·NO_3^-$	F_{000} = 496
M_r = 242.62	D_x = 1.679 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 1079 reflections
a = 7.4379 (3) Å	θ = 2.5–26.4°
b = 11.5518 (6) Å	μ = 0.40 mm^{-1}
c = 11.2288 (5) Å	T = 123 K
β = 95.831 (3)°	Prism, yellow

V = 959.80 (8) Å^3
Z = 4

0.20 × 0.05 × 0.05 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Monochromator: graphite
T = 123 K
ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
T_{min} = 0.925, T_{max} = 0.980
6472 measured reflections

2196 independent reflections
1574 reflections with $I > 2\tilde{}I$)
R_{int} = 0.049
θ_{max} = 27.5º
θ_{min} = 2.5º
h = −9→9
k = −15→12
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.192
S = 1.07
2196 reflections
153 parameters
2 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1164P)^2 + 0.1797P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}
Extinction correction: none

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	−0.30656 (11)	0.30338 (8)	0.76985 (8)	0.0321 (3)
O1	0.3429 (3)	0.4706 (2)	0.5632 (2)	0.0279 (6)
H1O	0.350 (6)	0.5423 (11)	0.553 (4)	0.042 (12)*
O2	0.5861 (3)	0.30211 (18)	0.4592 (2)	0.0261 (6)
O3	0.6434 (3)	0.12125 (19)	0.4947 (2)	0.0325 (6)
O4	0.8190 (3)	0.2201 (2)	0.3904 (2)	0.0286 (6)
N1	0.2829 (4)	0.2424 (2)	0.5774 (2)	0.0203 (6)
H1N	0.385 (3)	0.265 (4)	0.552 (3)	0.037 (11)*
N2	0.6849 (4)	0.2125 (2)	0.4472 (2)	0.0220 (6)
C1	0.2635 (4)	0.1291 (3)	0.5806 (3)	0.0250 (7)
H1A	0.3516	0.0806	0.5504	0.030*
C2	0.1155 (5)	0.0794 (3)	0.6278 (3)	0.0277 (7)
H2	0.1012	−0.0023	0.6295	0.033*

C3	−0.0094 (4)	0.1513 (3)	0.6720 (3)	0.0265 (7)
H3	−0.1093	0.1185	0.7063	0.032*
C4	0.0084 (4)	0.2727 (3)	0.6672 (3)	0.0213 (7)
C5	−0.1144 (4)	0.3518 (3)	0.7089 (3)	0.0254 (7)
C6	−0.0849 (5)	0.4687 (3)	0.7003 (3)	0.0325 (8)
H6	−0.1694	0.5213	0.7282	0.039*
C7	0.0666 (5)	0.5118 (3)	0.6513 (3)	0.0297 (8)
H7	0.0832	0.5931	0.6460	0.036*
C8	0.1920 (4)	0.4385 (3)	0.6108 (3)	0.0231 (7)
C9	0.1613 (4)	0.3178 (3)	0.6184 (3)	0.0204 (7)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0219 (5)	0.0405 (6)	0.0359 (5)	−0.0012 (3)	0.0128 (3)	−0.0003 (4)
O1	0.0275 (13)	0.0173 (12)	0.0413 (14)	0.0001 (9)	0.0147 (10)	0.0025 (10)
O2	0.0276 (13)	0.0138 (11)	0.0388 (13)	0.0032 (9)	0.0124 (10)	−0.0010 (9)
O3	0.0337 (14)	0.0145 (11)	0.0513 (16)	−0.0003 (9)	0.0135 (12)	0.0044 (10)
O4	0.0270 (13)	0.0265 (13)	0.0342 (13)	0.0027 (9)	0.0121 (10)	−0.0009 (10)
N1	0.0173 (14)	0.0205 (13)	0.0235 (14)	0.0010 (10)	0.0046 (11)	0.0003 (10)
N2	0.0214 (14)	0.0175 (13)	0.0274 (14)	0.0005 (10)	0.0038 (11)	−0.0025 (10)
C1	0.0241 (17)	0.0199 (16)	0.0320 (18)	0.0020 (12)	0.0073 (13)	−0.0003 (13)
C2	0.0278 (17)	0.0205 (16)	0.0356 (18)	−0.0003 (13)	0.0066 (14)	0.0058 (14)
C3	0.0227 (17)	0.0272 (17)	0.0300 (17)	−0.0079 (13)	0.0043 (13)	0.0027 (13)
C4	0.0208 (16)	0.0253 (16)	0.0180 (15)	−0.0034 (12)	0.0034 (12)	0.0000 (12)
C5	0.0182 (16)	0.0337 (19)	0.0252 (16)	−0.0003 (13)	0.0074 (12)	0.0025 (13)
C6	0.0271 (18)	0.0279 (18)	0.045 (2)	0.0074 (14)	0.0137 (15)	−0.0051 (15)
C7	0.0291 (19)	0.0210 (17)	0.0405 (19)	0.0021 (13)	0.0111 (15)	−0.0024 (14)
C8	0.0240 (16)	0.0201 (16)	0.0258 (16)	−0.0012 (12)	0.0058 (12)	0.0006 (12)
C9	0.0239 (17)	0.0171 (15)	0.0203 (15)	0.0021 (12)	0.0021 (12)	0.0009 (11)

Geometric parameters (Å, °)

Cl1—C5	1.739 (3)	C2—H2	0.9500
O1—C8	1.343 (4)	C3—C4	1.410 (5)
O1—H1O	0.84 (1)	C3—H3	0.9500
O2—N2	1.285 (3)	C4—C5	1.405 (4)
O3—N2	1.234 (3)	C4—C9	1.411 (4)
O4—N2	1.240 (4)	C5—C6	1.374 (5)
N1—C1	1.318 (4)	C6—C7	1.395 (5)
N1—C9	1.368 (4)	C6—H6	0.9500
N1—H1N	0.88 (1)	C7—C8	1.371 (4)
C1—C2	1.393 (5)	C7—H7	0.9500
C1—H1A	0.9500	C8—C9	1.417 (4)
C2—C3	1.375 (5)		
C8—O1—H1O	113 (3)	C5—C4—C3	124.6 (3)
C1—N1—C9	123.0 (3)	C9—C4—C3	117.7 (3)
C1—N1—H1N	114 (3)	C6—C5—C4	120.1 (3)

C9—N1—H1N	123 (3)	C6—C5—Cl1	119.2 (3)
O3—N2—O4	122.1 (3)	C4—C5—Cl1	120.7 (3)
O3—N2—O2	118.2 (3)	C5—C6—C7	121.3 (3)
O4—N2—O2	119.7 (3)	C5—C6—H6	119.3
N1—C1—C2	120.8 (3)	C7—C6—H6	119.3
N1—C1—H1A	119.6	C8—C7—C6	120.9 (3)
C2—C1—H1A	119.6	C8—C7—H7	119.5
C3—C2—C1	118.5 (3)	C6—C7—H7	119.5
C3—C2—H2	120.7	O1—C8—C7	125.8 (3)
C1—C2—H2	120.7	O1—C8—C9	116.3 (3)
C2—C3—C4	121.2 (3)	C7—C8—C9	118.0 (3)
C2—C3—H3	119.4	N1—C9—C4	118.8 (3)
C4—C3—H3	119.4	N1—C9—C8	119.3 (3)
C5—C4—C9	117.8 (3)	C4—C9—C8	121.9 (3)
C9—N1—C1—C2	−0.5 (5)	C6—C7—C8—O1	−179.4 (3)
N1—C1—C2—C3	−0.5 (5)	C6—C7—C8—C9	1.0 (5)
C1—C2—C3—C4	1.6 (5)	C1—N1—C9—C4	0.5 (4)
C2—C3—C4—C5	179.2 (3)	C1—N1—C9—C8	−179.6 (3)
C2—C3—C4—C9	−1.7 (5)	C5—C4—C9—N1	179.8 (3)
C9—C4—C5—C6	0.8 (5)	C3—C4—C9—N1	0.6 (4)
C3—C4—C5—C6	179.9 (3)	C5—C4—C9—C8	−0.2 (4)
C9—C4—C5—Cl1	179.8 (2)	C3—C4—C9—C8	−179.4 (3)
C3—C4—C5—Cl1	−1.1 (5)	O1—C8—C9—N1	−0.4 (4)
C4—C5—C6—C7	−0.6 (5)	C7—C8—C9—N1	179.3 (3)
Cl1—C5—C6—C7	−179.5 (3)	O1—C8—C9—C4	179.6 (3)
C5—C6—C7—C8	−0.4 (6)	C7—C8—C9—C4	−0.7 (5)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1o···O2^{i}	0.84 (1)	1.87 (1)	2.695 (3)	169 (4)
N1—H1n···O2	0.88 (1)	1.95 (1)	2.816 (3)	167 (4)

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1.

Fig. 1