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3-Acetyl-2,4-dimethylquinolin-1-ium chloride

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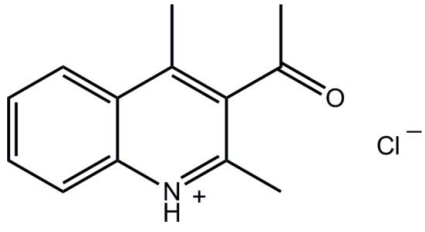
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.035; wR factor = 0.099; data-to-parameter ratio = 17.1.

In the title salt, $C_{13}H_{14}NO^{+}\cdot Cl^{-}$, the dihedral angle between the fused ring system (r.m.s. deviation = 0.039 Å) and the attached aldehyde group is 75.27 (16)°. In the crystal, the cation and anion are linked by an N—H···Cl hydrogen bond and the resulting pairs are connected into four-ion aggregates by π–π interactions between the C_6 and pyridinium rings [3.6450 (9) Å] of inversion-related quinolinium residues.

Related literature

For background details and biological applications of quinoline and quinoline chalcones, see: Joshi *et al.* (2011); Prasath *et al.* (2013*a*). For a related structure, see: Prasath *et al.* (2013*b*).



Experimental

Crystal data

$C_{13}H_{14}NO^{+}\cdot Cl^{-}$
M_r = 235.70
Orthorhombic, *Pbca*
a = 12.8221 (6) Å
b = 10.7281 (4) Å
c = 16.3785 (6) Å
V = 2252.97 (16) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.32 mm^{-1}
T = 100 K
0.50 × 0.40 × 0.30 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2013)
T_{min} = 0.956, T_{max} = 1.000
8404 measured reflections
2597 independent reflections
2207 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.099
S = 1.04
2597 reflections
152 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···Cl1	0.91 (2)	2.13 (2)	3.0374 (13)	175.4 (17)

Data collection: *CrysAlis PRO* (Agilent, 2013); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB7097).

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supplementary materials

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3-Acetyl-2,4-dimethylquinolin-1-ium chloride

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Comment

Nitrogen-containing heterocyclic analogues are found to be valuable intermediates in organic synthesis and exhibit a multitude of photophysical properties. In particular, quinoline analogues have received significant attention owing to their bio-activity such as anti-bacterial, anti-fungal, anti-malarial and anti-cancer activities (Prasath *et al.*, 2013*a*; Joshi *et al.*, 2011). As a continuation of structural studies in this area (Prasath *et al.*, 2013*b*), the title salt, (I), was investigated.

The fused-ring system of the cation in (I), Fig. 1, is almost planar with the r.m.s. deviation of the fitted atoms being 0.039 Å; maximum deviations are 0.051 (1) Å for the C3 atom and -0.044 (2) Å for the C5 atom. The aldehyde group is almost perpendicular to this plane, forming a C10—C9—C12—O1 torsion angle of 73.64 (18)°.

In the crystal, ions are linked by a N—H···Cl hydrogen bond, Table 1, and connected into four-ion aggregates by π—π interactions between the C_6 and pyridinium rings [inter-centorid distance 3.6450 (9) Å for symmetry operation 1 - *x*, 1 - *y*, 1 - *z*] of centrosymmetrically related quinolinyl residues, Fig. 2. These pack with no specific interactions between them.

Experimental

A mixture of 2-aminoacetophenone (0.68 g, 0.005 *M*), acetylacetone (0.5 g, 0.005 *M*) and 1 N HCl (20 ml) was stirred at 363 K for 45 minutes. To the resulting mixture, chloroform (20 ml) was added and the organic layer was passed through anhydrous Na_2SO_4. Re-crystallization was by slow evaporation of chloroform solution of (I) which yielded yellow blocks. *M*.pt. 393–395 K. Yield: 90%.

Refinement

The C-bound H atoms were geometrically placed (C—H = 0.95–0.98 Å) and refined as riding with U_{iso}(H) = 1.2–1.5U_{eq}(C). The N-bound H atom was refined freely.

Computing details

Data collection: *CrysAlis PRO* (Agilent, 2013); cell refinement: *CrysAlis PRO* (Agilent, 2013); data reduction: *CrysAlis PRO* (Agilent, 2013); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

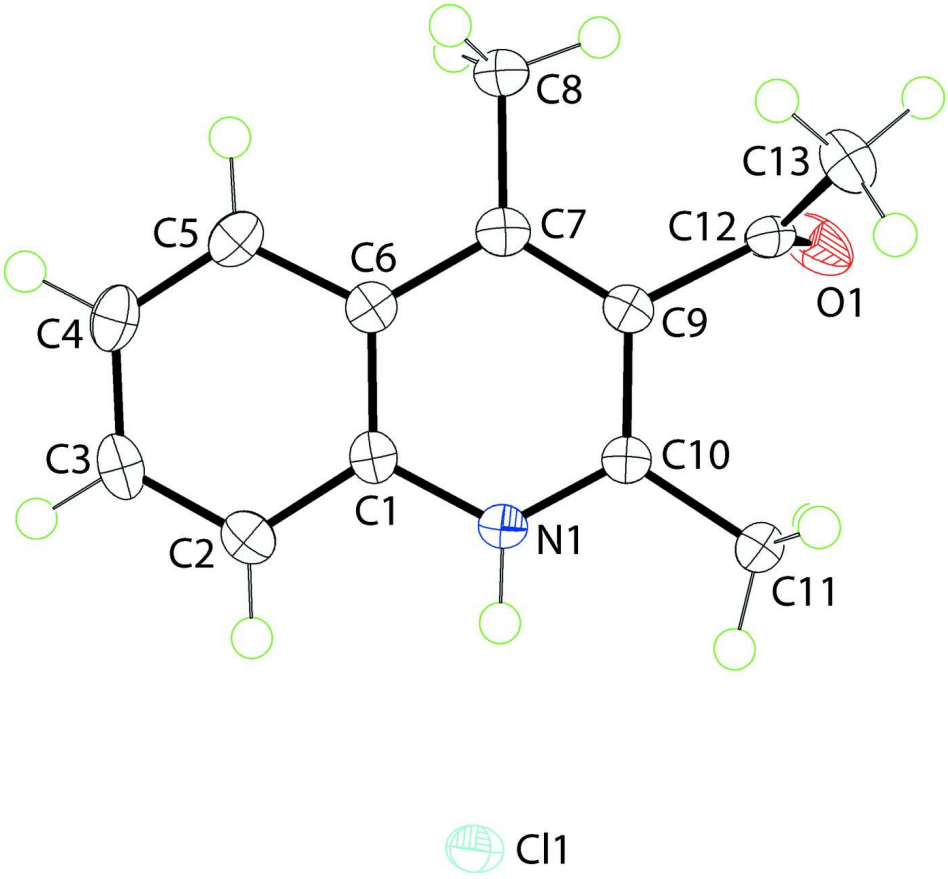

Figure 1

The molecular structures of the ions in (I) showing displacement ellipsoids at the 70% probability level.

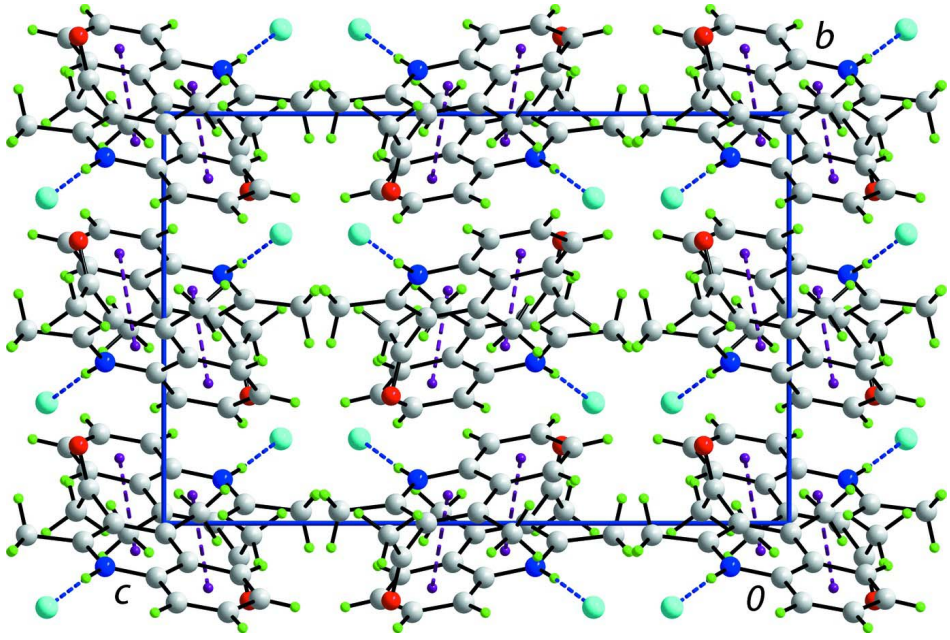

Figure 2

A view in projection down the *a* axis of the unit-cell contents of (I). The N—H···Cl and π—π interactions are shown as blue and purple dashed lines, respectively.

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Crystal data

$C_{13}H_{14}NO^{+}\cdot Cl^{-}$
M_r = 235.70
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
a = 12.8221 (6) Å
b = 10.7281 (4) Å
c = 16.3785 (6) Å
V = 2252.97 (16) Å^3
Z = 8
F(000) = 992
D_x = 1.390 Mg m^{-3}
Mo *Kα* radiation, λ = 0.71073 Å
Cell parameters from 3252 reflections
θ = 3.0–27.5°
μ = 0.32 mm^{-1}
T = 100 K
Block, yellow
0.50 × 0.40 × 0.30 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Radiation source: SuperNova (Mo) X-ray Source
Mirror monochromator
Detector resolution: 10.4041 pixels mm^{-1}
ω scan
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2013)
T_{min} = 0.956, T_{max} = 1.000
8404 measured reflections
2597 independent reflections
2207 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 27.6°, θ_{min} = 3.0°
h = −11→16
k = −13→10
l = −19→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.099$
$S = 1.04$
2597 reflections
152 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0514P)^2 + 0.7427P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -0.29$ e Å^{-3}

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	0.38171 (3)	0.29410 (3)	0.31279 (2)	0.01712 (13)
O1	0.78490 (9)	0.68933 (10)	0.36335 (7)	0.0257 (3)
N1	0.56888 (10)	0.39336 (11)	0.40603 (7)	0.0127 (3)
H1	0.5145 (17)	0.3655 (19)	0.3755 (12)	0.035 (5)*
C1	0.56522 (12)	0.36700 (12)	0.48826 (8)	0.0130 (3)
C2	0.47740 (12)	0.30581 (13)	0.52035 (8)	0.0156 (3)
H2	0.4216	0.2811	0.4858	0.019*
C3	0.47406 (13)	0.28251 (13)	0.60294 (9)	0.0177 (3)
H3	0.4141	0.2441	0.6260	0.021*
C4	0.55806 (13)	0.31479 (13)	0.65333 (9)	0.0185 (3)
H4	0.5555	0.2950	0.7098	0.022*
C5	0.64371 (13)	0.37435 (14)	0.62260 (8)	0.0174 (3)
H5	0.6999	0.3956	0.6577	0.021*
C6	0.64893 (12)	0.40465 (13)	0.53813 (8)	0.0140 (3)
C7	0.73191 (11)	0.47481 (13)	0.50274 (8)	0.0139 (3)
C8	0.81628 (12)	0.52547 (14)	0.55698 (9)	0.0192 (3)
H8A	0.8670	0.5715	0.5239	0.029*
H8B	0.8515	0.4564	0.5848	0.029*
H8C	0.7855	0.5815	0.5976	0.029*
C9	0.72892 (11)	0.49922 (13)	0.41992 (8)	0.0134 (3)
C10	0.64623 (11)	0.45412 (12)	0.37091 (8)	0.0125 (3)
C11	0.64361 (12)	0.47075 (13)	0.28065 (8)	0.0159 (3)
H11A	0.5797	0.4334	0.2587	0.024*
H11B	0.7045	0.4300	0.2562	0.024*
H11C	0.6449	0.5599	0.2676	0.024*

C12	0.80915 (12)	0.58304 (13)	0.37971 (8)	0.0155 (3)
C13	0.91308 (13)	0.52994 (14)	0.35969 (9)	0.0205 (3)
H13A	0.9622	0.5978	0.3485	0.031*
H13B	0.9072	0.4764	0.3114	0.031*
H13C	0.9384	0.4806	0.4060	0.031*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0144 (2)	0.0191 (2)	0.01791 (19)	−0.00141 (14)	−0.00107 (14)	−0.00348 (12)
O1	0.0198 (6)	0.0158 (5)	0.0415 (6)	−0.0027 (5)	−0.0034 (5)	0.0074 (5)
N1	0.0120 (6)	0.0124 (5)	0.0138 (5)	0.0001 (5)	−0.0011 (5)	−0.0007 (4)
C1	0.0140 (7)	0.0103 (6)	0.0148 (6)	0.0020 (5)	0.0018 (6)	−0.0002 (5)
C2	0.0150 (8)	0.0120 (6)	0.0197 (7)	0.0007 (6)	0.0010 (6)	−0.0012 (5)
C3	0.0186 (8)	0.0133 (6)	0.0211 (7)	0.0005 (6)	0.0071 (6)	0.0020 (5)
C4	0.0247 (9)	0.0154 (6)	0.0153 (6)	0.0041 (6)	0.0035 (7)	0.0023 (5)
C5	0.0206 (8)	0.0171 (7)	0.0146 (6)	0.0029 (6)	−0.0016 (6)	0.0003 (5)
C6	0.0145 (7)	0.0125 (6)	0.0150 (6)	0.0022 (6)	0.0005 (6)	−0.0012 (5)
C7	0.0129 (7)	0.0127 (6)	0.0162 (6)	0.0030 (6)	−0.0006 (6)	−0.0019 (5)
C8	0.0168 (8)	0.0247 (8)	0.0162 (6)	−0.0040 (7)	−0.0024 (6)	−0.0006 (6)
C9	0.0124 (8)	0.0121 (6)	0.0157 (6)	0.0014 (5)	0.0001 (6)	−0.0006 (5)
C10	0.0122 (7)	0.0111 (6)	0.0142 (6)	0.0020 (6)	−0.0004 (5)	−0.0004 (5)
C11	0.0168 (8)	0.0177 (7)	0.0132 (6)	−0.0016 (6)	−0.0005 (6)	0.0008 (5)
C12	0.0160 (8)	0.0174 (7)	0.0131 (6)	−0.0038 (6)	−0.0033 (6)	−0.0005 (5)
C13	0.0173 (8)	0.0192 (7)	0.0249 (7)	−0.0023 (6)	0.0036 (7)	0.0023 (6)

Geometric parameters (Å, º)

O1—C12	1.2119 (18)	C7—C9	1.3821 (18)
N1—C10	1.3188 (19)	C7—C8	1.502 (2)
N1—C1	1.3769 (17)	C8—H8A	0.9800
N1—H1	0.91 (2)	C8—H8B	0.9800
C1—C2	1.405 (2)	C8—H8C	0.9800
C1—C6	1.408 (2)	C9—C10	1.415 (2)
C2—C3	1.3762 (19)	C9—C12	1.517 (2)
C2—H2	0.9500	C10—C11	1.4894 (18)
C3—C4	1.400 (2)	C11—H11A	0.9800
C3—H3	0.9500	C11—H11B	0.9800
C4—C5	1.367 (2)	C11—H11C	0.9800
C4—H4	0.9500	C12—C13	1.486 (2)
C5—C6	1.4228 (18)	C13—H13A	0.9800
C5—H5	0.9500	C13—H13B	0.9800
C6—C7	1.426 (2)	C13—H13C	0.9800
C10—N1—C1	123.63 (13)	H8A—C8—H8B	109.5
C10—N1—H1	120.0 (12)	C7—C8—H8C	109.5
C1—N1—H1	116.4 (12)	H8A—C8—H8C	109.5
N1—C1—C2	119.28 (13)	H8B—C8—H8C	109.5
N1—C1—C6	118.85 (13)	C7—C9—C10	120.84 (13)
C2—C1—C6	121.86 (12)	C7—C9—C12	121.31 (13)

C3—C2—C1	118.51 (14)	C10—C9—C12	117.68 (12)
C3—C2—H2	120.7	N1—C10—C9	119.01 (12)
C1—C2—H2	120.7	N1—C10—C11	118.37 (12)
C2—C3—C4	120.69 (14)	C9—C10—C11	122.61 (13)
C2—C3—H3	119.7	C10—C11—H11A	109.5
C4—C3—H3	119.7	C10—C11—H11B	109.5
C5—C4—C3	121.10 (13)	H11A—C11—H11B	109.5
C5—C4—H4	119.4	C10—C11—H11C	109.5
C3—C4—H4	119.4	H11A—C11—H11C	109.5
C4—C5—C6	120.19 (14)	H11B—C11—H11C	109.5
C4—C5—H5	119.9	O1—C12—C13	122.82 (14)
C6—C5—H5	119.9	O1—C12—C9	118.68 (14)
C1—C6—C7	118.97 (12)	C13—C12—C9	118.47 (12)
C1—C6—C5	117.56 (13)	C12—C13—H13A	109.5
C7—C6—C5	123.42 (13)	C12—C13—H13B	109.5
C9—C7—C6	118.56 (13)	H13A—C13—H13B	109.5
C9—C7—C8	122.14 (13)	C12—C13—H13C	109.5
C6—C7—C8	119.21 (12)	H13A—C13—H13C	109.5
C7—C8—H8A	109.5	H13B—C13—H13C	109.5
C7—C8—H8B	109.5		

C10—N1—C1—C2	177.60 (13)	C5—C6—C7—C8	−3.1 (2)
C10—N1—C1—C6	−1.1 (2)	C6—C7—C9—C10	−0.9 (2)
N1—C1—C2—C3	−178.66 (12)	C8—C7—C9—C10	−177.57 (13)
C6—C1—C2—C3	0.0 (2)	C6—C7—C9—C12	174.34 (13)
C1—C2—C3—C4	−2.4 (2)	C8—C7—C9—C12	−2.3 (2)
C2—C3—C4—C5	2.5 (2)	C1—N1—C10—C9	−2.3 (2)
C3—C4—C5—C6	−0.1 (2)	C1—N1—C10—C11	176.77 (12)
N1—C1—C6—C7	3.45 (19)	C7—C9—C10—N1	3.3 (2)
C2—C1—C6—C7	−175.19 (13)	C12—C9—C10—N1	−172.12 (12)
N1—C1—C6—C5	−179.02 (12)	C7—C9—C10—C11	−175.71 (13)
C2—C1—C6—C5	2.3 (2)	C12—C9—C10—C11	8.8 (2)
C4—C5—C6—C1	−2.3 (2)	C7—C9—C12—O1	−101.78 (17)
C4—C5—C6—C7	175.13 (13)	C10—C9—C12—O1	73.64 (18)
C1—C6—C7—C9	−2.4 (2)	C7—C9—C12—C13	80.14 (17)
C5—C6—C7—C9	−179.80 (13)	C10—C9—C12—C13	−104.45 (15)
C1—C6—C7—C8	174.32 (13)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···Cl1	0.91 (2)	2.13 (2)	3.0374 (13)	175.4 (17)