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4-Acetylanilinium perchlorate

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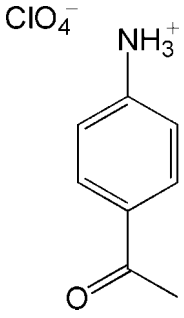
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.003 Å; R factor = 0.044; wR factor = 0.137; data-to-parameter ratio = 14.9.

In the title compound, $C_8H_{10}NO^+\cdot ClO_4^-$, the ions are connected in a three-dimensional hydrogen-bonded network *via* N—H···O hydrogen bonds, with four characteristic graph-set motifs: $C(8)$, $C_2^2(6)$, $R_4^2(10)$ and $R_4^4(12)$.

Related literature

For related literature, see: Bernstein *et al.* (1995); Athimoolam & Natarajan (2006); Cinčić & Kaitner (2007). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_8H_{10}NO^+\cdot ClO_4^-$
M_r = 235.62
Monoclinic, $P2_1/c$
a = 8.0287 (2) Å
b = 18.9091 (3) Å
c = 7.1327 (2) Å
β = 108.248 (2)°
V = 1028.40 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.37 mm^{-1}
T = 295 K
0.55 × 0.25 × 0.17 mm

Data collection

Oxford Diffraction Xcalibur CCD diffractometer
Absorption correction: analytical (Alcock, 1970)
T_{min} = 0.845, T_{max} = 0.943
10153 measured reflections
2240 independent reflections
1879 reflections with $I > 2\sigma(I)$
R_{int} = 0.013

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.137
S = 1.12
2240 reflections
150 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N31—H31B···O2	0.87 (3)	2.11 (3)	2.957 (3)	163 (3)
N31—H31A···O5^{i}	0.86 (3)	1.92 (3)	2.775 (2)	173 (3)
N31—H31C···O1^{ii}	0.84 (3)	2.54 (3)	3.015 (3)	116 (2)
N31—H31C···O3^{iii}	0.84 (3)	2.16 (3)	2.915 (3)	149 (3)

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2003); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2003); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PARST97* (Nardelli, 1995).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LW2041).

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supplementary materials

Acta Cryst. (2007). E**63**, o4672 [**doi:10.1107/S1600536807050751**]

4-Acetylanilinium perchlorate

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Comment

The molecular structure of compound (I), $C_8H_{10}NO^+$ ClO_4^-, is shown in Figure 1. The asymmetric unit consists of 4-acetylanilinium cation with protonated amino group and perchlorate anion. All bond lengths and bond angles correspond to the geometry parameters expected for atom types and the type of hybridization (Allen *et al.*, 1987).

The ions are connected in three-dimensional hydrogen-bonded network *via* N–H···O hydrogen bonds. All ammonium group H atoms are involved in the hydrogen bonding with three O-atoms of neighbouring perchlorate anion and O-atom of carbonyl group of neighbouring cation, with four-centred geometry motif observed. Four characteristic graph-set motifs can be recognized: $C_1^1(8)$, $C_2^2(6)$, $R_4^2(10)$ and $R_4^4(12)$ in the notation of Bernstein *et al.* (1995). Both ten- and twelve-membered ring moieties [$R_4^2(10)$ and $R_4^4(12)$] are formed of two 4-acetylanilinium cations and two perchlorate anion. The centre of twelve-membered ring is situated on a crystallographic centre of symmetry. The aggregation of two ring moieties results in infinite one-dimensional chains spreading along the *a* axis, with intercalated array of perchlorate ions, Fig. 2. Two infinite one-dimensional chains are detected with the donor participations of H31A in $C(8)$ motif and H31B and H31C in $C_2^2(6)$ graph-set motif.

Experimental

Single crystals of compound (I) were obtained by slow evaporation method. A solution of 100 mg 4'-aminoacetophenone dissolved in 2 ml of 2-propanol was heated at 70 °C. The clear solution was obtained, added to the 3 ml of perchloric acid (60%) and then left at room temperature. The crystals of (I) were collected by vacuum filtration, washed with cold acetone and dried in air.

Refinement

All N-bound H atoms were located in difference Fourier map and their positions and isotropic parameters were refined. Aromatic and methyl H atoms were placed in calculated positions and treated as riding on their parent C atoms, with C—H = 0.93 Å, $U_{iso}(H) = 1.2U_{eq}(C)$ for Csp^2 and C—H = 0.96 Å, $U_{iso}(H) = 1.5U_{eq}(C)$ for Csp^3, respectively.

Figures

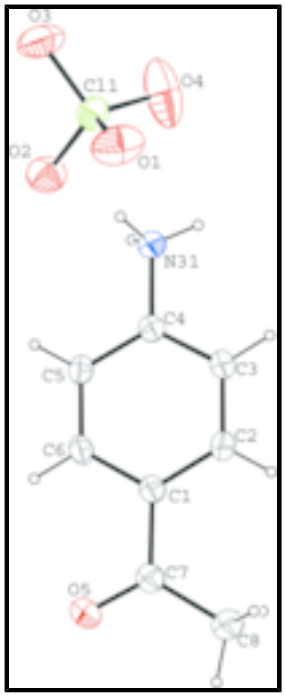

Fig. 1. The asymetric unit of (I), showing the crystallographic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radius.

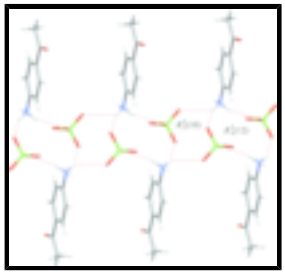

Fig. 2. A view of the one-dimensional hydrogen-bonded chain along *c* axis showing the aggregation of two different hydrogen-bonding motifs, $R_4^2(10)$ and $R_4^4(12)$. Hydrogen bonds are drawn as red dotted lines.

4-Acetylanilinium perchlorate

Crystal data

$C_8H_{10}NO^+·ClO_4^-$	$Z = 4$
$M_r = 235.62$	$F_{000} = 488$
Monoclinic, $P2_1/c$	$D_x = 1.522$ Mg m^{-3}
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 8.0287$ (2) Å	$\mu = 0.37$ mm^{-1}
$b = 18.9091$ (3) Å	$T = 295$ K
$c = 7.1327$ (2) Å	Prisms, colourles
$\beta = 108.248$ (2)º	0.55 × 0.25 × 0.17 mm
$V = 1028.40$ (4) Å^3	

Data collection

Oxford Diffraction Xcalibur CCD diffractometer	2240 independent reflections
Radiation source: fine-focus sealed tube	1879 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.013$
$T = 295$ K	$\theta_{max} = 27.0^o$
ω scans	$\theta_{min} = 4.0^o$
Absorption correction: analytical (Alcock, 1970)	$h = -10 \rightarrow 10$
$T_{min} = 0.845$, $T_{max} = 0.943$	$k = -24 \rightarrow 23$
10153 measured reflections	$l = -9 \rightarrow 9$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.044$	$w = 1/[\sigma^2(F_o^2) + (0.0754P)^2 + 0.4245P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.137$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.12$	$\Delta\rho_{max} = 0.42$ e Å^{-3}
2240 reflections	$\Delta\rho_{min} = -0.32$ e Å^{-3}
150 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.049 (6)
Secondary atom site location: difference Fourier map	

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 $F^2 > 2\text{sigma}(F^2)$ 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	0.35257 (7)	0.05725 (3)	0.73397 (7)	0.0458 (2)
O1	0.3640 (3)	0.11005 (10)	0.8786 (3)	0.0785 (6)

O2	0.5188 (2)	0.04870 (10)	0.6992 (3)	0.0637 (5)
O3	0.3092 (3)	−0.00828 (10)	0.8051 (4)	0.0811 (7)
O4	0.2255 (4)	0.07531 (19)	0.5542 (4)	0.1239 (12)
O5	1.18693 (19)	0.30662 (9)	0.6715 (3)	0.0641 (5)
N31	0.4986 (2)	0.11924 (9)	0.3228 (3)	0.0416 (4)
C1	0.8949 (2)	0.27357 (9)	0.5168 (3)	0.0357 (4)
C2	0.7219 (2)	0.29362 (10)	0.4321 (3)	0.0398 (4)
H2	0.6932	0.3414	0.4174	0.048*
C3	0.5910 (2)	0.24323 (10)	0.3689 (3)	0.0404 (4)
H3	0.4746	0.2567	0.3128	0.048*
C4	0.6366 (2)	0.17271 (9)	0.3909 (2)	0.0346 (4)
C5	0.8088 (2)	0.15104 (10)	0.4759 (3)	0.0398 (4)
H5	0.8370	0.1032	0.4901	0.048*
C6	0.9370 (2)	0.20189 (10)	0.5388 (3)	0.0396 (4)
H6	1.0531	0.1882	0.5967	0.048*
C7	1.0388 (2)	0.32664 (10)	0.5850 (3)	0.0417 (4)
C8	1.0006 (3)	0.40387 (11)	0.5470 (4)	0.0550 (6)
H8A	1.1086	0.4300	0.5855	0.082*
H8B	0.9275	0.4199	0.6222	0.082*
H8C	0.9412	0.4112	0.4090	0.082*
H31A	0.397 (4)	0.1386 (18)	0.273 (5)	0.082 (10)*
H31B	0.494 (4)	0.0911 (16)	0.418 (4)	0.064 (8)*
H31C	0.525 (4)	0.0918 (17)	0.244 (4)	0.066 (8)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0520 (3)	0.0391 (3)	0.0475 (3)	0.00303 (18)	0.0174 (2)	−0.00315 (18)
O1	0.0997 (15)	0.0607 (11)	0.0927 (14)	−0.0199 (10)	0.0556 (12)	−0.0335 (10)
O2	0.0633 (11)	0.0677 (11)	0.0711 (11)	−0.0006 (8)	0.0368 (9)	0.0008 (8)
O3	0.0843 (14)	0.0478 (10)	0.1317 (19)	−0.0144 (9)	0.0631 (14)	−0.0071 (11)
O4	0.113 (2)	0.171 (3)	0.0716 (15)	0.075 (2)	0.0057 (14)	0.0108 (16)
O5	0.0356 (8)	0.0511 (9)	0.0917 (13)	−0.0021 (6)	0.0002 (8)	0.0031 (8)
N31	0.0382 (8)	0.0370 (8)	0.0492 (10)	−0.0004 (7)	0.0131 (7)	0.0003 (7)
C1	0.0327 (8)	0.0371 (9)	0.0369 (8)	0.0021 (7)	0.0103 (7)	0.0008 (7)
C2	0.0377 (9)	0.0326 (9)	0.0487 (10)	0.0065 (7)	0.0128 (8)	0.0039 (7)
C3	0.0319 (8)	0.0386 (9)	0.0485 (10)	0.0053 (7)	0.0095 (7)	0.0033 (8)
C4	0.0339 (8)	0.0367 (9)	0.0335 (8)	0.0009 (7)	0.0108 (7)	0.0008 (7)
C5	0.0389 (9)	0.0336 (9)	0.0446 (10)	0.0067 (7)	0.0096 (8)	0.0030 (7)
C6	0.0323 (9)	0.0394 (9)	0.0434 (10)	0.0080 (7)	0.0063 (7)	0.0018 (7)
C7	0.0365 (9)	0.0412 (10)	0.0475 (10)	0.0001 (7)	0.0132 (8)	−0.0006 (8)
C8	0.0468 (11)	0.0400 (11)	0.0767 (15)	−0.0015 (8)	0.0172 (11)	−0.0025 (10)

Geometric parameters (Å, °)

Cl1—O4	1.408 (2)	C2—C3	1.385 (3)
Cl1—O1	1.4180 (18)	C2—H2	0.9300
Cl1—O3	1.4228 (19)	C3—C4	1.379 (3)
Cl1—O2	1.4404 (18)	C3—H3	0.9300

O5—C7	1.216 (2)	C4—C5	1.386 (2)
N31—C4	1.466 (2)	C5—C6	1.378 (3)
N31—H31A	0.86 (3)	C5—H5	0.9300
N31—H31B	0.88 (3)	C6—H6	0.9300
N31—H31C	0.84 (3)	C7—C8	1.499 (3)
C1—C2	1.383 (2)	C8—H8A	0.9600
C1—C6	1.394 (3)	C8—H8B	0.9600
C1—C7	1.492 (3)	C8—H8C	0.9600
O4—Cl1—O1	110.80 (16)	C2—C3—H3	120.6
O4—Cl1—O3	110.34 (19)	C3—C4—C5	121.91 (17)
O1—Cl1—O3	108.72 (13)	C3—C4—N31	118.89 (16)
O4—Cl1—O2	108.46 (15)	C5—C4—N31	119.19 (16)
O1—Cl1—O2	110.60 (12)	C6—C5—C4	118.53 (17)
O3—Cl1—O2	107.87 (11)	C6—C5—H5	120.7
C4—N31—H31A	111 (2)	C4—C5—H5	120.7
C4—N31—H31B	112.0 (19)	C5—C6—C1	120.78 (16)
H31A—N31—H31B	108 (3)	C5—C6—H6	119.6
C4—N31—H31C	109 (2)	C1—C6—H6	119.6
H31A—N31—H31C	113 (3)	O5—C7—C1	119.37 (18)
H31B—N31—H31C	103 (3)	O5—C7—C8	120.48 (18)
C2—C1—C6	119.40 (17)	C1—C7—C8	120.16 (17)
C2—C1—C7	121.82 (17)	C7—C8—H8A	109.5
C6—C1—C7	118.78 (16)	C7—C8—H8B	109.5
C1—C2—C3	120.62 (17)	H8A—C8—H8B	109.5
C1—C2—H2	119.7	C7—C8—H8C	109.5
C3—C2—H2	119.7	H8A—C8—H8C	109.5
C4—C3—C2	118.75 (16)	H8B—C8—H8C	109.5
C4—C3—H3	120.6		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N31—H31B···O2	0.87 (3)	2.11 (3)	2.957 (3)	163 (3)
N31—H31A···O5^{i}	0.86 (3)	1.92 (3)	2.775 (2)	173 (3)
N31—H31C···O1^{ii}	0.84 (3)	2.54 (3)	3.015 (3)	116 (2)
N31—H31C···O3^{iii}	0.84 (3)	2.16 (3)	2.915 (3)	149 (3)

Symmetry codes: (i) *x*−1, −*y*+1/2, *z*−1/2; (ii) *x*, *y*, *z*−1; (iii) −*x*+1, −*y*, −*z*+1.

Fig. 1

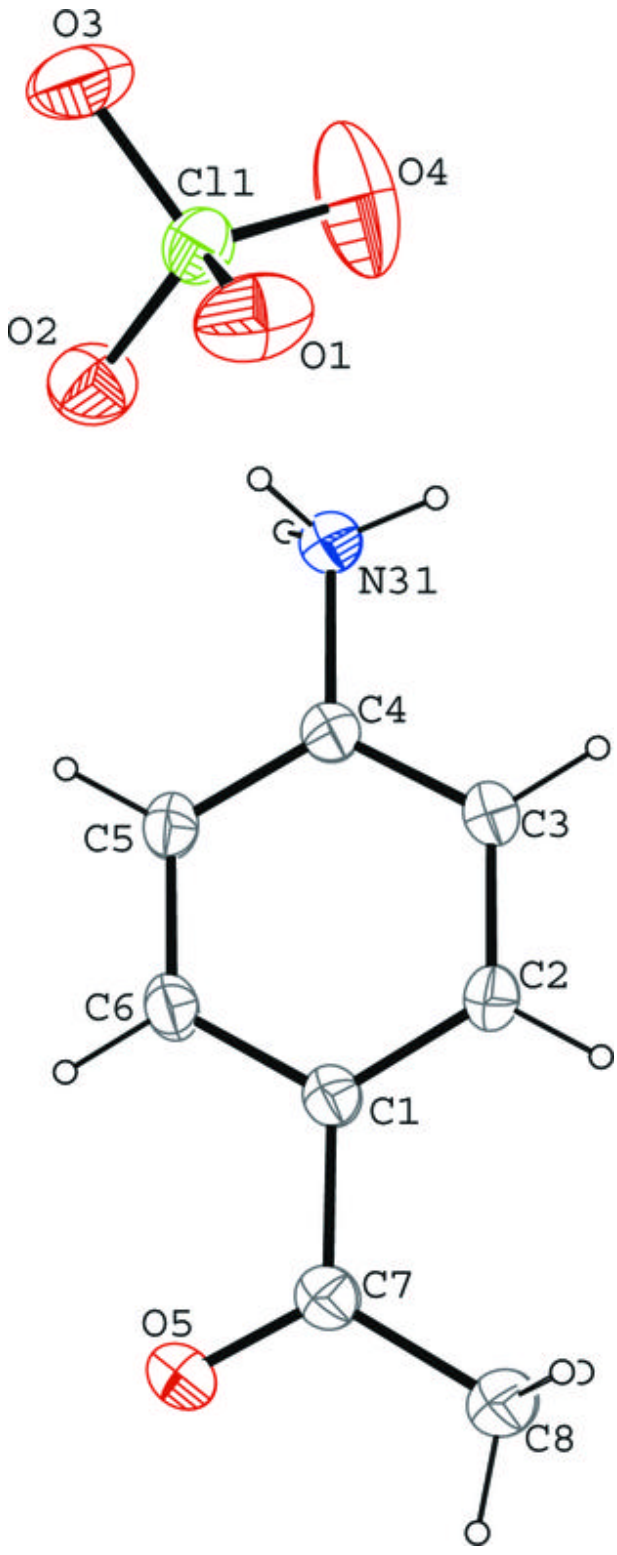

Fig. 2