Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-Chloroanilinium perchlorate

Benhua Zhou* and Jin Cai

School of Chemistry and Chemical Engineering, Southeast University, Nanjing 210096, People's Republic of China
Correspondence e-mail: cpu_cj@hotmail.com

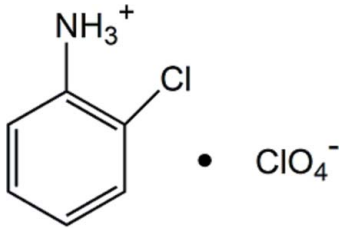
Received 26 April 2012; accepted 25 May 2012

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.046; wR factor = 0.089; data-to-parameter ratio = 17.3.

In the crystal of the title compound, $C_6H_7ClN^+\cdot ClO_4^-$, a layer-like structure parallel to the bc plane is formed through N—H···O hydrogen bonds between the cations and anions. These layers are connected by weak C—H···O interactions, forming a three-dimensional network.

Related literature

For general background to ferroelectric organic frameworks, see: Gray *et al.* (2002); Fu *et al.* (2007); Ye *et al.* (2009). For phase transitions of ferroelectric materials, see: Ye *et al.* (2006); Zhang *et al.* (2008); Zhao *et al.* (2008). For related structures, see: Gray *et al.* (2002); Balamurugan *et al.* (2010).



Experimental

Crystal data

$C_6H_7ClN^+\cdot ClO_4^-$
M_r = 228.03
Monoclinic, $P2_1/c$
a = 11.069 (2) Å
b = 7.3093 (15) Å
c = 13.718 (5) Å
β = 125.737 (19)°
V = 900.9 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.70 mm^{-1}
T = 293 K
0.20 × 0.20 × 0.20 mm

Data collection

Rigaku SCXmini diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.869, T_{max} = 0.869
8912 measured reflections
2060 independent reflections
1749 reflections with $I > 2\sigma(I)$
R_{int} = 0.040

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.089
S = 2.26
2060 reflections
119 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O2^{i}	0.89	2.21	2.978 (2)	145
N1—H1A···O4	0.89	2.63	3.333 (3)	136
N1—H1B···O3^{ii}	0.89	2.04	2.911 (2)	168
N1—H1C···O4^{iii}	0.89	2.29	3.022 (2)	140
N1—H1C···O2	0.89	2.51	3.039 (3)	118
C3—H3···O1^{iv}	0.93	2.70	3.331 (3)	126

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

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2005); software used to prepare material for publication: *SHELXL97*.

The authors are grateful to the Starter Fund of Southeast University, Nanjing, China.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZQ2165).

References

Balamurugan, P., Jagan, R. & Sivakumar, K. (2010). *Acta Cryst.* C**66**, o109–o113.
Brandenburg, K. & Putz, H. (2005). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Fu, D.-W., Song, Y.-M., Wang, G.-X., Ye, Q., Xiong, R.-G., Akutagawa, T., Nakamura, T., Chan, P. W. H. & Huang, S. D. (2007). *J. Am. Chem. Soc.* **129**, 5346–5347.
Gray, L. & Jones, P. G. (2002). *Z. Naturforsch. Teil B*, **57**, 61–72.
Rigaku (2005). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Ye, H.-Y., Fu, D.-W., Zhang, Y., Zhang, W., Xiong, R.-G. & Huang, S. D. (2009). *J. Am. Chem. Soc.* **131**, 42–43.
Ye, Q., Song, Y.-M., Wang, G.-X., Fu, D.-W., Chen, K., Chan, P. W. H., Zhu, J.-S., Huang, D. S. & Xiong, R.-G. (2006). *J. Am. Chem. Soc.* **128**, 6554–6555.
Zhang, W., Xiong, R.-G. & Huang, S.-P. D. (2008). *J. Am. Chem. Soc.* **130**, 10468–10469.
Zhao, H., Qu, Z.-R., Ye, H.-Y. & Xiong, R.-G. (2008). *Chem. Soc. Rev.* **37**, 84–100.

supporting information

Acta Cryst. (2012). E**68**, o2113 [https://doi.org/10.1107/S1600536812023963]

2-Chloroanilinium perchlorate

Benhua Zhou and Jin Cai

S1. Comment

The study of ferroelectric materials has received much attention and some materials have predominantly dielectric–ferroelectric performance (Ye *et al.*, 2006; Fu *et al.*, 2007; Zhao *et al.* 2008; Zhang *et al.*, 2008; Ye *et al.*, 2009). As a part of our work to obtain potential ferroelectric phase-transition materials, we report herein the crystal structure of title compound. Unluckily, the title compound exhibited no dielectric anomalies in the temperature range 93 – 453 K, suggesting that it might be only a paraelectric material.

The title compound, $C_6H_7ClN^+.ClO_4^-$, exhibits a two-dimensional layer-like structure parallel to the *bc* plane through intermolecular N—H···O hydrogen bonds between cations and anions (Fig. 1 & 2). Furthermore, the crystal structure is stabilized by weak C—H···O interactions which connect the two-dimensional layers.

The cation, $C_6H_7ClN^+$, is reported in the literature with different counter-ions (Gray *et al.*, 2002; Balamurugan *et al.*, 2010).

S2. Experimental

For the preparation of the title compound, a water solution of perchloric acid (1 g) was added to the ethanol solution of 2-chlorobenzenamine. The resulting precipitate was filtered. Colorless crystals suitable for X-ray analysis were formed after several weeks by slow evaporation of the solvent at room temperature.

S3. Refinement

All H atoms were calculated geometrically and allowed to ride on the parent atom with C–H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C), and with N–H = 0.89 Å U_{iso}(H) = 1.2U_{eq}(N).

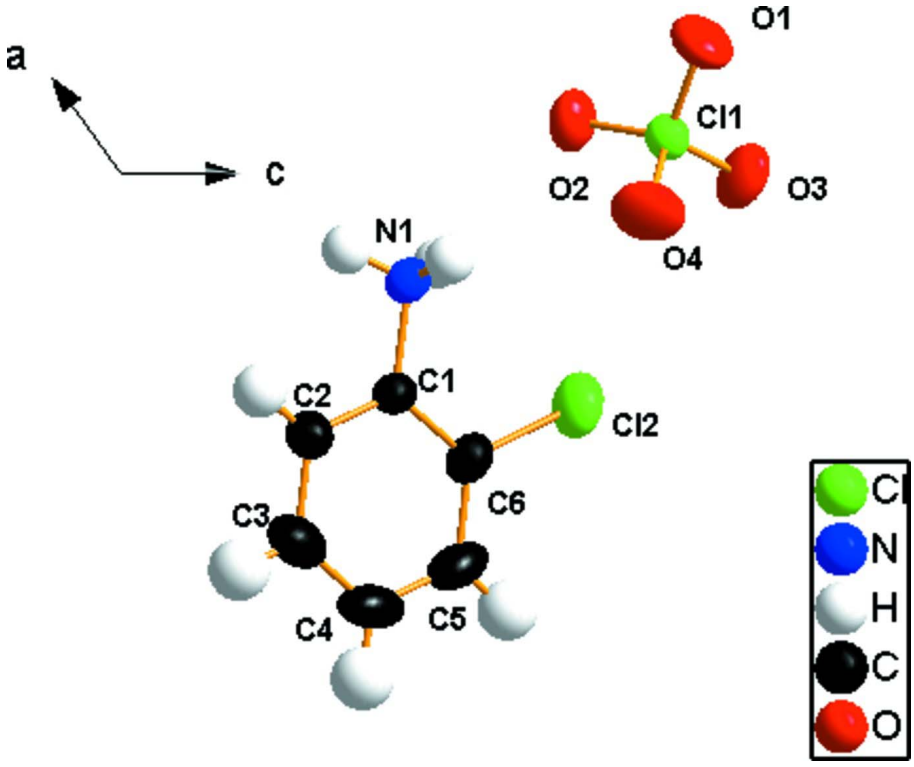


Figure 1

The molecular structure of the title compound, with the atomic numbering scheme and displacement ellipsoids drawn at the 30% probability level.

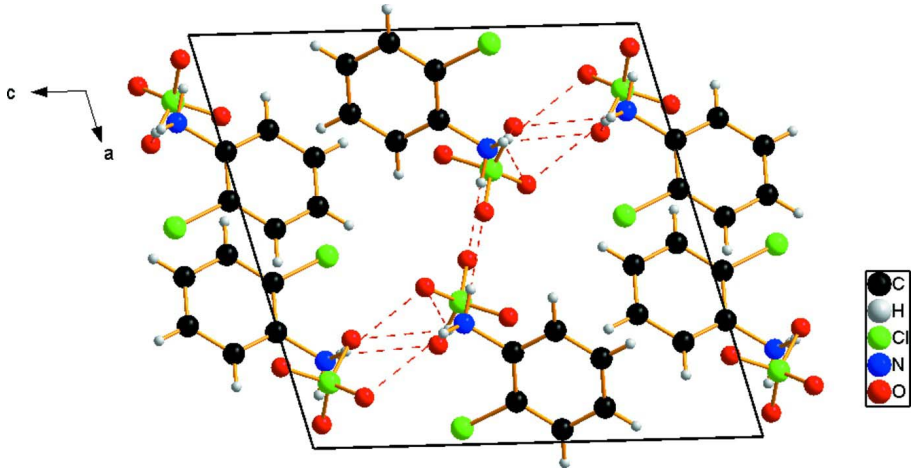


Figure 2

A view of the packing of the title compound, stacking along the *b* axis. Dashed lines indicate hydrogen bonds.

2-Chloroanilinium perchlorate

Crystal data

$C_6H_7ClN^+·ClO_4^-$
M_r = 228.03
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 11.069 (2) Å
b = 7.3093 (15) Å

c = 13.718 (5) Å
β = 125.737 (19)°
V = 900.9 (4) Å^3
Z = 4
F(000) = 464
D_x = 1.681 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å

Cell parameters from 2060 reflections
θ = 3.3–27.5°
μ = 0.70 mm^{-1}
T = 293 K
Prism, colourless
0.20 × 0.20 × 0.20 mm

Data collection

Rigaku SCXmini diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 13.6612 pixels mm^{-1}
CCD_Profile_fitting scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.869, T_{max} = 0.869

8912 measured reflections
2060 independent reflections
1749 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
θ_{max} = 27.5°, θ_{min} = 3.3°
h = −14→14
k = −9→9
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.089
S = 2.26
2060 reflections
119 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.010P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}
Extinction correction: *SHELXL*, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.231 (7)

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Cl2	−0.30288 (8)	−0.34063 (9)	−0.34203 (6)	0.0620 (3)
N1	−0.12505 (19)	−0.1679 (2)	−0.41599 (16)	0.0359 (5)
H1A	−0.0953	−0.0767	−0.3637	0.043*
H1B	−0.0808	−0.1583	−0.4530	0.043*
H1C	−0.1008	−0.2743	−0.3773	0.043*
C1	−0.2866 (2)	−0.1583 (3)	−0.50492 (19)	0.0316 (5)
C2	−0.3452 (3)	−0.0700 (3)	−0.6122 (2)	0.0421 (6)
H2	−0.2824	−0.0175	−0.6287	0.051*

C3	−0.4974 (3)	−0.0596 (3)	−0.6954 (2)	0.0556 (7)
H3	−0.5375	−0.0005	−0.7683	0.067*
C4	−0.5902 (3)	−0.1370 (3)	−0.6702 (3)	0.0584 (8)
H4	−0.6928	−0.1305	−0.7267	0.070*
C5	−0.5324 (3)	−0.2237 (3)	−0.5624 (3)	0.0536 (7)
H5	−0.5954	−0.2742	−0.5455	0.064*
C6	−0.3789 (3)	−0.2350 (3)	−0.4790 (2)	0.0391 (5)
Cl1	0.07943 (6)	−0.17797 (7)	−0.07815 (5)	0.0349 (2)
O1	0.21439 (19)	−0.0998 (3)	0.01753 (15)	0.0676 (6)
O2	0.10292 (19)	−0.2780 (2)	−0.15577 (14)	0.0545 (5)
O3	0.0235 (2)	−0.3048 (2)	−0.03403 (16)	0.0582 (5)
O4	−0.0262 (2)	−0.0368 (2)	−0.14591 (16)	0.0658 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl2	0.0771 (6)	0.0615 (5)	0.0639 (5)	−0.0132 (3)	0.0506 (5)	0.0054 (3)
N1	0.0367 (11)	0.0319 (10)	0.0425 (11)	−0.0002 (8)	0.0251 (10)	0.0026 (8)
C1	0.0303 (12)	0.0283 (12)	0.0360 (12)	−0.0023 (8)	0.0192 (11)	−0.0060 (9)
C2	0.0420 (14)	0.0435 (14)	0.0394 (13)	−0.0055 (10)	0.0229 (12)	−0.0035 (10)
C3	0.0513 (17)	0.0539 (17)	0.0420 (14)	0.0032 (12)	0.0163 (14)	−0.0065 (12)
C4	0.0341 (15)	0.0604 (18)	0.0604 (19)	−0.0024 (12)	0.0161 (14)	−0.0211 (14)
C5	0.0423 (16)	0.0518 (16)	0.0750 (19)	−0.0159 (12)	0.0389 (15)	−0.0235 (14)
C6	0.0457 (15)	0.0315 (12)	0.0499 (14)	−0.0057 (10)	0.0335 (13)	−0.0077 (10)
Cl1	0.0385 (3)	0.0308 (3)	0.0353 (3)	−0.0012 (2)	0.0214 (3)	0.0015 (2)
O1	0.0539 (12)	0.0806 (13)	0.0458 (10)	−0.0245 (10)	0.0163 (10)	−0.0160 (9)
O2	0.0699 (13)	0.0548 (11)	0.0535 (11)	0.0007 (9)	0.0443 (11)	−0.0075 (8)
O3	0.0762 (13)	0.0476 (11)	0.0782 (13)	−0.0014 (8)	0.0606 (12)	0.0120 (9)
O4	0.0618 (12)	0.0375 (10)	0.0780 (13)	0.0180 (8)	0.0294 (11)	0.0155 (9)

Geometric parameters (Å, º)

Cl2—C6	1.729 (2)	C3—H3	0.9300
N1—C1	1.464 (3)	C4—C5	1.376 (4)
N1—H1A	0.8900	C4—H4	0.9300
N1—H1B	0.8899	C5—C6	1.390 (3)
N1—H1C	0.8900	C5—H5	0.9300
C1—C2	1.374 (3)	Cl1—O1	1.4107 (17)
C1—C6	1.381 (3)	Cl1—O4	1.4237 (16)
C2—C3	1.379 (3)	Cl1—O3	1.4309 (15)
C2—H2	0.9300	Cl1—O2	1.4350 (16)
C3—C4	1.382 (4)		
C1—N1—H1A	109.4	C5—C4—C3	120.7 (2)
C1—N1—H1B	109.4	C5—C4—H4	119.6
H1A—N1—H1B	109.5	C3—C4—H4	119.6
C1—N1—H1C	109.6	C4—C5—C6	119.3 (2)
H1A—N1—H1C	109.5	C4—C5—H5	120.4

H1B—N1—H1C	109.5	C6—C5—H5	120.4
C2—C1—C6	120.6 (2)	C1—C6—C5	119.8 (2)
C2—C1—N1	119.84 (19)	C1—C6—Cl2	119.82 (18)
C6—C1—N1	119.5 (2)	C5—C6—Cl2	120.40 (19)
C1—C2—C3	119.8 (2)	O1—Cl1—O4	109.51 (12)
C1—C2—H2	120.1	O1—Cl1—O3	110.70 (11)
C3—C2—H2	120.1	O4—Cl1—O3	110.62 (11)
C2—C3—C4	119.9 (2)	O1—Cl1—O2	110.14 (11)
C2—C3—H3	120.1	O4—Cl1—O2	108.70 (11)
C4—C3—H3	120.1	O3—Cl1—O2	107.13 (11)
C6—C1—C2—C3	−0.7 (3)	N1—C1—C6—C5	179.00 (19)
N1—C1—C2—C3	−179.39 (19)	C2—C1—C6—Cl2	−178.46 (16)
C1—C2—C3—C4	0.3 (3)	N1—C1—C6—Cl2	0.2 (3)
C2—C3—C4—C5	0.4 (4)	C4—C5—C6—C1	0.5 (3)
C3—C4—C5—C6	−0.8 (4)	C4—C5—C6—Cl2	179.23 (18)
C2—C1—C6—C5	0.3 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O2^{i}	0.89	2.21	2.978 (2)	145
N1—H1*A*···O4	0.89	2.63	3.333 (3)	136
N1—H1*B*···O3^{ii}	0.89	2.04	2.911 (2)	168
N1—H1*C*···O4^{iii}	0.89	2.29	3.022 (2)	140
N1—H1*C*···O2	0.89	2.51	3.039 (3)	118
C3—H3···O1^{iv}	0.93	2.70	3.331 (3)	126

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