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2-Cyanoanilinium perchlorate

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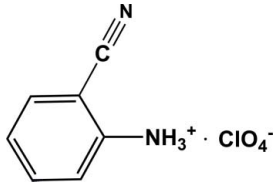
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.044; wR factor = 0.117; data-to-parameter ratio = 16.2.

In the title compound, $C_7H_7N_2^+\cdot ClO_4^-$, the cation is almost planar (r.m.s. deviation = 0.042 Å). In the crystal structure, the cations and anions are linked into a two-dimensional network parallel to (100) by N—H···O hydrogen bonds.

Related literature

For the crystal structure of 2-cyanoanilinium chloride, see: Oueslati *et al.* (2005). For Cl—O distances, see: Messai *et al.* (2009).



Experimental

Crystal data

$C_7H_7N_2^+\cdot ClO_4^-$
M_r = 218.60
Monoclinic, $P2_1/c$
a = 11.089 (2) Å
b = 7.4561 (15) Å
c = 13.872 (5) Å
β = 128.454 (18)°
V = 898.2 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.42 mm^{-1}
T = 298 K
0.40 × 0.05 × 0.05 mm

Data collection

Rigaku Mercury2 diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.90, T_{max} = 1.00
9026 measured reflections
2070 independent reflections
1761 reflections with $I > 2\sigma(I)$
R_{int} = 0.041

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.117
S = 1.11
2070 reflections
128 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O4^{i}	0.89	2.14	2.936 (2)	148
N2—H2B···O4^{ii}	0.89	2.24	3.007 (3)	144
N2—H2C···O1^{iii}	0.89	1.98	2.842 (2)	161

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

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: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI5023).

References

Messai, A., Direm, A., Benali-Cherif, N., Luneau, D. & Jeanneau, E. (2009). *Acta Cryst.* E**65**, o460.
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supporting information

Acta Cryst. (2010). E**66**, o467 [https://doi.org/10.1107/S1600536810003077]

2-Cyanoanilinium perchlorate

Li-Jing Cui and Xin-Yuan Chen

S1. Comment

Aniline derivatives attracted more attention as phase transition dielectric materials for their applications in micro-electronics and memory storage. With the purpose of obtaining phase transition crystals of 2-aminobenzonitrile salts, its interaction with various acids has been studied and we have obtained a series of new materials with this organic molecule. In this paper, we describe the crystal structure of the title compound, 2-cyanoanilinium perchlorate.

The asymmetric unit is composed of a 2-cyanoanilinium cation and a perchlorate anion (Fig.1). The anion displays a typical tetrahedral geometry around Cl atom and the Cl—O distances compare well with previously reported values (Messai *et al.*, 2009). The cation is almost planar (r.m.s. deviation 0.042 Å; maximum atomic deviation from coplanarity is 0.073 (2) Å by atom N1). The C—NH_3 [1.466 (2) Å] and C≡N [1.143 (3) Å] distances in the 2-cyanoanilinium cation are longer compared to the corresponding distances in the crystal structure of 2-cyanoanilinium chloride (1.457 (4) Å, 1.137 (4) Å; Oueslati *et al.*, 2005).

In the crystal structure, all the amine group H atoms are involved in N—H···O hydrogen bonds (Table 1). The N—H···O hydrogen bonds link the ionic units into a two-dimensional network parallel to the *ac* plane (Fig. 2).

S2. Experimental

The commercial 2-aminobenzonitrile (3 mmol, 324 mg) was dissolved in a water-$HClO_4$ (50:1 v/v) solution. The solvent was slowly evaporated in air affording colourless crystals of the title compound suitable for X-ray analysis.

While the permittivity measurement shows that there is no phase transition within the temperature range (from 100 K to 400 K), and the permittivity is 7.8 at 1 MHz at room temperature.

S3. Refinement

All H atoms were initially located in a difference Fourier map. They were then constrained to an ideal geometry, with C–H = 0.93 Å, N–H = 0.89 Å and U_{iso}(H) = 1.2U_{eq}(C) and 1.5U_{eq}(N). A rotating-group model was used for the -NH_3 group.

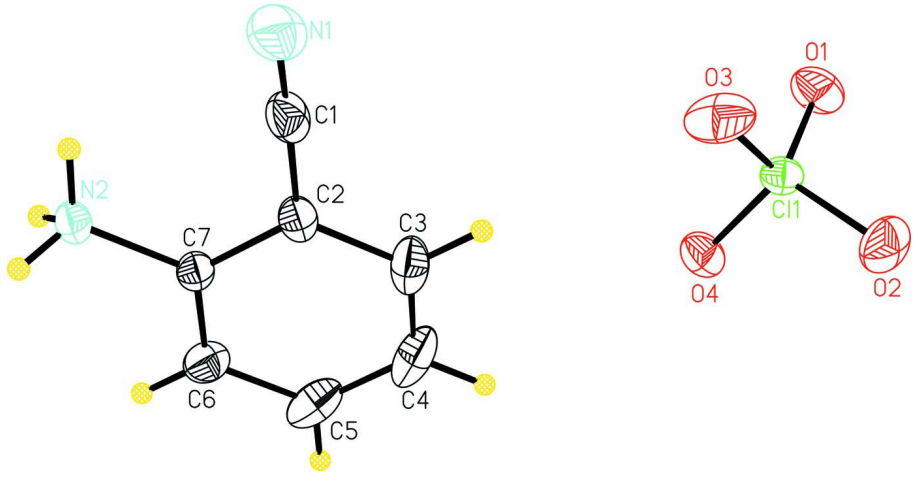

Figure 1

The asymmetric unit of the title compound with the atomic numbering scheme. Displacement ellipsoids were drawn at the 30% probability level.

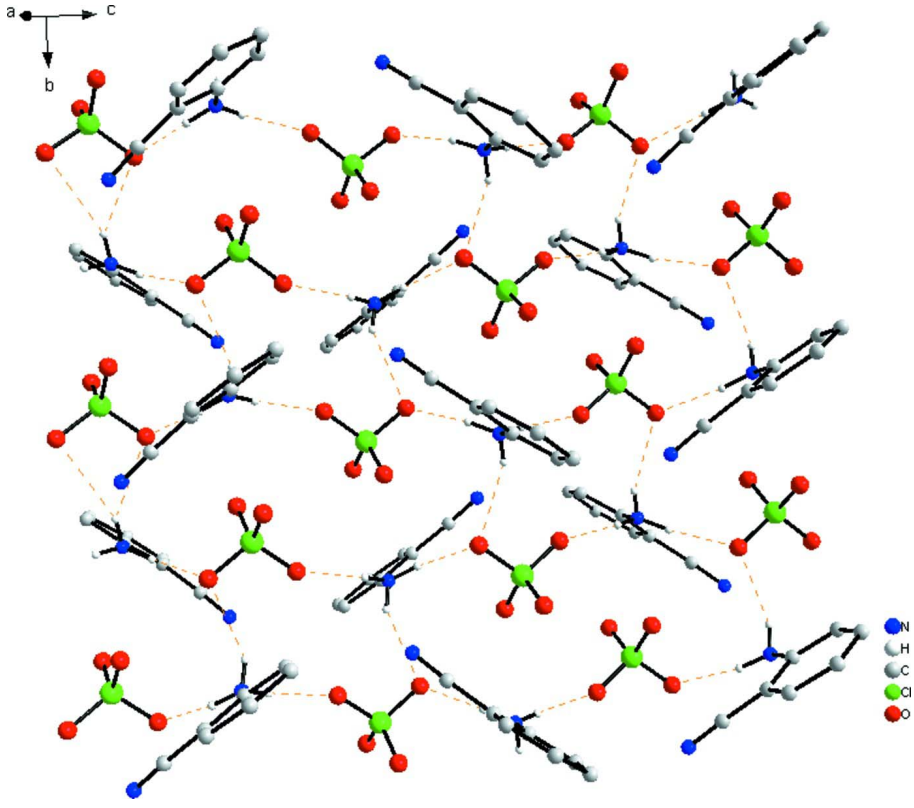

Figure 2

The crystal packing of the title compound, showing a two-dimensional network parallel to the (100). H atoms not involved in hydrogen bonding (dashed line) have been omitted for clarity.

2-Cyanoanilinium perchlorate

Crystal data

$C_7H_7N_2^+·ClO_4^-$
M_r = 218.60
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 11.089 (2) Å
b = 7.4561 (15) Å
c = 13.872 (5) Å
β = 128.454 (18)°
V = 898.2 (4) Å^3
Z = 4
$F(000)$ = 448
D_x = 1.617 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1761 reflections
θ = 3.3–27.5°
μ = 0.42 mm^{-1}
T = 298 K
Needle, colourless
0.40 × 0.05 × 0.05 mm

Data collection

Rigaku Mercury2 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} = 0.90, T_{max} = 1.00
9026 measured reflections
2070 independent reflections
1761 reflections with $I > 2\sigma(I)$
R_{int} = 0.041
θ_{max} = 27.5°, θ_{min} = 3.3°
h = −14→14
k = −9→9
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.117
S = 1.11
2070 reflections
128 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
$w = 1/[\sigma^2(F_o^2) + (0.0456P)^2 + 0.522P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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}
N2	0.10356 (18)	0.1622 (2)	0.40630 (16)	0.0351 (4)
H2A	0.0666	0.0523	0.3784	0.053*
H2B	0.0743	0.2332	0.3435	0.053*
H2C	0.0675	0.2053	0.4438	0.053*

N1	0.2247 (3)	0.4407 (3)	0.2725 (2)	0.0647 (7)
C7	0.2722 (2)	0.1553 (3)	0.49390 (19)	0.0327 (4)
C1	0.2853 (3)	0.3504 (3)	0.3573 (2)	0.0472 (6)
C5	0.5007 (3)	0.0559 (4)	0.6835 (2)	0.0629 (8)
H5	0.5493	−0.0064	0.7572	0.075*
C2	0.3588 (2)	0.2429 (3)	0.4665 (2)	0.0386 (5)
C6	0.3415 (3)	0.0628 (3)	0.6013 (2)	0.0469 (6)
H6	0.2827	0.0054	0.6189	0.056*
C4	0.5883 (3)	0.1403 (4)	0.6574 (3)	0.0662 (8)
H4	0.6952	0.1343	0.7133	0.079*
C3	0.5183 (3)	0.2326 (4)	0.5496 (3)	0.0547 (7)
H3	0.5775	0.2886	0.5320	0.066*
Cl1	0.91401 (6)	0.33466 (7)	0.57188 (4)	0.03477 (17)
O4	0.8898 (2)	0.2197 (2)	0.64175 (15)	0.0471 (4)
O3	0.7769 (2)	0.4305 (3)	0.48297 (16)	0.0681 (6)
O2	1.0336 (2)	0.4577 (2)	0.65476 (18)	0.0610 (5)
O1	0.9559 (2)	0.2236 (3)	0.51274 (17)	0.0574 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N2	0.0340 (9)	0.0349 (9)	0.0403 (10)	−0.0018 (7)	0.0250 (8)	−0.0023 (7)
N1	0.0733 (16)	0.0653 (15)	0.0617 (14)	−0.0195 (13)	0.0451 (13)	−0.0017 (12)
C7	0.0326 (10)	0.0306 (10)	0.0365 (10)	−0.0049 (8)	0.0223 (9)	−0.0081 (8)
C1	0.0499 (13)	0.0460 (13)	0.0576 (15)	−0.0166 (11)	0.0393 (13)	−0.0111 (12)
C5	0.0518 (15)	0.0571 (16)	0.0415 (13)	0.0008 (13)	0.0101 (12)	−0.0004 (12)
C2	0.0391 (11)	0.0351 (11)	0.0491 (12)	−0.0064 (9)	0.0311 (10)	−0.0094 (9)
C6	0.0476 (13)	0.0474 (13)	0.0384 (12)	−0.0070 (11)	0.0232 (11)	−0.0029 (10)
C4	0.0314 (12)	0.0587 (16)	0.0718 (19)	−0.0030 (12)	0.0140 (13)	−0.0145 (15)
C3	0.0393 (13)	0.0495 (14)	0.0762 (18)	−0.0111 (11)	0.0364 (14)	−0.0166 (13)
Cl1	0.0407 (3)	0.0312 (3)	0.0334 (3)	0.0017 (2)	0.0235 (2)	−0.00068 (19)
O4	0.0561 (10)	0.0436 (9)	0.0532 (10)	−0.0028 (8)	0.0397 (9)	0.0023 (8)
O3	0.0669 (12)	0.0745 (14)	0.0424 (10)	0.0332 (11)	0.0238 (9)	0.0151 (9)
O2	0.0660 (12)	0.0423 (10)	0.0638 (11)	−0.0212 (9)	0.0349 (10)	−0.0126 (8)
O1	0.0724 (12)	0.0587 (11)	0.0635 (11)	0.0041 (9)	0.0534 (11)	−0.0085 (9)

Geometric parameters (Å, º)

N2—C7	1.466 (2)	C5—H5	0.93
N2—H2A	0.89	C2—C3	1.388 (3)
N2—H2B	0.89	C6—H6	0.93
N2—H2C	0.89	C4—C3	1.367 (4)
N1—C1	1.143 (3)	C4—H4	0.93
C7—C6	1.364 (3)	C3—H3	0.93
C7—C2	1.396 (3)	Cl1—O3	1.4181 (18)
C1—C2	1.437 (4)	Cl1—O2	1.4233 (18)
C5—C4	1.381 (4)	Cl1—O1	1.4315 (17)
C5—C6	1.385 (4)	Cl1—O4	1.4385 (17)

C7—N2—H2A	109.5	C7—C6—C5	118.8 (2)
C7—N2—H2B	109.5	C7—C6—H6	120.6
H2A—N2—H2B	109.5	C5—C6—H6	120.6
C7—N2—H2C	109.5	C3—C4—C5	120.2 (2)
H2A—N2—H2C	109.5	C3—C4—H4	119.9
H2B—N2—H2C	109.5	C5—C4—H4	119.9
C6—C7—C2	121.2 (2)	C4—C3—C2	120.0 (2)
C6—C7—N2	118.99 (19)	C4—C3—H3	120.0
C2—C7—N2	119.78 (19)	C2—C3—H3	120.0
N1—C1—C2	177.1 (3)	O3—Cl1—O2	109.53 (13)
C4—C5—C6	120.8 (3)	O3—Cl1—O1	110.35 (12)
C4—C5—H5	119.6	O2—Cl1—O1	111.38 (12)
C6—C5—H5	119.6	O3—Cl1—O4	109.75 (12)
C3—C2—C7	119.0 (2)	O2—Cl1—O4	108.07 (11)
C3—C2—C1	120.1 (2)	O1—Cl1—O4	107.72 (11)
C7—C2—C1	120.8 (2)		

C6—C7—C2—C3	1.0 (3)	C4—C5—C6—C7	−0.3 (4)
N2—C7—C2—C3	−179.0 (2)	C6—C5—C4—C3	0.2 (4)
C6—C7—C2—C1	−175.6 (2)	C5—C4—C3—C2	0.5 (4)
N2—C7—C2—C1	4.4 (3)	C7—C2—C3—C4	−1.1 (4)
C2—C7—C6—C5	−0.3 (4)	C1—C2—C3—C4	175.5 (2)
N2—C7—C6—C5	179.7 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O4^{i}	0.89	2.14	2.936 (2)	148
N2—H2*B*···O4^{ii}	0.89	2.24	3.007 (3)	144
N2—H2*C*···O1^{iii}	0.89	1.98	2.842 (2)	161

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