

## 4-Cyanoanilinium perchlorate

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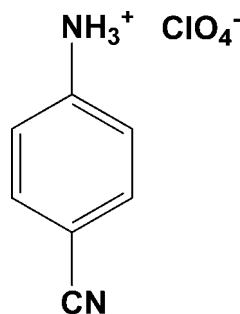
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Key indicators: single-crystal X-ray study;  $T = 298$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.036;  $wR$  factor = 0.097; data-to-parameter ratio = 16.6.

The title compound,  $\text{C}_7\text{H}_7\text{N}_2^+\cdot\text{ClO}_4^-$ , comprises discrete ions which are interconnected by  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds, leading to a neutral one-dimensional network along the [100] direction.

## Related literature

For the chemistry of nitrile derivatives, see: Xiong *et al.* (2002); Jin *et al.* (1994); Brewis *et al.* (2003); Fu *et al.* (2008); Duncia *et al.* (1991); Fu & Zhao (2007); Dai & Fu (2008); Smith *et al.* (2000).



## Experimental

## Crystal data

$\text{C}_7\text{H}_7\text{N}_2^+\cdot\text{ClO}_4^-$   
 $M_r = 218.60$   
Triclinic,  $P\bar{1}$   
 $a = 4.9905$  (10) Å  
 $b = 6.9465$  (14) Å  
 $c = 13.998$  (3) Å  
 $\alpha = 94.87$  (3)°  
 $\beta = 95.68$  (3)°

$\gamma = 103.99$  (3)°  
 $V = 465.57$  (17) Å<sup>3</sup>  
 $Z = 2$   
Mo  $K\alpha$  radiation  
 $\mu = 0.40$  mm<sup>-1</sup>  
 $T = 298$  (2) K  
 $0.25 \times 0.15 \times 0.15$  mm

## Data collection

Rigaku Mercury2 diffractometer  
Absorption correction: multi-scan  
(*CrystalClear*; Rigaku, 2005)  
 $T_{\min} = 0.941$ ,  $T_{\max} = 1.000$   
(expected range = 0.886–0.942)

4861 measured reflections  
2126 independent reflections  
1851 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.023$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.036$   
 $wR(F^2) = 0.097$   
 $S = 1.05$   
2126 reflections

128 parameters  
H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.25$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.34$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1A}\cdots\text{O2}^{\text{i}}$	0.89	2.04	2.881 (2)	158
$\text{N1}-\text{H1B}\cdots\text{O3}^{\text{ii}}$	0.89	1.98	2.855 (2)	166
$\text{N1}-\text{H1C}\cdots\text{O4}^{\text{iii}}$	0.89	2.04	2.871 (2)	156

Symmetry codes: (i)  $x, y, z - 1$ ; (ii)  $-x + 1, -y, -z + 1$ ; (iii)  $-x + 1, -y + 1, -z + 1$ .

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: *SHELXTL* (Sheldrick, 2008) and *PLATON* (Spek, 2003); 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: BX2181).

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**supplementary materials**

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## 4-Cyanoanilinium perchlorate

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### Comment

Nitrile derivatives have found wide range of applications in industry and coordination chemistry as ligands. For example, phthalonitriles have been used as starting materials for phthalocyanines (Jin *et al.*, 1994), which are important components for dyes, pigments, gas sensors, optical limiters and liquid crystals, and which are also used in medicine, as singlet oxygen photosensitisers for photodynamic therapy (Brewis *et al.*, 2003). And nitrile compounds are the precursor of tetrazole complexes (Duncia *et al.*, 1991; Xiong *et al.*, 2002; Fu *et al.*, 2008). Recently, a series of benzonitrile compounds have been reported (Fu & Zhao, 2007; Dai & Fu, 2008; Smith *et al.*, 2000). As an extension of these work on the structural characterization, we report here the crystal structure of the title compound *p*-cyanoanilinium perchloride. The crystal data show that in the title compound, the N1 atom of the amine group is protonated. The nitrile group and the benzene ring are essentially coplanar. The C1≡N2 bond length of 1.135 (3) Å is within the normal range (Fig. 1). The crystal packing is stabilized by cation–anion N—H⋯O hydrogen bonds, building an infinite one-dimensional chain parallel to the *a* axis. (Table 1, Fig. 2).

### Experimental

*p*-cyanoaniline (3 mmol, 354 mg) was dissolved in the solution of distilled water (10 ml) and perchloride acid (0.5 ml), and evaporated in the air affording colorless block crystals of this compound suitable for X-ray analysis were obtained.

### Refinement

All H atoms attached to C and N atoms were fixed geometrically and treated as riding with C—H = 0.93 Å (aromatic) and N—H = 0.89 Å with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C or N})$ .

### Figures

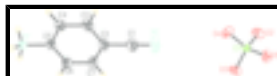


Fig. 1. A view of the title compound with the atomic numbering scheme. Displacement ellipsoids were drawn at the 30% probability level.

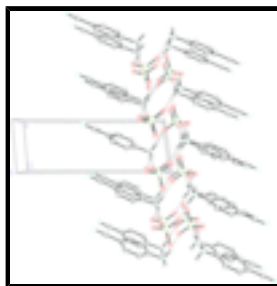


Fig. 2. The crystal packing of the title compound viewed along the *b* axis and all hydrogen atoms not involved in hydrogen bonding (dashed lines) were omitted for clarity.

## 4-Cyanoanilinium perchlorate

### Crystal data

$C_7H_7N_2^+ClO_4^-$	$Z = 2$
$M_r = 218.60$	$F_{000} = 224$
Triclinic, $P\bar{1}$	$D_x = 1.559 \text{ Mg m}^{-3}$
Hall symbol: -P 1	Mo $K\alpha$ radiation
$a = 4.9905 (10) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$b = 6.9465 (14) \text{ \AA}$	Cell parameters from 1806 reflections
$c = 13.998 (3) \text{ \AA}$	$\theta = 3.0\text{--}27.5^\circ$
$\alpha = 94.87 (3)^\circ$	$\mu = 0.40 \text{ mm}^{-1}$
$\beta = 95.68 (3)^\circ$	$T = 298 (2) \text{ K}$
$\gamma = 103.99 (3)^\circ$	Block, colourless
$V = 465.57 (17) \text{ \AA}^3$	$0.25 \times 0.15 \times 0.15 \text{ mm}$

### Data collection

Rigaku Mercury2 diffractometer	2126 independent reflections
Radiation source: fine-focus sealed tube	1851 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.023$
Detector resolution: $13.6612 \text{ pixels mm}^{-1}$	$\theta_{\text{max}} = 27.5^\circ$
$T = 298(2) \text{ K}$	$\theta_{\text{min}} = 3.0^\circ$
$\omega$ scans	$h = -6 \rightarrow 6$
Absorption correction: multi-scan (CrystalClear; Rigaku, 2005)	$k = -8 \rightarrow 8$
$T_{\text{min}} = 0.941$ , $T_{\text{max}} = 1.000$	$l = -18 \rightarrow 18$
4861 measured reflections	

### Refinement

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.036$	H-atom parameters constrained
$wR(F^2) = 0.097$	$w = 1/[\sigma^2(F_o^2) + (0.0483P)^2 + 0.1253P]$
$S = 1.05$	where $P = (F_o^2 + 2F_c^2)/3$
2126 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
128 parameters	$\Delta\rho_{\text{max}} = 0.25 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.34 \text{ e \AA}^{-3}$
	Extinction correction: none

*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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.6157 (3)	0.2508 (2)	0.12597 (11)	0.0399 (4)
H1A	0.4771	0.2405	0.0791	0.048*
H1B	0.6876	0.1460	0.1180	0.048*
H1C	0.7468	0.3623	0.1238	0.048*
N2	0.0727 (7)	0.2647 (4)	0.5503 (2)	0.1058 (10)
C1	0.1672 (6)	0.2642 (4)	0.47983 (19)	0.0743 (8)
C2	0.2877 (5)	0.2620 (3)	0.38998 (16)	0.0549 (5)
C3	0.2722 (5)	0.4100 (3)	0.33016 (17)	0.0557 (5)
H3	0.1860	0.5101	0.3478	0.067*
C4	0.3859 (4)	0.4064 (3)	0.24466 (15)	0.0458 (4)
H4	0.3789	0.5049	0.2041	0.055*
C5	0.5097 (3)	0.2566 (3)	0.21961 (13)	0.0362 (4)
C6	0.5293 (4)	0.1094 (3)	0.27829 (14)	0.0464 (5)
H6	0.6169	0.0104	0.2604	0.056*
C7	0.4158 (5)	0.1127 (4)	0.36408 (16)	0.0574 (6)
H7	0.4252	0.0145	0.4046	0.069*
C11	-0.00044 (8)	0.22266 (6)	0.91125 (3)	0.03315 (14)
O1	-0.2849 (3)	0.1812 (2)	0.92711 (11)	0.0552 (4)
O2	0.1712 (3)	0.3237 (2)	0.99794 (12)	0.0598 (4)
O3	0.0665 (3)	0.0398 (2)	0.88369 (13)	0.0593 (4)
O4	0.0420 (4)	0.3478 (2)	0.83610 (12)	0.0668 (5)

*Atomic displacement parameters ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
N1	0.0442 (8)	0.0392 (8)	0.0403 (9)	0.0162 (7)	0.0100 (6)	0.0048 (6)
N2	0.141 (3)	0.0996 (19)	0.0746 (17)	0.0122 (17)	0.0625 (17)	-0.0038 (14)
C1	0.0839 (18)	0.0741 (17)	0.0597 (16)	0.0059 (14)	0.0296 (13)	-0.0047 (13)
C2	0.0545 (12)	0.0599 (13)	0.0450 (12)	0.0039 (10)	0.0167 (9)	-0.0053 (10)
C3	0.0560 (13)	0.0514 (12)	0.0617 (14)	0.0159 (10)	0.0210 (10)	-0.0056 (10)
C4	0.0485 (11)	0.0424 (10)	0.0506 (12)	0.0168 (8)	0.0126 (9)	0.0046 (8)
C5	0.0328 (8)	0.0375 (9)	0.0366 (9)	0.0068 (7)	0.0046 (7)	0.0004 (7)

## supplementary materials

C6	0.0547 (11)	0.0455 (10)	0.0441 (11)	0.0205 (9)	0.0097 (9)	0.0072 (8)
C7	0.0714 (15)	0.0609 (13)	0.0425 (12)	0.0163 (11)	0.0126 (10)	0.0140 (10)
C11	0.0306 (2)	0.0284 (2)	0.0426 (3)	0.00948 (15)	0.00733 (15)	0.00697 (15)
O1	0.0333 (7)	0.0667 (9)	0.0627 (10)	0.0079 (6)	0.0146 (6)	-0.0046 (7)
O2	0.0531 (9)	0.0534 (9)	0.0661 (10)	0.0147 (7)	-0.0150 (7)	-0.0090 (7)
O3	0.0595 (9)	0.0396 (7)	0.0839 (12)	0.0271 (7)	0.0058 (8)	-0.0036 (7)
O4	0.0880 (12)	0.0539 (9)	0.0623 (11)	0.0127 (8)	0.0217 (9)	0.0293 (8)

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

N1—C5	1.462 (2)	C4—C5	1.371 (3)
N1—H1A	0.8900	C4—H4	0.9300
N1—H1B	0.8900	C5—C6	1.380 (3)
N1—H1C	0.8900	C6—C7	1.378 (3)
N2—C1	1.135 (3)	C6—H6	0.9300
C1—C2	1.447 (3)	C7—H7	0.9300
C2—C7	1.385 (3)	C11—O4	1.4202 (15)
C2—C3	1.392 (3)	C11—O3	1.4215 (14)
C3—C4	1.375 (3)	C11—O1	1.4222 (14)
C3—H3	0.9300	C11—O2	1.4346 (16)
C5—N1—H1A	109.5	C4—C5—C6	122.22 (18)
C5—N1—H1B	109.5	C4—C5—N1	118.25 (16)
H1A—N1—H1B	109.5	C6—C5—N1	119.50 (16)
C5—N1—H1C	109.5	C7—C6—C5	118.39 (19)
H1A—N1—H1C	109.5	C7—C6—H6	120.8
H1B—N1—H1C	109.5	C5—C6—H6	120.8
N2—C1—C2	179.6 (3)	C6—C7—C2	120.1 (2)
C7—C2—C3	120.66 (19)	C6—C7—H7	120.0
C7—C2—C1	119.9 (2)	C2—C7—H7	120.0
C3—C2—C1	119.4 (2)	O4—C11—O3	109.51 (11)
C4—C3—C2	119.1 (2)	O4—C11—O1	109.25 (11)
C4—C3—H3	120.4	O3—C11—O1	108.89 (10)
C2—C3—H3	120.4	O4—C11—O2	109.01 (11)
C5—C4—C3	119.54 (19)	O3—C11—O2	110.69 (10)
C5—C4—H4	120.2	O1—C11—O2	109.47 (10)
C3—C4—H4	120.2		
C7—C2—C3—C4	0.0 (3)	C4—C5—C6—C7	1.1 (3)
C1—C2—C3—C4	-179.7 (2)	N1—C5—C6—C7	-176.89 (19)
C2—C3—C4—C5	0.6 (3)	C5—C6—C7—C2	-0.4 (3)
C3—C4—C5—C6	-1.2 (3)	C3—C2—C7—C6	-0.1 (4)
C3—C4—C5—N1	176.83 (18)	C1—C2—C7—C6	179.6 (2)

### Hydrogen-bond geometry ( $\text{\AA}$ , $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1A $\cdots$ O2 <sup>i</sup>	0.89	2.04	2.881 (2)	158
N1—H1B $\cdots$ O3 <sup>ii</sup>	0.89	1.98	2.855 (2)	166
N1—H1C $\cdots$ O4 <sup>iii</sup>	0.89	2.04	2.871 (2)	156

Symmetry codes: (i)  $x, y, z-1$ ; (ii)  $-x+1, -y, -z+1$ ; (iii)  $-x+1, -y+1, -z+1$ .

Fig. 1

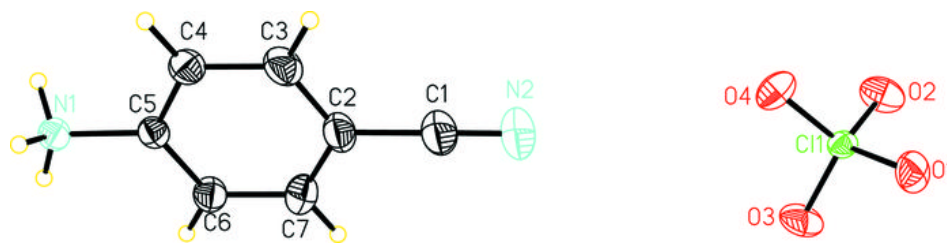


Fig. 2

