Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4-Cyanoanilinium perchlorate

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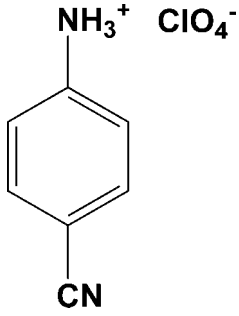
Received 13 September 2008; accepted 23 September 2008

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.036; wR factor = 0.097; data-to-parameter ratio = 16.6.

The title compound, $C_7H_7N_2^+\cdot ClO_4^-$, comprises discrete ions which are interconnected by N—H···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

$C_7H_7N_2^+\cdot ClO_4^-$
M_r = 218.60
Triclinic, $P\overline{1}$
a = 4.9905 (10) Å
b = 6.9465 (14) Å
c = 13.998 (3) Å
α = 94.87 (3)°
β = 95.68 (3)°
γ = 103.99 (3)°
V = 465.57 (17) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.40 mm^{-1}
T = 298 (2) K
0.25 × 0.15 × 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_{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
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 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	2.04	2.881 (2)	158
N1—H1B···O3^{ii}	0.89	1.98	2.855 (2)	166
N1—H1C···O4^{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*.

This work was supported by a Start-up Grant from Southeast University to Professor Ren-Gen Xiong.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BX2181).

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supporting information

Acta Cryst. (2008). E**64**, o2025 [doi:10.1107/S1600536808030687]

4-Cyanoanilinium perchlorate

Jing Dai

S1. 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 stablized by cation–anion N—H···O hydrogen bonds, building an infinite one-dimensional chain parallel to the *a* axis. (Table 1, Fig. 2).

S2. 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.

S3. 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_{iso}(H) = 1.2Ueq(C or N).

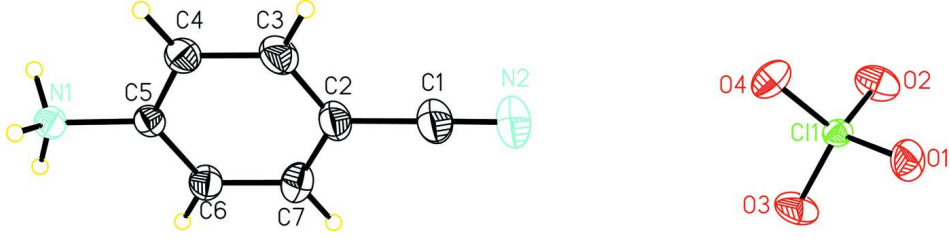

Figure 1

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

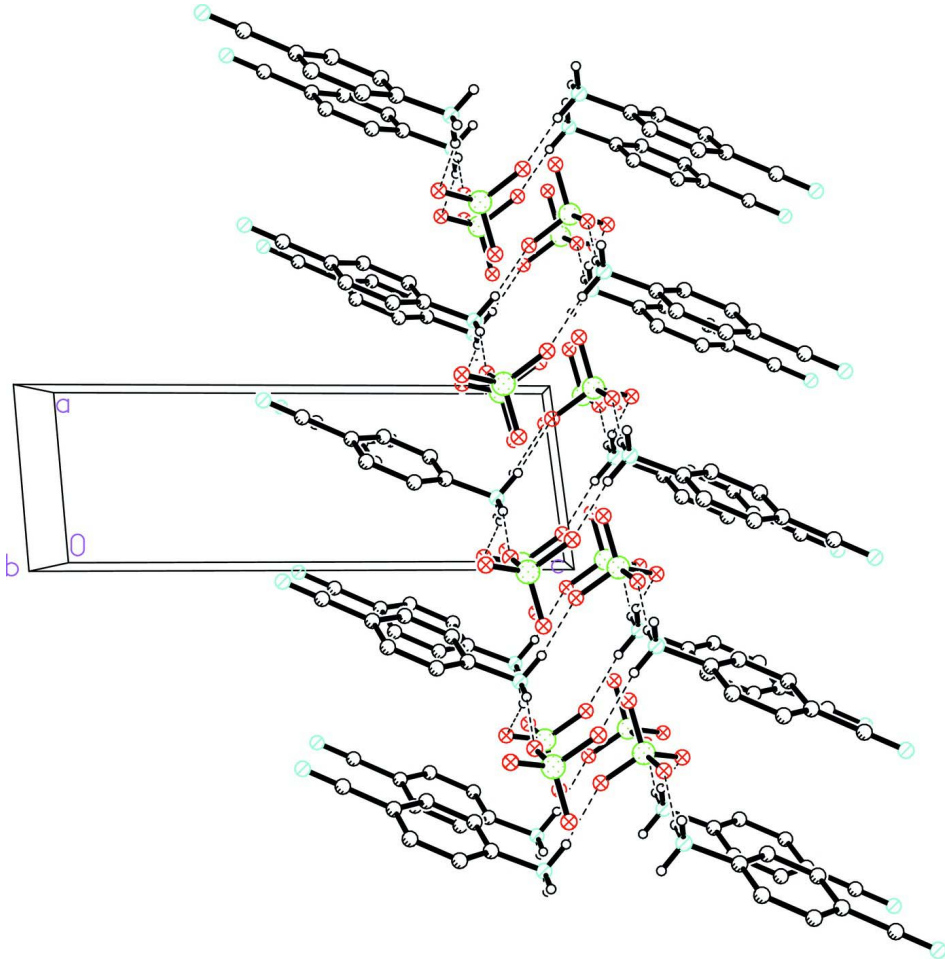

Figure 2

The crystal packing of the title compound 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

$C_7H_7N_2^+·ClO_4^-$
M_r = 218.60
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 4.9905 (10) Å
b = 6.9465 (14) Å
c = 13.998 (3) Å
α = 94.87 (3)°
β = 95.68 (3)°
γ = 103.99 (3)°
V = 465.57 (17) Å^3

Z = 2
F(000) = 224
D_x = 1.559 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1806 reflections
θ = 3.0–27.5°
μ = 0.40 mm^{-1}
T = 298 K
Block, colourless
0.25 × 0.15 × 0.15 mm

Data collection

Rigaku Mercury2 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 13.6612 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.941, T_{max} = 1.000
4861 measured reflections
2126 independent reflections
1851 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 27.5°, θ_{min} = 3.0°
h = −6→6
k = −8→8
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.097
S = 1.05
2126 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
$w = 1/[\sigma^2(F_o^2) + (0.0483P)^2 + 0.1253P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 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 (Å^2)

	x	y	z	U_{iso}*/U_{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*

Cl1	−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 (Å²)

	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)
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)
Cl1	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 (Å, º)

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)	Cl1—O4	1.4202 (15)
C2—C3	1.392 (3)	Cl1—O3	1.4215 (14)
C3—C4	1.375 (3)	Cl1—O1	1.4222 (14)
C3—H3	0.9300	Cl1—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—Cl1—O3	109.51 (11)
C4—C3—C2	119.1 (2)	O4—Cl1—O1	109.25 (11)

C4—C3—H3	120.4	O3—Cl1—O1	108.89 (10)
C2—C3—H3	120.4	O4—Cl1—O2	109.01 (11)
C5—C4—C3	119.54 (19)	O3—Cl1—O2	110.69 (10)
C5—C4—H4	120.2	O1—Cl1—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 (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O2^{i}	0.89	2.04	2.881 (2)	158
N1—H1*B*···O3^{ii}	0.89	1.98	2.855 (2)	166
N1—H1*C*···O4^{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.