

## 3-Cyanoanilinium nitrate

Bo Wang

Ordered Matter Science Research Center, College of Chemistry and Chemical Engineering, Southeast University, Nanjing 210096, People's Republic of China  
Correspondence e-mail: fudavid88@yahoo.com.cn

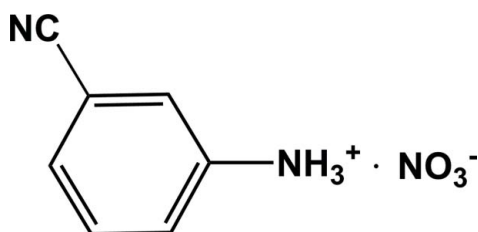
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Key indicators: single-crystal X-ray study;  $T = 298$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.052;  $wR$  factor = 0.141; data-to-parameter ratio = 15.6.

In the cation of the title compound,  $\text{C}_7\text{H}_7\text{N}_2^+\cdot\text{NO}_3^-$ , the nitrile group and the benzene ring are almost coplanar (r.m.s. deviation = 0.006 Å). In the crystal, the ions are connected by bifurcated  $\text{N}-\text{H}\cdots(\text{O},\text{O})$  hydrogen bonds, forming a two-dimensional network parallel to (001).

### Related literature

For the applications of metal-organic coordination compounds, see: Fu *et al.* (2007); Chen *et al.* (2001); Fu & Xiong (2008); Xiong *et al.* (1999); Xie *et al.* (2003); Zhao *et al.* (2004). For nitrile derivatives, see: Fu *et al.* (2008); Wang *et al.* 2002.



### Experimental

#### Crystal data

 $\text{C}_7\text{H}_7\text{N}_2^+\cdot\text{NO}_3^-$ 
 $M_r = 181.16$ 

 Orthorhombic, *Pbca*
 $a = 10.210$  (2) Å

 $b = 10.812$  (2) Å

 $c = 15.398$  (3) Å

 $V = 1699.8$  (6) Å<sup>3</sup>
 $Z = 8$ 

 Mo  $K\alpha$  radiation

 $\mu = 0.11$  mm<sup>-1</sup>
 $T = 298$  K

 $0.40 \times 0.25 \times 0.20$  mm

#### Data collection

Rigaku Mercury2 diffractometer  
Absorption correction: multi-scan  
(*CrystalClear*; Rigaku, 2005)  
 $T_{\min} = 0.94$ ,  $T_{\max} = 1.00$

15905 measured reflections  
1871 independent reflections  
1456 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.062$

#### Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.052$ 
 $wR(F^2) = 0.141$ 
 $S = 1.14$ 

1871 reflections

120 parameters

H-atom parameters constrained

 $\Delta\rho_{\max} = 0.21$  e Å<sup>-3</sup>
 $\Delta\rho_{\min} = -0.19$  e Å<sup>-3</sup>
**Table 1**

Hydrogen-bond geometry (Å, °).

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
N2—H2A $\cdots$ O3 <sup>i</sup>	0.89	2.22	3.104 (2)	173
N2—H2A $\cdots$ O1 <sup>i</sup>	0.89	2.44	3.107 (2)	133
N2—H2B $\cdots$ O2 <sup>ii</sup>	0.89	2.06	2.859 (2)	150
N2—H2B $\cdots$ O3 <sup>ii</sup>	0.89	2.25	3.049 (2)	149
N2—H2C $\cdots$ O2	0.89	1.85	2.738 (2)	172
N2—H2C $\cdots$ O1	0.89	2.56	3.090 (2)	119

Symmetry codes: (i)  $-x + \frac{1}{2}, y + \frac{1}{2}, z$ ; (ii)  $x + \frac{1}{2}, y, -z + \frac{3}{2}$ .

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*.

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: CI2892).

### References

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**supplementary materials**

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### 3-Cyanoanilinium nitrate

B. Wang

#### Comment

The construction of metal-organic coordination compounds has attracted much attention owing to potential functions, such as permittivity, fluorescence, magnetism and optical properties (Fu *et al.*, 2007; Chen *et al.*, 2001; Fu & Xiong, 2008; Xie *et al.*, 2003; Zhao *et al.*, 2004; Xiong *et al.*, 1999). Nitrile derivatives are a class of excellent ligands for the construction of novel metal-organic frameworks (Wang *et al.* 2002; Fu *et al.*, 2008). We report here the crystal structure of the title compound, 3-cyanoanilinium nitrate.

In the 3-cyanoanilinium cation (Fig.1), the nitrile group and the benzene ring are coplanar. The nitrile group C1≡N1 bond length of 1.102 (3) Å is within the normal range.

In the crystal structure, all the amine group H atoms are involved in N—H···O hydrogen bonds (Table 1) with O atoms of the NO<sub>3</sub><sup>-</sup> anion. These hydrogen bonds link the ionic units into a two-dimensional network (Fig. 2) parallel to the (001) plane.

#### Experimental

The commercial 3-aminobenzonitrile (3 mmol, 0.55 g) and HNO<sub>3</sub> (0.5 ml) were dissolved in ethanol (20 ml). Colourless block-shaped crystals of the title compound suitable for X-ray analysis were obtained by slow evaporation at room temperature.

#### Refinement

H atoms were positioned geometrically and treated as riding, with C-H = 0.93 Å, N-H = 0.89 Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  and  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{N})$ . A rotating-group model was used for the -NH<sub>3</sub> group.

#### Figures

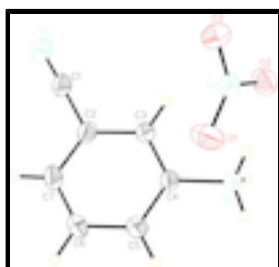


Fig. 1. A view of the title compound with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

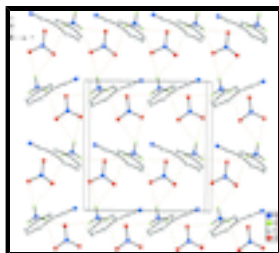
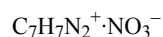


Fig. 2. The crystal packing of the title compound, viewed along the *c* axis, showing N—H...O hydrogen bonds (dashed lines). H atoms not involved in hydrogen bonding have been omitted for clarity.

## 3-Cyanoanilinium nitrate

### Crystal data



$$M_r = 181.16$$

Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

$$a = 10.210 (2) \text{ \AA}$$

$$b = 10.812 (2) \text{ \AA}$$

$$c = 15.398 (3) \text{ \AA}$$

$$V = 1699.8 (6) \text{ \AA}^3$$

$$Z = 8$$

$$F_{000} = 752$$

$$D_x = 1.416 \text{ Mg m}^{-3}$$

Mo *K* $\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 1456 reflections

$$\theta = 3.1\text{--}27.5^\circ$$

$$\mu = 0.11 \text{ mm}^{-1}$$

$$T = 298 \text{ K}$$

Block, colourless

$$0.40 \times 0.25 \times 0.20 \text{ mm}$$

### Data collection

Rigaku Mercury2  
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

Detector resolution: 13.6612 pixels  $\text{mm}^{-1}$

$$T = 298 \text{ K}$$

CCD profile fitting scans

Absorption correction: multi-scan  
(CrystalClear; Rigaku, 2005)

$$T_{\min} = 0.94, T_{\max} = 1.00$$

15905 measured reflections

1871 independent reflections

1456 reflections with  $I > 2\sigma(I)$

$$R_{\text{int}} = 0.062$$

$$\theta_{\max} = 27.5^\circ$$

$$\theta_{\min} = 3.1^\circ$$

$$h = -13 \rightarrow 13$$

$$k = -13 \rightarrow 13$$

$$l = -19 \rightarrow 19$$

### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$$R[F^2 > 2\sigma(F^2)] = 0.052$$

$$wR(F^2) = 0.141$$

$$S = 1.14$$

1871 reflections

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.059P)^2 + 0.3685P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} = 0.001$$

$$\Delta\rho_{\max} = 0.21 \text{ e \AA}^{-3}$$

$$\Delta\rho_{\min} = -0.19 \text{ e \AA}^{-3}$$

120 parameters

Extinction correction: SHELXL97 (Sheldrick, 2008),

$$F_c^* = kF_c [1 + 0.001 \times F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$$

Primary atom site location: structure-invariant direct methods

Extinction coefficient: 0.021 (3)

Secondary atom site location: difference Fourier map

### 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  $F^2 > 2\sigma(F^2)$  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 ( $\text{\AA}^2$ )

	x	y	z	$U_{iso}^*/U_{eq}$
N1	0.06292 (19)	0.5067 (2)	0.39061 (13)	0.0724 (6)
N2	0.39497 (14)	0.44355 (16)	0.69503 (9)	0.0435 (4)
H2A	0.4110	0.5239	0.7016	0.065*
H2B	0.4620	0.4001	0.7158	0.065*
H2C	0.3224	0.4236	0.7238	0.065*
C1	0.1482 (2)	0.4737 (2)	0.42791 (13)	0.0526 (5)
C2	0.25733 (17)	0.43017 (19)	0.47337 (11)	0.0443 (5)
C3	0.27221 (17)	0.45958 (18)	0.56150 (11)	0.0420 (5)
H3	0.2108	0.5080	0.5904	0.050*
C4	0.37764 (16)	0.41559 (17)	0.60241 (11)	0.0382 (4)
C5	0.46585 (19)	0.34385 (19)	0.55934 (12)	0.0488 (5)
H5	0.5382	0.3131	0.5890	0.059*
C6	0.4502 (2)	0.3155 (2)	0.47200 (13)	0.0568 (6)
H6	0.5119	0.2669	0.4436	0.068*
C7	0.3461 (2)	0.3585 (2)	0.42905 (12)	0.0538 (5)
H7	0.3339	0.3403	0.3706	0.065*
O1	0.23542 (15)	0.20195 (16)	0.71257 (11)	0.0701 (5)
O2	0.16047 (13)	0.37804 (14)	0.76798 (10)	0.0594 (5)
O3	0.04000 (13)	0.21817 (14)	0.73494 (10)	0.0592 (5)
N3	0.14567 (15)	0.26571 (16)	0.73782 (10)	0.0449 (4)

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
N1	0.0625 (12)	0.1071 (17)	0.0476 (11)	0.0072 (12)	-0.0138 (9)	-0.0010 (11)
N2	0.0413 (8)	0.0563 (10)	0.0328 (8)	0.0053 (7)	-0.0013 (6)	-0.0047 (7)
C1	0.0517 (11)	0.0722 (14)	0.0339 (10)	-0.0038 (10)	-0.0043 (9)	-0.0017 (9)
C2	0.0432 (10)	0.0547 (12)	0.0351 (9)	-0.0056 (8)	-0.0034 (7)	0.0006 (8)

## supplementary materials

C3	0.0382 (9)	0.0536 (11)	0.0341 (9)	0.0008 (8)	0.0015 (7)	-0.0030 (8)
C4	0.0401 (9)	0.0440 (10)	0.0306 (9)	-0.0018 (8)	0.0004 (7)	-0.0021 (7)
C5	0.0473 (10)	0.0588 (12)	0.0403 (10)	0.0103 (9)	0.0008 (8)	-0.0038 (9)
C6	0.0572 (13)	0.0703 (14)	0.0429 (11)	0.0126 (11)	0.0061 (9)	-0.0134 (10)
C7	0.0612 (12)	0.0685 (14)	0.0318 (9)	-0.0020 (11)	0.0013 (9)	-0.0094 (9)
O1	0.0487 (9)	0.0727 (11)	0.0889 (12)	0.0073 (8)	0.0163 (8)	-0.0161 (9)
O2	0.0548 (9)	0.0635 (10)	0.0598 (9)	-0.0069 (7)	0.0087 (7)	-0.0173 (8)
O3	0.0395 (8)	0.0752 (10)	0.0628 (10)	-0.0077 (7)	-0.0046 (6)	-0.0065 (7)
N3	0.0440 (9)	0.0596 (11)	0.0310 (8)	-0.0007 (8)	-0.0012 (6)	0.0000 (7)

### Geometric parameters (Å, °)

N1—C1	1.102 (3)	C4—C5	1.361 (3)
N2—C4	1.469 (2)	C5—C6	1.389 (3)
N2—H2A	0.89	C5—H5	0.93
N2—H2B	0.89	C6—C7	1.335 (3)
N2—H2C	0.89	C6—H6	0.93
C1—C2	1.397 (3)	C7—H7	0.93
C2—C7	1.374 (3)	O1—N3	1.211 (2)
C2—C3	1.402 (3)	O2—N3	1.309 (2)
C3—C4	1.335 (2)	O3—N3	1.196 (2)
C3—H3	0.93		
C4—N2—H2A	109.5	C3—C4—N2	118.82 (15)
C4—N2—H2B	109.5	C5—C4—N2	120.72 (16)
H2A—N2—H2B	109.5	C4—C5—C6	121.42 (18)
C4—N2—H2C	109.5	C4—C5—H5	119.3
H2A—N2—H2C	109.5	C6—C5—H5	119.3
H2B—N2—H2C	109.5	C7—C6—C5	119.66 (19)
N1—C1—C2	178.6 (2)	C7—C6—H6	120.2
C7—C2—C1	117.84 (17)	C5—C6—H6	120.2
C7—C2—C3	122.50 (17)	C6—C7—C2	118.37 (17)
C1—C2—C3	119.66 (17)	C6—C7—H7	120.8
C4—C3—C2	117.60 (17)	C2—C7—H7	120.8
C4—C3—H3	121.2	O3—N3—O1	115.22 (17)
C2—C3—H3	121.2	O3—N3—O2	121.07 (16)
C3—C4—C5	120.45 (17)	O1—N3—O2	123.69 (16)

### Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N2—H2A...O3 <sup>i</sup>	0.89	2.22	3.104 (2)	173
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Symmetry codes: (i)  $-x+1/2, y+1/2, z$ ; (ii)  $x+1/2, y, -z+3/2$ .

Fig. 1

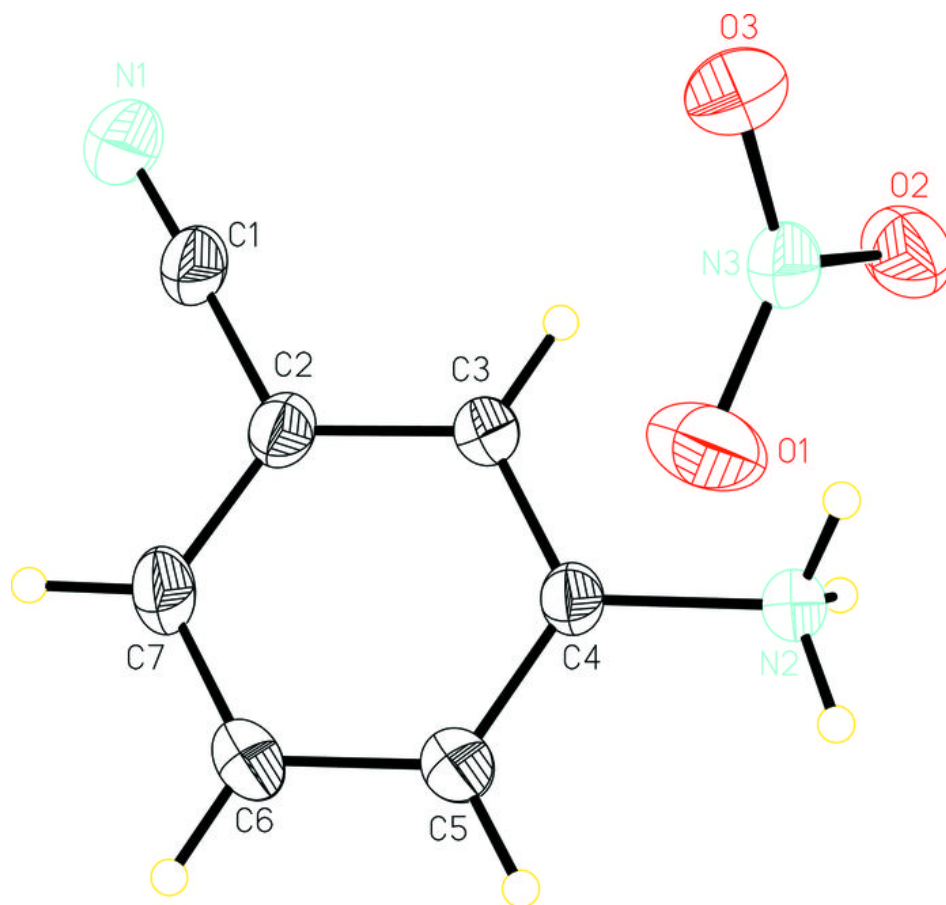


Fig. 2

