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3-Cyanoanilinium bromide

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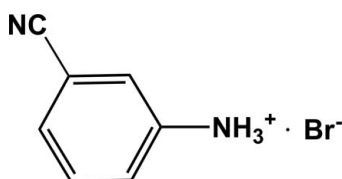
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.007$ Å; R factor = 0.052; wR factor = 0.134; data-to-parameter ratio = 18.7.

In the cation of the title compound, $\text{C}_7\text{H}_7\text{N}_2^+\cdot\text{Br}^-$, all non-H atoms are essentially coplanar [r.m.s. deviation = 0.010 (5) Å]. The compound is isomorphous with the chloride analogue. In the crystal, the cations and anions are connected by $\text{N}-\text{H}\cdots\text{Br}$ hydrogen bonds.

Related literature

For 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. For the chloride analogue, see: Wen (2008).



Experimental

Crystal data

$\text{C}_7\text{H}_7\text{N}_2^+\cdot\text{Br}^-$
 $M_r = 199.06$
Triclinic, $P\bar{1}$
 $a = 4.6396$ (9) Å
 $b = 6.1757$ (12) Å
 $c = 13.542$ (3) Å
 $\alpha = 93.07$ (3)°
 $\beta = 96.22$ (3)°

$\gamma = 97.33$ (3)°
 $V = 381.68$ (13) Å³
 $Z = 2$
Mo $K\alpha$ radiation
 $\mu = 5.31$ mm⁻¹
 $T = 298$ K
 $0.40 \times 0.05 \times 0.05$ mm

Data collection

Rigaku Mercury2 diffractometer
Absorption correction: multi-scan
(*CrystalClear*; Rigaku, 2005)
 $T_{\min} = 0.90$, $T_{\max} = 1.00$

3777 measured reflections
1716 independent reflections
1378 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.063$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.052$
 $wR(F^2) = 0.134$
 $S = 1.10$
1716 reflections

92 parameters
H-atom parameters constrained
 $\Delta\rho_{\max} = 0.71$ e Å⁻³
 $\Delta\rho_{\min} = -0.75$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N2}-\text{H2A}\cdots\text{Br1}^{\text{i}}$	0.89	2.59	3.434 (4)	159
$\text{N2}-\text{H2B}\cdots\text{Br1}^{\text{ii}}$	0.89	2.46	3.337 (4)	169
$\text{N2}-\text{H2C}\cdots\text{Br1}$	0.89	2.45	3.299 (4)	160

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

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); 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: BX2236).

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supporting information

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3-Cyanoanilinium bromide

Bo Wang

S1. 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, which is isomorphous with the chloride analogue (Wen, 2008). In the cation all non-H atoms are essentially coplanar [r.m.s. deviation 0.010 (5) Å]. In the crystal structure, the organic cations and bromide ions are connected by N—H \cdots Br hydrogen bonds along *b* axis, (Table 1), (Fig. 2).

S2. Experimental

The commercial 3-aminobenzonitrile (3 mmol, 0.55 g) and HBr (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.

S3. Refinement

All H atoms attached to C and N 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 -NH₃ group.

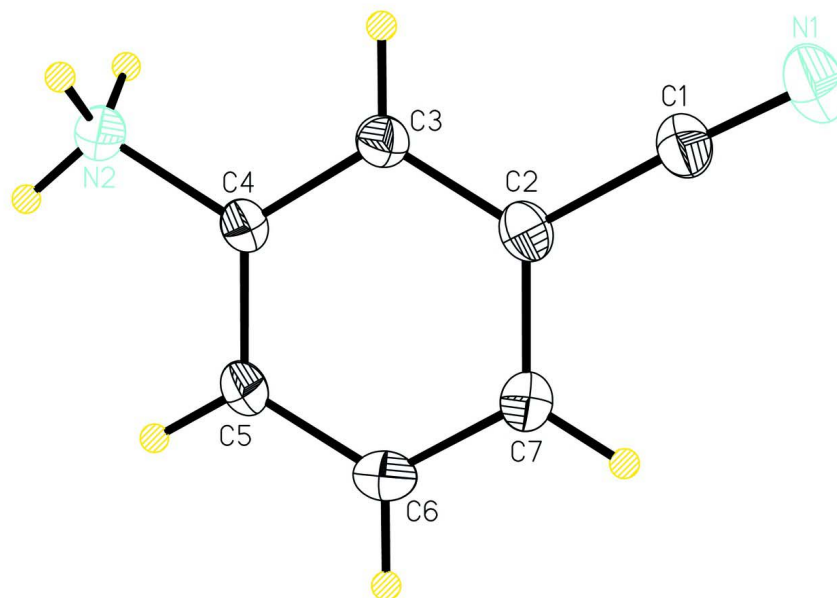
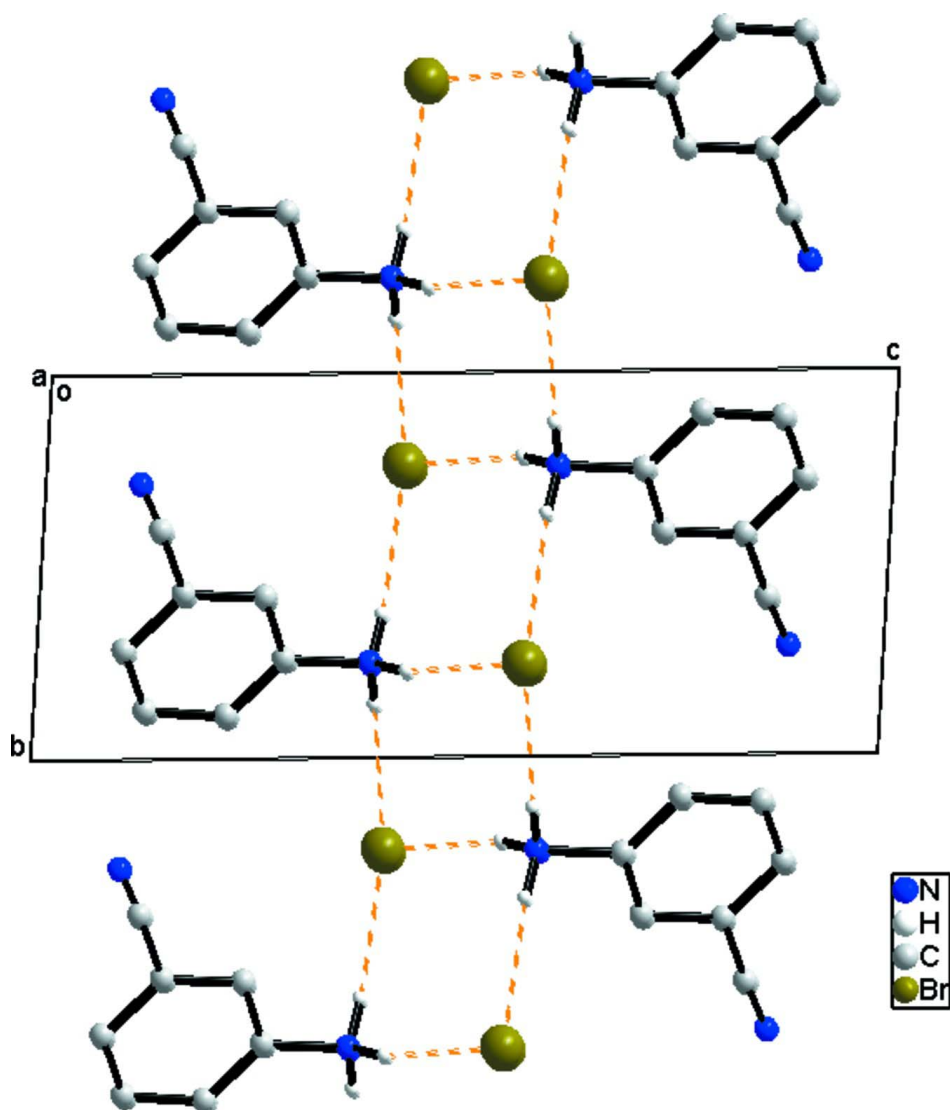


Figure 1

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

**Figure 2**

The crystal packing of the title compound, viewed along the *a* axis showing the N—H···Br interactions (dotted line) in the title compound. H atoms not involved in hydrogen bonding (dashed lines) have been omitted for clarity.

3-Cyanoanilinium bromide

Crystal data

$C_7H_7N_2^+ \cdot Br^-$

$M_r = 199.06$

Triclinic, $P\bar{1}$

Hall symbol: $-P\ 1$

$a = 4.6396$ (9) Å

$b = 6.1757$ (12) Å

$c = 13.542$ (3) Å

$\alpha = 93.07$ (3)°

$\beta = 96.22$ (3)°

$\gamma = 97.33$ (3)°

$V = 381.68$ (13) Å³

$Z = 2$

$F(000) = 196$

$D_x = 1.732$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 1378 reflections

$\theta = 3.0$ – 27.5 °

$\mu = 5.31$ mm⁻¹

$T = 298$ K

Block, colourless

$0.40 \times 0.05 \times 0.05$ 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)
 $T_{\min} = 0.90$, $T_{\max} = 1.00$

3777 measured reflections
1716 independent reflections
1378 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.063$
 $\theta_{\max} = 27.5^\circ$, $\theta_{\min} = 3.0^\circ$
 $h = -6 \rightarrow 5$
 $k = -8 \rightarrow 8$
 $l = -17 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.052$
 $wR(F^2) = 0.134$
 $S = 1.10$
1716 reflections
92 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
 $w = 1/[\sigma^2(F_o^2) + (0.053P)^2 + 0.0394P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.71 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.75 \text{ e } \text{Å}^{-3}$

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 $F^2 > 2\sigma(F^2)$ 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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N2	0.6256 (9)	0.2461 (6)	0.6051 (3)	0.0419 (10)
H2A	0.7437	0.1446	0.5990	0.063*
H2B	0.7175	0.3755	0.5927	0.063*
H2C	0.4653	0.2118	0.5619	0.063*
N1	-0.0113 (11)	0.7158 (8)	0.8871 (4)	0.0575 (13)
C4	0.5438 (10)	0.2565 (7)	0.7064 (4)	0.0339 (10)
C3	0.3783 (10)	0.4177 (7)	0.7330 (3)	0.0349 (10)
H3	0.3215	0.5166	0.6877	0.042*
C2	0.3001 (10)	0.4273 (7)	0.8286 (4)	0.0350 (10)
C5	0.6331 (11)	0.1113 (7)	0.7719 (4)	0.0389 (11)
H5	0.7446	0.0048	0.7527	0.047*
C7	0.3878 (12)	0.2814 (8)	0.8962 (4)	0.0429 (12)
H7	0.3345	0.2889	0.9604	0.051*
C6	0.5540 (12)	0.1259 (8)	0.8677 (4)	0.0473 (13)
H6	0.6146	0.0287	0.9132	0.057*
C1	0.1216 (11)	0.5910 (8)	0.8593 (4)	0.0422 (12)

Br1	0.09268 (11)	0.23870 (7)	0.42210 (4)	0.0448 (2)
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Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N2	0.053 (3)	0.044 (2)	0.032 (2)	0.0176 (19)	0.0038 (19)	0.0003 (18)
N1	0.058 (3)	0.052 (3)	0.067 (3)	0.020 (2)	0.016 (3)	-0.004 (2)
C4	0.040 (3)	0.028 (2)	0.033 (3)	0.0073 (18)	0.004 (2)	-0.0017 (18)
C3	0.039 (3)	0.033 (2)	0.032 (3)	0.010 (2)	0.000 (2)	0.0010 (19)
C2	0.030 (2)	0.034 (2)	0.041 (3)	0.0057 (18)	0.005 (2)	-0.002 (2)
C5	0.049 (3)	0.034 (2)	0.037 (3)	0.018 (2)	0.007 (2)	-0.001 (2)
C7	0.056 (3)	0.039 (3)	0.035 (3)	0.011 (2)	0.010 (2)	0.000 (2)
C6	0.062 (4)	0.040 (3)	0.045 (3)	0.015 (2)	0.010 (3)	0.014 (2)
C1	0.045 (3)	0.040 (3)	0.044 (3)	0.012 (2)	0.011 (2)	0.000 (2)
Br1	0.0573 (4)	0.0399 (3)	0.0423 (4)	0.0214 (2)	0.0112 (3)	0.0037 (2)

Geometric parameters (Å, °)

N2—C4	1.463 (6)	C3—H3	0.9300
N2—H2A	0.8900	C2—C7	1.384 (7)
N2—H2B	0.8900	C2—C1	1.457 (6)
N2—H2C	0.8900	C5—C6	1.388 (7)
N1—C1	1.121 (6)	C5—H5	0.9300
C4—C5	1.365 (6)	C7—C6	1.371 (7)
C4—C3	1.388 (6)	C7—H7	0.9300
C3—C2	1.383 (7)	C6—H6	0.9300
C4—N2—H2A	109.5	C3—C2—C1	120.2 (4)
C4—N2—H2B	109.5	C7—C2—C1	119.1 (5)
H2A—N2—H2B	109.5	C4—C5—C6	118.6 (4)
C4—N2—H2C	109.5	C4—C5—H5	120.7
H2A—N2—H2C	109.5	C6—C5—H5	120.7
H2B—N2—H2C	109.5	C6—C7—C2	119.4 (5)
C5—C4—C3	122.0 (4)	C6—C7—H7	120.3
C5—C4—N2	119.8 (4)	C2—C7—H7	120.3
C3—C4—N2	118.2 (4)	C7—C6—C5	121.0 (5)
C2—C3—C4	118.2 (4)	C7—C6—H6	119.5
C2—C3—H3	120.9	C5—C6—H6	119.5
C4—C3—H3	120.9	N1—C1—C2	177.0 (6)
C3—C2—C7	120.7 (4)		
C5—C4—C3—C2	-0.9 (7)	N2—C4—C5—C6	179.6 (5)
N2—C4—C3—C2	179.8 (4)	C3—C2—C7—C6	0.0 (7)
C4—C3—C2—C7	0.7 (7)	C1—C2—C7—C6	179.5 (5)
C4—C3—C2—C1	-178.8 (4)	C2—C7—C6—C5	-0.6 (8)
C3—C4—C5—C6	0.3 (7)	C4—C5—C6—C7	0.5 (8)

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 Br1 ⁱ	0.89	2.59	3.434 (4)	159
N2—H2B \cdots Br1 ⁱⁱ	0.89	2.46	3.337 (4)	169
N2—H2C \cdots Br1	0.89	2.45	3.299 (4)	160

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