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4-Cyanoanilinium bromide

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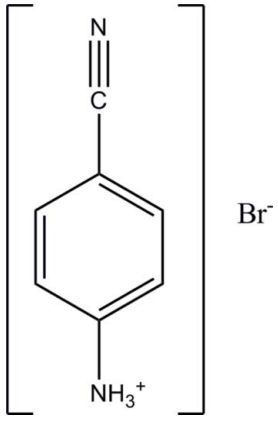
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.020; wR factor = 0.051; data-to-parameter ratio = 20.6.

In the crystal structure of the title compound, $C_7H_7N_2^+\cdot Br^-$, the cations are associated into inversion dimers through weak pairwise C—H···N hydrogen bonds. The dimers further form stepped sheets *via* weak pairwise C—H···N hydrogen bonds. In the sheets, the spacing between the mean planes of the laterally displaced aromatic rings in adjacent dimers is 1.124 (6) Å. Three N—H···Br interactions and two weak C—H···Br interactions per cation tie the sheets together.

Related literature

For the structure of 4-cyanoanilinium choride, see: Colapietro *et al.* (1981). For the structure of 4-cyanoanilinium iodide, see: Mague *et al.* (2012). For the structure of anilinium bromide, see: Schweiss *et al.* (1983). For a discussion of C—H and N—H hydrogen bonding to halide ions, see: Steiner (1998).



Experimental

Crystal data

$C_7H_7N_2^+\cdot Br^-$
M_r = 199.06
Triclinic, $P\overline{1}$
a = 4.3102 (10) Å
b = 6.1076 (13) Å
c = 14.510 (3) Å
α = 91.719 (3)°
β = 93.290 (3)°
γ = 101.428 (3)°
V = 373.46 (14) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 5.42 mm^{-1}
T = 100 K
0.20 × 0.19 × 0.16 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: numerical (*SADABS*; Sheldrick, 2009)
T_{min} = 0.631, T_{max} = 0.837
6534 measured reflections
1874 independent reflections
1802 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.020
$wR(F^2)$ = 0.051
S = 1.06
1874 reflections
91 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.86 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···Br1	0.88	2.47	3.3209 (16)	162
C2—H2···Br1^{i}	0.95	2.87	3.7316 (18)	151
C3—H3···N2^{ii}	0.95	2.62	3.466 (2)	149
C5—H5···N2^{iii}	0.95	2.69	3.517 (2)	146
C6—H6···Br1^{iv}	0.95	3.00	3.8063 (18)	144
N1—H1B···Br1^{iv}	0.88	2.54	3.4174 (16)	175
N1—H1C···Br1^{v}	0.88	2.49	3.3400 (16)	162

Symmetry codes: (i) $-x, -y+2, -z+1$; (ii) $-x, -y+2, -z+2$; (iii) $-x+1, -y+1, -z+2$; (iv) $-x+1, -y+1, -z+1$; (v) $-x+1, -y+2, -z+1$.

Data collection: *APEX2* (Bruker, 2010); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXM* (Sheldrick, 1998, 2004); 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: JJ2147).

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supporting information

Acta Cryst. (2012). E**68**, o2884 [https://doi.org/10.1107/S1600536812037014]

4-Cyanoanilinium bromide

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S1. Comment

In the title compound, $[C_7H_7N_2]^+$ Br^-, the cations are associated into dimers through weak, pairwise C3—H3···N2 intermolecular interactions (Fig. 1). The dimers further form stepped sheets *via* weak, pairwise C5—H5···N2 intermolecular interactions. In these sheets the spacing between the mean planes of the aromatic rings in adjacent dimers is 1.124 (6) Å (Table 1). The three hydrogen atoms of the anilinium group make contacts with the surrounding anions of 2.47 - 2.54 Å. These distances compare well with the mean value of 2.49 (2) Å for an N^+—H···Br^- hydrogen bond (Steiner, 1998) and serve, together with weak C2—H2···Br1 and C6—H6···Br1 interactions, to tie the stepped sheets into a layer structure (Fig. 2) with the layers 3.493 (7) Å apart and forming rectangular channels of width *ca* 12.8 Å (Fig. 3).

S2. Experimental

0.55 g of 4-cyanoaniline and 2.5 ml of aquous hydrobromic acid (2 *M*) were combined in 10 ml of ethanol. This solution was slowly evaporated to dryness under ambient conditions to form crystals of the title compound.

S3. Refinement

H-atoms attached to C were placed in calculated positions (C—H = 0.95 - 0.98 Å) while those attached to N were placed in sites determined from a difference map and their coordinates adjusted to give N—H = 0.88 Å. All H-atoms were included as riding contributions with isotropic displacement parameters 1.2 times those of the attached atoms.

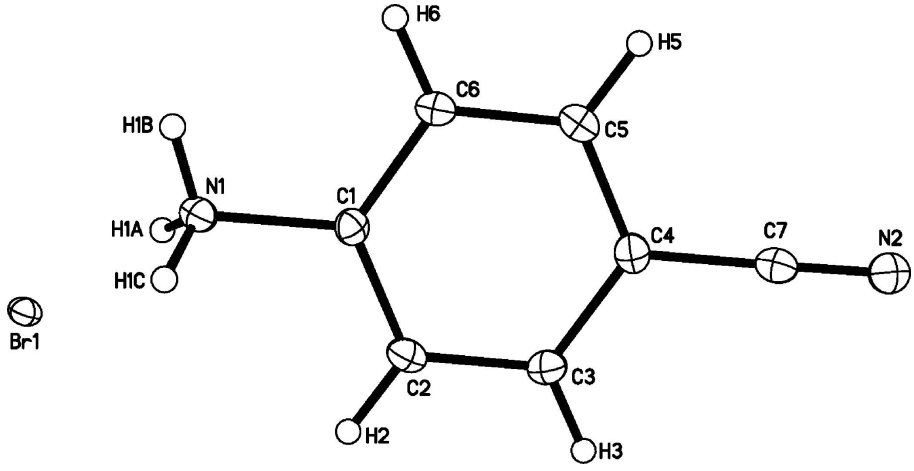

Figure 1

Perspective view of the asymmetric unit with displacement ellipsoids drawn at the 50% probability level

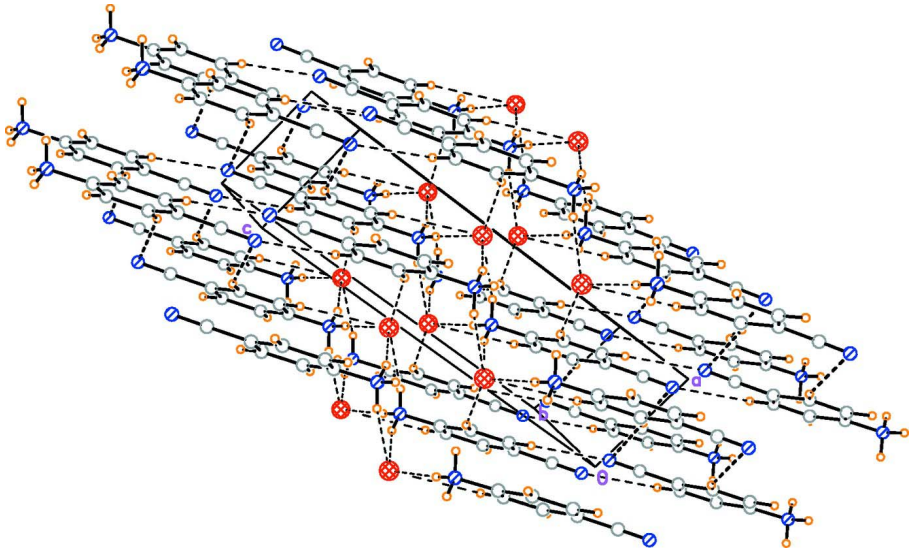

Figure 2

Packing showing the stepped layer structure. N—H···Br, C—H···N and C—H···Br interactions are shown as dashed lines. Color key: C = gray, H = orange, Br = red, N = blue.

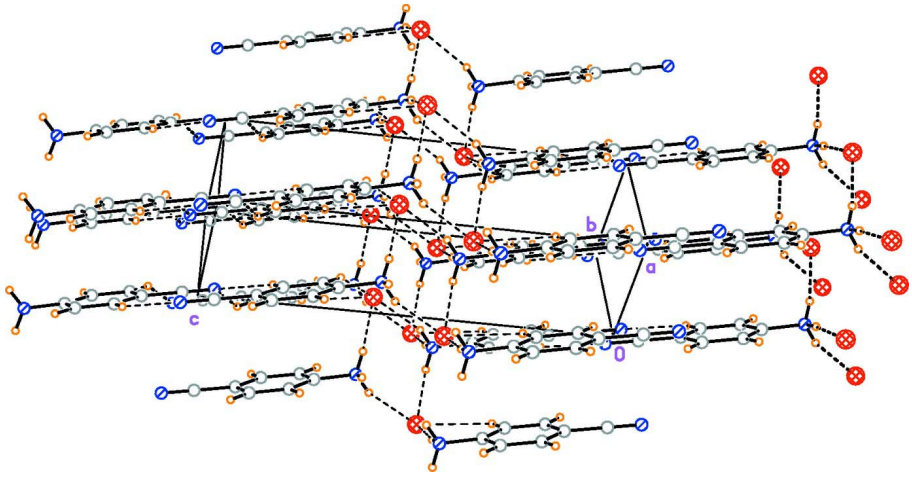

Figure 3

Packing showing the rectangular channels. N—H···Br, C—H···N and C—H···Br interactions are shown as dashed lines. Color key: C = gray, H = orange, Br = red, N = blue.

4-Cyanoanilinium bromide

Crystal data

$C_7H_7N_2^+·Br^-$
M_r = 199.06
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 4.3102 (10) Å
b = 6.1076 (13) Å
c = 14.510 (3) Å
α = 91.719 (3)°
β = 93.290 (3)°
γ = 101.428 (3)°
V = 373.46 (14) Å^3
Z = 2
F(000) = 196
D_x = 1.770 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5589 reflections
θ = 2.8–29.1°
μ = 5.42 mm^{-1}

T = 100 K
Block, colourless

0.20 × 0.19 × 0.16 mm

Data collection

Bruker SMART APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: numerical (*SADABS*; Sheldrick, 2009)
T_{min} = 0.631, T_{max} = 0.837

6534 measured reflections
1874 independent reflections
1802 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 29.2°, θ_{min} = 2.8°
h = −5→5
k = −8→8
l = −19→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.020
$wR(F^2)$ = 0.051
S = 1.06
1874 reflections
91 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
$w = 1/[\sigma^2(F_o^2) + (0.0248P)^2 + 0.1891P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.86 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

Special details

Experimental. The diffraction data were obtained from 3 sets of 400 frames, each of width 0.5 °. in omega, collected at phi = 0.00, 90.00 and 180.00 °. and 2 sets of 800 frames, each of width 0.45 ° in phi, collected at omega = -30.00 and 210.00 °. The scan time was 10 sec/frame.

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. H-atoms attached to carbon were placed in calculated positions (C—H = 0.95 Å) while those attached to nitrogen were placed in locations derived from a difference map and then their coordinates adjusted to give an N—H distance of 0.88 Å. All were included as riding contributions with isotropic displacement parameters 1.2 times those of the attached atoms.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
Br1	0.08172 (3)	0.73901 (2)	0.423553 (10)	0.01208 (7)
N1	0.5934 (3)	0.7244 (2)	0.60221 (10)	0.0128 (3)
H1A	0.4298	0.7029	0.5615	0.015*
H1B	0.6868	0.6102	0.5940	0.015*
H1C	0.7187	0.8527	0.5922	0.015*
N2	0.1311 (4)	0.7765 (3)	1.04130 (11)	0.0215 (3)
C1	0.4868 (4)	0.7327 (3)	0.69615 (11)	0.0117 (3)
C2	0.3482 (4)	0.9098 (3)	0.72253 (12)	0.0142 (3)
H2	0.3199	1.0208	0.6801	0.017*

C3	0.2514 (4)	0.9219 (3)	0.81193 (12)	0.0148 (3)
H3	0.1545	1.0409	0.8312	0.018*
C4	0.2980 (4)	0.7575 (3)	0.87320 (12)	0.0140 (3)
C5	0.4367 (4)	0.5796 (3)	0.84545 (12)	0.0158 (3)
H5	0.4650	0.4679	0.8875	0.019*
C6	0.5326 (4)	0.5674 (3)	0.75594 (12)	0.0142 (3)
H6	0.6278	0.4481	0.7361	0.017*
C7	0.2031 (4)	0.7694 (3)	0.96694 (13)	0.0168 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.01341 (9)	0.01043 (10)	0.01308 (10)	0.00377 (6)	0.00081 (6)	0.00256 (6)
N1	0.0141 (6)	0.0118 (7)	0.0130 (7)	0.0032 (5)	0.0006 (5)	0.0018 (5)
N2	0.0304 (9)	0.0179 (8)	0.0179 (8)	0.0073 (7)	0.0055 (7)	0.0020 (6)
C1	0.0118 (7)	0.0123 (8)	0.0102 (7)	0.0013 (6)	−0.0010 (6)	−0.0002 (6)
C2	0.0159 (8)	0.0124 (8)	0.0149 (8)	0.0040 (6)	0.0000 (6)	0.0033 (6)
C3	0.0167 (8)	0.0127 (8)	0.0157 (8)	0.0050 (7)	0.0009 (6)	0.0001 (6)
C4	0.0141 (8)	0.0150 (8)	0.0122 (8)	0.0017 (6)	0.0007 (6)	0.0008 (6)
C5	0.0185 (8)	0.0143 (8)	0.0152 (8)	0.0047 (7)	0.0007 (6)	0.0038 (6)
C6	0.0165 (8)	0.0118 (8)	0.0152 (8)	0.0045 (6)	0.0008 (6)	0.0018 (6)
C7	0.0201 (8)	0.0125 (8)	0.0181 (9)	0.0038 (7)	0.0011 (7)	0.0026 (6)

Geometric parameters (Å, º)

N1—C1	1.466 (2)	C2—H2	0.9500
N1—H1A	0.8800	C3—C4	1.397 (2)
N1—H1B	0.8801	C3—H3	0.9500
N1—H1C	0.8800	C4—C5	1.399 (2)
N2—C7	1.142 (3)	C4—C7	1.447 (2)
C1—C6	1.387 (2)	C5—C6	1.390 (2)
C1—C2	1.389 (2)	C5—H5	0.9500
C2—C3	1.390 (2)	C6—H6	0.9500
C1—N1—H1A	110.3	C2—C3—H3	120.3
C1—N1—H1B	110.7	C4—C3—H3	120.3
H1A—N1—H1B	106.0	C3—C4—C5	120.97 (16)
C1—N1—H1C	108.9	C3—C4—C7	120.11 (16)
H1A—N1—H1C	108.7	C5—C4—C7	118.91 (16)
H1B—N1—H1C	112.2	C6—C5—C4	119.56 (16)
C6—C1—C2	122.32 (16)	C6—C5—H5	120.2
C6—C1—N1	119.28 (15)	C4—C5—H5	120.2
C2—C1—N1	118.38 (15)	C1—C6—C5	118.79 (16)
C1—C2—C3	118.97 (16)	C1—C6—H6	120.6
C1—C2—H2	120.5	C5—C6—H6	120.6
C3—C2—H2	120.5	N2—C7—C4	178.9 (2)
C2—C3—C4	119.38 (16)		

C6—C1—C2—C3	−0.1 (3)	C7—C4—C5—C6	−179.23 (17)
N1—C1—C2—C3	−178.82 (15)	C2—C1—C6—C5	−0.1 (3)
C1—C2—C3—C4	0.5 (3)	N1—C1—C6—C5	178.66 (15)
C2—C3—C4—C5	−0.8 (3)	C4—C5—C6—C1	−0.2 (3)
C2—C3—C4—C7	179.08 (16)	C3—C4—C7—N2	−162 (11)
C3—C4—C5—C6	0.7 (3)	C5—C4—C7—N2	18 (12)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···Br1	0.88	2.47	3.3209 (16)	162
C2—H2···Br1^{i}	0.95	2.87	3.7316 (18)	151
C3—H3···N2^{ii}	0.95	2.62	3.466 (2)	149
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N1—H1*B*···Br1^{iv}	0.88	2.54	3.4174 (16)	175
N1—H1*C*···Br1^{v}	0.88	2.49	3.3400 (16)	162

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