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2,4,6-Trimethylanilinium bromide

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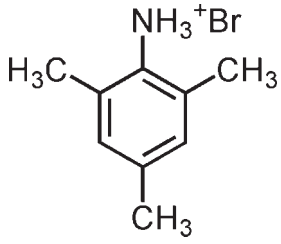
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.007 Å; R factor = 0.050; wR factor = 0.122; data-to-parameter ratio = 20.3.

In the title compound, $C_9H_{14}N^+\cdot Br^-$, an intramolecular N—H···Br interaction links the anion to the cation. In the crystal structure, intermolecular N—H···Br interactions link the molecules into a three-dimensional network.

Related literature

For related structures, see: Lemmerer & Billing (2007); Long *et al.* (2007).



Experimental

Crystal data

$C_9H_{14}N^+\cdot Br^-$ M_r = 216.11
Orthorhombic, *Pbca*
a = 10.399 (2) Å
b = 18.720 (4) Å
c = 10.282 (2) Å
V = 2001.6 (7) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 4.05 mm^{-1}
T = 294 K
0.2 × 0.2 × 0.2 mm

Data collection

Rigaku SCXmini diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.88, T_{max} = 1.000
18944 measured reflections
2292 independent reflections
1627 reflections with $I > 2\sigma(I)$
R_{int} = 0.099

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.122
S = 1.00
2292 reflections
113 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.37 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^{i}	0.84 (5)	2.58 (5)	3.376 (4)	157 (4)
N1—H1B···Br1	0.94 (6)	2.47 (6)	3.391 (4)	168 (4)
N1—H1C···Br1^{ii}	1.01 (5)	2.32 (5)	3.292 (4)	162 (4)

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

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: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2763).

References

Lemmerer, A. & Billing, D. G. (2007). *Acta Cryst.* E**63**, o929–o931.
Long, S., Siegler, M. & Li, T. (2007). *Acta Cryst.* E**63**, o3080.
Rigaku (2005). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
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supporting information

Acta Cryst. (2009). E**65**, o2376 [doi:10.1107/S1600536809034497]

2,4,6-Trimethylanilinium bromide

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S1. Comment

The crystal structure of the title compound is reported herein as part of a study of 2,4,6-trimethylanilinium halide salts. The other halide salts have been reported, previously (Lemmerer & Billing, 2007; Long *et al.*, 2007).

The asymmetric unit of the title compound, (Fig. 1), contains one 2,4,6 -trimethylbenzenaminium cation and one bromide anion. The intramolecular N-H···Br interaction (Table 1) links the anion to the cation.

In the crystal structure, intra- and intermolecular N-H···Br interactions (Table 1) link the molecules into a three-dimensional network.

S2. Experimental

For the preparation of the title compound, 2,4,6-trimethylaniline (3 mmol) was dissolved in ethanol (6 ml), and concentrated hydrobromic acid was added dropwise to dissolve the solid phase persisting in a mixture of bismuth tricbromide (3 mmol) and water (5 ml). The two solutions were then mixed and stirred for 15 min. The resulting precipitate was filtered off and dissolved in hydrobromic acid. Colorless crystals suitable for X-ray analysis were formed after several weeks by slow evaporation of the solvent at room temperature.

S3. Refinement

Atoms H1A, H1B and H1C (for NH_3) are located in a difference Fourier map and refined isotropically. The remaining H atoms were positioned geometrically with C-H = 0.93 and 0.96 Å for aromatic and methyl H atoms, respectively, and constrained to ride on their parent atoms, with $U_{iso}(H) = xU_{eq}(C)$, where x = 1.5 for methyl H and x = 1.2 for aromatic H atoms.

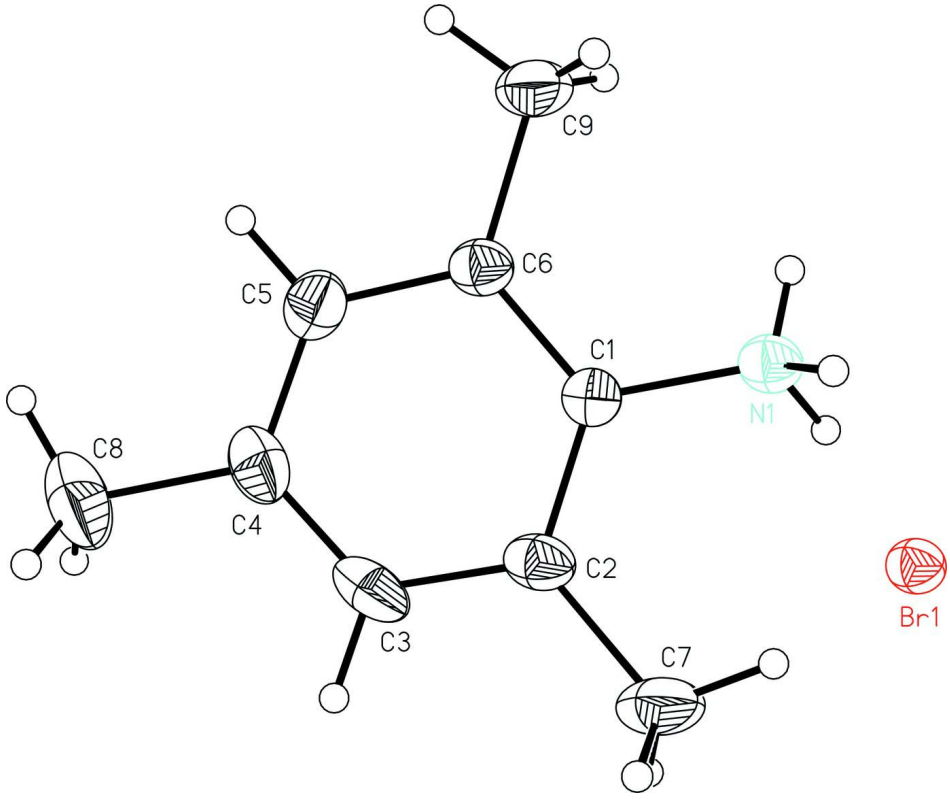

Figure 1

The molecular structure of the title molecule, with the atom-numbering scheme. Hydrogen bond is shown as dashed line.

2,4,6-Trimethylanilinium bromide

Crystal data

$C_9H_{14}N^+\cdot Br^-$
$M_r = 216.11$
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
$a = 10.399$ (2) Å
$b = 18.720$ (4) Å
$c = 10.282$ (2) Å
$V = 2001.6$ (7) Å^3
$Z = 8$
$F(000) = 880$
$D_x = 1.434$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1647 reflections
$\theta = 3.0$–$27.6°$
$\mu = 4.05$ mm^{-1}
$T = 294$ K
Prism, colorless
0.2 × 0.2 × 0.2 mm

Data collection

Rigaku SCXmini 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)
$T_{min} = 0.88$, $T_{max} = 1.000$
18944 measured reflections
2292 independent reflections
1627 reflections with $I > 2\sigma(I)$
$R_{int} = 0.099$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.0°$
$h = -13\rightarrow13$
$k = -24\rightarrow24$
$l = -13\rightarrow13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.122$
$S = 1.00$
2292 reflections
113 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0437P)^2 + 2.8318P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.37$ e Å^{-3}
$\Delta\rho_{min} = -0.41$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0097 (8)

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}
Br1	0.10008 (4)	0.00542 (2)	0.69604 (4)	0.0471 (2)
N1	0.1859 (4)	0.0634 (2)	0.3959 (4)	0.0420 (9)
H1A	0.113 (4)	0.058 (2)	0.361 (5)	0.053 (14)*
H1B	0.171 (5)	0.042 (3)	0.477 (6)	0.085 (18)*
H1C	0.249 (4)	0.033 (3)	0.346 (4)	0.052 (12)*
C1	0.2218 (4)	0.1388 (2)	0.4055 (4)	0.0364 (9)
C2	0.3168 (4)	0.1578 (2)	0.4945 (4)	0.0466 (11)
C3	0.3457 (4)	0.2299 (3)	0.5046 (4)	0.0563 (13)
H3A	0.4063	0.2440	0.5659	0.068*
C4	0.2896 (5)	0.2813 (3)	0.4289 (5)	0.0545 (12)
C5	0.2005 (4)	0.2595 (2)	0.3376 (5)	0.0523 (11)
H5A	0.1630	0.2935	0.2837	0.063*
C6	0.1650 (4)	0.1883 (2)	0.3237 (4)	0.0416 (10)
C7	0.3892 (4)	0.1028 (3)	0.5721 (5)	0.0747 (17)
H7A	0.3565	0.0561	0.5525	0.112*
H7B	0.3786	0.1123	0.6633	0.112*
H7C	0.4789	0.1050	0.5503	0.112*
C8	0.3257 (6)	0.3597 (3)	0.4419 (6)	0.0892 (19)
H8A	0.2766	0.3874	0.3811	0.134*
H8B	0.4157	0.3655	0.4240	0.134*
H8C	0.3077	0.3756	0.5287	0.134*
C9	0.0696 (5)	0.1680 (3)	0.2198 (5)	0.0656 (14)
H9A	0.0422	0.2102	0.1744	0.098*

H9B	−0.0035	0.1454	0.2592	0.098*
H9C	0.1093	0.1356	0.1596	0.098*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0419 (3)	0.0519 (3)	0.0473 (3)	−0.00391 (19)	−0.00179 (18)	0.0025 (2)
N1	0.035 (2)	0.048 (2)	0.043 (2)	−0.0016 (17)	−0.0005 (18)	0.0009 (18)
C1	0.033 (2)	0.041 (2)	0.035 (2)	−0.0008 (17)	0.0048 (17)	0.0004 (18)
C2	0.037 (2)	0.065 (3)	0.038 (2)	−0.011 (2)	−0.0016 (19)	0.005 (2)
C3	0.052 (3)	0.078 (3)	0.039 (3)	−0.027 (3)	0.001 (2)	−0.012 (2)
C4	0.061 (3)	0.050 (3)	0.053 (3)	−0.016 (2)	0.012 (2)	−0.006 (2)
C5	0.050 (2)	0.049 (3)	0.058 (3)	0.001 (2)	0.002 (2)	0.010 (2)
C6	0.035 (2)	0.050 (2)	0.040 (2)	−0.0016 (19)	−0.0005 (18)	0.0021 (19)
C7	0.050 (3)	0.100 (4)	0.074 (4)	−0.025 (3)	−0.024 (3)	0.037 (3)
C8	0.108 (5)	0.059 (3)	0.101 (5)	−0.031 (3)	0.005 (4)	−0.016 (3)
C9	0.060 (3)	0.078 (3)	0.058 (3)	−0.018 (3)	−0.024 (2)	0.018 (3)

Geometric parameters (Å, º)

N1—C1	1.464 (5)	C6—C1	1.383 (5)
N1—H1A	0.84 (5)	C6—C5	1.391 (6)
N1—H1B	0.94 (6)	C6—C9	1.507 (6)
N1—H1C	1.01 (5)	C7—H7A	0.9600
C2—C3	1.387 (6)	C7—H7B	0.9600
C2—C1	1.393 (5)	C7—H7C	0.9600
C2—C7	1.504 (6)	C8—H8A	0.9600
C3—H3A	0.9300	C8—H8B	0.9600
C4—C3	1.368 (6)	C8—H8C	0.9600
C4—C8	1.521 (7)	C9—H9A	0.9600
C5—C4	1.380 (6)	C9—H9B	0.9600
C5—H5A	0.9300	C9—H9C	0.9600
C1—N1—H1A	112 (3)	C1—C6—C5	117.8 (4)
C1—N1—H1B	114 (3)	C1—C6—C9	122.9 (4)
C1—N1—H1C	114 (3)	C5—C6—C9	119.3 (4)
H1A—N1—H1B	100 (4)	C2—C7—H7A	109.5
H1A—N1—H1C	108 (4)	C2—C7—H7B	109.5
H1B—N1—H1C	108 (4)	C2—C7—H7C	109.5
C2—C1—N1	118.1 (4)	H7A—C7—H7B	109.5
C6—C1—N1	119.7 (4)	H7A—C7—H7C	109.5
C6—C1—C2	122.1 (4)	H7B—C7—H7C	109.5
C1—C2—C7	122.0 (4)	C4—C8—H8A	109.5
C3—C2—C1	116.8 (4)	C4—C8—H8B	109.5
C3—C2—C7	121.1 (4)	C4—C8—H8C	109.5
C2—C3—H3A	118.3	H8A—C8—H8B	109.5
C4—C3—C2	123.3 (4)	H8A—C8—H8C	109.5
C4—C3—H3A	118.3	H8B—C8—H8C	109.5

C3—C4—C5	117.8 (4)	C6—C9—H9A	109.5
C3—C4—C8	121.5 (5)	C6—C9—H9B	109.5
C5—C4—C8	120.7 (5)	C6—C9—H9C	109.5
C4—C5—C6	122.0 (4)	H9A—C9—H9B	109.5
C4—C5—H5A	119.0	H9A—C9—H9C	109.5
C6—C5—H5A	119.0	H9B—C9—H9C	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···Br1^{i}	0.84 (5)	2.58 (5)	3.376 (4)	157 (4)
N1—H1*B*···Br1	0.94 (6)	2.47 (6)	3.391 (4)	168 (4)
N1—H1*C*···Br1^{ii}	1.01 (5)	2.32 (5)	3.292 (4)	162 (4)

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