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4-Bromo-2,6-dimethylanilinium bromide monohydrate

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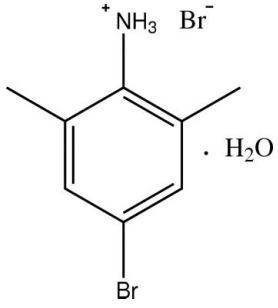
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.008 Å; R factor = 0.045; wR factor = 0.096; data-to-parameter ratio = 17.3.

In the title compound, $C_8H_{11}BrN^+\cdot Br^-\cdot H_2O$, a network of N—H···O, N—H···Br and O—H···Br hydrogen bonds helps to consolidate the crystal packing.

Related literature

For related literature, see: Hirao & Fukuhara (1998); MacDiamid *et al.* (1998); Wakayama (1998); Wang *et al.* (2002).



Experimental

Crystal data

$C_8H_{11}BrN^+\cdot Br^-\cdot H_2O$
M_r = 298.99
Monoclinic, $P2_1/n$
a = 7.1630 (14) Å
b = 18.649 (4) Å
c = 8.4770 (17) Å
β = 109.98 (3)°
V = 1064.2 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 7.58 mm^{-1}
T = 293 (2) K
0.40 × 0.20 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.152, T_{max} = 0.221
2077 measured reflections
2077 independent reflections
1346 reflections with $I > 2\sigma(I)$
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.096
S = 0.97
2077 reflections
120 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.52 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H2···Br2^{i}	0.84 (2)	2.56 (3)	3.380 (5)	169 (3)
O1—H1···Br2	0.84 (2)	2.50 (2)	3.322 (5)	174 (3)
N1—H1*C*···O1	0.89	1.87	2.754 (6)	170
N1—H1*B*···Br2^{ii}	0.89	2.53	3.388 (5)	163
N1—H1*A*···Br2^{iii}	0.89	2.71	3.523 (4)	153

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXL97*; software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LW2046).

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supporting information

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4-Bromo-2,6-dimethylanilinium bromide monohydrate

Jun-jie Tang, Jing Chen, Jin-tang Wang, Ai-hua Lu and You-sheng Chen

S1. Comment

aniline is a novel,useful intermediate,which can be used in various areas. Some derivatives of aniline have improving anticorrosion ability for metals (Wang *et al.*, 2002). Some show high efficacy as chemical sensors (MacDiamid *et al.*,1998) and catalitic oxidation (Hirao & Fukuhara, 1998). We report here the crystal structure of the title compound, (I). A network of intermolecular N—H···Br and O—H···Br hydrogen bonds helps to establish the crystal packing, Fig. 1 and Fig. 2.

S2. Experimental

The title compound is synthesized according to the literature(Wakayama *et al.*, 1998). Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution.

S3. Refinement

All H atoms bonded to the C atoms were placed geometrically at the distances of 0.93–0.96 Å and included in the refinement in riding motion approximation with U_{iso}(H) = 1.2 or 1.5U_{eq} of the carrier atom.

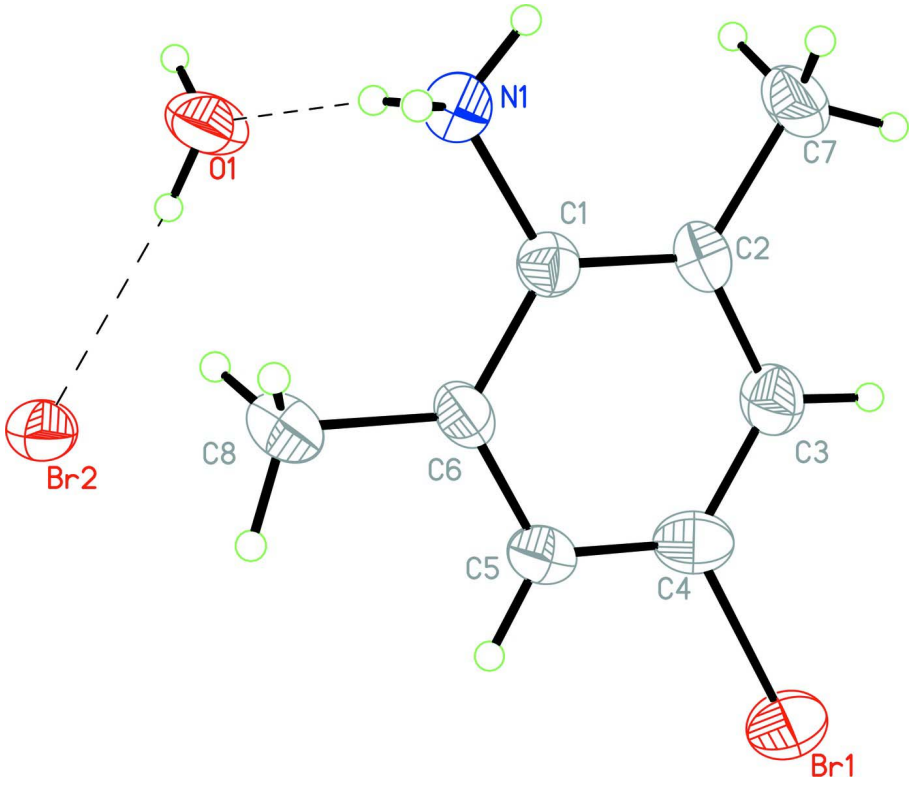

Figure 1

A view of the molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level The crystal structure of (I).

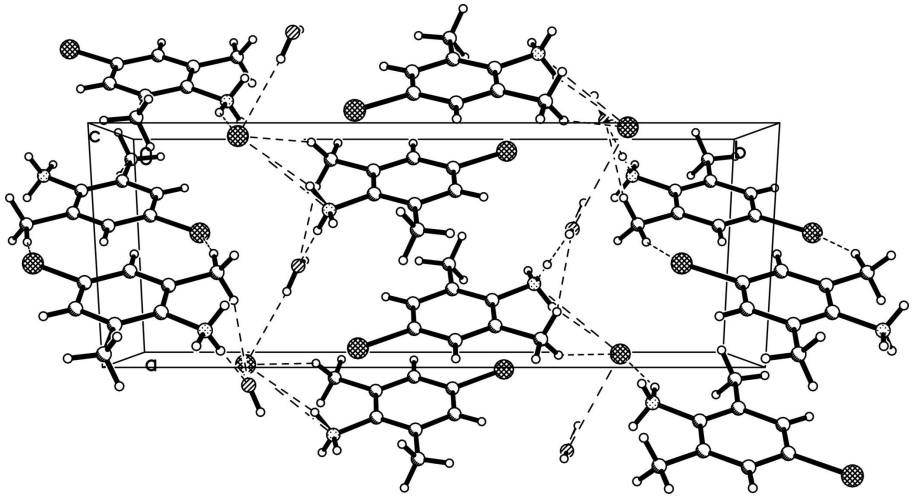

Figure 2

A packing diagram of the title molecular structure, with dash line indicates intermolecularintermolecular N—H···O, N—H···Br and O—H···Br hydrogen bonds

4-Bromo-2,6-dimethylanilinium bromide monohydrate

Crystal data

$C_8H_{11}BrN^+\cdot Br^-\cdot H_2O$
$M_r = 298.99$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 7.1630\ (14)$ Å
$b = 18.649\ (4)$ Å
$c = 8.4770\ (17)$ Å
$\beta = 109.98\ (3)°$
$V = 1064.2\ (4)$ Å^3
$Z = 4$
$F(000) = 584$
$D_x = 1.866$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9–13°$
$\mu = 7.58$ mm^{-1}
$T = 293$ K
Block, colorless
0.40 × 0.20 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.152$, $T_{max} = 0.221$
2077 measured reflections
2077 independent reflections
1346 reflections with $I > 2\sigma(I)$
$R_{int} = 0.000$
$\theta_{max} = 26.0°$, $\theta_{min} = 2.2°$
$h = -8\rightarrow8$
$k = 0\rightarrow22$
$l = 0\rightarrow10$
3 standard reflections every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.096$
$S = 0.97$
2077 reflections
120 parameters
2 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0437P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.42$ e Å^{-3}
$\Delta\rho_{min} = -0.52$ 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}
Br1	0.93285 (11)	0.38204 (3)	0.77315 (8)	0.0490 (2)
Br2	0.47862 (10)	0.69675 (3)	0.95867 (8)	0.0407 (2)
C1	0.7335 (8)	0.5937 (3)	0.4804 (7)	0.0276 (13)

C2	0.6858 (8)	0.5297 (3)	0.3931 (7)	0.0310 (13)
C3	0.7483 (8)	0.4664 (3)	0.4828 (7)	0.0346 (14)
H3	0.7195	0.4224	0.4282	0.041*
C4	0.8528 (9)	0.4691 (3)	0.6521 (7)	0.0368 (15)
C5	0.9007 (8)	0.5334 (3)	0.7373 (7)	0.0330 (14)
H5	0.9728	0.5338	0.8517	0.040*
C6	0.8412 (8)	0.5966 (3)	0.6519 (6)	0.0298 (13)
C7	0.5756 (9)	0.5250 (3)	0.2060 (6)	0.0410 (16)
H7A	0.4469	0.5466	0.1799	0.062*
H7B	0.5605	0.4755	0.1723	0.062*
H7C	0.6494	0.5497	0.1472	0.062*
C8	0.8945 (9)	0.6665 (3)	0.7470 (7)	0.0420 (16)
H8A	0.7752	0.6910	0.7436	0.063*
H8B	0.9682	0.6960	0.6964	0.063*
H8C	0.9739	0.6569	0.8616	0.063*
N1	0.6621 (7)	0.6625 (2)	0.3935 (5)	0.0336 (12)
H1A	0.6039	0.6545	0.2841	0.050*
H1B	0.7645	0.6921	0.4099	0.050*
H1C	0.5749	0.6822	0.4343	0.050*
O1	0.4192 (8)	0.7175 (3)	0.5552 (6)	0.0494 (12)
H1	0.442 (3)	0.710 (4)	0.657 (3)	0.05 (2)*
H2	0.306 (3)	0.736 (4)	0.518 (3)	0.09 (3)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0650 (5)	0.0365 (4)	0.0427 (4)	0.0084 (4)	0.0147 (3)	0.0129 (3)
Br2	0.0445 (4)	0.0422 (4)	0.0337 (3)	0.0045 (3)	0.0113 (3)	0.0053 (3)
C1	0.030 (3)	0.028 (3)	0.027 (3)	−0.001 (2)	0.012 (3)	0.000 (2)
C2	0.035 (3)	0.033 (3)	0.027 (3)	−0.001 (3)	0.014 (3)	−0.006 (3)
C3	0.040 (4)	0.027 (3)	0.034 (3)	−0.005 (3)	0.011 (3)	−0.005 (3)
C4	0.038 (4)	0.039 (4)	0.033 (3)	−0.001 (3)	0.011 (3)	0.010 (3)
C5	0.037 (4)	0.034 (3)	0.024 (3)	0.002 (3)	0.006 (3)	0.002 (3)
C6	0.031 (3)	0.034 (3)	0.023 (3)	−0.001 (3)	0.008 (3)	−0.006 (2)
C7	0.054 (4)	0.038 (4)	0.025 (3)	−0.006 (3)	0.006 (3)	−0.009 (3)
C8	0.045 (4)	0.048 (4)	0.027 (3)	0.003 (3)	0.003 (3)	−0.004 (3)
N1	0.037 (3)	0.032 (3)	0.033 (3)	0.002 (2)	0.012 (2)	0.000 (2)
O1	0.052 (3)	0.069 (4)	0.026 (3)	0.019 (3)	0.012 (2)	0.005 (2)

Geometric parameters (Å, º)

Br1—C4	1.901 (6)	C7—H7A	0.9600
C1—C2	1.384 (7)	C7—H7B	0.9600
C1—C6	1.395 (7)	C7—H7C	0.9600
C1—N1	1.481 (6)	C8—H8A	0.9600
C2—C3	1.393 (7)	C8—H8B	0.9600
C2—C7	1.513 (7)	C8—H8C	0.9600
C3—C4	1.374 (7)	N1—H1A	0.8900

C3—H3	0.9300	N1—H1B	0.8900
C4—C5	1.381 (8)	N1—H1C	0.8900
C5—C6	1.373 (7)	O1—H1	0.84 (2)
C5—H5	0.9300	O1—H2	0.84 (2)
C6—C8	1.511 (7)		

C2—C1—C6	122.6 (5)	C2—C7—H7B	109.5
C2—C1—N1	120.0 (5)	H7A—C7—H7B	109.5
C6—C1—N1	117.3 (5)	C2—C7—H7C	109.5
C1—C2—C3	117.6 (5)	H7A—C7—H7C	109.5
C1—C2—C7	123.8 (5)	H7B—C7—H7C	109.5
C3—C2—C7	118.5 (5)	C6—C8—H8A	109.5
C4—C3—C2	119.9 (5)	C6—C8—H8B	109.5
C4—C3—H3	120.1	H8A—C8—H8B	109.5
C2—C3—H3	120.1	C6—C8—H8C	109.5
C3—C4—C5	121.8 (5)	H8A—C8—H8C	109.5
C3—C4—Br1	119.3 (5)	H8B—C8—H8C	109.5
C5—C4—Br1	118.9 (4)	C1—N1—H1A	109.5
C6—C5—C4	119.5 (5)	C1—N1—H1B	109.5
C6—C5—H5	120.2	H1A—N1—H1B	109.5
C4—C5—H5	120.2	C1—N1—H1C	109.5
C5—C6—C1	118.5 (5)	H1A—N1—H1C	109.5
C5—C6—C8	118.9 (5)	H1B—N1—H1C	109.5
C1—C6—C8	122.6 (5)	H1—O1—H2	106 (3)
C2—C7—H7A	109.5		

C6—C1—C2—C3	0.4 (8)	C3—C4—C5—C6	0.7 (9)
N1—C1—C2—C3	−176.9 (5)	Br1—C4—C5—C6	−179.0 (4)
C6—C1—C2—C7	−177.7 (5)	C4—C5—C6—C1	0.0 (8)
N1—C1—C2—C7	5.0 (8)	C4—C5—C6—C8	179.9 (5)
C1—C2—C3—C4	0.2 (9)	C2—C1—C6—C5	−0.6 (8)
C7—C2—C3—C4	178.5 (5)	N1—C1—C6—C5	176.8 (5)
C2—C3—C4—C5	−0.8 (9)	C2—C1—C6—C8	179.5 (6)
C2—C3—C4—Br1	178.9 (4)	N1—C1—C6—C8	−3.1 (8)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H2···Br2^{i}	0.84 (2)	2.56 (3)	3.380 (5)	169 (3)
O1—H1···Br2	0.84 (2)	2.50 (2)	3.322 (5)	174 (3)
N1—H1*C*···O1	0.89	1.87	2.754 (6)	170
N1—H1*B*···Br2^{ii}	0.89	2.53	3.388 (5)	163
N1—H1*A*···Br2^{iii}	0.89	2.71	3.523 (4)	153

Symmetry codes: (i) *x*−1/2, −*y*+3/2, *z*−1/2; (ii) *x*+1/2, −*y*+3/2, *z*−1/2; (iii) *x*, *y*, *z*−1.