Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Sodium *N*-bromobenzenesulfonamidate sesquihydrate

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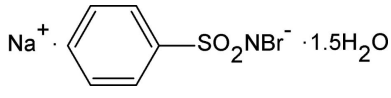
Received 14 May 2007; accepted 23 May 2007

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.011 Å; R factor = 0.034; wR factor = 0.088; data-to-parameter ratio = 14.2.

In the title compound, $Na^+\cdot C_6H_5BrNO_2S^-\cdot 1.5H_2O$, there is no interaction between the N atom and the Na^+ cation, and the Na cation exhibits octahedral coordination by three O atoms from water molecules and by three sulfonyl O atoms of three different *N*-bromobenzenesulfonamide anions. The S—N distance of 1.578 (4) Å is consistent with an S=N double bond, similar to the distance of 1.582 (5) Å observed for the corresponding *N*-chloro compound. A two-dimensional polymeric layer runs parallel to the *ab* plane. The water molecules participate in O—H···N hydrogen bonds.

Related literature

For related literature, see: George *et al.* (2000); Gowda & Shetty (2004); Gowda & Usha (2003); Gowda *et al.* (2005); Gowda, Foro *et al.* (2007); Gowda, Jyothi *et al.* (2007); Gowda, Kozisek *et al.* (2007); Gowda, Savitha *et al.* (2007); Gowda, Srilatha *et al.* (2007); Gowda *et al.* (2003); Olmstead & Power (1986); Usha & Gowda (2006).



Experimental

Crystal data

$Na^+\cdot C_6H_5BrNO_2S^-\cdot 1.5H_2O$
M_r = 285.10
Monoclinic, $C2$
a = 10.521 (3) Å
b = 6.760 (2) Å
c = 14.853 (4) Å
β = 103.97 (2)°
V = 1025.1 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 4.24 mm^{-1}
T = 293 (2) K
0.50 × 0.50 × 0.10 mm

Data collection

Oxford Diffraction Xcalibur diffractometer
Absorption correction: analytical (Clark & Reid, 1995)
T_{min} = 0.155, T_{max} = 0.652
2957 measured reflections
1880 independent reflections
1704 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.088
S = 1.05
1880 reflections
132 parameters
5 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.44 e Å^{-3}
Absolute structure: Flack (1983), 778 Friedel pairs
Flack parameter: −0.018 (12)

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

D—H···A	D—H	H···A	D···A	D—H···A
O3W—H31···N1^{i}	0.85 (4)	2.12 (4)	2.943 (5)	167 (5)
O4W—H42···N1^{ii}	0.85 (4)	2.07 (2)	2.878 (5)	161 (5)

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2003); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2003); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 2002); software used to prepare material for publication: *SHELXL97*, *PLATON* (Spek, 2003) and *WinGX* (Farrugia, 1999).

BTG thanks the Alexander von Humboldt Foundation, Bonn, Germany, for extensions to his research fellowship. JK and MT thank the Grant Agency of the Slovak Republic (grant No. 1/2449/05).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2177).

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 doi:10.1107/S1600536807025184

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supplementary materials

Acta Cryst. (2007). E**63**, m1739-m1740 [**doi:10.1107/S1600536807025184**]

Sodium *N*-bromobenzenesulfonamidate sesquihydrate

B. T. Gowda, K. M. Usha, J. Kozísek, M. Tokarcík and H. Fuess

Comment

The chemistry of arylsulfonamides and their N-halo compounds is of interest as they show diverse physical, chemical and biological properties. Thus N-halo arylsulfonamides are of interest in synthetic, mechanistic, analytical and biological chemistry (Gowda *et al.*, 2005; Gowda & Shetty, 2004; Usha & Gowda, 2006). In the present work, the structure of sodium *N*-bromo- benzenesulfonamde (NaNBBSA) has been determined to explore the effects substitution on the solid state structures of sulfonamides and N-halo arylsulfonamides (Gowda *et al.*, 2003; Gowda, Foro *et al.*, 2007; Gowda, Jyothi *et al.*, 2007; Gowda, Kozisek *et al.*, 2007; Gowda, Savitha *et al.*, 2007; Gowda, Srilatha *et al.*, 2007).

The structure of NaNBBSA (Fig. 1) resembles those of sodium *N*-chloro- arylsulfonamdes (George *et al.*, 2000; Gowda, Foro *et al.*, 2007; Gowda, Jyothi *et al.*, 2007; Gowda, Savitha *et al.*, 2007; Gowda, Srilatha *et al.*, 2007; Olmstead & Power, 1986). NaNBBSA is the parent or unsubstituted *N*-bromo-arylsulphonamide. The structure confirms that there is no interaction between the nitrogen and sodium atoms. The sodium ion exhibits octahedral coordination by three O atoms from water molecules and by three sulfonyl O atoms of three different *N*-bromobenzenesulfonamide anions. The S—N distance of 1.578 (4)Å is consistent with a S—N double bond, similar to the distance of 1.582 (5)Å observed with the corresponding *N*-chloro compound. Packing diagram of the title compound involving hydrogen bonds is shown in Fig.2.

Experimental

The title compound was prepared according to the literature method (Gowda & Usha, 2003). The purity of the compound was checked by determining its melting point. It was characterized by recording its infrared and NMR spectra (Gowda & Usha, 2003). Single crystals of the title compound were obtained from its aqueous solution and used for X-ray diffraction studies at room temperature.

Refinement

H atoms of the benzene ring were positioned geometrically and refined using a riding model with C—H = 0.93 Å and with U_{iso}(H) = 1.2 U_{eq}(C). H atoms of the water molecules (O3w, O4w) were visible in difference map and were subsequently treated as riding with O—H bond length restrained to 0.85 (1) Å and mutual distance of H atoms 1.35 (1) Å. No restraints were applied for non-hydrogen atoms.

Figures

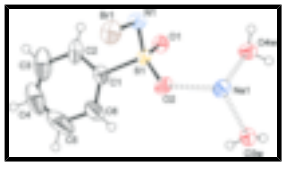

Fig. 1. View of the asymmetric unit showing the atom labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radii.

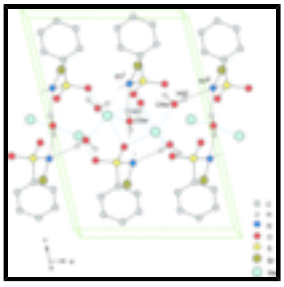

Fig. 2. Partial packing view of the title compound showing hydrogen bonds network. Hydrogen atoms bonded to benzene C atoms have been omitted.. Symmetry codes: (i) −*x* + 3/2, *y* − 1/2,-*z* + 1; (ii) −*x* + 2, *y*, −*z* + 1].

Sodium *N*-bromobenzenesulfonamidate sesquihydrate

Crystal data

$Na^+ \cdot C_6H_5BrNO_2S^- \cdot 1.5H_2O$	F_{000} = 564
M_r = 285.10	D_x = 1.847 Mg m^{-3}
Monoclinic, *C*2	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: C 2y	Cell parameters from 2235 reflections
a = 10.521 (3) Å	θ = 2.5–26.4º
b = 6.760 (2) Å	μ = 4.24 mm^{-1}
c = 14.853 (4) Å	*T* = 293 (2) K
β = 103.97 (2)º	Plate, yellow
V = 1025.1 (5) Å^3	0.50 × 0.50 × 0.10 mm
Z = 4	

Data collection

Oxford Diffraction Xcalibur diffractometer	1704 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.032
T = 293(2) K	θ_{max} = 26.0º
ω and φ scans	θ_{min} = 4.6º
Absorption correction: analytical (Clark & Reid, 1995)	*h* = −12→9
T_{min} = 0.155, T_{max} = 0.652	*k* = −8→8
2957 measured reflections	*l* = −18→18
1880 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)]$ = 0.034	$w = 1/[\sigma^2(F_o^2) + (0.0547P)^2 + 0.3614P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.088	$(\Delta/\sigma)_{max}$ = 0.002

S = 1.05	$\Delta\rho_{max}$ = 0.32 e Å^{-3}
1880 reflections	$\Delta\rho_{min}$ = −0.44 e Å^{-3}
132 parameters	Extinction correction: none
5 restraints	Absolute structure: Flack (1983), 778 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: −0.018 (12)
Secondary atom site location: difference Fourier map	

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 $F^2 > 2\text{sigma}(F^2)$ 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}
C1	0.8171 (4)	0.2054 (7)	0.2212 (3)	0.0356 (9)
C2	0.9260 (6)	0.1722 (13)	0.1867 (3)	0.0563 (13)
H2	1.0085	0.2091	0.2213	0.068*
C3	0.9128 (10)	0.0846 (11)	0.1011 (5)	0.087 (3)
H3	0.9862	0.0645	0.0777	0.105*
C4	0.7953 (12)	0.0290 (12)	0.0518 (5)	0.092 (3)
H4	0.7869	−0.0257	−0.0068	0.111*
C5	0.6873 (11)	0.0508 (15)	0.0859 (6)	0.101 (3)
H5	0.607	0.0026	0.0524	0.122*
C6	0.6962 (6)	0.1473 (11)	0.1726 (4)	0.0657 (18)
H6	0.6222	0.1695	0.1951	0.079*
S1	0.84086 (9)	0.31275 (15)	0.33360 (7)	0.0271 (2)
O1	0.9150 (3)	0.1703 (6)	0.39966 (19)	0.0350 (6)
O2	0.7112 (3)	0.3566 (6)	0.3454 (2)	0.0437 (8)
O3W	0.5	0.2619 (7)	0.5	0.0364 (10)
H31	0.505 (5)	0.178 (6)	0.543 (3)	0.044*
O4W	0.8026 (3)	0.3538 (5)	0.5824 (2)	0.0415 (8)
H41	0.758 (4)	0.348 (9)	0.623 (2)	0.05*
H42	0.870 (3)	0.421 (8)	0.607 (3)	0.05*
N1	0.9361 (4)	0.4949 (5)	0.3388 (3)	0.0347 (8)
Br1	0.86056 (5)	0.68675 (6)	0.24930 (3)	0.05208 (18)
Na1	0.63738 (16)	0.5271 (3)	0.46276 (12)	0.0365 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.053 (2)	0.024 (2)	0.0294 (19)	0.002 (2)	0.0084 (17)	0.0042 (19)
C2	0.081 (3)	0.059 (3)	0.038 (2)	0.010 (4)	0.031 (2)	0.002 (3)
C3	0.154 (8)	0.060 (4)	0.062 (4)	0.024 (5)	0.053 (5)	−0.006 (4)
C4	0.169 (9)	0.057 (4)	0.046 (4)	−0.009 (6)	0.019 (5)	−0.017 (3)
C5	0.115 (7)	0.094 (6)	0.071 (5)	−0.037 (5)	−0.023 (5)	−0.020 (5)
C6	0.072 (3)	0.078 (5)	0.041 (3)	−0.022 (3)	0.001 (2)	−0.009 (3)
S1	0.0296 (4)	0.0250 (5)	0.0281 (5)	0.0014 (4)	0.0094 (4)	0.0013 (4)
O1	0.0403 (14)	0.0319 (15)	0.0323 (14)	0.0045 (15)	0.0078 (11)	0.0090 (15)
O2	0.0321 (15)	0.053 (2)	0.0496 (19)	0.0076 (15)	0.0175 (13)	−0.0002 (17)
O3W	0.048 (2)	0.027 (2)	0.035 (2)	0	0.011 (2)	0
O4W	0.0332 (14)	0.042 (2)	0.048 (2)	0.0001 (14)	0.0090 (14)	−0.0032 (15)
N1	0.0377 (18)	0.0267 (18)	0.038 (2)	−0.0028 (15)	0.0053 (15)	0.0008 (15)
Br1	0.0708 (3)	0.0315 (2)	0.0569 (3)	0.0070 (3)	0.0214 (2)	0.0136 (3)
Na1	0.0365 (8)	0.0316 (9)	0.0442 (10)	0.0041 (7)	0.0156 (7)	−0.0005 (7)

Geometric parameters (Å, °)

C1—C6	1.360 (7)	S1—N1	1.578 (4)
C1—C2	1.382 (7)	O1—Na1^{i}	2.441 (3)
C1—S1	1.781 (4)	O1—Na1^{ii}	2.496 (3)
C2—C3	1.379 (9)	O2—Na1	2.372 (4)
C2—H2	0.93	O3W—Na1	2.448 (4)
C3—C4	1.330 (12)	O3W—H31	0.85 (4)
C3—H3	0.93	O4W—Na1^{i}	2.436 (4)
C4—C5	1.359 (13)	O4W—Na1	2.461 (4)
C4—H4	0.93	O4W—H41	0.85 (4)
C5—C6	1.427 (12)	O4W—H42	0.85 (4)
C5—H5	0.93	N1—Br1	1.890 (4)
C6—H6	0.93	Na1—Na1^{iii}	3.335 (3)
S1—O2	1.447 (3)	Na1—Na1^{iv}	4.123 (2)
S1—O1	1.459 (3)		
C6—C1—C2	120.6 (5)	S1—N1—Br1	110.3 (2)
C6—C1—S1	121.1 (4)	O2—Na1—O4W^{iv}	94.90 (14)
C2—C1—S1	118.2 (4)	O2—Na1—O1^{iv}	171.17 (14)
C3—C2—C1	120.3 (7)	O4W^{iv}—Na1—O1^{iv}	89.82 (14)
C3—C2—H2	119.9	O2—Na1—O3W	97.26 (13)
C1—C2—H2	119.9	O4W^{iv}—Na1—O3W	158.86 (12)
C4—C3—C2	120.2 (8)	O1^{iv}—Na1—O3W	80.58 (12)
C4—C3—H3	119.9	O2—Na1—O4W	90.00 (13)
C2—C3—H3	119.9	O4W^{iv}—Na1—O4W	116.45 (11)
C3—C4—C5	120.9 (7)	O1^{iv}—Na1—O4W	81.21 (12)
C3—C4—H4	119.5	O3W—Na1—O4W	80.85 (11)

C5—C4—H4	119.5	O2—Na1—O1^{v}	110.86 (13)
C4—C5—C6	120.5 (7)	O4W^{iv}—Na1—O1^{v}	80.02 (13)
C4—C5—H5	119.8	O1^{iv}—Na1—O1^{v}	77.29 (13)
C6—C5—H5	119.8	O3W—Na1—O1^{v}	79.51 (11)
C1—C6—C5	117.4 (7)	O4W—Na1—O1^{v}	152.89 (13)
C1—C6—H6	121.3	O2—Na1—Na1^{iii}	135.06 (11)
C5—C6—H6	121.3	O4W^{iv}—Na1—Na1^{iii}	113.23 (9)
O2—S1—O1	114.88 (19)	O1^{iv}—Na1—Na1^{iii}	48.20 (8)
O2—S1—N1	116.1 (2)	O3W—Na1—Na1^{iii}	47.07 (8)
O1—S1—N1	104.6 (2)	O4W—Na1—Na1^{iii}	106.15 (11)
O2—S1—C1	105.9 (2)	O1^{v}—Na1—Na1^{iii}	46.82 (8)
O1—S1—C1	107.0 (2)	O2—Na1—Na1^{iv}	109.05 (11)
N1—S1—C1	107.9 (2)	O4W^{iv}—Na1—Na1^{iv}	32.85 (8)
S1—O1—Na1^{i}	129.23 (17)	O1^{iv}—Na1—Na1^{iv}	71.44 (10)
S1—O1—Na1^{ii}	144.00 (18)	O3W—Na1—Na1^{iv}	150.70 (10)
Na1^{i}—O1—Na1^{ii}	84.98 (12)	O4W—Na1—Na1^{iv}	86.36 (11)
S1—O2—Na1	132.4 (2)	O1^{v}—Na1—Na1^{iv}	102.05 (10)
Na1^{iii}—O3W—Na1	85.86 (16)	Na1^{iii}—Na1—Na1^{iv}	113.52 (6)
Na1^{iii}—O3W—H31	105 (4)	O2—Na1—Na1^{i}	61.96 (9)
Na1—O3W—H31	137 (4)	O4W^{iv}—Na1—Na1^{i}	130.31 (11)
Na1^{i}—O4W—Na1	114.68 (14)	O1^{iv}—Na1—Na1^{i}	109.38 (11)
Na1^{i}—O4W—H41	112 (4)	O3W—Na1—Na1^{i}	70.83 (7)
Na1—O4W—H41	97 (4)	O4W—Na1—Na1^{i}	32.46 (9)
Na1^{i}—O4W—H42	111 (4)	O1^{v}—Na1—Na1^{i}	147.63 (11)
Na1—O4W—H42	116 (4)	Na1^{iv}—Na1—Na1^{i}	110.13 (8)
H41—O4W—H42	106 (4)		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3W—H31···N1^{i}	0.85 (4)	2.12 (4)	2.943 (5)	167 (5)
O4W—H42···N1^{vi}	0.85 (4)	2.07 (2)	2.878 (5)	161 (5)

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

Fig. 1

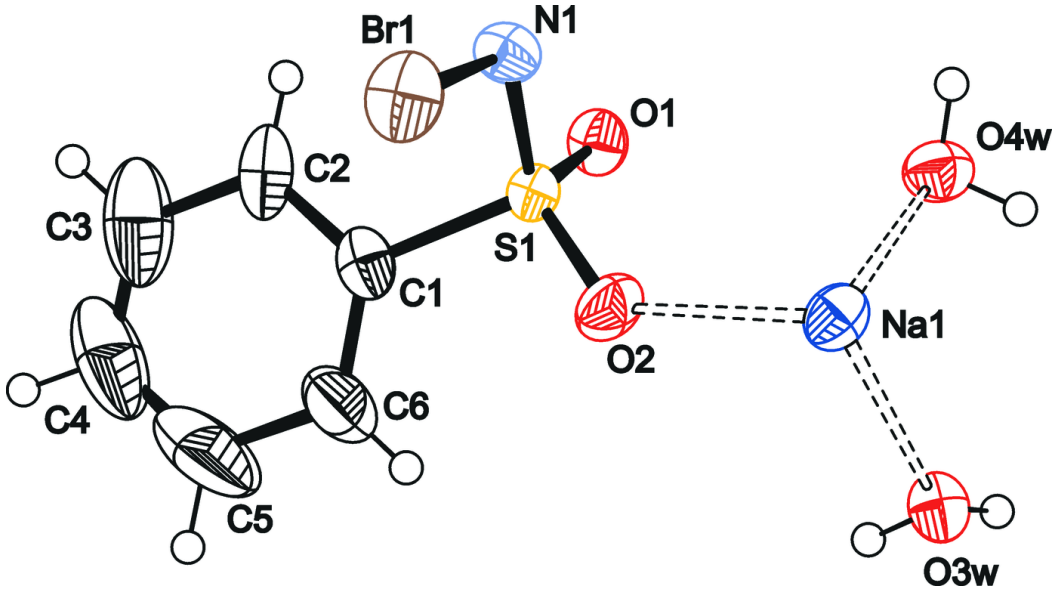

Fig. 2