Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Sodium *N*-bromo-4-chloro-2-methylbenzenesulfonamidate sesquihydrate

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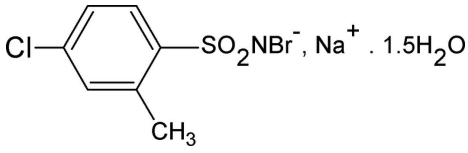
Received 2 June 2007; accepted 14 June 2007

Key indicators: single-crystal X-ray study; T = 300 K; mean σ(C–C) = 0.007 Å; R factor = 0.070; wR factor = 0.091; data-to-parameter ratio = 14.4.

The title compound, $Na^+\cdot C_7H_6BrClNO_2S^-\cdot 1.5H_2O$, crystallizes with two cations, two anions and three water molecules in the asymmetric unit; its structure is similar to that of sodium *N*-bromobenzenesulfonamidate and sodium *N*-bromo-4-chlorobenzenesulfonamidate. The sodium ion shows octahedral coordination by three O atoms of water molecules and by three sulfonyl O atoms of three different *N*-bromo-4-chloro-2-methylbenzenesulfonamide anions. There is no interaction between the N atoms and sodium ions in the structure. The S—N distance of 1.584 (4) Å is consistent with an S=N double bond. The crystal structure is stabilized by O—H···N hydrogen bonds.

Related literature

For related literature, see: George *et al.* (2000); Gowda & Usha (2003); Gowda, Jyothi *et al.* (2007); Gowda, Kožíšek *et al.* (2007); Gowda, Usha *et al.* (2007); Gowda, Srilatha *et al.* (2007); Usha & Gowda (2006).



Experimental

Crystal data

$Na^+\cdot C_7H_6BrClNO_2S^-\cdot 1.5H_2O$
M_r = 333.56
Monoclinic, $C2/c$
a = 11.055 (2) Å
b = 6.7804 (14) Å
c = 30.727 (6) Å
β = 98.84 (3)°
V = 2275.9 (8) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 4.06 mm^{-1}
T = 300 (2) K
0.58 × 0.48 × 0.10 mm

Data collection

Oxford Diffraction Xcalibur CCD diffractometer
Absorption correction: analytical (*CrysAlis RED*; Oxford Diffraction, 2003) using a multifaceted crystal model (Clark & Reid, 1995)
T_{min} = 0.196, T_{max} = 0.632
6929 measured reflections
2231 independent reflections
2066 reflections with $I > 2\sigma(I)$
R_{int} = 0.067

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.070
$wR(F^2)$ = 0.091
S = 1.21
2049 reflections
142 parameters
4 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.68 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3W—H31···N1^{i}	0.82	2.16	2.927 (5)	156
O4W—H41···N1^{ii}	0.83	2.19	3.010 (5)	171

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

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 of 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: DN2194).

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 doi:10.1107/S1600536807029248

supplementary materials

Acta Cryst. (2007). E**63**, m1957 [**doi:10.1107/S1600536807029248**]

Sodium *N*-bromo-4-chloro-2-methylbenzenesulfonamidate sesquihydrate

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Comment

The chemistry of *N*-halo-arylsulfonamides is of interest due to their diverse characteristics (Usha & Gowda, 2006). In the present work, the structure of sodium *N*-bromo-2-methyl-4-chloro-benzenesulfonamidate (NaNB2M4CBSA) has been determined to explore the substituent effects on the solid state structures of arylsulfonamides and N-halo arylsulfonamidates (Gowda *et al.*, 2007*a*, *b*, *c*, *d*). The structure of NaNB2M4CBSA (Fig. 1) resembles those of sodium *N*-chloro-2-methyl-4-chloro-benzenesulfonamidate (NaNC2M4CBSA)(Gowda, Srilatha *et al.*, 2007), sodium *N*-bromo-benzenesulfonamidate (NaNBBSA) (Gowda, Usha *et al.*, 2007) and sodium *N*-bromo-4-chloro-benzenesulfonamidate (NaNB4CBSA)(Gowda, Kožíšek *et al.*, 2007) and other sodium *N*-chloro-arylsulfonamidates (George *et al.*, 2000; Gowda, Jyothi *et al.*, 2007). NaNB2M4CBSA crystallizes with two cations, two anions and three water molecules in the asymmetric unit. The sodium ion shows octahedral coordination by three O atoms of water molecules and by three sulfonyl O atoms of three different *N*-bromo-2-methyl- 4-chloro-benzenesulfonamide anions. There is no interaction between the nitrogen and sodium ions in the molecule. The S—N distances of N1—S1, 1.584 (4)Å is consistent with a S—N double bond and in agreement with those observed with NaNBBSA, NaNB4CBSA and NaNC2M4CBSA. O—H···N hydrogen bonding interactions result in the formation of a polymeric layer structure running parallel to the (0 0 1) plane (Table 1, 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

All H atoms attached to C and O atoms were positioned geometrically and treated as riding with C—H = 0.93Å (aromatic) and C—H = 0.96Å (methyl). H atoms of water molecules were refined using restraint on O—H bond length 0.85 (3)Å and restraint on their mutual distance 1.45 (4) Å. All H atoms have U_{iso}(H) = 1.2 U_{eq}(C).

Regarding the discrepancy with reflection numbers, the number of reflections used in the refinement (2049) differs from the total number of reflections (2231) because of applying resolution shel 0.82 to 3.5 Å.

Figures

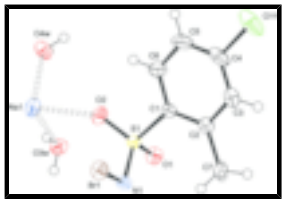

Fig. 1. *ORTEP* view of the title compound showing the atom labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. Na O interactions are shown as dashed lines. H atoms are represented as small spheres of arbitrary radii.

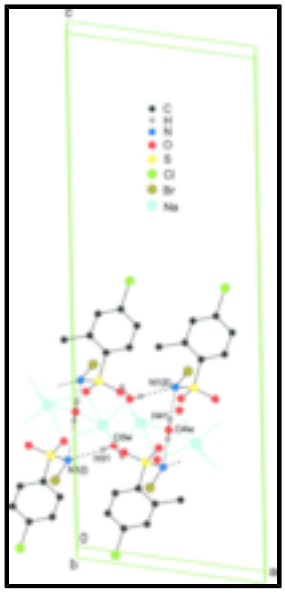

Fig. 2. Partial packing diagram of (I) showing hydrogen bonds O3w—H31···N1(i) and O4w—-H41···N1(ii). Symmetry codes: (i) −*x*, *y*, −*z* + 1/2; (ii) *x* + 1/2, *y* − 1/2, *z*. H atoms not involved in hydrogen bonds have been omitted for clarity.

Sodium *N*-bromo-4-chloro-2-methylbenzenesulfonamidate sesquihydrate

Crystal data

$Na^+ \cdot C_7H_6BrClNO_2S^- \cdot 1.5H_2O$	F_{000} = 1320
M_r = 333.56	D_x = 1.929 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 2337 reflections
a = 11.055 (2) Å	θ = 2.5–26.4º
b = 6.7804 (14) Å	μ = 4.06 mm^{-1}
c = 30.727 (6) Å	*T* = 300 (2) K
β = 98.84 (3)º	Block, pale yellow
V = 2275.9 (8) Å^3	0.58 × 0.48 × 0.10 mm
Z = 8	

Data collection

Oxford Diffraction Xcalibur CCD diffractometer	2066 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.067
T = 300(2) K	θ_{max} = 26.4º

Rotation method data acquisition using ωand φ scans	θ_{min} = 4.6º
Absorption correction: analytical (CrysAlis RED; Oxford Diffraction, 2003) using a multifaceted crystal model (Clark & Reid, 1995)	h = −13→13
T_{min} = 0.196, T_{max} = 0.632	k = −5→8
6929 measured reflections	l = −38→38
2231 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.070	H-atom parameters constrained
$wR(F^2)$ = 0.091	$w = 1/[\sigma^2(F_o^2) + (0.0001P)^2 + 21.0707P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.21	$(\Delta/\sigma)_{max} < 0.001$
2049 reflections	$\Delta\rho_{max}$ = 0.46 e Å^{-3}
142 parameters	$\Delta\rho_{min}$ = −0.68 e Å^{-3}
4 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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	0.6736 (4)	−0.3263 (7)	0.38143 (14)	0.0199 (10)
C2	0.5880 (4)	−0.3838 (7)	0.40799 (15)	0.0236 (11)
C3	0.6320 (5)	−0.4536 (8)	0.45013 (17)	0.0331 (13)
H3	0.5773	−0.4954	0.4684	0.040*
C4	0.7558 (6)	−0.4611 (8)	0.46494 (18)	0.0371 (14)
C5	0.8403 (5)	−0.4086 (9)	0.43893 (19)	0.0399 (14)
H5	0.9237	−0.4177	0.4493	0.048*
C6	0.7983 (5)	−0.3415 (9)	0.39678 (18)	0.0328 (12)
H6	0.8543	−0.3060	0.3785	0.039*
C7	0.4501 (4)	−0.3698 (8)	0.39352 (17)	0.0286 (12)

H7A	0.4086	−0.4212	0.4163	0.043*
H7B	0.4274	−0.4450	0.3671	0.043*
H7C	0.4274	−0.2343	0.3881	0.043*
N1	0.5374 (4)	−0.0524 (6)	0.32821 (13)	0.0242 (9)
O1	0.5613 (3)	−0.3788 (5)	0.30020 (11)	0.0292 (8)
O2	0.7460 (3)	−0.1762 (6)	0.31310 (12)	0.0339 (9)
O3W	0.7076 (3)	−0.1898 (6)	0.20927 (12)	0.0341 (9)
H31	0.6490	−0.1190	0.2001	0.051*
H32	0.7490	−0.2170	0.1886	0.051*
O4W	1.0000	−0.2798 (8)	0.2500	0.0337 (12)
H41	1.0070	−0.3652	0.2695	0.050*
S1	0.63097 (10)	−0.22859 (19)	0.32722 (4)	0.0210 (3)
Cl11	0.80599 (19)	−0.5332 (3)	0.51941 (5)	0.0600 (5)
Br1	0.60784 (5)	0.15106 (9)	0.366127 (17)	0.03129 (16)
Na1	0.85419 (18)	−0.0126 (3)	0.26251 (7)	0.0323 (5)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.023 (2)	0.016 (3)	0.020 (2)	0.000 (2)	−0.0012 (18)	−0.003 (2)
C2	0.028 (2)	0.020 (3)	0.022 (2)	0.000 (2)	0.004 (2)	−0.002 (2)
C3	0.045 (3)	0.029 (3)	0.026 (3)	−0.003 (3)	0.008 (2)	0.004 (2)
C4	0.052 (4)	0.023 (3)	0.030 (3)	0.001 (3)	−0.017 (3)	0.002 (2)
C5	0.030 (3)	0.041 (4)	0.043 (3)	0.007 (3)	−0.011 (3)	0.003 (3)
C6	0.026 (2)	0.032 (3)	0.039 (3)	0.004 (3)	0.002 (2)	0.001 (3)
C7	0.027 (2)	0.030 (3)	0.031 (3)	−0.006 (2)	0.009 (2)	0.004 (2)
N1	0.025 (2)	0.024 (2)	0.0217 (19)	0.0010 (19)	−0.0035 (16)	0.0016 (18)
O1	0.0325 (18)	0.031 (2)	0.0236 (17)	−0.0036 (17)	0.0021 (14)	−0.0051 (15)
O2	0.0286 (18)	0.042 (2)	0.034 (2)	−0.0017 (18)	0.0140 (16)	0.0030 (18)
O3W	0.0277 (18)	0.039 (3)	0.035 (2)	0.0013 (17)	0.0058 (15)	−0.0010 (18)
O4W	0.049 (3)	0.027 (3)	0.024 (2)	0.000	0.001 (2)	0.000
S1	0.0214 (6)	0.0237 (7)	0.0179 (5)	0.0006 (5)	0.0031 (4)	0.0003 (5)
Cl11	0.0932 (13)	0.0454 (11)	0.0324 (8)	−0.0005 (10)	−0.0190 (8)	0.0083 (7)
Br1	0.0338 (3)	0.0274 (3)	0.0330 (3)	−0.0024 (3)	0.0062 (2)	−0.0056 (2)
Na1	0.0316 (11)	0.0338 (12)	0.0330 (11)	−0.0065 (10)	0.0101 (9)	−0.0003 (10)

Geometric parameters (Å, °)

C1—C6	1.390 (6)	N1—Br1	1.894 (4)
C1—C2	1.397 (6)	O1—S1	1.457 (4)
C1—S1	1.788 (5)	O1—Na1^{i}	2.441 (4)
C2—C3	1.394 (7)	O2—S1	1.449 (3)
C2—C7	1.524 (7)	O2—Na1	2.377 (4)
C3—C4	1.375 (8)	O3W—Na1	2.436 (4)
C3—H3	0.9300	O3W—Na1^{i}	2.488 (4)
C4—C5	1.366 (8)	O3W—H31	0.8208
C4—Cl11	1.750 (5)	O3W—H32	0.8585
C5—C6	1.384 (8)	O4W—Na1	2.494 (4)

C5—H5	0.9300	O4W—H41	0.8283
C6—H6	0.9300	S1—Na1^{i}	3.388 (2)
C7—H7A	0.9600	Na1—O1^{ii}	2.568 (4)
C7—H7B	0.9600	Na1—S1^{iii}	3.388 (2)
C7—H7C	0.9600	Na1—Na1^{iv}	3.427 (4)
N1—S1	1.584 (4)	Na1—Na1^{i}	4.105 (3)
C6—C1—C2	120.4 (4)	C1—S1—Na1^{i}	120.60 (16)
C6—C1—S1	116.7 (4)	O2—Na1—O3W	81.90 (14)
C2—C1—S1	122.9 (3)	O2—Na1—O1^{iii}	168.96 (16)
C3—C2—C1	117.8 (4)	O3W—Na1—O1^{iii}	87.06 (14)
C3—C2—C7	118.9 (4)	O2—Na1—O3W^{iii}	89.42 (14)
C1—C2—C7	123.3 (4)	O3W—Na1—O3W^{iii}	118.56 (11)
C4—C3—C2	120.5 (5)	O1^{iii}—Na1—O3W^{iii}	96.15 (14)
C4—C3—H3	119.7	O2—Na1—O4W	99.44 (14)
C2—C3—H3	119.7	O3W—Na1—O4W	85.24 (12)
C5—C4—C3	122.1 (5)	O1^{iii}—Na1—O4W	79.25 (12)
C5—C4—Cl11	119.2 (4)	O3W^{iii}—Na1—O4W	155.70 (14)
C3—C4—Cl11	118.7 (5)	O2—Na1—O1^{ii}	111.94 (14)
C4—C5—C6	118.1 (5)	O3W—Na1—O1^{ii}	158.82 (14)
C4—C5—H5	120.9	O1^{iii}—Na1—O1^{ii}	78.58 (14)
C6—C5—H5	120.9	O3W^{iii}—Na1—O1^{ii}	78.81 (13)
C5—C6—C1	120.9 (5)	O4W—Na1—O1^{ii}	76.89 (12)
C5—C6—H6	119.5	O2—Na1—S1^{iii}	152.84 (12)
C1—C6—H6	119.5	O3W—Na1—S1^{iii}	81.03 (11)
C2—C7—H7A	109.5	O3W^{iii}—Na1—S1^{iii}	80.44 (10)
C2—C7—H7B	109.5	O4W—Na1—S1^{iii}	100.03 (9)
H7A—C7—H7B	109.5	O1^{ii}—Na1—S1^{iii}	90.86 (10)
C2—C7—H7C	109.5	O2—Na1—Na1^{iv}	137.06 (13)
H7A—C7—H7C	109.5	O3W—Na1—Na1^{iv}	113.64 (12)
H7B—C7—H7C	109.5	O1^{iii}—Na1—Na1^{iv}	48.38 (10)
S1—N1—Br1	110.7 (2)	O3W^{iii}—Na1—Na1^{iv}	113.35 (10)
S1—O1—Na1^{i}	118.48 (19)	O4W—Na1—Na1^{iv}	46.60 (10)
S1—O1—Na1^{v}	149.3 (2)	O1^{ii}—Na1—Na1^{iv}	45.30 (9)
Na1^{i}—O1—Na1^{v}	86.31 (13)	S1^{iii}—Na1—Na1^{iv}	69.67 (6)
S1—O2—Na1	149.7 (2)	O2—Na1—Na1^{i}	54.02 (10)
Na1—O3W—Na1^{i}	112.95 (15)	O3W—Na1—Na1^{i}	33.93 (10)
Na1—O3W—H31	110.6	O1^{iii}—Na1—Na1^{i}	115.51 (12)
Na1^{i}—O3W—H31	112.9	O3W^{iii}—Na1—Na1^{i}	127.99 (12)
Na1—O3W—H32	103.2	O4W—Na1—Na1^{i}	74.25 (9)
Na1^{i}—O3W—H32	106.0	O1^{ii}—Na1—Na1^{i}	144.31 (12)
H31—O3W—H32	110.7	S1^{iii}—Na1—Na1^{i}	114.39 (9)

Na1^{iv}—O4W—Na1	86.81 (19)	Na1^{iv}—Na1—Na1^{i}	118.57 (6)
Na1^{iv}—O4W—H41	129.0	O2—Na1—Na1^{iii}	100.58 (12)
Na1—O4W—H41	112.8	O3W—Na1—Na1^{iii}	89.16 (13)
O2—S1—O1	114.3 (2)	O1^{iii}—Na1—Na1^{iii}	79.46 (10)
O2—S1—N1	115.3 (2)	O3W^{iii}—Na1—Na1^{iii}	33.12 (9)
O1—S1—N1	104.4 (2)	O4W—Na1—Na1^{iii}	158.22 (11)
O2—S1—C1	104.6 (2)	O1^{ii}—Na1—Na1^{iii}	103.23 (10)
O1—S1—C1	108.2 (2)	S1^{iii}—Na1—Na1^{iii}	58.26 (5)
N1—S1—C1	109.9 (2)	Na1^{iv}—Na1—Na1^{iii}	118.57 (6)
O2—S1—Na1^{i}	75.00 (16)	Na1^{i}—Na1—Na1^{iii}	111.37 (10)
N1—S1—Na1^{i}	123.91 (16)		

Symmetry codes: (i) −*x*+3/2, *y*−1/2, −*z*+1/2; (ii) *x*+1/2, *y*+1/2, *z*; (iii) −*x*+3/2, *y*+1/2, −*z*+1/2; (iv) −*x*+2, *y*, −*z*+1/2; (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^{vi}	0.82	2.16	2.927 (5)	156
O4W—H41···N1^{vii}	0.83	2.19	3.010 (5)	171

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

Fig. 1

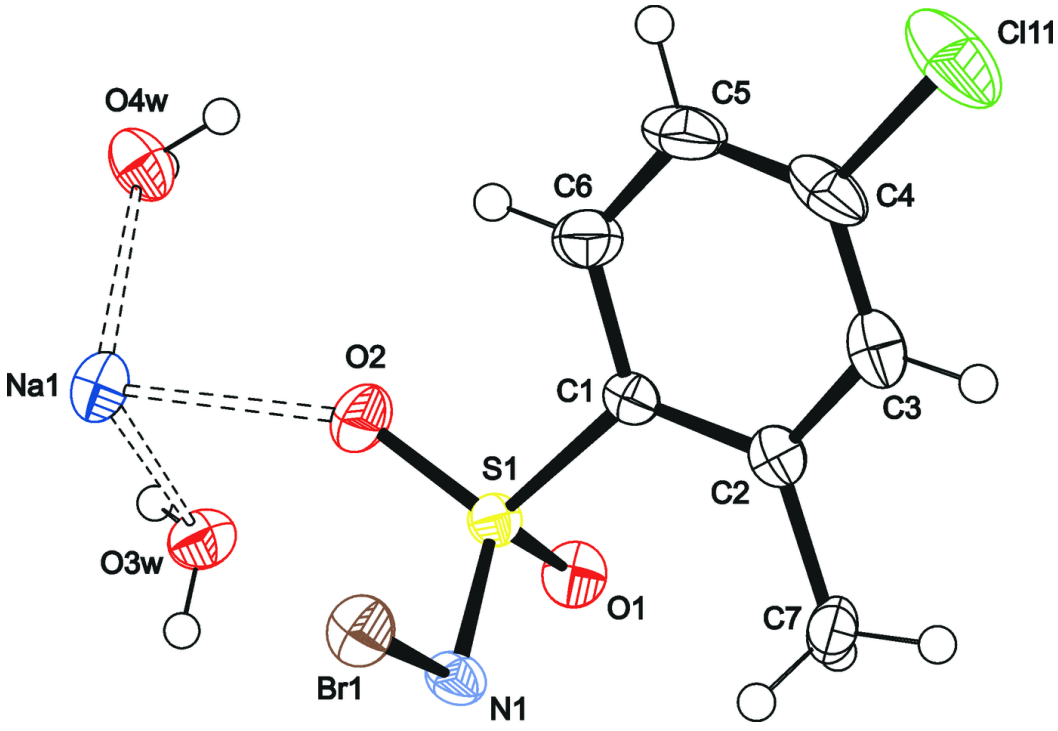

Fig. 2