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Sodium *N*,2-dichlorobenzenesulfonamidate sesquihydrate

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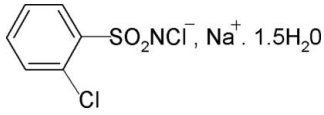
Key indicators: single-crystal X-ray study; T = 299 K; mean σ(C–C) = 0.003 Å; R factor = 0.029; wR factor = 0.069; data-to-parameter ratio = 14.7.

In the title compound, $Na^+\cdot C_6H_4Cl_2NO_2S^-\cdot 1.5H_2O$, one of the water molecules lies on a twofold axis. There is no interaction between the N atom and the sodium ion. The sodium ion exhibits a pseudo-octahedral coordination defined by three water O atoms and three sulfonyl O atoms from three different anions. The S—N distance of 1.588 (2) Å is consistent with an S=N double bond. The crystal structure is stabilized by O—H···N and O—H···Cl hydrogen bonds.

Related literature

For background to *N*-haloarylsulfonamides, see: Gowda *et al.* (2005). For related structures, see: Gowda *et al.* (2007, 2009); George *et al.* (2000); Olmstead & Power (1986).



Experimental

Crystal data

$Na^+\cdot C_6H_4Cl_2NO_2S^-\cdot 1.5H_2O$
M_r = 275.08
Monoclinic, $C2/c$
a = 11.1288 (7) Å
b = 6.6724 (4) Å
c = 28.144 (2) Å
β = 102.274 (6)°
V = 2042.1 (2) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.87 mm^{-1}
T = 299 K
0.46 × 0.36 × 0.28 mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)
T_{min} = 0.691, T_{max} = 0.794
6590 measured reflections
2076 independent reflections
1944 reflections with $I > 2\sigma(I)$
R_{int} = 0.014

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.069
S = 1.15
2076 reflections
141 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H31···N1^{i}	0.79 (2)	2.15 (2)	2.926 (2)	166 (3)
O3—H32···Cl1^{ii}	0.81 (2)	2.67 (2)	3.4782 (16)	171 (2)
O4—H41···N1^{ii}	0.81 (2)	2.19 (2)	3.005 (2)	176 (2)

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2009); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BX2268).

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supporting information

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Sodium *N*,2-dichlorobenzenesulfonamidate sesquihydrate

B. Thimme Gowda, Sabine Foro, K. Shakuntala and Hartmut Fuess

S1. Comment

In the present work, as a part of exploring the substituent effects on the solid-state structures of N-halo aryl-sulfonamidates (Gowda et al., 2005; 2007; 2009), the structure of sodium N-chloro-2-chloro- benzenesulfonamidate (I) has been determined (Fig. 1). The structure of (I) resembles the sodium salts of N-chloro-4-chlorobenzenesulfonamidate (Gowda et al., 2007), N-chloro-2-methylbenzenesulfonamidate (Gowda et al., 2009), and other sodium N-chloro- aryl-sulfonamidates (George et al., 2000; Olmstead & Power, 1986).

The sodium ion shows pseudo-octahedral coordination defined by three water-O atoms and by three sulfonyl-O atoms derived from three different anions. There is no interaction between the nitrogen and sodium ions. The S—N distance of 1.588 (2)Å is consistent with a S—N double bond and is in agreement with those observed with related N-chloro aryl-sulfonamides.

The Packing diagram consists of a two-dimensional polymeric layer running parallel to the ac plane (Fig. 2). The molecular packing is stabilized by N-H···O and O-H···Cl hydrogen bonds (Table 1)

S2. Experimental

The title compound was prepared according to the literature method (Gowda *et al.*, 2005; 2007). The purity of the compound was checked by determining its melting point. It was characterized by recording its infrared and NMR spectra. Single crystals of the title compound used in X-ray diffraction studies were obtained from a slow evaporation of its chloroform solution at room temperature.

S3. Refinement

The O-bound H atoms were located in difference map and later restrained to O—H = 0.82 (2) Å. The other H atoms were positioned with idealized geometry using a riding model with C—H = 0.93 Å. All H atoms were refined with isotropic displacement parameters (set to 1.2 times of the U_{eq} of the parent atom).

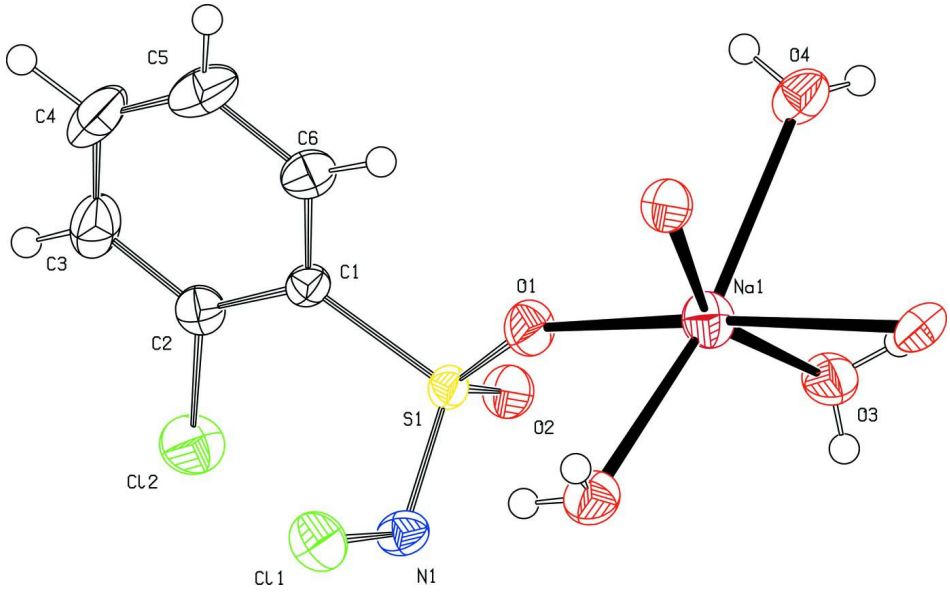

Figure 1

Molecular structure of the title compound, showing the atom labelling scheme. The displacement ellipsoids are drawn at the 50% probability level. The H atoms are represented as small spheres of arbitrary radii.

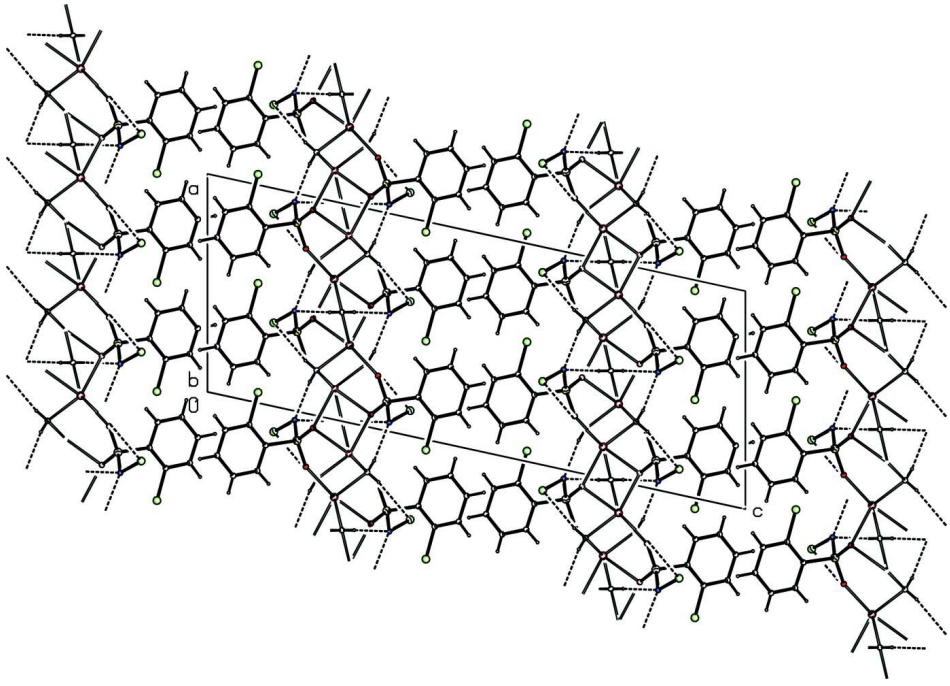

Figure 2

Molecular packing of the title compound with hydrogen bonding shown as dashed lines.

Sodium *N*,2-dichlorobenzenesulfonamidate sesquihydrate

Crystal data

$Na^+ \cdot C_6H_4Cl_2NO_2S^- \cdot 1.5H_2O$
$M_r = 275.08$
Monoclinic, $C2/c$
Hall symbol: -C 2yc
$a = 11.1288$ (7) Å
$b = 6.6724$ (4) Å
$c = 28.144$ (2) Å
$\beta = 102.274$ (6)°
$V = 2042.1$ (2) Å^3
$Z = 8$
$F(000) = 1112$
$D_x = 1.789$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2816 reflections
$\theta = 3.0$–$27.9°$
$\mu = 0.87$ mm^{-1}
$T = 299$ K
Prism, colourless
0.46 × 0.36 × 0.28 mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector
Radiation source: fine-focus sealed tube
Graphite monochromator
Rotation method data acquisition using ω and phi scans
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)
$T_{min} = 0.691$, $T_{max} = 0.794$
6590 measured reflections
2076 independent reflections
1944 reflections with $I > 2\sigma(I)$
$R_{int} = 0.014$
$\theta_{max} = 26.4°$, $\theta_{min} = 3.0°$
$h = -13 \rightarrow 13$
$k = -8 \rightarrow 6$
$l = -33 \rightarrow 35$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.069$
$S = 1.15$
2076 reflections
141 parameters
3 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
$w = 1/[\sigma^2(F_o^2) + (0.0246P)^2 + 3.4504P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.006$
$\Delta\rho_{max} = 0.36$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}

Special details

Experimental. (CrysAlis RED; Oxford Diffraction, 2009) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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}
C1	0.31355 (16)	0.8280 (3)	0.10792 (6)	0.0208 (4)

C2	0.39097 (18)	0.8966 (3)	0.07759 (7)	0.0257 (4)
C3	0.3434 (2)	0.9809 (3)	0.03289 (8)	0.0376 (5)
H3	0.3951	1.0261	0.0132	0.045*
C4	0.2181 (2)	0.9965 (4)	0.01810 (8)	0.0454 (6)
H4	0.1843	1.0514	−0.0122	0.054*
C5	0.1413 (2)	0.9312 (4)	0.04792 (9)	0.0431 (6)
H5	0.0566	0.9446	0.0375	0.052*
C6	0.18813 (18)	0.8472 (3)	0.09260 (7)	0.0301 (4)
H6	0.1358	0.8038	0.1122	0.036*
Cl1	0.38325 (5)	0.35684 (8)	0.123234 (19)	0.03468 (14)
Cl2	0.54842 (5)	0.88282 (9)	0.09392 (2)	0.04180 (16)
N1	0.45784 (14)	0.5415 (2)	0.16212 (6)	0.0255 (3)
Na1	0.14395 (7)	0.50352 (13)	0.23560 (3)	0.03065 (19)
O1	0.25555 (13)	0.6627 (2)	0.18286 (5)	0.0329 (3)
O2	0.44039 (12)	0.8680 (2)	0.19636 (5)	0.0295 (3)
O3	0.29191 (13)	0.6793 (2)	0.29703 (5)	0.0333 (3)
H31	0.3538 (18)	0.627 (4)	0.3106 (9)	0.040*
H32	0.258 (2)	0.718 (4)	0.3182 (8)	0.040*
O4	0.0000	0.7742 (3)	0.2500	0.0336 (5)
H41	0.013 (2)	0.852 (3)	0.2729 (7)	0.040*
S1	0.36626 (4)	0.71995 (7)	0.166416 (15)	0.02023 (12)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0244 (9)	0.0166 (8)	0.0201 (8)	0.0008 (7)	0.0022 (7)	−0.0003 (7)
C2	0.0290 (10)	0.0207 (9)	0.0277 (9)	−0.0013 (8)	0.0068 (8)	−0.0009 (8)
C3	0.0540 (14)	0.0311 (11)	0.0293 (11)	−0.0020 (10)	0.0129 (10)	0.0059 (9)
C4	0.0605 (15)	0.0406 (13)	0.0279 (11)	0.0060 (12)	−0.0068 (10)	0.0108 (10)
C5	0.0355 (12)	0.0442 (13)	0.0412 (12)	0.0054 (10)	−0.0106 (9)	0.0065 (11)
C6	0.0239 (9)	0.0331 (11)	0.0310 (10)	0.0007 (8)	0.0008 (8)	0.0010 (9)
Cl1	0.0399 (3)	0.0266 (3)	0.0380 (3)	−0.0038 (2)	0.0092 (2)	−0.0080 (2)
Cl2	0.0281 (3)	0.0487 (3)	0.0520 (3)	−0.0023 (2)	0.0163 (2)	0.0120 (3)
N1	0.0243 (8)	0.0226 (8)	0.0269 (8)	0.0010 (7)	−0.0002 (6)	−0.0014 (7)
Na1	0.0281 (4)	0.0325 (4)	0.0328 (4)	−0.0052 (3)	0.0097 (3)	0.0006 (3)
O1	0.0272 (7)	0.0430 (9)	0.0309 (7)	−0.0009 (6)	0.0116 (6)	0.0065 (7)
O2	0.0295 (7)	0.0308 (8)	0.0252 (7)	−0.0014 (6)	−0.0007 (5)	−0.0081 (6)
O3	0.0245 (7)	0.0408 (9)	0.0333 (8)	0.0014 (7)	0.0035 (6)	−0.0031 (7)
O4	0.0432 (12)	0.0244 (11)	0.0291 (11)	0.000	−0.0015 (9)	0.000
S1	0.0197 (2)	0.0233 (2)	0.0173 (2)	−0.00026 (17)	0.00302 (15)	0.00009 (17)

Geometric parameters (Å, º)

C1—C6	1.376 (3)	Na1—O2^{i}	2.4710 (15)
C1—C2	1.412 (3)	Na1—O2^{ii}	2.4759 (15)
C1—S1	1.7786 (18)	Na1—O4	2.5035 (18)
C2—C3	1.377 (3)	Na1—O3^{ii}	2.5120 (18)
C2—Cl2	1.717 (2)	Na1—S1^{ii}	3.3661 (9)

C3—C4	1.372 (3)	O1—S1	1.4562 (14)
C3—H3	0.9300	O2—S1	1.4390 (14)
C4—C5	1.389 (4)	O2—Na1^{iii}	2.4710 (15)
C4—H4	0.9300	O2—Na1^{iv}	2.4759 (15)
C5—C6	1.374 (3)	O3—Na1^{iv}	2.5120 (18)
C5—H5	0.9300	O3—H31	0.792 (16)
C6—H6	0.9300	O3—H32	0.811 (16)
Cl1—N1	1.7376 (16)	O4—Na1^{v}	2.5035 (18)
N1—S1	1.5883 (16)	O4—H41	0.814 (16)
Na1—O1	2.3785 (15)	S1—Na1^{iv}	3.3661 (9)
Na1—O3	2.4220 (17)		

C6—C1—C2	119.28 (17)	O2^{ii}—Na1—O3^{ii}	98.75 (6)
C6—C1—S1	116.14 (14)	O4—Na1—O3^{ii}	157.17 (5)
C2—C1—S1	124.58 (14)	O1—Na1—S1^{ii}	151.07 (5)
C3—C2—C1	121.30 (19)	O3—Na1—S1^{ii}	79.85 (4)
C3—C2—Cl2	116.02 (16)	O2^{i}—Na1—S1^{ii}	88.45 (4)
C1—C2—Cl2	122.68 (15)	O2^{ii}—Na1—S1^{ii}	22.58 (3)
C4—C3—C2	118.5 (2)	O4—Na1—S1^{ii}	97.97 (3)
C4—C3—H3	120.8	O3^{ii}—Na1—S1^{ii}	82.95 (4)
C2—C3—H3	120.8	S1—O1—Na1	154.80 (9)
C3—C4—C5	120.6 (2)	S1—O2—Na1^{iii}	150.45 (9)
C3—C4—H4	119.7	S1—O2—Na1^{iv}	116.06 (8)
C5—C4—H4	119.7	Na1^{iii}—O2—Na1^{iv}	89.02 (5)
C6—C5—C4	121.3 (2)	Na1—O3—Na1^{iv}	111.04 (6)
C6—C5—H5	119.4	Na1—O3—H31	121.4 (19)
C4—C5—H5	119.4	Na1^{iv}—O3—H31	105.4 (19)
C5—C6—C1	119.1 (2)	Na1—O3—H32	108.9 (19)
C5—C6—H6	120.5	Na1^{iv}—O3—H32	102.1 (19)
C1—C6—H6	120.5	H31—O3—H32	106 (3)
S1—N1—Cl1	110.56 (9)	Na1^{v}—O4—Na1	87.67 (8)
O1—Na1—O3	82.14 (6)	Na1^{v}—O4—H41	109.7 (18)
O1—Na1—O2^{i}	115.80 (6)	Na1—O4—H41	125.3 (18)
O3—Na1—O2^{i}	156.33 (6)	O2—S1—O1	114.36 (9)
O1—Na1—O2^{ii}	168.48 (6)	O2—S1—N1	105.17 (8)
O3—Na1—O2^{ii}	86.38 (6)	O1—S1—N1	115.30 (9)
O2^{i}—Na1—O2^{ii}	75.50 (6)	O2—S1—C1	107.39 (9)
O1—Na1—O4	102.47 (6)	O1—S1—C1	105.41 (8)
O3—Na1—O4	84.05 (5)	N1—S1—C1	108.91 (8)
O2^{i}—Na1—O4	77.23 (5)	O2—S1—Na1^{iv}	41.36 (6)
O2^{ii}—Na1—O4	77.14 (5)	O1—S1—Na1^{iv}	73.08 (6)
O1—Na1—O3^{ii}	85.97 (6)	N1—S1—Na1^{iv}	128.14 (6)
O3—Na1—O3^{ii}	118.37 (5)	C1—S1—Na1^{iv}	117.93 (6)
O2^{i}—Na1—O3^{ii}	79.99 (5)		

C6—C1—C2—C3	−0.5 (3)	O2^{ii}—Na1—O4—Na1^{v}	−38.86 (3)
S1—C1—C2—C3	−179.26 (16)	O3^{ii}—Na1—O4—Na1^{v}	43.09 (13)
C6—C1—C2—Cl2	178.96 (16)	S1^{ii}—Na1—O4—Na1^{v}	−47.647 (17)

S1—C1—C2—Cl2	0.2 (2)	Na1^{iii}—O2—S1—O1	141.67 (17)
C1—C2—C3—C4	−0.2 (3)	Na1^{iv}—O2—S1—O1	−3.82 (12)
Cl2—C2—C3—C4	−179.70 (18)	Na1^{iii}—O2—S1—N1	14.2 (2)
C2—C3—C4—C5	0.9 (4)	Na1^{iv}—O2—S1—N1	−131.33 (9)
C3—C4—C5—C6	−0.8 (4)	Na1^{iii}—O2—S1—C1	−101.75 (18)
C4—C5—C6—C1	0.1 (4)	Na1^{iv}—O2—S1—C1	112.77 (9)
C2—C1—C6—C5	0.5 (3)	Na1^{iii}—O2—S1—Na1^{iv}	145.5 (2)
S1—C1—C6—C5	179.42 (17)	Na1—O1—S1—O2	−71.7 (2)
O3—Na1—O1—S1	50.3 (2)	Na1—O1—S1—N1	50.4 (3)
O2^{i}—Na1—O1—S1	−145.9 (2)	Na1—O1—S1—C1	170.6 (2)
O2^{ii}—Na1—O1—S1	45.6 (5)	Na1—O1—S1—Na1^{iv}	−74.3 (2)
O4—Na1—O1—S1	132.4 (2)	Cl1—N1—S1—O2	−175.92 (9)
O3^{ii}—Na1—O1—S1	−69.0 (2)	Cl1—N1—S1—O1	57.14 (12)
S1^{ii}—Na1—O1—S1	−1.5 (3)	Cl1—N1—S1—C1	−61.07 (11)
O1—Na1—O3—Na1^{iv}	31.28 (6)	Cl1—N1—S1—Na1^{iv}	144.96 (6)
O2^{i}—Na1—O3—Na1^{iv}	−109.93 (14)	C6—C1—S1—O2	−118.21 (15)
O2^{ii}—Na1—O3—Na1^{iv}	−149.66 (7)	C2—C1—S1—O2	60.61 (18)
O4—Na1—O3—Na1^{iv}	−72.23 (6)	C6—C1—S1—O1	4.11 (17)
O3^{ii}—Na1—O3—Na1^{iv}	112.35 (9)	C2—C1—S1—O1	−177.07 (16)
S1^{ii}—Na1—O3—Na1^{iv}	−171.45 (6)	C6—C1—S1—N1	128.38 (15)
O1—Na1—O4—Na1^{v}	152.95 (5)	C2—C1—S1—N1	−52.80 (18)
O3—Na1—O4—Na1^{v}	−126.49 (5)	C6—C1—S1—Na1^{iv}	−74.61 (16)
O2^{i}—Na1—O4—Na1^{v}	38.93 (4)	C2—C1—S1—Na1^{iv}	104.21 (15)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H31···N1^{vi}	0.79 (2)	2.15 (2)	2.926 (2)	166 (3)
O3—H32···Cl1^{iv}	0.81 (2)	2.67 (2)	3.4782 (16)	171 (2)
O4—H41···N1^{iv}	0.81 (2)	2.19 (2)	3.005 (2)	176 (2)

Symmetry codes: (iv) $-x+1/2, y+1/2, -z+1/2$; (vi) $-x+1, y, -z+1/2$.