

Poly[octa- μ -aqua-tetraaquabis(μ_4 -5-sulfonatobenzene-1,3-dicarboxylato)-nickel(II)tetrasodium]

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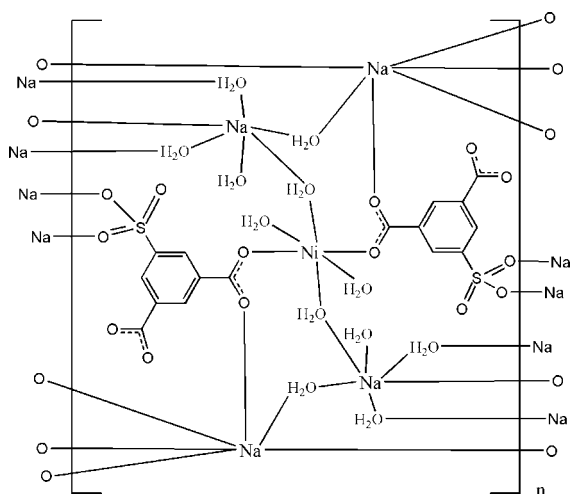
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 Key indicators: single-crystal X-ray study; $T = 295$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.025; wR factor = 0.071; data-to-parameter ratio = 13.9.

In the crystal structure of the title compound, $[\text{Na}_4\text{Ni}(\text{C}_8\text{H}_3\text{O}_7\text{S})_2(\text{H}_2\text{O})_{12}]_n$, the Ni^{II} cation occupies an inversion centre and is coordinated by the carboxyl groups of the sulfoisophthalate trianions and water molecules in a distorted octahedral geometry. Two independent Na^{I} atoms are connected by the carboxyl and sulfonate groups of the sulfoisophthalate ligands and water molecules in a distorted octahedral geometry. The sulfoisophthalate ligands and coordinated water molecules bridge the Ni^{II} and Na^{I} cations, forming a three-dimensional polymeric structure. Weak π - π stacking is present between parallel benzene rings [centroid-centroid distance = 3.9349 (10) Å]. Extensive O—H...O and C—H...O hydrogen bonding helps to stabilize the crystal structure.

Related literature

For general background, see: Su & Xu (2004); Pan *et al.* (2006). For the isotopic structure of the Co analogue, see: Zhang *et al.* (2009).



Experimental

Crystal data

 $[\text{Na}_4\text{Ni}(\text{C}_8\text{H}_3\text{O}_7\text{S})_2(\text{H}_2\text{O})_{12}]$
 $M_r = 853.19$

 Monoclinic, $P2_1/n$
 $a = 7.8770$ (9) Å

 $b = 17.229$ (2) Å

 $c = 11.7474$ (13) Å

 $\beta = 93.292$ (4)°

 $V = 1591.7$ (3) Å³
 $Z = 2$

 Mo $K\alpha$ radiation

 $\mu = 0.90$ mm⁻¹
 $T = 295$ K

 $0.30 \times 0.22 \times 0.20$ mm

Data collection

Rigaku R-Axis RAPID IP

diffractometer

Absorption correction: multi-scan

 (*ABSCOR*; Higashi, 1995)

 $T_{\text{min}} = 0.770$, $T_{\text{max}} = 0.835$

17574 measured reflections

3107 independent reflections

 2866 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.023$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.025$
 $wR(F^2) = 0.071$
 $S = 1.08$

3107 reflections

223 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.42$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.40$ e Å⁻³
Table 1

Selected bond lengths (Å).

Ni—O1	2.0252 (11)	Ni—O9	2.0727 (11)
Ni—O8	2.0731 (14)		

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O8—H8 <i>A</i> ...O13 ⁱ	0.84	2.03	2.861 (2)	173
O8—H8 <i>B</i> ...O4 ⁱⁱ	0.85	1.99	2.8130 (19)	162
O9—H9 <i>A</i> ...O7 ⁱⁱⁱ	0.85	2.16	2.9854 (17)	163
O9—H9 <i>B</i> ...O2	0.84	1.82	2.6168 (17)	159
O10—H10 <i>A</i> ...O7 ⁱⁱ	0.83	2.04	2.8553 (19)	167
O10—H10 <i>B</i> ...O3 ^{iv}	0.85	1.83	2.6615 (19)	165
O11—H11 <i>A</i> ...O7 ⁱⁱⁱ	0.89	1.90	2.7659 (18)	167
O11—H11 <i>B</i> ...O3 ^{iv}	0.87	1.92	2.783 (2)	175
O12—H12 <i>A</i> ...O1 ^v	0.84	2.11	2.9470 (18)	173
O12—H12 <i>B</i> ...O4 ^{vi}	0.89	2.04	2.8994 (19)	163
O13—H13 <i>A</i> ...O4 ⁱⁱ	0.84	1.94	2.733 (2)	157
O13—H13 <i>B</i> ...O6 ^{vii}	0.88	2.21	2.9486 (19)	141
C7—H7...O11 ^{viii}	0.93	2.50	3.371 (2)	157

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

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MS, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2561).

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supplementary materials

Acta Cryst. (2009). E65, m429-m430 [doi:10.1107/S1600536809009489]

Poly[octa- μ -aqua-tetraaquabis(μ_4 -5-sulfonatobenzene-1,3-dicarboxylato)nickel(II)tetrasodium]

B.-Y. Zhang, J.-J. Nie and D.-J. Xu

Comment

As a part of investigation on π - π stacking between aromatic rings (Su & Xu, 2004; Pan *et al.*, 2006), the title Ni^{II} compound has recently been prepared in our laboratory, and its crystal structure is reported here.

A part of the three dimensional polymeric structure of the title compound is shown in Fig. 1. The Ni^{II} compound is isomorphous with the Co^{II} compound (Zhang *et al.*, 2009). The Ni atom occupies a special position in an inversion centre and assumes a distorted NiO₆ octahedral geometry, The Ni—O bond distances (Table 1) are about 0.03 Å shorter than corresponding Co—O bond distances found in the isomorphous Co^{II} compound. Both crystallographically independent Na^I atoms are in distorted octahedral coordination geometry. The sulfoisophthalate trianions and water molecules bridge the metal atoms to form the polymeric structure.

The extensive O—H \cdots O hydrogen bonding network presents in the crystal structure (Table 2), weak C—H \cdots O hydrogen bonding also helps to stabilize the crystal structure. The distance between parallel the C2-benzene plane and C2^v-benzene plane is 3.551 (9) Å [symmetry code: (v) $1-x, 1-y, -z$], and the centroids distance between the benzene rings is 3.9349 (10) Å. These findings suggest a weak π - π stacking involving sulfoisophthalate ligand.

Experimental

A water-ethanol solution (25 ml, 3:2) containing monosodium 5-sulfoisophthalate (0.270 g, 1 mmol), Na₂CO₃ (0.212 g, 2 mmol) and NiCl₂·6H₂O (0.600 g, 2.5 mmol) was refluxed for 8 h and filtered after cooling to room temperature. The single crystals of the title compound were obtained from the filtrate after 3 weeks.

Refinement

Water H atoms were located in a difference Fourier map and refined as riding in as-found relative positions, with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$. Other H atoms were placed in calculated positions with C—H = 0.93 Å and refined in riding mode with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Figures

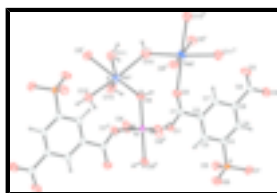


Fig. 1. A part of the polymeric structure of the title compound with 50% probability displacement (arbitrary spheres for H atoms) [symmetry codes: (i) $-x + 3/2, +y - 1/2, -z + 1/2$; (ii) $-x + 1/2, +y - 1/2, -z + 1/2$; (iii) $-x + 1, -y + 1, -z + 1$; (iv) $x - 1/2, -y + 1/2, +z - 1/2$; (v) $-x + 1, -y + 1, -z$].

Poly[octa- μ -aqua-tetraaquabis(μ_4 -5-sulfonatobenzene-1,3-dicarboxylato)nickel(II)tetrasodium]

Crystal data

$[\text{Na}_4\text{Ni}(\text{C}_8\text{H}_3\text{O}_7\text{S})_2(\text{H}_2\text{O})_{12}]$	$F_{000} = 876$
$M_r = 853.19$	$D_x = 1.780 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
Hall symbol: -P 2yn	$\lambda = 0.71073 \text{ \AA}$
$a = 7.8770 (9) \text{ \AA}$	Cell parameters from 2356 reflections
$b = 17.229 (2) \text{ \AA}$	$\theta = 2.5\text{--}25.0^\circ$
$c = 11.7474 (13) \text{ \AA}$	$\mu = 0.90 \text{ mm}^{-1}$
$\beta = 93.292 (4)^\circ$	$T = 295 \text{ K}$
$V = 1591.7 (3) \text{ \AA}^3$	Prism, green
$Z = 2$	$0.30 \times 0.22 \times 0.20 \text{ mm}$

Data collection

Rigaku R-Axis RAPID IP diffractometer	3107 independent reflections
Radiation source: fine-focus sealed tube	2866 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.023$
Detector resolution: $10.0 \text{ pixels mm}^{-1}$	$\theta_{\text{max}} = 26.0^\circ$
$T = 295 \text{ K}$	$\theta_{\text{min}} = 2.1^\circ$
ω scans	$h = -9 \rightarrow 9$
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	$k = -21 \rightarrow 21$
$T_{\text{min}} = 0.770$, $T_{\text{max}} = 0.835$	$l = -14 \rightarrow 14$
17574 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.025$	H-atom parameters constrained
$wR(F^2) = 0.071$	$w = 1/[\sigma^2(F_o^2) + (0.0384P)^2 + 0.7337P]$
$S = 1.08$	where $P = (F_o^2 + 2F_c^2)/3$
3107 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
223 parameters	$\Delta\rho_{\text{max}} = 0.42 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.40 \text{ e \AA}^{-3}$
	Extinction correction: none

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Ni	0.5000	0.5000	0.5000	0.01887 (10)
Na1	0.82068 (9)	0.33240 (4)	0.45637 (6)	0.02989 (18)
Na2	0.40578 (9)	0.26823 (4)	0.20100 (6)	0.02845 (17)
S	0.28801 (5)	0.73027 (2)	0.01772 (3)	0.01908 (11)
O1	0.42320 (16)	0.52266 (7)	0.33588 (10)	0.0244 (3)
O2	0.42591 (19)	0.40594 (7)	0.25407 (10)	0.0335 (3)
O3	0.1627 (2)	0.37896 (8)	-0.12900 (12)	0.0456 (4)
O4	0.11757 (18)	0.47912 (8)	-0.24403 (11)	0.0332 (3)
O5	0.45777 (16)	0.74769 (7)	-0.01724 (11)	0.0279 (3)
O6	0.25488 (18)	0.76128 (7)	0.12908 (10)	0.0291 (3)
O7	0.15727 (16)	0.75328 (7)	-0.06942 (11)	0.0267 (3)
O8	0.75086 (17)	0.51710 (9)	0.46157 (11)	0.0362 (3)
H8A	0.8191	0.5403	0.5074	0.054*
H8B	0.7789	0.5274	0.3941	0.054*
O9	0.53288 (16)	0.38303 (6)	0.46643 (10)	0.0238 (3)
H9A	0.4645	0.3534	0.4997	0.036*
H9B	0.5021	0.3785	0.3975	0.036*
O10	0.70115 (18)	0.26289 (7)	0.28653 (11)	0.0336 (3)
H10A	0.7540	0.2629	0.2277	0.050*
H10B	0.6997	0.2152	0.3041	0.050*
O11	0.68483 (16)	0.21970 (8)	0.55884 (12)	0.0327 (3)
H11A	0.5764	0.2266	0.5724	0.049*
H11B	0.6788	0.1866	0.5033	0.049*
O12	0.87077 (17)	0.37265 (7)	0.65035 (11)	0.0312 (3)
H12A	0.7926	0.4047	0.6585	0.047*
H12B	0.9552	0.3964	0.6897	0.047*
O13	1.03972 (18)	0.40442 (8)	0.36780 (12)	0.0354 (3)
H13A	1.0040	0.4338	0.3148	0.053*
H13B	1.1122	0.3726	0.3378	0.053*
C1	0.4055 (2)	0.47741 (9)	0.25112 (14)	0.0205 (3)
C2	0.3474 (2)	0.51522 (9)	0.13980 (14)	0.0211 (3)
C3	0.3448 (2)	0.59532 (9)	0.12908 (13)	0.0210 (3)
H3	0.3862	0.6266	0.1889	0.025*

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C4	0.2800 (2)	0.62800 (9)	0.02841 (14)	0.0187 (3)
C5	0.2204 (2)	0.58282 (9)	-0.06300 (14)	0.0218 (3)
H5	0.1774	0.6060	-0.1301	0.026*
C6	0.2258 (2)	0.50242 (9)	-0.05294 (14)	0.0224 (4)
C7	0.2909 (2)	0.46959 (10)	0.04849 (14)	0.0244 (4)
H7	0.2966	0.4159	0.0551	0.029*
C8	0.1644 (2)	0.44968 (10)	-0.15014 (14)	0.0240 (4)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni	0.02441 (18)	0.01777 (16)	0.01396 (16)	0.00201 (11)	-0.00293 (12)	-0.00019 (10)
Na1	0.0270 (4)	0.0368 (4)	0.0256 (4)	0.0015 (3)	-0.0011 (3)	0.0022 (3)
Na2	0.0305 (4)	0.0314 (4)	0.0234 (4)	-0.0003 (3)	0.0012 (3)	-0.0038 (3)
S	0.0229 (2)	0.01610 (19)	0.0184 (2)	-0.00051 (15)	0.00256 (15)	0.00059 (14)
O1	0.0382 (7)	0.0198 (6)	0.0145 (6)	0.0029 (5)	-0.0053 (5)	-0.0005 (5)
O2	0.0610 (9)	0.0186 (6)	0.0196 (6)	0.0057 (6)	-0.0087 (6)	0.0003 (5)
O3	0.0870 (12)	0.0189 (7)	0.0285 (7)	-0.0022 (7)	-0.0168 (7)	-0.0035 (5)
O4	0.0503 (8)	0.0298 (7)	0.0181 (6)	0.0013 (6)	-0.0090 (6)	-0.0012 (5)
O5	0.0249 (7)	0.0297 (7)	0.0294 (7)	-0.0062 (5)	0.0045 (5)	-0.0013 (5)
O6	0.0448 (8)	0.0212 (6)	0.0221 (7)	-0.0007 (5)	0.0094 (6)	-0.0036 (5)
O7	0.0272 (7)	0.0243 (6)	0.0284 (7)	0.0031 (5)	-0.0006 (5)	0.0058 (5)
O8	0.0294 (7)	0.0570 (9)	0.0223 (7)	-0.0067 (6)	0.0020 (5)	-0.0017 (6)
O9	0.0336 (7)	0.0187 (6)	0.0185 (6)	0.0011 (5)	-0.0041 (5)	-0.0004 (5)
O10	0.0472 (8)	0.0290 (7)	0.0246 (7)	0.0009 (6)	0.0034 (6)	0.0035 (5)
O11	0.0276 (7)	0.0317 (7)	0.0381 (8)	0.0043 (5)	-0.0047 (6)	-0.0062 (6)
O12	0.0337 (7)	0.0291 (7)	0.0304 (7)	0.0028 (5)	-0.0013 (6)	-0.0073 (5)
O13	0.0381 (8)	0.0312 (7)	0.0370 (8)	0.0066 (6)	0.0037 (6)	0.0064 (6)
C1	0.0265 (9)	0.0194 (8)	0.0153 (8)	0.0006 (6)	-0.0020 (6)	0.0009 (6)
C2	0.0269 (9)	0.0205 (8)	0.0155 (8)	0.0012 (7)	-0.0012 (6)	0.0005 (6)
C3	0.0268 (9)	0.0203 (8)	0.0157 (8)	-0.0007 (6)	0.0002 (6)	-0.0017 (6)
C4	0.0213 (8)	0.0166 (7)	0.0185 (8)	-0.0010 (6)	0.0024 (6)	0.0004 (6)
C5	0.0276 (9)	0.0218 (8)	0.0156 (8)	0.0006 (7)	-0.0023 (6)	0.0023 (6)
C6	0.0290 (9)	0.0219 (8)	0.0161 (8)	-0.0005 (7)	-0.0021 (7)	-0.0012 (6)
C7	0.0360 (10)	0.0170 (8)	0.0198 (8)	-0.0003 (7)	-0.0024 (7)	-0.0005 (6)
C8	0.0312 (9)	0.0221 (8)	0.0182 (8)	-0.0002 (7)	-0.0027 (7)	-0.0026 (6)

Geometric parameters (\AA , $^\circ$)

Ni—O1 ⁱ	2.0252 (11)	O5—Na1 ^{vii}	2.3545 (14)
Ni—O1	2.0252 (11)	O5—Na2 ^{vi}	2.4814 (14)
Ni—O8 ⁱ	2.0731 (14)	O6—Na2 ^{viii}	2.4271 (14)
Ni—O8	2.0731 (14)	O8—H8A	0.8407
Ni—O9	2.0727 (11)	O8—H8B	0.8534
Ni—O9 ⁱ	2.0727 (11)	O9—H9A	0.8532
Na1—O5 ⁱⁱ	2.3545 (14)	O9—H9B	0.8358
Na1—O12	2.3930 (14)	O10—H10A	0.8273
Na1—O13	2.4095 (16)	O10—H10B	0.8481

Na1—O9	2.4382 (14)	O11—Na2 ⁱⁱⁱ	2.3511 (15)
Na1—O10	2.4669 (15)	O11—H11A	0.8860
Na1—O11	2.5519 (16)	O11—H11B	0.8656
Na1—Na2 ⁱⁱⁱ	3.3901 (10)	O12—Na2 ⁱⁱⁱ	2.5105 (15)
Na2—O11 ^{iv}	2.3511 (15)	O12—H12A	0.8368
Na2—O6 ^v	2.4271 (14)	O12—H12B	0.8876
Na2—O2	2.4561 (14)	O13—H13A	0.8386
Na2—O5 ^{vi}	2.4814 (14)	O13—H13B	0.8789
Na2—O10	2.4825 (16)	C1—C2	1.508 (2)
Na2—O12 ^{iv}	2.5105 (15)	C2—C7	1.383 (2)
Na2—Na1 ^{iv}	3.3901 (10)	C2—C3	1.386 (2)
S—O6	1.4505 (12)	C3—C4	1.381 (2)
S—O5	1.4525 (13)	C3—H3	0.9300
S—O7	1.4645 (13)	C4—C5	1.386 (2)
S—C4	1.7680 (16)	C5—C6	1.391 (2)
O1—C1	1.266 (2)	C5—H5	0.9300
O2—C1	1.242 (2)	C6—C7	1.390 (2)
O3—C8	1.244 (2)	C6—C8	1.517 (2)
O4—C8	1.250 (2)	C7—H7	0.9300
O1 ⁱ —Ni—O1	180.0	O7—S—C4	107.03 (7)
O1 ⁱ —Ni—O9	87.70 (5)	C1—O1—Ni	130.04 (11)
O1—Ni—O9	92.30 (5)	C1—O2—Na2	160.79 (11)
O1 ⁱ —Ni—O9 ⁱ	92.30 (5)	S—O5—Na1 ^{vii}	136.10 (8)
O1—Ni—O9 ⁱ	87.70 (5)	S—O5—Na2 ^{vi}	132.45 (8)
O9—Ni—O9 ⁱ	180.000 (1)	Na1 ^{vii} —O5—Na2 ^{vi}	88.98 (5)
O1 ⁱ —Ni—O8 ⁱ	90.09 (5)	S—O6—Na2 ^{viii}	152.77 (8)
O1—Ni—O8 ⁱ	89.91 (5)	Ni—O8—H8A	120.7
O9—Ni—O8 ⁱ	91.97 (5)	Ni—O8—H8B	122.2
O9 ⁱ —Ni—O8 ⁱ	88.03 (5)	H8A—O8—H8B	107.8
O1 ⁱ —Ni—O8	89.91 (5)	Ni—O9—Na1	118.93 (6)
O1—Ni—O8	90.09 (5)	Ni—O9—H9A	114.0
O9—Ni—O8	88.03 (5)	Na1—O9—H9A	114.9
O9 ⁱ —Ni—O8	91.97 (5)	Ni—O9—H9B	104.1
O8 ⁱ —Ni—O8	180.0	Na1—O9—H9B	98.0
O5 ⁱⁱ —Na1—O12	79.14 (5)	H9A—O9—H9B	103.4
O5 ⁱⁱ —Na1—O13	85.05 (5)	Na1—O10—Na2	127.93 (6)
O12—Na1—O13	100.31 (5)	Na1—O10—H10A	119.3
O5 ⁱⁱ —Na1—O9	151.89 (5)	Na2—O10—H10A	99.6
O12—Na1—O9	87.31 (5)	Na1—O10—H10B	106.5
O13—Na1—O9	121.84 (5)	Na2—O10—H10B	96.3
O5 ⁱⁱ —Na1—O10	100.75 (5)	H10A—O10—H10B	102.5
O12—Na1—O10	160.96 (6)	Na2 ⁱⁱⁱ —O11—Na1	87.39 (5)
O13—Na1—O10	98.64 (5)	Na2 ⁱⁱⁱ —O11—H11A	122.8
O9—Na1—O10	84.27 (5)	Na1—O11—H11A	114.7

supplementary materials

O5 ⁱⁱ —Na1—O11	73.06 (5)	Na2 ⁱⁱⁱ —O11—H11B	126.9
O12—Na1—O11	79.75 (5)	Na1—O11—H11B	98.8
O13—Na1—O11	157.76 (5)	H11A—O11—H11B	102.2
O9—Na1—O11	80.39 (5)	Na1—O12—Na2 ⁱⁱⁱ	87.44 (5)
O10—Na1—O11	82.02 (5)	Na1—O12—H12A	102.6
O5 ⁱⁱ —Na1—Na2 ⁱⁱⁱ	47.04 (4)	Na2 ⁱⁱⁱ —O12—H12A	133.0
O12—Na1—Na2 ⁱⁱⁱ	47.71 (4)	Na1—O12—H12B	135.0
O13—Na1—Na2 ⁱⁱⁱ	121.18 (4)	Na2 ⁱⁱⁱ —O12—H12B	104.7
O9—Na1—Na2 ⁱⁱⁱ	106.32 (4)	H12A—O12—H12B	99.8
O10—Na1—Na2 ⁱⁱⁱ	119.01 (4)	Na1—O13—H13A	114.4
O11—Na1—Na2 ⁱⁱⁱ	43.85 (3)	Na1—O13—H13B	110.5
O11 ^{iv} —Na2—O6 ^v	100.89 (5)	H13A—O13—H13B	106.1
O11 ^{iv} —Na2—O2	97.43 (5)	O2—C1—O1	125.48 (15)
O6 ^v —Na2—O2	82.41 (5)	O2—C1—C2	118.99 (14)
O11 ^{iv} —Na2—O5 ^{vi}	74.44 (5)	O1—C1—C2	115.47 (14)
O6 ^v —Na2—O5 ^{vi}	169.25 (5)	C7—C2—C3	119.56 (15)
O2—Na2—O5 ^{vi}	107.62 (5)	C7—C2—C1	119.68 (15)
O11 ^{iv} —Na2—O10	158.30 (6)	C3—C2—C1	120.73 (14)
O6 ^v —Na2—O10	100.73 (5)	C4—C3—C2	119.12 (15)
O2—Na2—O10	83.55 (5)	C4—C3—H3	120.4
O5 ^{vi} —Na2—O10	84.57 (5)	C2—C3—H3	120.4
O11 ^{iv} —Na2—O12 ^{iv}	81.40 (5)	C3—C4—C5	121.77 (15)
O6 ^v —Na2—O12 ^{iv}	95.27 (5)	C3—C4—S	117.02 (12)
O2—Na2—O12 ^{iv}	177.18 (6)	C5—C4—S	121.10 (12)
O5 ^{vi} —Na2—O12 ^{iv}	74.58 (5)	C4—C5—C6	119.09 (15)
O10—Na2—O12 ^{iv}	98.50 (5)	C4—C5—H5	120.5
O11 ^{iv} —Na2—Na1 ^{iv}	48.76 (4)	C6—C5—H5	120.5
O6 ^v —Na2—Na1 ^{iv}	125.80 (4)	C7—C6—C5	119.09 (15)
O2—Na2—Na1 ^{iv}	135.72 (4)	C7—C6—C8	119.18 (14)
O5 ^{vi} —Na2—Na1 ^{iv}	43.98 (3)	C5—C6—C8	121.73 (15)
O10—Na2—Na1 ^{iv}	117.34 (4)	C2—C7—C6	121.34 (15)
O12 ^{iv} —Na2—Na1 ^{iv}	44.84 (3)	C2—C7—H7	119.3
O6—S—O5	113.27 (8)	C6—C7—H7	119.3
O6—S—O7	112.03 (8)	O3—C8—O4	124.58 (16)
O5—S—O7	111.61 (8)	O3—C8—C6	116.32 (15)
O6—S—C4	107.10 (7)	O4—C8—C6	119.10 (15)
O5—S—C4	105.26 (8)		

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x+3/2, y-1/2, -z+1/2$; (iii) $x+1/2, -y+1/2, z+1/2$; (iv) $x-1/2, -y+1/2, z-1/2$; (v) $-x+1/2, y-1/2, -z+1/2$; (vi) $-x+1, -y+1, -z$; (vii) $-x+3/2, y+1/2, -z+1/2$; (viii) $-x+1/2, y+1/2, -z+1/2$.

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
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O8—H8A…O13 ^{ix}	0.84	2.03	2.861 (2)	173
O8—H8B…O4 ^{vi}	0.85	1.99	2.8130 (19)	162
O9—H9A…O7 ^v	0.85	2.16	2.9854 (17)	163
O9—H9B…O2	0.84	1.82	2.6168 (17)	159
O10—H10A…O7 ^{vi}	0.83	2.04	2.8553 (19)	167
O10—H10B…O3 ⁱⁱⁱ	0.85	1.83	2.6615 (19)	165
O11—H11A…O7 ^v	0.89	1.90	2.7659 (18)	167
O11—H11B…O3 ⁱⁱⁱ	0.87	1.92	2.783 (2)	175
O12—H12A…O1 ⁱ	0.84	2.11	2.9470 (18)	173
O12—H12B…O4 ^x	0.89	2.04	2.8994 (19)	163
O13—H13A…O4 ^{vi}	0.84	1.94	2.733 (2)	157
O13—H13B…O6 ⁱⁱ	0.88	2.21	2.9486 (19)	141
C7—H7…O11 ^{iv}	0.93	2.50	3.371 (2)	157

Symmetry codes: (ix) $-x+2, -y+1, -z+1$; (vi) $-x+1, -y+1, -z$; (v) $-x+1/2, y-1/2, -z+1/2$; (iii) $x+1/2, -y+1/2, z+1/2$; (i) $-x+1, -y+1, -z+1$; (x) $x+1, y, z+1$; (ii) $-x+3/2, y-1/2, -z+1/2$; (iv) $x-1/2, -y+1/2, z-1/2$.

