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# Crystal structures of $\text{Na}_2\text{SeO}_4 \cdot 1.5\text{H}_2\text{O}$ and $\text{Na}_2\text{SeO}_4 \cdot 10\text{H}_2\text{O}$

Matthias Weil<sup>a\*</sup> and Barbara Bonneau<sup>b</sup><sup>a</sup>Institute for Chemical Technologies and Analytics, Division of Structural Chemistry, Vienna University of Technology, Getreidemarkt 9/164-SC, A-1060 Vienna, Austria, and <sup>b</sup>IUT Bordeaux 1, 15 Rue Naudet, 33175 Gradignan, France.

\*Correspondence e-mail: mweil@mail.zserv.tuwien.ac.at

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The crystal structures of  $\text{Na}_2\text{SeO}_4 \cdot 1.5\text{H}_2\text{O}$  (sodium selenate sesquihydrate) and  $\text{Na}_2\text{SeO}_4 \cdot 10\text{H}_2\text{O}$  (sodium selenate decahydrate) are isotypic with those of  $\text{Na}_2\text{CrO}_4 \cdot 1.5\text{H}_2\text{O}$  and  $\text{Na}_2\text{XSeO}_4 \cdot 10\text{H}_2\text{O}$  ( $X = \text{S}, \text{Cr}$ ), respectively. The asymmetric unit of the sesquihydrate contains two  $\text{Na}^+$  cations, one  $\text{SeO}_4$  tetrahedron and one and a half water molecules, the other half being generated by twofold rotation symmetry. The coordination polyhedra of the cations are a distorted monocapped octahedron and a square pyramid; these  $[\text{NaO}_x]$  polyhedra are linked through common edges and corners into a three-dimensional framework structure, the voids of which are filled with the Se atoms of the  $\text{SeO}_4$  tetrahedra. The structure is consolidated by  $\text{O}—\text{H} \cdots \text{O}$  hydrogen bonds between coordinating water molecules and framework O atoms. The asymmetric unit of the decahydrate consists of two  $\text{Na}^+$  cations, one  $\text{SeO}_4$  tetrahedron and ten water molecules. Both  $\text{Na}^+$  cations are octahedrally surrounded by water molecules and by edge-sharing condensed into zigzag chains extending parallel to  $[001]$ . The  $\text{SeO}_4$  tetrahedra and two uncoordinating water molecules are situated between the chains and are connected to the chains through an intricate network of medium-strength  $\text{O}—\text{H} \cdots \text{O}$  hydrogen bonds.

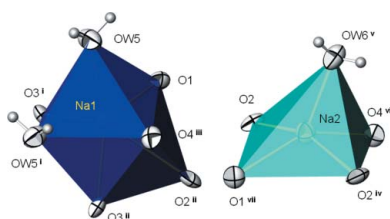
## 1. Chemical context

Based on recent studies in the system Na/Se/O/H that revealed dimorphism of the phases  $\text{NaHSeO}_4$  and  $\text{Na}_5\text{H}_3(\text{SeO}_4)_4 \cdot (\text{H}_2\text{O})_2$  (Pollitt & Weil, 2014), we became interested in the structure determination of hydrous phases of  $\text{Na}_2\text{SeO}_4$ . Although the first report of the decahydrate of  $\text{Na}_2\text{SeO}_4$  dates back to 1827 (Mitscherlich, 1827), a detailed structure report for this compound has not been published so far. Mitscherlich (1827) also recognized an isomorphic relationship of  $\text{Na}_2\text{SeO}_4 \cdot 10\text{H}_2\text{O}$  with  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$  (Glauber's salt or mirabilite as a mineral species). This relation was later confirmed by Rosický (1908) and by Ruben *et al.* (1961) on the basis of unit-cell determinations using diffraction methods. Another hydrous phase of  $\text{Na}_2\text{SeO}_4$  reported in the literature is the metastable heptahydrate that crystallized from super-saturated  $\text{Na}_2\text{SeO}_4$  solutions only when seeded with  $\text{Na}_2\text{SO}_4 \cdot 7\text{H}_2\text{O}$  nuclei below 293 K (Belarew, 1965).

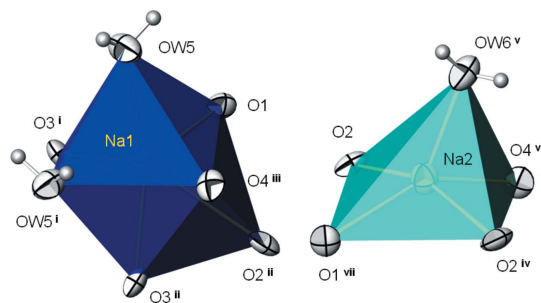
During crystallization studies of aqueous  $\text{Na}_2\text{SeO}_4$  solutions under different temperature conditions, we were able to isolate crystals not only of the decahydrate, but also of the sesquihydrate, the crystal structures of which are reported here.

## 2. Structural commentary

$\text{Na}_2\text{SeO}_4 \cdot 1.5\text{H}_2\text{O}$  is isotypic with the corresponding chromate (Kahlenberg, 2012) and is the second example of the



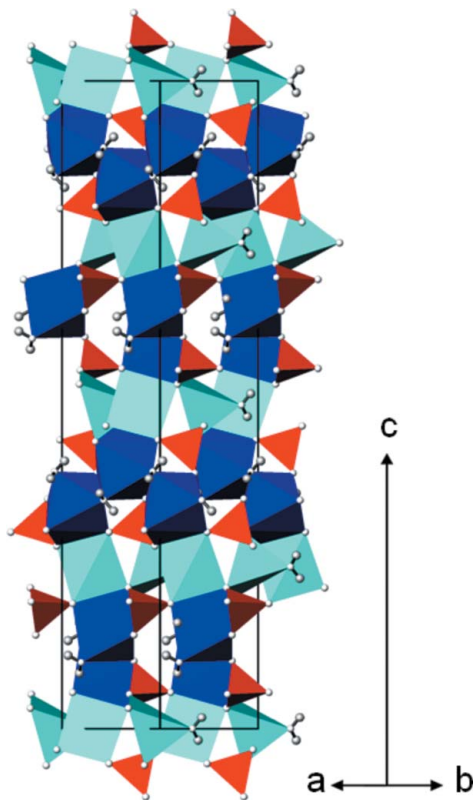
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**Figure 1**

The  $\text{NaO}_7$  and  $\text{NaO}_5$  polyhedra in the structure of  $\text{Na}_2\text{SO}_4 \cdot 1.5\text{H}_2\text{O}$ . Displacement parameters are drawn at the 99% probability level. [Symmetry codes: (i)  $x + \frac{1}{4}, y + \frac{1}{4}, -z + \frac{1}{4}$ ; (ii)  $x + \frac{1}{2}, y + \frac{1}{2}, z$ ; (iii)  $x + 1, y, z$ ; (iv)  $x - \frac{1}{2}, -y, -z + \frac{1}{2}$ ; (v)  $x - 1, y, z$ ; (vi)  $x, -y + \frac{1}{2}, -z + \frac{1}{2}$ ; (vii)  $x - \frac{1}{2}, y - \frac{1}{2}, z$ .]

$\text{Na}_2\text{XO}_4 \cdot 1.5\text{H}_2\text{O}$  structure family. The main building blocks of this structure type are distorted  $[\text{NaO}_5(\text{H}_2\text{O})_2]$  (Na1) mono-capped octahedra, distorted  $[\text{NaO}_4(\text{H}_2\text{O})]$  square pyramids (Na2) (Fig. 1) and rather regular  $\text{XO}_4$  ( $X = \text{Se}, \text{Cr}$ ) tetrahedra. These building blocks are linked through common corners and edges into a three-dimensional framework structure (Fig. 2). Hydrogen bonds of the type  $\text{O} - \text{H} \cdots \text{O}$  between the coordinating water molecules and parts of the framework O atoms provide additional stabilization (Table 1). The bond lengths (Table 2) and angles within the individual building blocks of



**Figure 2**

The crystal structure of  $\text{Na}_2\text{SO}_4 \cdot 1.5\text{H}_2\text{O}$  in a projection along  $[110]$ .  $\text{NaO}_5$  polyhedra are turquoise,  $\text{NaO}_7$  polyhedra are blue,  $\text{SeO}_4$  tetrahedra are red and H atoms are grey. Hydrogen bonds have been omitted for clarity.

**Table 1**

Hydrogen-bond geometry ( $\text{\AA}, ^\circ$ ) for  $1.5\text{-hydrate}$ .

$D - \text{H} \cdots A$	$D - \text{H}$	$\text{H} \cdots A$	$D \cdots A$	$D - \text{H} \cdots A$
$\text{OW5} - \text{H1} \cdots \text{O4}^{\text{viii}}$	0.82 (1)	2.13 (1)	2.922 (2)	164 (3)
$\text{OW5} - \text{H2} \cdots \text{O3}^{\text{ix}}$	0.82 (1)	2.08 (1)	2.891 (2)	169 (3)
$\text{OW6} - \text{H3} \cdots \text{O1}^{\text{vi}}$	0.82 (1)	1.90 (1)	2.703 (2)	167 (4)

Symmetry codes: (vi)  $x, -y + \frac{1}{2}, -z + \frac{1}{2}$ ; (viii)  $x + \frac{1}{2}, y - \frac{1}{2}, z$ ; (ix)  $x + \frac{3}{4}, y - \frac{1}{4}, -z + \frac{1}{4}$ .

**Table 2**

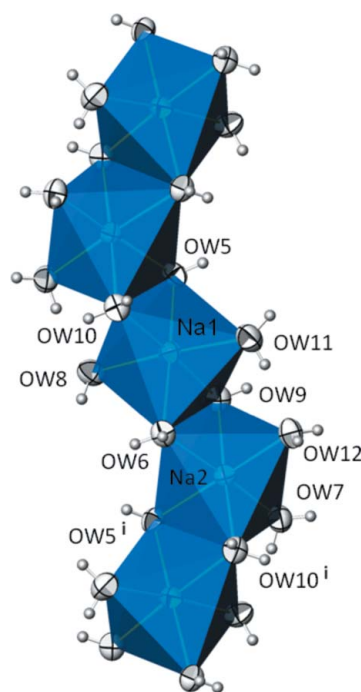
Selected bond lengths ( $\text{\AA}$ ) for  $1.5\text{-hydrate}$ .

$\text{Na1} - \text{OW5}$	2.3660 (18)	$\text{Na2} - \text{O2}^{\text{iv}}$	2.3301 (18)
$\text{Na1} - \text{O3}^{\text{i}}$	2.4157 (19)	$\text{Na2} - \text{OW6}^{\text{v}}$	2.3480 (18)
$\text{Na1} - \text{O1}$	2.4379 (18)	$\text{Na2} - \text{O4}^{\text{vi}}$	2.3651 (19)
$\text{Na1} - \text{O3}^{\text{ii}}$	2.4594 (16)	$\text{Na2} - \text{O1}^{\text{vii}}$	2.4103 (18)
$\text{Na1} - \text{OW5}^{\text{i}}$	2.465 (2)	$\text{Se1} - \text{O2}$	1.6350 (14)
$\text{Na1} - \text{O4}^{\text{iii}}$	2.6057 (19)	$\text{Se1} - \text{O3}$	1.6367 (14)
$\text{Na1} - \text{O2}^{\text{ii}}$	2.8475 (17)	$\text{Se1} - \text{O4}$	1.6451 (16)
$\text{Na2} - \text{O2}$	2.298 (2)	$\text{Se1} - \text{O1}$	1.6481 (15)

Symmetry codes: (i)  $x + \frac{1}{4}, y + \frac{1}{4}, -z + \frac{1}{4}$ ; (ii)  $x + \frac{1}{2}, y + \frac{1}{2}, z$ ; (iii)  $x + 1, y, z$ ; (iv)  $x - \frac{1}{2}, -y, -z + \frac{1}{2}$ ; (v)  $x - 1, y, z$ ; (vi)  $x, -y + \frac{1}{2}, -z + \frac{1}{2}$ ; (vii)  $x - \frac{1}{2}, y - \frac{1}{2}, z$ .

the selenate and chromate structures are more or less identical with mean distances of  $\text{SeO}_4 = 1.641$ ;  $\text{CrO}_4 = 1.651$ ;  $\text{Na1O}_7 = 2.514$  (selenate), 2.505 (chromate);  $\text{Na2O}_5 = 2.350$  (selenate), 2.360  $\text{\AA}$  (chromate).

Isotypism has been reported for several  $\text{Na}_2\text{XO}_4 \cdot 10\text{H}_2\text{O}$  ( $X = \text{S}, \text{Se}, \text{Cr}, \text{W}, \text{Mo}$ ) phases (Ruben *et al.*, 1961), but only the structures of  $X = \text{S}$  (Levy & Lisensky, 1978; Prescott *et al.*, 2001) and  $\text{Cr}$  (Kahlenberg, 2012) have been determined so far. As expected, the general structural set-up in the isotypic  $\text{Na}_2\text{XO}_4 \cdot 10\text{H}_2\text{O}$  structures is very similar. Each of the two  $\text{Na}^+$



**Figure 3**

A chain of edge-sharing  $\text{NaO}_6$  octahedra in the crystal structure of  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ . Displacement parameters are drawn at the 99% probability level. [Symmetry code: (i)  $x, -y - \frac{1}{2}, z - \frac{1}{2}$ .]

Table 3

Selected bond lengths (Å) for 10-hydrate.

Na1—OW5	2.3776 (6)	Na2—OW7	2.3935 (6)
Na1—OW6 <sup>i</sup>	2.4181 (6)	Na2—OW9	2.4325 (6)
Na1—OW11	2.4184 (6)	Na2—OW6	2.4415 (6)
Na1—OW10	2.4194 (6)	Na2—OW10 <sup>ii</sup>	2.4667 (6)
Na1—OW8 <sup>i</sup>	2.4473 (6)	Se1—O41	1.6335 (5)
Na1—OW9 <sup>i</sup>	2.4507 (6)	Se1—O31	1.6394 (5)
Na2—OW12	2.3814 (6)	Se1—O1	1.6398 (5)
Na2—OW5 <sup>ii</sup>	2.3891 (6)	Se1—O21	1.6421 (5)

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

cations is octahedrally surrounded [mean Na—O distance of the two octahedra is 2.420 Å (see Table 3); sulfate analogue (Prescott *et al.*, 2001): 2.415 Å; chromate analogue (Kahlenberg, 2012): 2.423 Å]. The [NaO<sub>6</sub>] octahedra are linked *via* edge-sharing into zigzag chains (Fig. 3) running parallel to [001]. These chains are linked with neighbouring chains and intermediate SeO<sub>4</sub> tetrahedra (mean Se—O distance 1.639; sulfate 1.488, chromate 1.647 Å) and non-coordinating lattice water molecules through O—H...O hydrogen bonds of medium strength (Table 4) to build up the crystal structure (Fig. 4). The most important difference between the structures of the three Na<sub>2</sub>XO<sub>4</sub>·10H<sub>2</sub>O ( $X = S, Se, Cr$ ) phases is the missing disorder of the XO<sub>4</sub> tetrahedron in the selenate compound that has been observed in the sulfate compound on the basis of single-crystal neutron data (Levy & Lisensky, 1978) and single-crystal X-ray data (Prescott *et al.*, 2001), or for the chromate compound on the basis of single-crystal X-ray data (Kahlenberg, 2012).

### 3. Synthesis and crystallization

Anhydrous Na<sub>2</sub>SeO<sub>4</sub> was prepared according to the method compiled by Brauer (1963) by adding a half-concentrated aqueous selenic acid solution (*ca* 60 wt%) to an excess of an Na<sub>2</sub>CO<sub>3</sub> solution. The resulting solution was heated until a

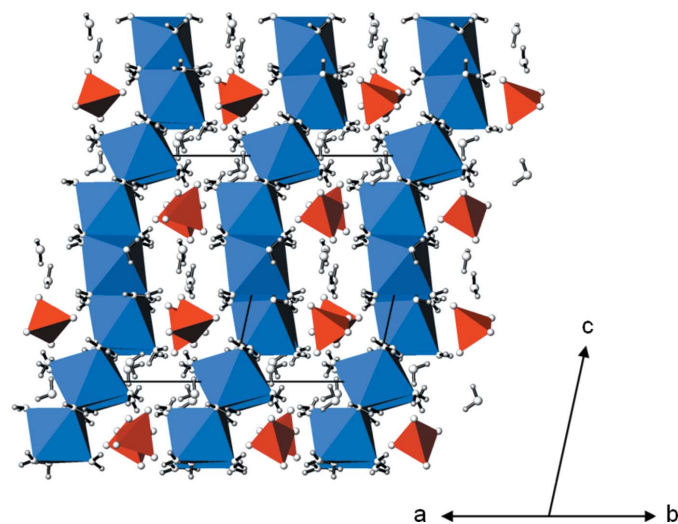


Figure 4

The crystal structure of Na<sub>2</sub>SO<sub>4</sub>·10H<sub>2</sub>O in a projection along [110]. NaO<sub>6</sub> polyhedra are light blue, SeO<sub>4</sub> tetrahedra are red, O atoms are white and H atoms are grey. Hydrogen bonds have been omitted for clarity.

Table 4

Hydrogen-bond geometry (Å, °) for 10-hydrate.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
OW5—H5A...O41	0.82 (1)	1.96 (1)	2.7570 (7)	164 (1)
OW5—H5B...OW13 <sup>iii</sup>	0.82 (1)	2.00 (1)	2.7980 (7)	165 (1)
OW6—H6A...OW14	0.82 (1)	2.02 (1)	2.8301 (7)	168 (1)
OW6—H6B...O41 <sup>ii</sup>	0.82 (1)	1.98 (1)	2.7791 (7)	166 (2)
OW7—H7A...O1 <sup>iv</sup>	0.82 (1)	1.97 (1)	2.7727 (7)	166 (1)
OW7—H7B...OW8 <sup>v</sup>	0.82 (1)	1.95 (1)	2.7542 (7)	168 (1)
OW8—H8A...O41 <sup>ii</sup>	0.82 (1)	1.95 (1)	2.7544 (7)	166 (1)
OW8—H8B...OW7 <sup>vi</sup>	0.82 (1)	1.99 (1)	2.8076 (7)	178 (1)
OW9—H9A...O1 <sup>vii</sup>	0.82 (1)	2.11 (1)	2.9152 (7)	168 (1)
OW9—H9B...OW13 <sup>viii</sup>	0.82 (1)	2.04 (1)	2.8596 (7)	177 (1)
OW10—H10A...OW14 <sup>ix</sup>	0.82 (1)	2.05 (1)	2.8686 (7)	178 (2)
OW10—H10B...O31 <sup>x</sup>	0.82 (1)	2.08 (1)	2.8920 (7)	174 (1)
OW11—H11A...O31	0.82 (1)	2.05 (1)	2.8604 (7)	171 (1)
OW11—H11B...OW12 <sup>i</sup>	0.82 (1)	1.96 (1)	2.7716 (8)	168 (1)
OW12—H12A...O21 <sup>iv</sup>	0.82 (1)	1.92 (1)	2.7359 (7)	179 (1)
OW12—H12B...OW11 <sup>viii</sup>	0.82 (1)	1.97 (1)	2.7818 (7)	173 (1)
OW13—H13A...O1 <sup>xi</sup>	0.82 (1)	1.98 (1)	2.7932 (7)	172 (1)
OW13—H13B...O21 <sup>x</sup>	0.82 (1)	1.98 (1)	2.7931 (7)	170 (1)
OW14—H14A...O21 <sup>xii</sup>	0.82 (1)	1.98 (1)	2.8002 (7)	174 (1)
OW14—H14B...O31 <sup>viii</sup>	0.82 (1)	2.00 (1)	2.8061 (7)	169 (1)

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

considerable amount of the neutralization product had crystallized. The crystal mush was then separated by suction filtration of the still-hot solution and dried in air. X-ray powder diffraction revealed a single-phase material. The Na<sub>2</sub>SeO<sub>4</sub> crystals were then dissolved in small amounts of water and kept at *ca* 300, 293 and 280 K until complete evaporation of the solvent. According to Rietveld refinements using TOPAS (Bruker, 2013) the product crystallized at 300 K consisted of Na<sub>2</sub>SeO<sub>4</sub> and Na<sub>2</sub>SeO<sub>4</sub>·1.5H<sub>2</sub>O in an approximate 9:1 weight ratio, the product crystallized at 290 K consisted of Na<sub>2</sub>SeO<sub>4</sub> and Na<sub>2</sub>SeO<sub>4</sub>·1.5H<sub>2</sub>O in an approximate 5:1 ratio, and the product crystallized at 280 K consisted of Na<sub>2</sub>SeO<sub>4</sub>, Na<sub>2</sub>SeO<sub>4</sub>·1.5H<sub>2</sub>O and Na<sub>2</sub>SeO<sub>4</sub>·10H<sub>2</sub>O in an approximate 5:4:1 ratio. The crystal forms of the three obtained phases were different and were used for separation. Crystals of the anhydrous phase had mainly a lath-like form, of the sesquihydrate a plate-like form, and of the decahydrate a pinacoidal form. All obtained hydrate phases tend to weather when stored under ambient conditions.

### 4. Refinement

Unit-cell determinations revealed isotopic relationships with the corresponding chromate phases (Kahlenberg, 2012). For better comparison of the isotopic structures, atom labels and the setting of the unit cells of the selenate compounds were retained, and the coordinates of the non-H atoms of the chromate structure were used as starting parameters for refinement [note that the unit cell of Na<sub>2</sub>CrO<sub>4</sub>·1.5H<sub>2</sub>O is given in the non-standard setting *F2dd* of space group No. 43 (standard setting *Fdd2*)]. The H atoms of the water molecules were located from difference maps and were refined with a common *U*<sub>iso</sub> parameter and a fixed O—H distance of 0.82 Å. Experimental details are given in Table 1.

**Table 5**  
Experimental details.

	1.5-hydrate	10-hydrate
Crystal data		
Chemical formula	Na <sub>2</sub> SeO <sub>4</sub> ·1.5H <sub>2</sub> O	Na <sub>2</sub> O <sub>4</sub> Se·10H <sub>2</sub> O
$M_r$	215.96	369.10
Crystal system, space group	Orthorhombic, $F2dd$	Monoclinic, $P2_1/c$
Temperature (K)	100	100
$a, b, c$ (Å)	6.7533 (8), 8.6299 (10), 35.206 (4)	11.5758 (6), 10.4911 (5), 12.9570 (7)
$\alpha, \beta, \gamma$ (°)	90, 90, 90	90, 107.995 (3), 90
$V$ (Å <sup>3</sup> )	2051.8 (4)	1496.56 (13)
$Z$	16	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$
$\mu$ (mm <sup>-1</sup> )	7.43	2.62
Crystal size (mm)	0.20 × 0.15 × 0.10	0.32 × 0.18 × 0.09
Data collection		
Diffractometer	Bruker SMART CCD	Bruker APEXII CCD
Absorption correction	Multi-scan ( <i>SADABS</i> ; Bruker, 2008)	Multi-scan ( <i>SADABS</i> ; Bruker, 2013)
$T_{\min}$ , $T_{\max}$	0.488, 0.584	0.642, 0.749
No. of measured, independent and observed [ $I > 2\sigma(I)$ ] reflections	8363, 1824, 1723	213856, 11218, 9196
$R_{\text{int}}$	0.032	0.054
$(\sin \theta/\lambda)_{\text{max}}$ (Å <sup>-1</sup> )	0.762	0.965
Refinement		
$R[F^2 > 2\sigma(F^2)]$ , $wR(F^2)$ , $S$	0.019, 0.042, 0.99	0.021, 0.046, 1.05
No. of reflections	1824	11218
No. of parameters	89	215
No. of restraints	4	20
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{\text{max}}$ , $\Delta\rho_{\text{min}}$ (e Å <sup>-3</sup> )	0.91, -0.37	0.48, -0.52
Absolute structure	Flack (1983), 823 Friedel pairs	—
Absolute structure parameter	0.025 (8)	—

Computer programs: *SMART*, *SAINT*, *SAINT-Plus* and *APEX2* (Bruker, 2013, 2013), *SHELXS97* and *SHELXL97* (Sheldrick, 2008), *ATOMS* (Dowty, 2006) and *pubCIF* (Westrip, 2010).

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## References

- Belarew, C. (1965). *Z. Anorg. Allg. Chem.* **336**, 92–95.
- Brauer, G. (1963). In *Handbook of Preparative Inorganic Chemistry*, Vol. 1, 2nd ed. New York, London: Academic Press.
- Bruker (2008). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bruker (2013). *APEX2*, *SAINT-Plus*, *SADABS* and *TOPAS*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Dowty, E. (2006). *ATOMS for Windows*. Shape Software, Kingsport, Tennessee, USA.
- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
- Kahlenberg, V. (2012). *Z. Kristallogr.* **227**, 621–628.
- Levy, H. A. & Lisensky, G. C. (1978). *Acta Cryst.* **B34**, 3502–3510.
- Mitscherlich, E. (1827). *Pogg. Ann.* **11**, 323–332.
- Pollitt, S. & Weil, M. (2014). *Z. Anorg. Allg. Chem.* doi:10.1002/zaac.201400068.
- Prescott, H. A., Troyanov, S. I. & Kemnitz, E. (2001). *J. Solid State Chem.* **156**, 415–421.
- Rosický, V. (1908). *Z. Kristallogr.* **45**, 473–489.
- Ruben, H. W., Templeton, D. H., Rosenstein, R. D. & Olovsson, I. (1961). *J. Am. Chem. Soc.* **83**, 820–824.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

## supporting information

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## Crystal structures of $\text{Na}_2\text{SeO}_4 \cdot 1.5\text{H}_2\text{O}$ and $\text{Na}_2\text{SeO}_4 \cdot 10\text{H}_2\text{O}$

Matthias Weil and Barbara Bonneau

### Computing details

Data collection: *SMART* (Bruker, 2008) for 1.5-hydrate; *APEX2* (Bruker, 2013) for 10-hydrate. Cell refinement: *SAINT* (Bruker, 2008) for 1.5-hydrate; *SAINT-Plus* (Bruker, 2013) for 10-hydrate. Data reduction: *SAINT* (Bruker, 2008) for 1.5-hydrate; *SAINT-Plus* (Bruker, 2013) for 10-hydrate. For both compounds, program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ATOMS* (Dowty, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

### (1.5-hydrate) Sodium selenate sesquihydrate

#### Crystal data

$\text{Na}_2\text{SeO}_4 \cdot 1.5\text{H}_2\text{O}$

$M_r = 215.96$

Orthorhombic, *F2dd*

Hall symbol: *F -2d 2*

$a = 6.7533$  (8) Å

$b = 8.6299$  (10) Å

$c = 35.206$  (4) Å

$V = 2051.8$  (4) Å<sup>3</sup>

$Z = 16$

$F(000) = 1648$

$D_x = 2.797$  Mg m<sup>-3</sup>

Mo *Kα* radiation,  $\lambda = 0.71073$  Å

Cell parameters from 4199 reflections

$\theta = 3.9\text{--}32.8^\circ$

$\mu = 7.43$  mm<sup>-1</sup>

$T = 100$  K

Fragment, colourless

$0.20 \times 0.15 \times 0.10$  mm

#### Data collection

Bruker SMART CCD  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\omega$  scan

Absorption correction: multi-scan

(*SADABS*; Bruker, 2008)

$T_{\min} = 0.488$ ,  $T_{\max} = 0.584$

8363 measured reflections

1824 independent reflections

1723 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.032$

$\theta_{\max} = 32.8^\circ$ ,  $\theta_{\min} = 2.3^\circ$

$h = -10 \rightarrow 9$

$k = -12 \rightarrow 12$

$l = -53 \rightarrow 53$

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.019$

$wR(F^2) = 0.042$

$S = 0.99$

1824 reflections

89 parameters

4 restraints

Primary atom site location: structure-invariant  
direct methods

Secondary atom site location: difference Fourier  
map

Hydrogen site location: inferred from  
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0148P)^2]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.91$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.37$  e Å<sup>-3</sup>

Absolute structure: Flack (1983), 823 Friedel pairs

Absolute structure parameter: 0.025 (8)

### 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 ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Se1	0.45783 (6)	0.26019 (2)	0.188204 (5)	0.00624 (5)
Na1	0.94725 (16)	0.40728 (9)	0.15499 (2)	0.01065 (16)
Na2	0.14581 (15)	0.04335 (11)	0.24656 (3)	0.01009 (18)
O1	0.6662 (2)	0.35383 (19)	0.19642 (4)	0.0091 (3)
O2	0.4372 (3)	0.11507 (17)	0.21780 (4)	0.0097 (3)
O3	0.4561 (3)	0.18930 (16)	0.14509 (4)	0.0096 (3)
O4	0.2752 (2)	0.38446 (19)	0.19358 (5)	0.0095 (3)
OW5	0.9382 (3)	0.13500 (18)	0.14734 (4)	0.0119 (3)
OW6	0.9204 (3)	0.2500	0.2500	0.0122 (5)
H1	0.898 (5)	0.076 (3)	0.1639 (7)	0.033 (6)*
H2	1.026 (4)	0.088 (4)	0.1364 (8)	0.033 (6)*
H3	0.844 (4)	0.232 (4)	0.2676 (7)	0.033 (6)*

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Se1	0.00707 (8)	0.00585 (8)	0.00581 (7)	−0.00002 (8)	0.00015 (9)	−0.00019 (7)
Na1	0.0128 (4)	0.0089 (4)	0.0103 (4)	−0.0009 (4)	0.0007 (4)	0.0004 (3)
Na2	0.0106 (4)	0.0103 (4)	0.0094 (4)	−0.0018 (3)	0.0014 (3)	−0.0007 (3)
O1	0.0081 (8)	0.0091 (7)	0.0100 (7)	−0.0028 (6)	0.0001 (6)	−0.0007 (6)
O2	0.0106 (8)	0.0081 (6)	0.0106 (6)	−0.0013 (6)	−0.0004 (6)	0.0046 (5)
O3	0.0128 (7)	0.0093 (6)	0.0068 (6)	−0.0010 (8)	−0.0005 (7)	−0.0033 (5)
O4	0.0101 (7)	0.0093 (7)	0.0091 (7)	0.0035 (6)	0.0010 (6)	−0.0011 (6)
OW5	0.0131 (9)	0.0096 (7)	0.0129 (7)	0.0004 (7)	0.0036 (7)	0.0014 (5)
OW6	0.0112 (15)	0.0156 (11)	0.0097 (9)	0.000	0.000	0.0036 (8)

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

Na1—OW5	2.3660 (18)	Na2—O2 <sup>iv</sup>	2.3301 (18)
Na1—O3 <sup>i</sup>	2.4157 (19)	Na2—OW6 <sup>v</sup>	2.3480 (18)
Na1—O1	2.4379 (18)	Na2—O4 <sup>vi</sup>	2.3651 (19)
Na1—O3 <sup>ii</sup>	2.4594 (16)	Na2—O1 <sup>vii</sup>	2.4103 (18)
Na1—OW5 <sup>i</sup>	2.465 (2)	Se1—O2	1.6350 (14)

Na1—O4 <sup>iii</sup>	2.6057 (19)	Se1—O3	1.6367 (14)
Na1—O2 <sup>ii</sup>	2.8475 (17)	Se1—O4	1.6451 (16)
Na2—O2	2.298 (2)	Se1—O1	1.6481 (15)
O2—Se1—O3	107.70 (7)	O2 <sup>iv</sup> —Na2—O4 <sup>vi</sup>	84.15 (6)
O2—Se1—O4	111.23 (9)	OW6 <sup>v</sup> —Na2—O4 <sup>vi</sup>	89.63 (6)
O3—Se1—O4	110.19 (9)	O2—Na2—O1 <sup>vii</sup>	79.12 (6)
O2—Se1—O1	109.66 (8)	O2 <sup>iv</sup> —Na2—O1 <sup>vii</sup>	91.76 (6)
O3—Se1—O1	110.61 (9)	OW6 <sup>v</sup> —Na2—O1 <sup>vii</sup>	126.16 (6)
O4—Se1—O1	107.47 (8)	O4 <sup>vi</sup> —Na2—O1 <sup>vii</sup>	144.12 (7)
OW5—Na1—O3 <sup>i</sup>	90.74 (6)	Se1—O1—Na2 <sup>ii</sup>	114.37 (8)
OW5—Na1—O1	81.96 (6)	Se1—O1—Na1	130.73 (9)
O3 <sup>i</sup> —Na1—O1	86.18 (7)	Na2 <sup>ii</sup> —O1—Na1	110.74 (7)
OW5—Na1—O3 <sup>ii</sup>	165.30 (6)	Se1—O2—Na2	124.06 (9)
O3 <sup>i</sup> —Na1—O3 <sup>ii</sup>	78.45 (5)	Se1—O2—Na2 <sup>viii</sup>	137.82 (10)
O1—Na1—O3 <sup>ii</sup>	106.93 (7)	Na2—O2—Na2 <sup>viii</sup>	97.03 (5)
OW5—Na1—OW5 <sup>i</sup>	81.63 (6)	Se1—O2—Na1 <sup>vii</sup>	89.17 (6)
O3 <sup>i</sup> —Na1—OW5 <sup>i</sup>	84.75 (6)	Na2—O2—Na1 <sup>vii</sup>	101.13 (6)
O1—Na1—OW5 <sup>i</sup>	161.11 (7)	Na2 <sup>viii</sup> —O2—Na1 <sup>vii</sup>	91.96 (6)
O3 <sup>ii</sup> —Na1—OW5 <sup>i</sup>	87.43 (6)	Se1—O3—Na1 <sup>ix</sup>	135.64 (11)
OW5—Na1—O4 <sup>iii</sup>	90.36 (7)	Se1—O3—Na1 <sup>vii</sup>	103.80 (7)
O3 <sup>i</sup> —Na1—O4 <sup>iii</sup>	164.16 (7)	Na1 <sup>ix</sup> —O3—Na1 <sup>vii</sup>	90.39 (6)
O1—Na1—O4 <sup>iii</sup>	109.61 (5)	Se1—O4—Na2 <sup>vi</sup>	123.44 (9)
O3 <sup>ii</sup> —Na1—O4 <sup>iii</sup>	97.35 (7)	Se1—O4—Na1 <sup>v</sup>	128.80 (9)
OW5 <sup>i</sup> —Na1—O4 <sup>iii</sup>	79.79 (6)	Na2 <sup>vi</sup> —O4—Na1 <sup>v</sup>	97.50 (6)
OW5—Na1—O2 <sup>ii</sup>	135.50 (6)	Na1—OW5—Na1 <sup>ix</sup>	91.42 (7)
O3 <sup>i</sup> —Na1—O2 <sup>ii</sup>	118.59 (6)	Na1—OW5—H1	123 (2)
O1—Na1—O2 <sup>ii</sup>	68.66 (5)	Na1 <sup>ix</sup> —OW5—H1	111 (2)
O3 <sup>ii</sup> —Na1—O2 <sup>ii</sup>	59.17 (5)	Na1—OW5—H2	122 (3)
OW5 <sup>i</sup> —Na1—O2 <sup>ii</sup>	130.17 (6)	Na1 <sup>ix</sup> —OW5—H2	100 (2)
O4 <sup>iii</sup> —Na1—O2 <sup>ii</sup>	70.31 (5)	H1—OW5—H2	106 (3)
O2—Na2—O2 <sup>iv</sup>	155.95 (7)	Na2 <sup>x</sup> —OW6—Na2 <sup>iii</sup>	99.15 (10)
O2—Na2—OW6 <sup>v</sup>	111.92 (6)	Na2 <sup>x</sup> —OW6—H3	121 (3)
O2 <sup>iv</sup> —Na2—OW6 <sup>v</sup>	91.48 (6)	Na2 <sup>iii</sup> —OW6—H3	108 (3)
O2—Na2—O4 <sup>vi</sup>	90.30 (7)		

Symmetry codes: (i)  $x+1/4, y+1/4, -z+1/4$ ; (ii)  $x+1/2, y+1/2, z$ ; (iii)  $x+1, y, z$ ; (iv)  $x-1/2, -y, -z+1/2$ ; (v)  $x-1, y, z$ ; (vi)  $x, -y+1/2, -z+1/2$ ; (vii)  $x-1/2, y-1/2, z$ ; (viii)  $x+1/2, -y, -z+1/2$ ; (ix)  $x-1/4, y-1/4, -z+1/4$ ; (x)  $x+1, -y+1/2, -z+1/2$ .

#### Hydrogen-bond geometry ( $\text{\AA}, ^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
OW5—H1 $\cdots$ O4 <sup>xi</sup>	0.82 (1)	2.13 (1)	2.922 (2)	164 (3)
OW5—H2 $\cdots$ O3 <sup>xii</sup>	0.82 (1)	2.08 (1)	2.891 (2)	169 (3)
OW6—H3 $\cdots$ O1 <sup>vi</sup>	0.82 (1)	1.90 (1)	2.703 (2)	167 (4)

Symmetry codes: (vi)  $x, -y+1/2, -z+1/2$ ; (xi)  $x+1/2, y-1/2, z$ ; (xii)  $x+3/4, y-1/4, -z+1/4$ .



**(10-hydrate) Sodium selenate decahydrate***Crystal data*Na<sub>2</sub>O<sub>4</sub>Se·10H<sub>2</sub>O $M_r = 369.10$ Monoclinic,  $P2_1/c$ 

Hall symbol: -P 2ybc

 $a = 11.5758$  (6) Å $b = 10.4911$  (5) Å $c = 12.9570$  (7) Å $\beta = 107.995$  (3)° $V = 1496.56$  (13) Å<sup>3</sup> $Z = 4$  $F(000) = 752$  $D_x = 1.638$  Mg m<sup>-3</sup>Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 9719 reflections

 $\theta = 2.7\text{--}40.1^\circ$  $\mu = 2.62$  mm<sup>-1</sup> $T = 100$  K

Fragment, colourless

 $0.32 \times 0.18 \times 0.09$  mm*Data collection*

Bruker APEXII CCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 $\omega$  and  $\phi$  scans

Absorption correction: multi-scan

(SADABS; Bruker, 2013)

 $T_{\min} = 0.642$ ,  $T_{\max} = 0.749$ 

213856 measured reflections

11218 independent reflections

9196 reflections with  $I > 2\sigma(I)$  $R_{\text{int}} = 0.054$  $\theta_{\max} = 43.3^\circ$ ,  $\theta_{\min} = 1.9^\circ$  $h = -22 \rightarrow 22$  $k = -20 \rightarrow 20$  $l = -24 \rightarrow 24$ *Refinement*Refinement on  $F^2$ 

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.021$  $wR(F^2) = 0.046$  $S = 1.05$ 

11218 reflections

215 parameters

20 restraints

Primary atom site location: structure-invariant

direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.017P)^2 + 0.2899P]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\max} = 0.006$  $\Delta\rho_{\max} = 0.48$  e Å<sup>-3</sup> $\Delta\rho_{\min} = -0.52$  e Å<sup>-3</sup>*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<sup>2</sup>)*

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
Se1	0.752121 (5)	0.139467 (5)	0.740658 (4)	0.00723 (1)
Na1	0.74307 (2)	-0.24486 (3)	0.97851 (2)	0.01112 (5)
Na2	0.75630 (2)	-0.11228 (3)	0.23666 (2)	0.01081 (4)
O1	0.86910 (4)	0.19770 (5)	0.83551 (4)	0.01211 (7)



O21	0.73195 (4)	0.21974 (4)	0.62765 (4)	0.01245 (8)
O31	0.63136 (4)	0.15034 (5)	0.78083 (4)	0.01257 (7)
O41	0.77699 (5)	−0.00971 (4)	0.71826 (4)	0.01330 (8)
OW5	0.85363 (4)	−0.21683 (5)	0.85304 (4)	0.01236 (7)
OW6	0.64473 (4)	−0.27987 (5)	0.11583 (4)	0.01269 (8)
OW7	0.87675 (5)	0.03985 (5)	0.36196 (4)	0.01443 (8)
OW8	0.87052 (5)	−0.42968 (5)	0.05311 (4)	0.01472 (8)
OW9	0.88112 (4)	−0.10907 (5)	0.11623 (4)	0.01306 (8)
OW10	0.61813 (4)	−0.39325 (5)	0.84832 (4)	0.01310 (8)
OW11	0.61541 (5)	−0.06034 (5)	0.91589 (4)	0.01584 (9)
OW12	0.63321 (5)	0.04187 (5)	0.11747 (4)	0.01397 (8)
OW13	0.09783 (5)	−0.14961 (5)	0.94520 (4)	0.01473 (8)
OW14	0.39997 (5)	−0.34873 (5)	0.08502 (4)	0.01467 (8)
H5A	0.8345 (13)	−0.1472 (6)	0.8240 (11)	0.0412 (9)*
H5B	0.9267 (3)	−0.2110 (14)	0.8845 (10)	0.0412 (9)*
H6A	0.5712 (2)	−0.2905 (14)	0.1003 (11)	0.0412 (9)*
H6B	0.6722 (13)	−0.3481 (7)	0.1436 (11)	0.0412 (9)*
H7A	0.8618 (13)	0.1162 (3)	0.3535 (12)	0.0412 (9)*
H7B	0.8709 (12)	0.0169 (13)	0.4206 (5)	0.0412 (9)*
H8A	0.8415 (11)	−0.4602 (12)	0.0978 (8)	0.0412 (9)*
H8B	0.9440 (2)	−0.4401 (13)	0.0788 (10)	0.0412 (9)*
H9A	0.9508 (4)	−0.1364 (12)	0.1394 (11)	0.0412 (9)*
H9B	0.8892 (13)	−0.0359 (5)	0.0973 (11)	0.0412 (9)*
H10A	0.6143 (13)	−0.4669 (4)	0.8683 (11)	0.0412 (9)*
H10B	0.5473 (4)	−0.3767 (12)	0.8146 (10)	0.0412 (9)*
H11A	0.6205 (12)	−0.0062 (10)	0.8719 (8)	0.0412 (9)*
H11B	0.6254 (12)	−0.0213 (11)	0.9728 (6)	0.0412 (9)*
H12A	0.6618 (12)	0.1138 (5)	0.1205 (12)	0.0412 (9)*
H12B	0.5615 (3)	0.0506 (13)	0.1131 (11)	0.0412 (9)*
H13A	0.1146 (13)	−0.1610 (13)	1.0108 (2)	0.0412 (9)*
H13B	0.1423 (10)	−0.1952 (11)	0.9226 (10)	0.0412 (9)*
H14A	0.3586 (11)	−0.3337 (13)	0.0224 (4)	0.0412 (9)*
H14B	0.3809 (12)	−0.2944 (10)	0.1223 (9)	0.0412 (9)*

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Se1	0.00796 (2)	0.00652 (2)	0.00716 (2)	0.00002 (2)	0.00228 (1)	0.00003 (2)
Na1	0.01190 (11)	0.01128 (11)	0.01052 (11)	−0.00036 (9)	0.00396 (9)	−0.00045 (8)
Na2	0.01176 (11)	0.01011 (10)	0.01050 (10)	0.00016 (9)	0.00333 (8)	0.00013 (8)
O1	0.00993 (17)	0.01300 (18)	0.01144 (17)	−0.00124 (14)	0.00041 (14)	−0.00188 (14)
O21	0.0162 (2)	0.01156 (18)	0.00963 (17)	0.00085 (15)	0.00404 (15)	0.00315 (14)
O31	0.00980 (17)	0.0156 (2)	0.01373 (18)	−0.00012 (15)	0.00571 (14)	−0.00071 (15)
O41	0.0190 (2)	0.00658 (16)	0.01498 (19)	0.00140 (15)	0.00617 (16)	−0.00054 (14)
OW5	0.01162 (18)	0.01215 (18)	0.01230 (18)	−0.00102 (15)	0.00222 (14)	0.00144 (14)
OW6	0.01137 (18)	0.01229 (19)	0.01431 (19)	−0.00005 (15)	0.00381 (15)	0.00107 (14)
OW7	0.0160 (2)	0.01212 (19)	0.01404 (19)	−0.00039 (16)	0.00291 (16)	0.00042 (15)
OW8	0.01307 (19)	0.0172 (2)	0.0149 (2)	0.00141 (16)	0.00583 (16)	0.00232 (16)

OW9	0.01073 (18)	0.01301 (18)	0.0154 (2)	0.00022 (14)	0.00393 (15)	0.00012 (15)
OW10	0.01081 (18)	0.01338 (18)	0.01408 (19)	−0.00065 (15)	0.00237 (15)	0.00038 (15)
OW11	0.0166 (2)	0.0150 (2)	0.0176 (2)	0.00205 (17)	0.00788 (17)	0.00367 (16)
OW12	0.01201 (19)	0.01201 (19)	0.0171 (2)	−0.00065 (15)	0.00339 (16)	0.00122 (15)
OW13	0.01312 (19)	0.0176 (2)	0.01342 (19)	0.00137 (16)	0.00410 (15)	0.00051 (16)
OW14	0.0148 (2)	0.0152 (2)	0.01333 (19)	0.00127 (16)	0.00335 (15)	−0.00077 (15)

*Geometric parameters (Å, °)*

Na1—OW5	2.3776 (6)	Na2—OW7	2.3935 (6)
Na1—OW6 <sup>i</sup>	2.4181 (6)	Na2—OW9	2.4325 (6)
Na1—OW11	2.4184 (6)	Na2—OW6	2.4415 (6)
Na1—OW10	2.4194 (6)	Na2—OW10 <sup>ii</sup>	2.4667 (6)
Na1—OW8 <sup>i</sup>	2.4473 (6)	Se1—O41	1.6335 (5)
Na1—OW9 <sup>i</sup>	2.4507 (6)	Se1—O31	1.6394 (5)
Na2—OW12	2.3814 (6)	Se1—O1	1.6398 (5)
Na2—OW5 <sup>ii</sup>	2.3891 (6)	Se1—O21	1.6421 (5)
O41—Se1—O31	109.72 (2)	Na1—OW5—H5A	107.3 (10)
O41—Se1—O1	109.88 (3)	Na2 <sup>iii</sup> —OW5—H5A	111.8 (10)
O31—Se1—O1	108.89 (2)	Na1—OW5—H5B	111.1 (10)
O41—Se1—O21	108.50 (2)	Na2 <sup>iii</sup> —OW5—H5B	125.7 (10)
O31—Se1—O21	110.31 (2)	H5A—OW5—H5B	104.6 (14)
O1—Se1—O21	109.53 (2)	Na1 <sup>iv</sup> —OW6—Na2	94.959 (19)
OW5—Na1—OW6 <sup>i</sup>	175.60 (2)	Na1 <sup>iv</sup> —OW6—H6A	122.0 (10)
OW5—Na1—OW11	94.230 (19)	Na2—OW6—H6A	124.0 (10)
OW6 <sup>i</sup> —Na1—OW11	89.467 (19)	Na1 <sup>iv</sup> —OW6—H6B	104.4 (10)
OW5—Na1—OW10	86.270 (19)	Na2—OW6—H6B	106.9 (10)
OW6 <sup>i</sup> —Na1—OW10	95.70 (2)	H6A—OW6—H6B	102.8 (13)
OW11—Na1—OW10	96.28 (2)	Na2—OW7—H7A	120.4 (10)
OW5—Na1—OW8 <sup>i</sup>	88.961 (19)	Na2—OW7—H7B	103.6 (10)
OW6 <sup>i</sup> —Na1—OW8 <sup>i</sup>	87.276 (19)	H7A—OW7—H7B	109.7 (14)
OW11—Na1—OW8 <sup>i</sup>	176.39 (2)	Na1 <sup>iv</sup> —OW8—H8A	105.1 (10)
OW10—Na1—OW8 <sup>i</sup>	85.59 (2)	Na1 <sup>iv</sup> —OW8—H8B	133.6 (10)
OW5—Na1—OW9 <sup>i</sup>	93.341 (19)	H8A—OW8—H8B	105.2 (13)
OW6 <sup>i</sup> —Na1—OW9 <sup>i</sup>	84.368 (19)	Na2—OW9—Na1 <sup>iv</sup>	94.36 (2)
OW11—Na1—OW9 <sup>i</sup>	88.42 (2)	Na2—OW9—H9A	118.2 (10)
OW10—Na1—OW9 <sup>i</sup>	175.30 (2)	Na1 <sup>iv</sup> —OW9—H9A	114.2 (10)
OW8 <sup>i</sup> —Na1—OW9 <sup>i</sup>	89.72 (2)	Na2—OW9—H9B	110.5 (10)
OW12—Na2—OW5 <sup>ii</sup>	171.87 (2)	Na1 <sup>iv</sup> —OW9—H9B	115.8 (10)
OW12—Na2—OW7	95.37 (2)	H9A—OW9—H9B	104.3 (13)
OW5 <sup>ii</sup> —Na2—OW7	90.57 (2)	Na1—OW10—Na2 <sup>iii</sup>	92.153 (19)
OW12—Na2—OW9	85.96 (2)	Na1—OW10—H10A	117.7 (10)
OW5 <sup>ii</sup> —Na2—OW9	99.063 (19)	Na2 <sup>iii</sup> —OW10—H10A	108.3 (10)
OW7—Na2—OW9	95.10 (2)	Na1—OW10—H10B	121.3 (10)
OW12—Na2—OW6	88.92 (2)	Na2 <sup>iii</sup> —OW10—H10B	113.9 (10)
OW5 <sup>ii</sup> —Na2—OW6	85.252 (19)	H10A—OW10—H10B	103.0 (13)
OW7—Na2—OW6	175.61 (2)	Na1—OW11—H11A	128.3 (10)

OW9—Na2—OW6	84.260 (19)	Na1—OW11—H11B	101.3 (10)
OW12—Na2—OW10 <sup>ii</sup>	89.884 (19)	H11A—OW11—H11B	105.1 (13)
OW5 <sup>ii</sup> —Na2—OW10 <sup>ii</sup>	84.965 (19)	Na2—OW12—H12A	116.2 (10)
OW7—Na2—OW10 <sup>ii</sup>	86.209 (19)	Na2—OW12—H12B	120.7 (10)
OW9—Na2—OW10 <sup>ii</sup>	175.74 (2)	H12A—OW12—H12B	106.6 (13)
OW6—Na2—OW10 <sup>ii</sup>	94.743 (19)	H13A—OW13—H13B	108.1 (13)
Na1—OW5—Na2 <sup>iii</sup>	95.178 (19)	H14A—OW14—H14B	105.5 (13)

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

### Hydrogen-bond geometry ( $\text{\AA}$ , $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
OW5—H5A $\cdots$ O41	0.82 (1)	1.96 (1)	2.7570 (7)	164 (1)
OW5—H5B $\cdots$ OW13 <sup>v</sup>	0.82 (1)	2.00 (1)	2.7980 (7)	165 (1)
OW6—H6A $\cdots$ OW14	0.82 (1)	2.02 (1)	2.8301 (7)	168 (1)
OW6—H6B $\cdots$ O41 <sup>ii</sup>	0.82 (1)	1.98 (1)	2.7791 (7)	166 (2)
OW7—H7A $\cdots$ O1 <sup>vi</sup>	0.82 (1)	1.97 (1)	2.7727 (7)	166 (1)
OW7—H7B $\cdots$ OW8 <sup>iii</sup>	0.82 (1)	1.95 (1)	2.7542 (7)	168 (1)
OW8—H8A $\cdots$ O41 <sup>ii</sup>	0.82 (1)	1.95 (1)	2.7544 (7)	166 (1)
OW8—H8B $\cdots$ OW7 <sup>vii</sup>	0.82 (1)	1.99 (1)	2.8076 (7)	178 (1)
OW9—H9A $\cdots$ O1 <sup>viii</sup>	0.82 (1)	2.11 (1)	2.9152 (7)	168 (1)
OW9—H9B $\cdots$ OW13 <sup>ix</sup>	0.82 (1)	2.04 (1)	2.8596 (7)	177 (1)
OW10—H10A $\cdots$ OW14 <sup>x</sup>	0.82 (1)	2.05 (1)	2.8686 (7)	178 (2)
OW10—H10B $\cdots$ O31 <sup>xi</sup>	0.82 (1)	2.08 (1)	2.8920 (7)	174 (1)
OW11—H11A $\cdots$ O31	0.82 (1)	2.05 (1)	2.8604 (7)	171 (1)
OW11—H11B $\cdots$ OW12 <sup>i</sup>	0.82 (1)	1.96 (1)	2.7716 (8)	168 (1)
OW12—H12A $\cdots$ O21 <sup>vi</sup>	0.82 (1)	1.92 (1)	2.7359 (7)	179 (1)
OW12—H12B $\cdots$ OW11 <sup>ix</sup>	0.82 (1)	1.97 (1)	2.7818 (7)	173 (1)
OW13—H13A $\cdots$ O1 <sup>xii</sup>	0.82 (1)	1.98 (1)	2.7932 (7)	172 (1)
OW13—H13B $\cdots$ O21 <sup>xi</sup>	0.82 (1)	1.98 (1)	2.7931 (7)	170 (1)
OW14—H14A $\cdots$ O21 <sup>xiii</sup>	0.82 (1)	1.98 (1)	2.8002 (7)	174 (1)
OW14—H14B $\cdots$ O31 <sup>ix</sup>	0.82 (1)	2.00 (1)	2.8061 (7)	169 (1)

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