# Re-refinement of sodium ammonium sulfate dihydrate at 170 K

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The title compound, sodium ammonium sulfate dihydrate (SASD), $NaNH_4SO_4\cdot 2H_2O$, a synthetic analogue of the mineral lecontite, is a well known ferroelectric. The crystal structure of the paraelectric phase has been re-refined at 170 K on the basis of single-crystal X-ray data, improving the previous study [Arzt & Glazer (1994). *Acta Cryst.* B**50**, 425–431] in terms of accuracy regarding hydrogen-atom positions and thus details of the hydrogen bonding. $O-H\cdots O$ and $N-H\cdots O$ hydrogen bonds between the principal building units [$Na(OH_2)_4O_2$ octahedra, $SO_4$ tetrahedra and ammonium cations] constitute a three-dimensional network structure.

**3D view**
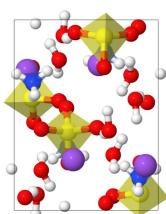


## Structure description

The title compound, sodium ammonium sulfate dihydrate (SASD), $NaNH_4SO_4\cdot 2H_2O$, is the synthetic analogue of the mineral lecontite (Hawthorne *et al.*, 2000), as revealed by Faust & Bloss (1963) through a diffractometry study of both synthetic and natural material. The crystal structure of SASD was first determined by Corazza *et al.* (1967) from equi-inclination Weissenberg photographs at room temperature. Arzt & Glazer (1994) redetermined the crystal structure at room temperature based on serial detector data. Properties of the well-known ferroelectric SASD have been widely studied (Arzt & Glazer, 1994; Fawcett *et al.*, 1975; Genin & O'Reilly, 1969; Hilczer *et al.*, 1991, 1992, 1993; Kanesaka & Ozaki, 1994; Kassem & Hedewy, 1988; Kloprogge *et al.*, 2006; Lipinski *et al.*, 2003; Lipinski & Kuriata, 2005; Ono *et al.*, 1993; Osaka, 1978; Osaka & Makita, 1970; Ribeiro *et al.*, 2006). Kloprogge *et al.* (2006) also reported a Rietveld refinement of the structure of SASD at room temperature, thereby confirming the results of the previous single-crystal X-ray analyses. We have now re-refined the crystal structure of the para-lectric phase of SASD at 170 K on the basis of single-crystal X-ray diffraction data.

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| Na1—O2$^i$ | 2.3229 (15) | Na1—O4$^i$ | 2.4546 (14) |
| Na1—O4 | 2.3733 (14) | S1—O5 | 1.4628 (14) |
| Na1—O2 | 2.4054 (16) | S1—O4 | 1.4721 (12) |
| Na1—O1 | 2.4087 (15) | S1—O3 | 1.4728 (15) |
| Na1—O1$^i$ | 2.4389 (16) | S1—O6 | 1.4740 (15) |
| | | | |
| O2$^i$—Na1—O4 | 111.05 (5) | O4—Na1—O4$^i$ | 167.55 (5) |
| O2$^i$—Na1—O2 | 166.61 (6) | O2—Na1—O4$^i$ | 86.58 (5) |
| O4—Na1—O2 | 81.31 (5) | O1—Na1—O4$^i$ | 92.00 (5) |
| O2$^i$—Na1—O1 | 103.97 (6) | O1$^i$—Na1—O4$^i$ | 81.23 (5) |
| O4—Na1—O1 | 83.54 (5) | O5—S1—O4 | 109.42 (8) |
| O2—Na1—O1 | 81.97 (5) | O5—S1—O3 | 109.02 (11) |
| O2$^i$—Na1—O1$^i$ | 83.03 (5) | O4—S1—O3 | 110.07 (8) |
| O4—Na1—O1$^i$ | 101.42 (5) | O5—S1—O6 | 109.17 (11) |
| O2—Na1—O1$^i$ | 89.58 (5) | O4—S1—O6 | 109.83 (8) |
| O1—Na1—O1$^i$ | 169.50 (3) | O3—S1—O6 | 109.31 (9) |
| O2$^i$—Na1—O4$^i$ | 81.28 (5) | | |

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

**Table 2**
Hydrogen-bond geometry (Å, °).

| $D$—H$\cdots A$ | $D$—H | H$\cdots A$ | $D\cdots A$ | $D$—H$\cdots A$ |
|---|---|---|---|---|
| O1—H1$A\cdots$O6$^{ii}$ | 0.84 (2) | 1.99 (2) | 2.812 (2) | 165 (3) |
| O1—H1$B\cdots$O5$^{iii}$ | 0.85 (2) | 1.89 (2) | 2.7439 (19) | 175 (3) |
| O2—H2$A\cdots$O3$^{iv}$ | 0.81 (2) | 1.98 (2) | 2.781 (2) | 171 (3) |
| O2—H2$B\cdots$O6$^i$ | 0.81 (2) | 1.98 (2) | 2.781 (2) | 175 (3) |
| N1—H1$C\cdots$O4 | 0.81 (2) | 2.14 (2) | 2.948 (2) | 172 (3) |
| N1—H1$D\cdots$O3$^{iv}$ | 0.83 (2) | 2.03 (2) | 2.854 (2) | 172 (3) |
| N1—H1$E\cdots$O3$^v$ | 0.81 (2) | 2.23 (2) | 2.977 (2) | 152 (3) |
| N1—H1$E\cdots$O5$^v$ | 0.81 (2) | 2.60 (3) | 3.324 (3) | 149 (3) |
| N1—H1$F\cdots$O5$^{vi}$ | 0.81 (2) | 2.58 (3) | 3.245 (3) | 140 (3) |
| N1—H1$F\cdots$O6$^{vi}$ | 0.81 (2) | 2.13 (2) | 2.897 (3) | 157 (3) |

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

As shown in Fig. 1, the sodium cation is hexa-coordinated with a considerably distorted octahedral coordination sphere formed by four water molecules in the equatorial plane and two sulfate O atoms in the apical positions. Selected bond lengths and angles are listed in Table 1. Each of the ligands links two sodium cations in a $\mu$-coordination mode, resulting in chains along the [100] direction with the Na cations located near to a $2_1$ screw axis. Na1$\cdots$Na1$^i$ and Na1$\cdots$Na1$^{ii}$ are separated by 3.1317 (2) and 3.1316 (2) Å, respectively [symmetry codes: (i) $x - \frac{1}{2}, -y + \frac{3}{2}, -z + 1$; (ii) $x + \frac{1}{2}, -y + \frac{3}{2}, -z + 1$]. The chains can be described as consisting of NaO$_6$ octahedra sharing one face (Fig. 2) defined by atoms O1, O2 and O4. The sulfate anion exhibits the typical tetrahedral shape with an r.m.s. deviation from exact $T_d$ symmetry of only

0.0092 Å, as calculated with MOLSYM in *PLATON* (Spek, 2020). In the chains, the SO$_4$ tetrahedra have one O atom in common with a pair of NaO$_6$ octahedra. Chain motifs are encountered in the structures of many other sulfates (Gorogotskaya & Bokii, 1973).

The crystal structure features hydrogen bonds of the O—H$\cdots$O and N—H$\cdots$O type (Table 2). The water molecules form medium–strong and nearly linear intra- and interchain hydrogen bonds to sulfate oxygen atoms. The interstices between the [Na($\mu$-SO$_4$)($\mu$-H$_2$O)$_2$]$_n^-$ chains accommodate the ammonium cations, which form hydrogen bonds to sulfate oxygen atoms, thus establishing a three-dimensional network. The positions of the ammonium hydrogen atoms determined in the current study appear to be more accurate than those in the room-temperature structure reported by Arzt & Glazer (1994). Note that details of hydrogen bonding were not discussed in the latter report; based on the reported structure data (Arzt & Glazer, 1994), N—H distances range between 0.73 and 0.99 Å. Corazza *et al.* (1967) did not refine hydrogen-



**Figure 1**
Section of the crystal structure of SASD, viewed approximately along the *c*-axis direction towards the origin. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen atoms are represented by small spheres of arbitrary radius. Thick dashed lines represent hydrogen bonds. [Symmetry codes: (i) $x + \frac{1}{2}, -y + \frac{3}{2}, -z + 1$; (ii) $x - \frac{1}{2}, -y + \frac{3}{2}, -z + 1$.]
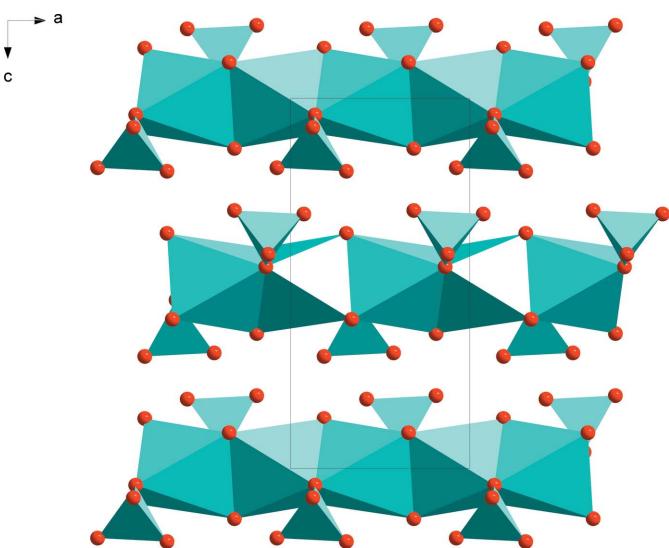


**Figure 2**
The crystal structure of SASD viewed down the *b*-axis direction, showing the chains featuring face-sharing NaO$_6$ octahedra with appended SO$_4$ tetrahedra. Ammonium ions are omitted for clarity.

atom parameters in the original room-temperature structure determination but included their presumed positions in the structure-factor calculation for the final refinement of the non-hydrogen atoms. In the current study, semi-free refinement applying only similarity restraints on the 1,2-distances involving hydrogen atoms resulted in reasonable hydrogen-atom parameters and a sensible hydrogen-bonding scheme.

The absolute structure of the crystal was established by anomalous-dispersion effects in the diffraction data, as indicated by a Flack $x$ parameter close to zero with a reasonably small standard uncertainty (Table 3). The Hooft $y$ parameter (Hooft *et al.*, 2008), as calculated with *PLATON*, is 0.07 (2). Interestingly, the structure exhibits chirality opposite to the previously reported room temperature structures (Corazza *et al.*, 1967; Arzt & Glazer, 1994).

## Synthesis and crystallization

A crystal of the title compound suitable for single-crystal X-ray analysis was obtained unintentionally from a solution in an acetonitrile/water mixture after synthesis of an organic compound. Ammonium ions and sodium sulfate in this mixture originated from an employed reagent and the drying agent, respectively.

## Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3.

## Acknowledgements

## Funding information

**Table 3**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | NaNH₄SO₄·2H₂O |
| $M_r$ | 173.12 |
| Crystal system, space group | Orthorhombic, $P2_12_12_1$ |
| Temperature (K) | 170 |
| $a, b, c$ (Å) | 6.2001 (2), 8.1917 (3), 12.8121 (6) |
| $V$ (Å³) | 650.72 (4) |
| $Z$ | 4 |
| Radiation type | Mo $K\alpha$ |
| $\mu$ (mm⁻¹) | 0.53 |
| Crystal size (mm) | 0.50 × 0.48 × 0.28 |
| | |
| Data collection | |
| Diffractometer | Stoe IPDS 2T |
| Absorption correction | Multi-scan [*MULABS* (Blessing, 1995) in *PLATON* (Spek, 2020)] |
| $T_{min}$, $T_{max}$ | 0.728, 1.117 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 14489, 1754, 1690 |
| $R_{int}$ | 0.038 |
| | |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.023, 0.058, 1.09 |
| No. of reflections | 1754 |
| No. of parameters | 114 |
| No. of restraints | 12 |
| H-atom treatment | All H-atom parameters refined |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å⁻³) | 0.20, −0.34 |
| Absolute structure | Flack $x$ determined using 672 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013) |
| Absolute structure parameter | −0.05 (2) |

Computer programs: *X-AREA* and *X-RED* (Stoe & Cie, 2016), *SHELXT* (Sheldrick, 2015a), *SHELXL2018* (Sheldrick, 2015b), *DIAMOND* (Brandenburg, 2018), *enCIFer* (Allen *et al.*, 2004) and *publCIF* (Westrip, 2010).

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# full crystallographic data

## Re-refinement of sodium ammonium sulfate dihydrate at 170 K

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**Ammonium sodium sulfate dihydrate**

*Crystal data*
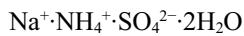
| | |
|---|---|
| $Na^+ \cdot NH_4^+ \cdot SO_4^{2-} \cdot 2H_2O$ | $D_x = 1.767$ Mg m$^{-3}$ |
| $M_r = 173.12$ | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| Orthorhombic, $P2_12_12_1$ | Cell parameters from 15574 reflections |
| $a = 6.2001$ (2) Å | $\theta = 3.0–29.5°$ |
| $b = 8.1917$ (3) Å | $\mu = 0.53$ mm$^{-1}$ |
| $c = 12.8121$ (6) Å | $T = 170$ K |
| $V = 650.72$ (4) Å$^3$ | Prism, colourless |
| $Z = 4$ | $0.50 \times 0.48 \times 0.28$ mm |
| $F(000) = 360$ | |

*Data collection*

| | |
|---|---|
| STOE IPDS 2T diffractometer | $T_{min} = 0.728$, $T_{max} = 1.117$ |
| Radiation source: sealed X-ray tube, Incoatec I$\mu$s | 14489 measured reflections |
| | 1754 independent reflections |
| Detector resolution: 6.67 pixels mm$^{-1}$ | 1690 reflections with $I > 2\sigma(I)$ |
| $\omega$ scans | $R_{int} = 0.038$ |
| Absorption correction: multi-scan [*MULABS* (Blessing, 1995) in *PLATON* (Spek, 2020)] | $\theta_{max} = 29.2°$, $\theta_{min} = 3.0°$ |
| | $h = -8 \rightarrow 7$ |
| | $k = -11 \rightarrow 11$ |
| | $l = -17 \rightarrow 17$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Hydrogen site location: difference Fourier map |
| Least-squares matrix: full | All H-atom parameters refined |
| $R[F^2 > 2\sigma(F^2)] = 0.023$ | $w = 1/[\sigma^2(F_o^2) + (0.0357P)^2 + 0.0942P]$ |
| $wR(F^2) = 0.058$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.09$ | $(\Delta/\sigma)_{max} < 0.001$ |
| 1754 reflections | $\Delta\rho_{max} = 0.20$ e Å$^{-3}$ |
| 114 parameters | $\Delta\rho_{min} = -0.34$ e Å$^{-3}$ |
| 12 restraints | Absolute structure: Flack $x$ determined using 672 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013) |
| Primary atom site location: dual | |
| Secondary atom site location: difference Fourier map | Absolute structure parameter: $-0.05$ (2) |

*Special details*

**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**. Hydrogen-atom positions were located in a difference Fourier map, and their parameters were refined with standard similarity restraints on 1,2-distances for O—H and and N—H bonds (with a standard uncertainty of 0.02 Å). $U_{iso}$(H) values were refined freely.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

|      | *x*          | *y*          | *z*          | $U_{iso}$*/$U_{eq}$ |
|------|--------------|--------------|--------------|---------------------|
| Na1  | 0.08933 (10) | 0.73476 (8)  | 0.51432 (5)  | 0.01968 (17)        |
| S1   | 0.37520 (7)  | 0.41221 (5)  | 0.62681 (3)  | 0.01762 (11)        |
| O1   | 0.3414 (2)   | 0.91744 (17) | 0.59691 (11) | 0.0248 (3)          |
| H1A  | 0.368 (6)    | 0.903 (4)    | 0.6609 (18)  | 0.060 (10)*         |
| H1B  | 0.351 (5)    | 1.020 (3)    | 0.587 (2)    | 0.050 (9)*          |
| O2   | 0.3121 (2)   | 0.79007 (17) | 0.36474 (11) | 0.0228 (3)          |
| H2A  | 0.303 (5)    | 0.721 (3)    | 0.320 (2)    | 0.040 (8)*          |
| H2B  | 0.250 (5)    | 0.873 (3)    | 0.348 (2)    | 0.035 (7)*          |
| O3   | 0.1880 (3)   | 0.4256 (2)   | 0.69708 (12) | 0.0326 (3)          |
| O4   | 0.3627 (2)   | 0.53757 (14) | 0.54471 (10) | 0.0212 (3)          |
| O5   | 0.3756 (4)   | 0.25020 (16) | 0.57880 (12) | 0.0390 (4)          |
| O6   | 0.5751 (2)   | 0.4335 (2)   | 0.68758 (12) | 0.0337 (4)          |
| N1   | 0.3688 (3)   | 0.3331 (2)   | 0.35538 (13) | 0.0244 (3)          |
| H1C  | 0.356 (5)    | 0.394 (3)    | 0.4051 (19)  | 0.042 (8)*          |
| H1D  | 0.364 (6)    | 0.401 (3)    | 0.308 (2)    | 0.051 (9)*          |
| H1E  | 0.483 (4)    | 0.284 (4)    | 0.354 (3)    | 0.056 (10)*         |
| H1F  | 0.269 (4)    | 0.269 (3)    | 0.358 (3)    | 0.050 (9)*          |

*Atomic displacement parameters (Å²)*

|     | $U^{11}$      | $U^{22}$      | $U^{33}$      | $U^{12}$        | $U^{13}$       | $U^{23}$       |
|-----|---------------|---------------|---------------|-----------------|----------------|----------------|
| Na1 | 0.0170 (3)    | 0.0211 (3)    | 0.0209 (3)    | 0.0009 (2)      | 0.0010 (3)     | −0.0001 (2)    |
| S1  | 0.02186 (19)  | 0.01448 (17)  | 0.01652 (18)  | −0.00021 (15)   | 0.00033 (15)   | 0.00224 (14)   |
| O1  | 0.0334 (7)    | 0.0183 (6)    | 0.0226 (6)    | −0.0006 (6)     | −0.0037 (5)    | −0.0011 (5)    |
| O2  | 0.0253 (6)    | 0.0237 (6)    | 0.0195 (6)    | 0.0023 (5)      | −0.0034 (5)    | 0.0005 (5)     |
| O3  | 0.0308 (7)    | 0.0383 (8)    | 0.0288 (7)    | 0.0050 (6)      | 0.0113 (6)     | 0.0072 (6)     |
| O4  | 0.0259 (6)    | 0.0170 (5)    | 0.0206 (5)    | −0.0001 (5)     | −0.0020 (5)    | 0.0056 (4)     |
| O5  | 0.0715 (11)   | 0.0150 (6)    | 0.0304 (7)    | 0.0014 (8)      | 0.0006 (8)     | −0.0004 (5)    |
| O6  | 0.0311 (7)    | 0.0402 (8)    | 0.0298 (7)    | −0.0088 (6)     | −0.0106 (6)    | 0.0138 (6)     |
| N1  | 0.0263 (8)    | 0.0266 (7)    | 0.0201 (7)    | 0.0006 (7)      | 0.0007 (7)     | −0.0009 (6)    |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Na1—O2$^i$   | 2.3229 (15) | S1—O3     | 1.4728 (15) |
| Na1—O4       | 2.3733 (14) | S1—O6     | 1.4740 (15) |
| Na1—O2       | 2.4054 (16) | O1—H1A    | 0.84 (2)    |
| Na1—O1       | 2.4087 (15) | O1—H1B    | 0.85 (2)    |
| Na1—O1$^i$   | 2.4389 (16) | O2—H2A    | 0.81 (2)    |
| Na1—O4$^i$   | 2.4546 (14) | O2—H2B    | 0.81 (2)    |
| Na1—Na1$^{ii}$ | 3.1316 (2) | N1—H1C    | 0.814 (19)  |
| Na1—Na1$^i$  | 3.1317 (2)  | N1—H1D    | 0.828 (19)  |

| | | | |
|---|---|---|---|
| Na1—H2B | 2.61 (3) | N1—H1E | 0.81 (2) |
| S1—O5 | 1.4628 (14) | N1—H1F | 0.81 (2) |
| S1—O4 | 1.4721 (12) | | |

| | | | |
|---|---|---|---|
| O2$^i$—Na1—O4 | 111.05 (5) | O1$^i$—Na1—H2B | 89.2 (6) |
| O2$^i$—Na1—O2 | 166.61 (6) | O4$^i$—Na1—H2B | 68.8 (5) |
| O4—Na1—O2 | 81.31 (5) | Na1$^{ii}$—Na1—H2B | 59.5 (6) |
| O2$^i$—Na1—O1 | 103.97 (6) | Na1$^i$—Na1—H2B | 104.3 (6) |
| O4—Na1—O1 | 83.54 (5) | O5—S1—O4 | 109.42 (8) |
| O2—Na1—O1 | 81.97 (5) | O5—S1—O3 | 109.02 (11) |
| O2$^i$—Na1—O1$^i$ | 83.03 (5) | O4—S1—O3 | 110.07 (8) |
| O4—Na1—O1$^i$ | 101.42 (5) | O5—S1—O6 | 109.17 (11) |
| O2—Na1—O1$^i$ | 89.58 (5) | O4—S1—O6 | 109.83 (8) |
| O1—Na1—O1$^i$ | 169.50 (3) | O3—S1—O6 | 109.31 (9) |
| O2$^i$—Na1—O4$^i$ | 81.28 (5) | Na1—O1—Na1$^{ii}$ | 80.48 (4) |
| O4—Na1—O4$^i$ | 167.55 (5) | Na1—O1—H1A | 118 (2) |
| O2—Na1—O4$^i$ | 86.58 (5) | Na1$^{ii}$—O1—H1A | 112 (2) |
| O1—Na1—O4$^i$ | 92.00 (5) | Na1—O1—H1B | 127 (2) |
| O1$^i$—Na1—O4$^i$ | 81.23 (5) | Na1$^{ii}$—O1—H1B | 112 (2) |
| O2$^i$—Na1—Na1$^{ii}$ | 144.84 (5) | H1A—O1—H1B | 105 (3) |
| O4—Na1—Na1$^{ii}$ | 50.70 (4) | Na1$^{ii}$—O2—Na1 | 82.93 (4) |
| O2—Na1—Na1$^{ii}$ | 47.40 (4) | Na1$^{ii}$—O2—H2A | 118 (2) |
| O1—Na1—Na1$^{ii}$ | 50.18 (4) | Na1—O2—H2A | 113 (2) |
| O1$^i$—Na1—Na1$^{ii}$ | 126.60 (5) | Na1$^{ii}$—O2—H2B | 127 (2) |
| O4$^i$—Na1—Na1$^{ii}$ | 118.04 (5) | Na1—O2—H2B | 95 (2) |
| O2$^i$—Na1—Na1$^i$ | 49.66 (4) | H2A—O2—H2B | 111 (3) |
| O4—Na1—Na1$^i$ | 141.29 (5) | S1—O4—Na1 | 129.03 (8) |
| O2—Na1—Na1$^i$ | 117.40 (5) | S1—O4—Na1$^{ii}$ | 136.05 (8) |
| O1—Na1—Na1$^i$ | 130.11 (5) | Na1—O4—Na1$^{ii}$ | 80.86 (4) |
| O1$^i$—Na1—Na1$^i$ | 49.34 (4) | H1C—N1—H1D | 99 (3) |
| O4$^i$—Na1—Na1$^i$ | 48.44 (4) | H1C—N1—H1E | 114 (3) |
| Na1$^{ii}$—Na1—Na1$^i$ | 163.71 (5) | H1D—N1—H1E | 110 (3) |
| O2$^i$—Na1—H2B | 149.9 (5) | H1C—N1—H1F | 107 (3) |
| O4—Na1—H2B | 99.0 (5) | H1D—N1—H1F | 116 (3) |
| O2—Na1—H2B | 18.0 (5) | H1E—N1—H1F | 110 (3) |
| O1—Na1—H2B | 80.9 (6) | | |

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

*Hydrogen-bond geometry (Å, °)*

| $D$—H⋯$A$ | $D$—H | H⋯$A$ | $D$⋯$A$ | $D$—H⋯$A$ |
|---|---|---|---|---|
| O1—H1$A$⋯O6$^{iii}$ | 0.84 (2) | 1.99 (2) | 2.812 (2) | 165 (3) |
| O1—H1$B$⋯O5$^{iv}$ | 0.85 (2) | 1.89 (2) | 2.7439 (19) | 175 (3) |
| O2—H2$A$⋯O3$^v$ | 0.81 (2) | 1.98 (2) | 2.781 (2) | 171 (3) |
| O2—H2$B$⋯O6$^i$ | 0.81 (2) | 1.98 (2) | 2.781 (2) | 175 (3) |
| N1—H1$C$⋯O4 | 0.81 (2) | 2.14 (2) | 2.948 (2) | 172 (3) |
| N1—H1$D$⋯O3$^v$ | 0.83 (2) | 2.03 (2) | 2.854 (2) | 172 (3) |

| | | | | |
|---|---|---|---|---|
| N1—H1*E*···O3<sup>vi</sup> | 0.81 (2) | 2.23 (2) | 2.977 (2) | 152 (3) |
| N1—H1*E*···O5<sup>vi</sup> | 0.81 (2) | 2.60 (3) | 3.324 (3) | 149 (3) |
| N1—H1*F*···O5<sup>vii</sup> | 0.81 (2) | 2.58 (3) | 3.245 (3) | 140 (3) |
| N1—H1*F*···O6<sup>vii</sup> | 0.81 (2) | 2.13 (2) | 2.897 (3) | 157 (3) |

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