



$(\text{NH}_4)\text{Mg}(\text{HSO}_4)(\text{SO}_4)(\text{H}_2\text{O})_2$ and $\text{NaSc}(\text{CrO}_4)_2(\text{H}_2\text{O})_2$, two crystal structures comprising kröhnkite-type chains, and the temperature-induced phase transition $(\text{NH}_4)\text{Mg}(\text{HSO}_4)(\text{SO}_4)(\text{H}_2\text{O})_2 \rightleftharpoons (\text{NH}_4)\text{MgH}(\text{SO}_4)_2(\text{H}_2\text{O})_2$

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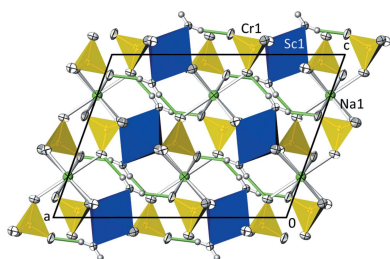
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The crystal structure of the mineral kröhnkite, $\text{Na}_2\text{Cu}(\text{SO}_4)_2(\text{H}_2\text{O})_2$, contains infinite chains composed of $[\text{CuO}_4(\text{OH}_2)_2]$ octahedra corner-linked with SO_4 tetrahedra. Such or similar tetrahedral–octahedral ‘kröhnkite-type’ chains are present in the crystal structures of numerous compounds with the composition $A_nM(\text{XO}_4)_2(\text{H}_2\text{O})_2$. The title compounds, $(\text{NH}_4)\text{Mg}(\text{HSO}_4)(\text{SO}_4)(\text{H}_2\text{O})_2$, ammonium magnesium hydrogen sulfate sulfate dihydrate, and $\text{NaSc}(\text{CrO}_4)_2(\text{H}_2\text{O})_2$, sodium scandium bis(chromate) dihydrate, are members of the large family with such kröhnkite-type chains. At 100 K, $(\text{NH}_4)\text{Mg}(\text{HSO}_4)(\text{SO}_4)(\text{H}_2\text{O})_2$ has an unprecedented triclinic crystal structure and contains $[\text{MgO}_4(\text{OH}_2)_2]$ octahedra linked by $\text{SO}_3(\text{OH})$ and SO_4 tetrahedra into chains extending parallel to $[\bar{1}10]$. Adjacent chains are linked by very strong hydrogen bonds between $\text{SO}_3(\text{OH})$ and SO_4 tetrahedra into layers parallel to (111). Ammonium cations and water molecules connect adjacent layers through hydrogen-bonding interactions of medium-to-weak strength into a three-dimensional network. $(\text{NH}_4)\text{Mg}(\text{HSO}_4)(\text{SO}_4)(\text{H}_2\text{O})_2$ shows a reversible phase transition and crystallizes at room temperature in structure type **E** in the classification scheme for structures with kröhnkite-type chains, with half of the unit-cell volume for the resulting triclinic cell, and with disordered H atoms of the ammonium tetrahedron and the H atom between two symmetry-related sulfate groups. IR spectroscopic room-temperature data for the latter phase are provided. Monoclinic $\text{NaSc}(\text{CrO}_4)_2(\text{H}_2\text{O})_2$ adopts structure type **F1** in the classification scheme for structures with kröhnkite-type chains. Here, $[\text{ScO}_4(\text{OH}_2)_2]$ octahedra (point group symmetry $\bar{1}$) are linked by CrO_4 tetrahedra into chains parallel to [010]. The Na^+ cations (site symmetry 2) have a [6 + 2] coordination and connect adjacent chains into a three-dimensional framework that is consolidated by medium–strong hydrogen bonds involving the water molecules. Quantitative structural comparisons are made between $\text{NaSc}(\text{CrO}_4)_2(\text{H}_2\text{O})_2$ and its isotypic $\text{NaM}(\text{CrO}_4)_2(\text{H}_2\text{O})_2$ ($M = \text{Al}$ and Fe) analogues.



1. Introduction

Compounds comprising tetrahedral oxoanions (XO_4) and two types of cations, viz. a larger cation A and a smaller cation M , often exist as dihydrates with the general formula $A_nM(\text{XO}_4)_2(\text{H}_2\text{O})_2$ ($n = 1$ or 2) when crystallized from aqueous solutions or under hydrothermal conditions. Irrespective of the chemical nature of A , M or X , the crystal structures of $A_nM(\text{XO}_4)_2(\text{H}_2\text{O})_2$ compounds frequently comprise infinite chains composed of more or less distorted $[\text{MO}_4(\text{OH}_2)_2]$



Table 1
Comparative compilation of structure types with kröhnkite-type chains.

| Type | Generalized formula(e) | Space group | Z | Generalized unit-cell parameters (Å, °) | No. of representatives |
|-----------------------|---|-------------|---|---|------------------------|
| A | $M1^{II}M2^{II}(TO_4)_2(H_2O)_2$: $M1 = Ca, Sr$; $M^{II} = Mg, Mn, Fe, Co, Ni, Zn$; $T = P, As$. $M1^{II}M2^{II}(TO_4)_2(H_2O)_2$: $M1 = Na, K, NH_4$; $M^{II} = Mg, Fe, Co, Ni, Cu, Zn$; $T = S, Se, Cr, Mo, W$ | $P\bar{1}$ | 1 | $a \approx 5.7\text{--}7.1, b \approx 6.7\text{--}7.9, c \approx 5.3\text{--}6.0, \alpha \approx 93\text{--}105, \beta \approx 105\text{--}112, \gamma \approx 103\text{--}111$ | 32 |
| B | $M1^{II}M2^{II}(TO_4)_2(H_2O)_2$: $M1 = Ca$; $M2 = Mn, Fe$; $T = P$ | $P\bar{1}$ | 1 | $a \approx 5.8\text{--}6.0, b \approx 6.5\text{--}6.6, c \approx 5.5, \alpha \approx 102\text{--}103, \beta \approx 108\text{--}109, \gamma \approx 90\text{--}91$ | 2 |
| C | $M1^{II}M2^{II}(TO_4)_2(H_2O)_2$: $M1 = K, Rb$; $M2 = Mn, Cd$; $T = S, Se, Cr$ | $P\bar{1}$ | 2 | $a \approx 6.6\text{--}6.9, b \approx 7.3\text{--}7.7, c \approx 10.7\text{--}11.4, \alpha \approx 72\text{--}73, \beta \approx 74\text{--}75, \gamma \approx 70$ | 4 |
| C1 | $K_2Fe(SO_4)_2(H_2O)_2$ | $P1^a$ | 2 | $a \approx 6.6, b \approx 7.3, c \approx 10.7, \alpha \approx 73, \beta \approx 74, \gamma \approx 70$ | 1 |
| D | $M1^{II}M2^{II}(TO_4)_2(H_2O)_2$: $M1 = Ca$; $M2 = Mg, Mn, Co, Cu, Zn$; $T = As$. $M1^{II}M2^{II}(TO_4)_2(H_2O)_2$: $M1 = Na, NH_4, Rb$; $M2 = Cr, Mn, Fe, Cu, Cd$; $T = S, Se, Cr, Mo$ | $P2_1/c$ | 2 | $a \approx 5.8\text{--}6.8, b \approx 12.8\text{--}14.3, c \approx 5.4\text{--}5.9, \beta \approx 106\text{--}111$ | 14 |
| E | $M1^{II}M2^{II}H(TO_4)_2(H_2O)_2$: $M1 = K, NH_4, Cs$; $M2 = Mg, Mn, Fe, Co, Zn$; $T = S, Se$ | $P\bar{1}$ | 1 | $a \approx 4.6\text{--}4.8, b \approx 5.7\text{--}5.9, c \approx 8.1\text{--}8.6, \alpha \approx 103\text{--}104, \beta \approx 96\text{--}100, \gamma \approx 94\text{--}97$ | 8 |
| E1^b | $(NH_4)Mg(HSO_4)(SO_4)(H_2O)_2$ (at 100 K) | $P\bar{1}$ | 2 | $a \approx 7.1, b \approx 7.7, c \approx 8.3, \alpha \approx 84.6, \beta \approx 73.3, \gamma \approx 77.4$ | 1 |
| F1^c | $M1^{II}M2^{III}(TO_4)_2(H_2O)_2$: $M1 = Na$; $M2 = Al, Sc, Fe$; $T = Cr$ | $C2/c$ | 4 | $a \approx 14.1\text{--}14.5, b \approx 5.3\text{--}5.6, c \approx 10.7\text{--}10.8, \beta \approx 109\text{--}110$ | 3 |
| F2^c | $M1^{II}M2^{III}(TO_4)_2(H_2O)_2$: $M1 = Na, K, NH_4, Tl$; $M2 = Al, Fe, In$; $T = Cr$ | $C2/m$ | 2 | $a \approx 10.7\text{--}11.0, b \approx 5.4\text{--}5.6, c \approx 7.5\text{--}7.6, \beta \approx 114$ | 5 |
| G | $AgSc(CrO_4)_2(H_2O)_2$ | $P\bar{1}$ | 1 | $a \approx 5.6, b \approx 6.1, c \approx 7.4, \alpha \approx 111, \beta \approx 90, \gamma \approx 117$ | 1 |
| H^d | $K_2Zn(CrO_4)_2(H_2O)_2$ | $C2/c$ | 4 | $a \approx 15.0, b \approx 5.7, c \approx 12.3, \beta \approx 117$ | 1 |

Notes: (a) Type C1 is represented only by the low-temperature modification of $K_2Fe(SO_4)_2(H_2O)_2$ and has an uncertain space group. (b) Type E1 is described for the first time in the present work; it is an ordered variant of type E (see text). (c) Types F1 and F2 are closely related (see text). (d) Type H (Stoilova *et al.*, 2008) is closely related to type A.

octahedra corner-linked by XO_4 tetrahedra, a structural motif that is known from the mineral kröhnkite [$Na_2Cu(SO_4)_2(H_2O)_2$; Dahlgren, 1952]. An astonishingly large number of natural and synthetic hydrated oxysalts with this formula type is known to contain such ‘kröhnkite-type’ chains in their crystal structures. The widespread occurrence of this motif is associated with its flexible nature and assemblies of the corner-sharing octahedral–tetrahedral building units within a chain.

Reviews on natural and synthetic compounds with kröhnkite-type chains were given in four subsequent reports (Fleck *et al.*, 2002a; Fleck & Kolitsch, 2003; Kolitsch & Fleck, 2005, 2006). In general, compounds with the composition $A_nM(XO_4)_2(H_2O)_2$, where $A = Na^I, K^I, Rb^I, Cs^I, NH_4, H^I, Ca^{II}$ or Sr^{II} ; $M = Mg^{II}, Cr^{II}, Mn^{II}, Fe^{II}, Co^{II}, Ni^{II}, Cu^{II}, Zn^{II}, Cd^{II}, Al^{III}, Fe^{III}, Sc^{III}, In^{III}$ or Tl^{III} and $X = P^V, As^V, S^{VI}, Se^{VI}, Cr^{VI}, Mo^{VI}$ or W^{VI} , containing kröhnkite-type chains, can be subdivided into eight major structure types denoted as **A–H**, for which more than 70 representatives are known up to date. Table 1

compiles the most important parameters for these structure types, based on all representatives reported until the end of 2020, including the new type **E1** described herein.

We report here two new representatives of compounds with kröhnkite-type chains, *viz.* $(NH_4)Mg(HSO_4)(SO_4)(H_2O)_2$ and $NaSc(CrO_4)_2(H_2O)_2$.

2. Experimental

2.1. Synthesis and crystallization

2.1.1. $(NH_4)Mg(HSO_4)(SO_4)(H_2O)_2$. A stoichiometric mixture of $MgSO_4(H_2O)_7$, TeO_2 and KOH (ratio 2:1:2 mmol; all reagents from Merck) was placed in a Teflon container with 6 ml capacity that was filled to approximately two-thirds of its volume with water. The container was closed, placed in a steel autoclave and heated at 480 K under autogenous pressure for 4 d. After slow cooling to room temperature within 1 d, the colourless reaction product was filtered off, washed with water

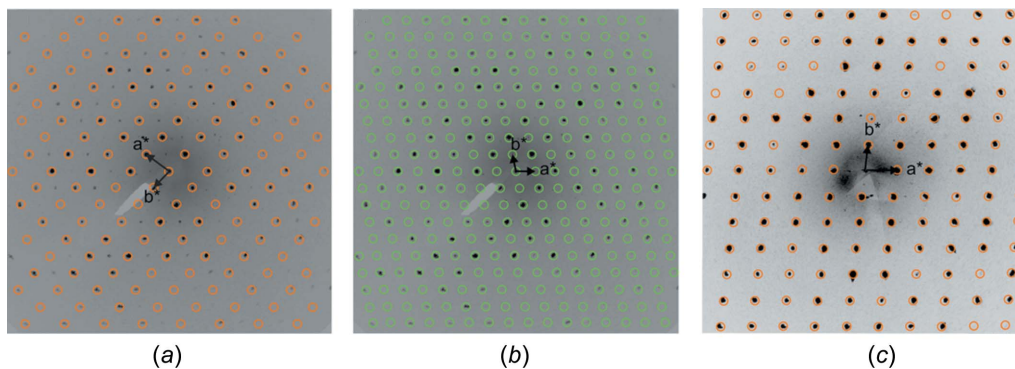


Figure 1
The $hk1$ plane of reciprocal space of $(NH_4)Mg(HSO_4)(SO_4)(H_2O)_2$ [100 K data in parts (a)/(b)] and of $(NH_4)MgH(SO_4)_2(H_2O)_2$ [296 K data in part (c)] reconstructed from CCD data. (a) The small cell with matching reflections is marked with orange circles; no superstructure reflections are visible. (b) The actual supercell with matching reflections is marked with green circles. (c) The actual small cell is marked with orange circles without superstructure reflections.

Table 2
Experimental details.

Experiments were carried out with Mo $K\alpha$ radiation. All H-atom parameters were refined.

| | (NH ₄)Mg(HSO ₄)(SO ₄)(H ₂ O) ₂ at 100 K | (NH ₄)MgH(SO ₄) ₂ (H ₂ O) ₂ at 296 K | NaSc(CrO ₄) ₂ (H ₂ O) ₂ |
|--|---|---|--|
| Crystal data | | | |
| Chemical formula | (NH ₄)Mg(HSO ₄)(SO ₄)(H ₂ O) ₂ | (NH ₄)Mg(HSO ₄)(SO ₄)(H ₂ O) ₂ | NaSc(CrO ₄) ₂ (H ₂ O) ₂ |
| M_r | 271.51 | 271.51 | 335.98 |
| Crystal system, space group | Triclinic, $P\bar{1}$ | Triclinic, $P\bar{1}$ | Monoclinic, $C2/c$ |
| Temperature (K) | 100 | 296 | 293 |
| a, b, c (Å) | 7.0631 (7), 7.7065 (7), 8.3372 (8) | 4.6771 (1), 5.7697 (1), 8.3697 (2) | 14.505 (3), 5.563 (1), 10.763 (2) |
| α, β, γ (°) | 84.603 (3), 73.339 (3), 77.387 (3) | 104.208 (1), 98.189 (1), 94.508 (1) | 90, 109.82 (3), 90 |
| V (Å ³) | 424.03 (7) | 215.20 (1) | 817.0 (3) |
| Z | 2 | 1 | 4 |
| μ (mm ⁻¹) | 0.75 | 0.73 | 3.51 |
| Crystal size (mm) | 0.12 × 0.09 × 0.02 | 0.12 × 0.09 × 0.01 | 0.17 × 0.10 × 0.03 |
| Data collection | | | |
| Diffractometer | Bruker APEXII CCD | Bruker APEXII CCD | Nonius KappaCCD |
| Absorption correction | Multi-scan (<i>SADABS</i> ; Krause <i>et al.</i> , 2015) | Multi-scan (<i>SADABS</i> ; Krause <i>et al.</i> , 2015) | Multi-scan (<i>SCALEPACK</i> ; Otwinowski <i>et al.</i> , 2003) |
| T_{\min}, T_{\max} | 0.708, 0.747 | 0.699, 0.747 | 0.755, 0.949 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 30998, 4431, 3120 | 11147, 1940, 1843 | 3418, 1783, 1372 |
| R_{int} | 0.051 | 0.024 | 0.017 |
| $(\sin \theta/\lambda)_{\text{max}}$ (Å ⁻¹) | 0.864 | 0.812 | 0.805 |
| Refinement | | | |
| $R[F^2 > 2\sigma(F^2)], wR(F^2), S$ | 0.033, 0.081, 1.02 | 0.019, 0.054, 1.18 | 0.027, 0.081, 1.08 |
| No. of reflections | 4431 | 1940 | 1783 |
| No. of parameters | 163 | 95 | 75 |
| No. of restraints | 0 | 0 | 2 |
| $\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å ⁻³) | 0.52, -0.54 | 0.30, -0.47 | 1.06, -0.69 |

Computer programs: *APEX2* (Bruker, 2016), *COLLECT* (Nonius, 2003), *DENZO* and *SCALEPACK* (Otwinowski *et al.*, 2003), *SHELXT* (Sheldrick, 2015a), *SHELXS97* (Sheldrick, 2008), *SHELXL2018* (Sheldrick, 2015b), *ATOMS* (Dowty, 2006) and *pubCIF* (Westrip, 2010).

and ethanol, and was dried in air. Inspection under a polarizing microscope revealed a microcrystalline solid with only very few crystals visible (diameter \simeq 0.1 mm). Powder X-ray diffraction (PXRD) of the bulk revealed spiroffite-type Mg₂Te₃O₈ (Lin *et al.*, 2013) as the main product, and MgTe₂O₅ (Weil, 2005) as a minor product. The grown crystals correspond to the title compound. Structure refinement showed NH₄⁺ cations present in the structure. The source of ammonium remains unclear; most probably, ammonium cations were left in the cracks of the Teflon container from previous reactions in ammonia solution.

For a directed synthesis of (NH₄)Mg(HSO₄)(SO₄)(H₂O)₂, equimolar aqueous solutions of NH₄HSO₄ and MgSO₄(H₂O)₇ were mixed at room temperature and stirred for homogeneity. The used NH₄HSO₄ was freshly prepared by slowly adding concentrated ammonia solution to concentrated sulfuric acid in stoichiometric amounts and recrystallization of the colourless product from water; its purity was checked by PXRD. The mixed NH₄HSO₄ and MgSO₄ solutions were evaporated to dryness at 353 K in a drying oven and also much more slowly at room temperature. Semi-quantitative phase analysis using the Rietveld method with *HighScore Plus* (Degen *et al.*, 2014) revealed a phase mixture of (NH₄)MgH(SO₄)₂(H₂O)₂ and synthetic boussingaultite [(NH₄)₂Mg(SO₄)₂(H₂O)₆] in a ratio of \simeq 94:6 wt% for the sample dried at 353 K, and in a ratio of 91:9 wt% for the sample dried at room temperature.

A mid-range IR spectrum was recorded at room temperature for selected crystals of (NH₄)MgH(SO₄)₂(H₂O)₂ in the attenuated total reflectance (ATR) technique in the range 4000–450 cm⁻¹ on a PerkinElmer Spectrum Two FT-IR spectrometer with a UATR accessory (diamond detector crystal) attached.

2.1.2. NaSc(CrO₄)₂(H₂O)₂. Small tabular orange crystals with a rhombus-shaped outline crystallized at room temperature from an acidic aqueous solution (pH about 4) containing dissolved reagent-grade Na₂CO₃ (Merck), Sc₂O₃ (99.99%, alphametall, Germany) and reagent-grade CrO₃ (Merck). The crystals were often arranged in radiating clusters. They were associated with pale orange–yellow blade-shaped crystals of Na₂Cr₂O₇·2H₂O (Casari *et al.*, 2007).

2.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

For the refinement of (NH₄)Mg(HSO₄)(SO₄)(H₂O)₂ (100 K data), all H atoms were located from difference Fourier maps and were refined freely. Reflections $\bar{2}03$, 102, 100 and 230 were obstructed by the beam stop and were therefore omitted from the refinement. For the structure analysis of (NH₄)MgH(SO₄)₂(H₂O)₂ (296 K data), a different crystal was measured (resulting in a different orienting matrix; see Fig. 1). For

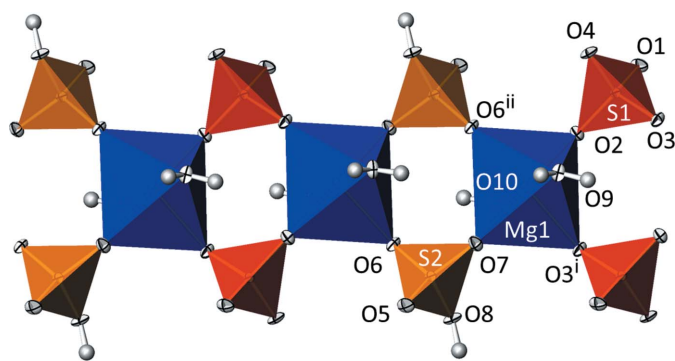


Figure 2

The kröhnkite-type chain in the crystal structure of $(\text{NH}_4)\text{Mg}(\text{HSO}_4)(\text{SO}_4)(\text{H}_2\text{O})_2$ at 100 K. $[\text{MgO}_4(\text{OH}_2)_2]$ octahedra are blue, $\text{SO}_3(\text{OH})$ tetrahedra are orange and SO_4 tetrahedra are red. Displacement ellipsoids are drawn at the 74% probability level and H atoms are given as grey spheres of arbitrary radius. [Symmetry codes: (i) $-x, -y + 1, -z + 1$; (ii) $-x + 1, -y, -z + 1$.]

refinement, the starting coordinates and labelling of atoms were adapted from the isotopic Fe compound (Heinicke *et al.*, 2004). In this structure, the H atoms (H1A–H1D) bonded to N atoms are all disordered over two equally occupied sites. The H1O atom located between two symmetry-related SO_4 tetrahedra was clearly discernible from a difference Fourier map; it is disordered across the inversion centre with an occupancy of 0.5 for the two H-atom sites. All H atoms in this structure were refined freely.

For refinement of $\text{NaSc}(\text{CrO}_4)_2(\text{H}_2\text{O})_2$, the coordinates and labelling of atoms were taken from isotopic $\text{NaFe}(\text{CrO}_4)_2(\text{H}_2\text{O})_2$ (Hardy & Graverneau, 1970). The H atoms of the water molecule were located from a difference Fourier map and were refined with a constraint of $\text{O}–\text{H} = 0.90 \pm 0.03 \text{ \AA}$.

3. Results and discussion

3.1. $(\text{NH}_4)\text{Mg}(\text{HSO}_4)(\text{SO}_4)(\text{H}_2\text{O})_2$

3.1.1. Structure analysis. $(\text{NH}_4)\text{Mg}(\text{HSO}_4)(\text{SO}_4)(\text{H}_2\text{O})_2$ was obtained serendipitously from a hydrothermal synthesis intended to crystallize a compound in the system $\text{Mg}–\text{S}^{\text{VI}}–\text{Te}^{\text{IV}}–\text{O}–\text{H}$ (Weil & Shir Khanlou, 2017). A subsequently performed directed synthesis yielded this material in >90% yield by evaporation of an aqueous solution containing equimolar amounts of NH_4HSO_4 and MgSO_4 .

$(\text{NH}_4)\text{Mg}(\text{HSO}_4)(\text{SO}_4)(\text{H}_2\text{O})_2$ is the fourth compound in the $\text{NH}_3–\text{MgO}–\text{SO}_3–\text{H}_2\text{O}$ system. The three other known members are the two minerals boussingaultite, *i.e.* $(\text{NH}_4)_2\text{Mg}(\text{SO}_4)_2(\text{H}_2\text{O})_6$ (Maslen *et al.*, 1988), and efremovite, *i.e.* $(\text{NH}_4)_2\text{Mg}_2(\text{SO}_4)_3$ (Shcherbakova & Bazhenova, 1989), and synthetic $(\text{NH}_4)_2\text{Mg}_3(\text{OH})_2(\text{SO}_4)_3(\text{H}_2\text{O})_2$ (Marri *et al.*, 2017). Monoclinic boussingaultite is a representative of the picromerite group and crystallizes isotypically with many other synthetic $A^{\text{I}}_2M^{\text{II}}(\text{XO}_4)_2(\text{H}_2\text{O})_6$ compounds ($A^{\text{I}} = \text{NH}_4, \text{K}, \text{Rb}, \text{Cs}$ or Tl ; $M^{\text{II}} = \text{Mg}, \text{V}, \text{Cr}, \text{Mn}, \text{Fe}, \text{Co}, \text{Ni}, \text{Cu}, \text{Zn}$ or Cd ; $X = \text{S}, \text{Se}$ or Cr), commonly known as Tutton's salts [crystal structure first determined by Hofmann (1931)]. Efremovite adopts the

cubic langbeinite structure type (Zemann & Zemann, 1957), and orthorhombic $(\text{NH}_4)_2\text{Mg}_3(\text{OH})_2(\text{SO}_4)_3(\text{H}_2\text{O})_2$ is isotopic with its cadmium analogue $(\text{NH}_4)_2\text{Cd}_3(\text{OH})_2(\text{SO}_4)_3(\text{H}_2\text{O})_2$ (Yin, 2011).

$(\text{NH}_4)\text{Mg}(\text{HSO}_4)(\text{SO}_4)(\text{H}_2\text{O})_2$ is an unprecedented member within the family of compounds with kröhnkite-type chains and crystallizes in a unique structure type at 100 K, here denoted as **E1** in order to conform to the classification of compounds with kröhnkite-type chains (Fleck *et al.*, 2002a; Table 1). All atoms in the triclinic structure are situated on general positions. $[\text{MgO}_4(\text{OH}_2)_2]$ octahedra are corner-linked by $\text{SO}_3(\text{OH})$ and SO_4 tetrahedra into chains running parallel to $[\bar{1}10]$ (Fig. 2). Adjacent chains are joined by hydrogen bonds between hydrogen sulfate and sulfate tetrahedra into sheets extending parallel to (111). Ammonium cations, situated between the sheets, and water molecules are also involved in hydrogen bonding and consolidate the three-dimensional network (Fig. 3).

The $\text{Mg}–\text{O}$ bond lengths in the $[\text{MgO}_4(\text{OH}_2)_2]$ octahedron scatter only slightly [range 2.0382 (9)–2.0715 (9) Å; Table 3], with the two *trans*-aligned water molecules (O9 and O19) in the axial positions. The mean $\text{Mg}–\text{O}$ distance of 2.061 Å fits well into the grand mean value of 2.09 (6) Å for six-coordinate Mg^{II} (Gagné & Hawthorne, 2016). The SO_4 tetrahedron (centred by atom S1) is slightly distorted, with bond lengths and angles in the ranges 1.4659 (9)–1.4901 (9) Å (mean 1.476 Å) and 106.87 (5)–111.52 (5)° (mean 109.5°), respectively. The bond-length values are in very good agreement with those given in a review on the sulfate group, for which the grand mean $\text{S}–\text{O}$ distance is 1.473 Å, with minimum and maximum $\text{S}–\text{O}$ distances of 1.430 and 1.501 Å, respectively (Hawthorne *et al.*, 2000). The longest bond in the S1O_4 tetrahedron is that to atom O4, acting as an acceptor atom for a hydrogen bond involving the OH group of the hydrogen

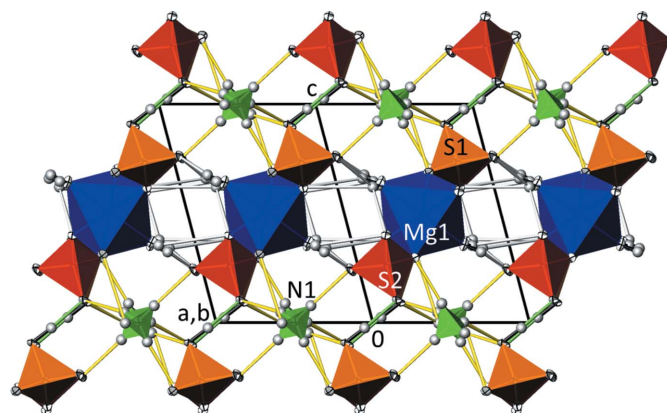


Figure 3

The crystal structure of $(\text{NH}_4)\text{Mg}(\text{HSO}_4)(\text{SO}_4)(\text{H}_2\text{O})_2$ at 100 K in a projection along $[\bar{1}10]$. $[\text{MgO}_4(\text{OH}_2)_2]$ octahedra are blue, SO_4 tetrahedra are red, $\text{SO}_3(\text{OH})$ tetrahedra are orange and ammonium groups are green. The strong hydrogen bond between the $\text{SO}_3(\text{OH})$ and SO_4 tetrahedra is indicated by green lines, hydrogen bonds involving water molecules by white lines and those involving the ammonium cations by yellow lines. Displacement ellipsoids are drawn at the 74% probability level.

Table 3

Selected bond lengths (Å) for (NH₄)Mg(HSO₄)(SO₄)(H₂O)₂ at 100 K.

| | | | |
|----------------------|-------------|-------|------------|
| Mg1—O2 | 2.0382 (9) | S1—O2 | 1.4685 (8) |
| Mg1—O7 | 2.0601 (9) | S1—O3 | 1.4775 (8) |
| Mg1—O3 ⁱ | 2.0630 (9) | S1—O4 | 1.4901 (9) |
| Mg1—O10 | 2.0645 (9) | S2—O5 | 1.4480 (9) |
| Mg1—O9 | 2.0660 (10) | S2—O6 | 1.4583 (8) |
| Mg1—O6 ⁱⁱ | 2.0715 (9) | S2—O7 | 1.4611 (8) |
| S1—O1 | 1.4659 (9) | S2—O8 | 1.5474 (9) |

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

sulfate group. The corresponding S₂O₃(OH) tetrahedron shows the typical S—O bond-length distribution where the bond to the OH group (O8) is considerably elongated. The S2—O8 bond of 1.5474 (9) Å is about 0.09 Å longer than the mean bond length (1.456 Å) of the remaining three bonds, in good agreement with other structures comprising a hydrogen sulfate anion, *e.g.* Mg(HSO₄)₂(H₂O) (Worzala *et al.*, 1991) or Th(HSO₄)₂(SO₄) (Betke & Wickleder, 2012). In the magnesium compound, with its two independent SO₃(OH) tetrahedra, mean values of 1.448 Å for the S—O and 1.550 Å for the S—OH bond lengths are found, and for the thorium compound, the corresponding mean values are 1.452 and 1.533 Å, respectively, for two independent SO₃(OH) tetrahedra; the SO₄ group in the thorium compound has a mean S—O bond length of 1.467 Å.

In the crystal structure of (NH₄)Mg(HSO₄)(SO₄)(H₂O)₂, the short hydrogen bond between the S₂O₃(OH) and the S₁O₄ tetrahedra [O8···O4ⁱⁱⁱ = 2.5048 (12) Å; Table 4] is linear [177 (3)°] and considered as strong (Jeffrey, 1997). In comparison, the other types of O—H···O hydrogen-bonding interactions are much weaker and are connected with the two water molecules. One of the water molecules (O9) is involved in a slightly bent hydrogen bond of medium strength to atom O1^{iv} and in a weak trifurcated hydrogen bond to O2ⁱ, O5^v and O7^v; numerical values of these interactions, as well as

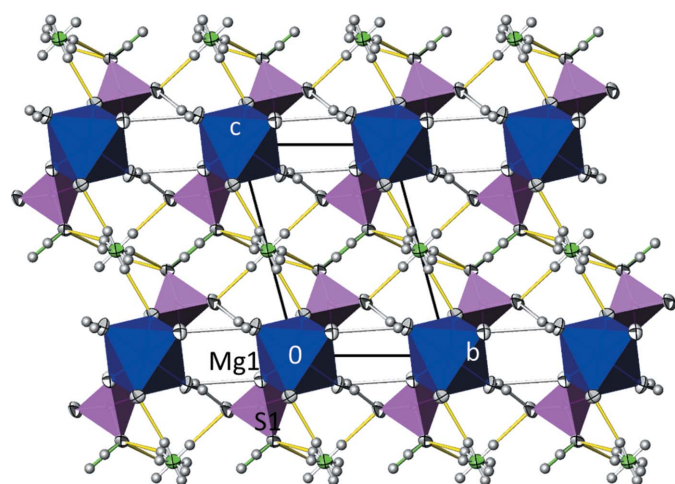


Figure 4
The crystal structure of (NH₄)MgH(SO₄)₂(H₂O)₂ at 296 K in a projection along [100]. Displacement ellipsoids and colour codes are as in Fig. 2, except for the SO₄ tetrahedra which are lilac. The disordered ammonium group and the H₁O atom disordered between two sulfate tetrahedra are shown.

Table 4

Hydrogen-bond geometry (Å, °) for (NH₄)Mg(HSO₄)(SO₄)(H₂O)₂ at 100 K.

| D—H···A | D—H | H···A | D···A | D—H···A |
|-----------------------------|------------|------------|-------------|------------|
| O8—H1O···O4 ⁱⁱⁱ | 0.91 (3) | 1.59 (3) | 2.5048 (12) | 177 (3) |
| O9—H1W···O1 ^{iv} | 0.81 (2) | 2.02 (2) | 2.7623 (13) | 154 (2) |
| O9—H2W···O2 ⁱ | 0.78 (3) | 2.56 (3) | 3.1278 (12) | 132 (2) |
| O9—H2W···O5 ^v | 0.78 (3) | 2.62 (3) | 3.2887 (12) | 146 (2) |
| O9—H2W···O7 ^v | 0.78 (3) | 2.55 (3) | 2.9578 (12) | 115 (2) |
| O10—H3W···O1 ^{vi} | 0.77 (2) | 2.38 (2) | 3.0970 (12) | 156 (2) |
| O10—H3W···O2 ^{vi} | 0.77 (2) | 2.64 (2) | 3.0615 (12) | 117 (2) |
| O10—H4W···O5 ^{vii} | 0.80 (2) | 1.95 (2) | 2.7125 (13) | 160.5 (19) |
| N1—H1N···O4 ^v | 0.896 (19) | 2.108 (19) | 2.9913 (14) | 168.2 (15) |
| N1—H2N···O1 ⁱ | 0.85 (2) | 2.04 (2) | 2.8920 (14) | 179.2 (18) |
| N1—H3N···O3 ⁱⁱⁱ | 0.868 (19) | 1.98 (2) | 2.8451 (14) | 170.5 (18) |
| N1—H4N···O8 ^{viii} | 0.870 (19) | 2.229 (19) | 3.0368 (14) | 154.4 (16) |

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

symmetry codes, are collated in Table 4. The other water molecule (O10) is the donor of one medium-strong and slightly bent hydrogen bond to O5^{vii}, and of a weak bifurcated hydrogen bond to O1^{vi} and O2^{vi}. As expected, the ammonium cation is also engaged in hydrogen bonding. All of its H atoms are accepted in a more or less linear manner [N—H···O angles range from 168.2 (15) to 179.2 (18)°] by the O atoms of the sulfate group (O4^v, O1ⁱ and O3ⁱⁱⁱ) and, interestingly, by the OH group of the hydrogen sulfate anion (O8^{viii}). The latter hydrogen bond is much more bent [154.4 (16)°], most probably due to steric reasons to avoid a too close contact with the H atom of the hydroxy group.

Bond-valence sums (BVSS; Brown, 2002), calculated with the parameters of Brese & O'Keefe (1991), amount to 2.22 valence units (v.u.) for Mg, 5.98 v.u. for S1 and 5.96 v.u. for S2, in good agreement with the formal charges of +II and +VI, respectively.

3.1.2. Phase transition. As mentioned above, at 100 K, (NH₄)Mg(HSO₄)(SO₄)(H₂O)₂ crystallizes in an own structure type, denoted as **E1**. Between 100 K and room temperature, the crystal is transformed into a triclinic structure corresponding to type **E** (space group $P\bar{1}$, $Z = 1$) in the classification of compounds with kröhnkite-type chains (Table 1). Next to the six isotypic sulfates KFeH(SO₄)₂(H₂O)₂ (Fleck *et al.*, 2002b), KMgH(SO₄)₂(H₂O)₂ (Maciček *et al.*, 1994), KZnH(SO₄)₂(H₂O)₂, KMnH(SO₄)₂(H₂O)₂, CsMnH(SO₄)₂(H₂O)₂ (Trojanov *et al.*, 2002) and NH₄FeH(SO₄)₂(H₂O)₂ (Heinicke *et al.*, 2004), and the selenate KMgH(SeO₄)₂(H₂O)₂ (Trojanov *et al.*, 2002), (NH₄)MgH(SO₄)(SO₄)(H₂O)₂, or more precisely (NH₄)MgH(SO₄)₂(H₂O)₂ at this temperature, is the eighth member of this structure type. The [M^{II}O₄(OH)₂] octahedron in these structures (Fig. 4 and Table 5) is located on an inversion centre, just like the A cation (for A = NH₄; the H sites are disordered). A peculiarity of type **E** pertains to the dynamically disordered H atom between two symmetry-related sulfate groups, defining a short asymmetrical hydrogen bond with O···O contacts around 2.5 Å (Table 6). In comparison, in the crystal structure of (NH₄)Mg(HSO₄)(SO₄)(H₂O)₂ at 100 K, the H atom is ordered between two sulfate tetrahedra, defining distinct SO₃OH and SO₄ groups. This

Table 5
Selected bond lengths (Å) for $(\text{NH}_4)\text{MgH}(\text{SO}_4)_2(\text{H}_2\text{O})_2$ at 296 K.

| | | | |
|---------------------|------------|-------|------------|
| Mg1—O2 | 2.0509 (6) | S1—O2 | 1.4604 (6) |
| Mg1—O4 ⁱ | 2.0720 (6) | S1—O4 | 1.4651 (6) |
| Mg1—O5 | 2.0731 (6) | S1—O1 | 1.5164 (6) |
| S1—O3 | 1.4525 (6) | | |

Symmetry code: (i) $x + 1, y, z$.

ordering is accompanied by a doubling of the unit-cell volume of the type **E1** relative. The bond lengths of the principal building units in the disordered room-temperature structure (Table 5; mean values for the Mg—O and S—O bond are 2.065 and 1.474 Å, respectively) are similar to those in the ordered low-temperature structure. Although the S—O(H) bond (O1) in the disordered structure is still the longest in the SO_4 tetrahedron, it is about 0.03 Å shorter than the S—OH bond (O8) in the ordered structure. On the other hand, the $\text{O1} \cdots \text{O1}^{\text{iv}}$ distance of the hydrogen bond with the disordered H1O atom [2.4790 (12) Å] is considerably shorter than the corresponding value in the ordered structure [2.5048 (12) Å], indicating a very strong hydrogen bond (Jeffrey, 1997) for $(\text{NH}_4)\text{MgH}(\text{SO}_4)_2(\text{H}_2\text{O})_2$.

The crystal structure of $(\text{NH}_4)\text{Mg}(\text{HSO}_4)(\text{SO}_4)(\text{H}_2\text{O})_2$ at 100 K represents a twofold superstructure with ordered H atoms for the ammonium and hydrogen sulfate groups relative to the crystal structure of $(\text{NH}_4)\text{MgH}(\text{SO}_4)_2(\text{H}_2\text{O})_2$ at 296 K with a halved unit-cell volume. The subcell of the latter is related to the doubled cell of the $(\text{NH}_4)\text{Mg}(\text{HSO}_4)(\text{SO}_4)(\text{H}_2\text{O})_2$ superstructure by application of the matrix $(\bar{1} \bar{1} 0, 1 \bar{1} 0, 0 0 1)$; the symmetry relationship between the substructure and the superstructure is of isomorphic type with index 2 (i2) (Müller, 2013). Fig. 1 shows the $hk1$ plane of reciprocal space of $(\text{NH}_4)\text{Mg}(\text{HSO}_4)(\text{SO}_4)(\text{H}_2\text{O})_2$ and the relation of the subcell (Fig. 1a) and the cell of the actual superstructure (Fig. 1b); the missing reflections for the subcell clearly indicate that the doubled cell is correct at this temperature. Fig. 1(c)

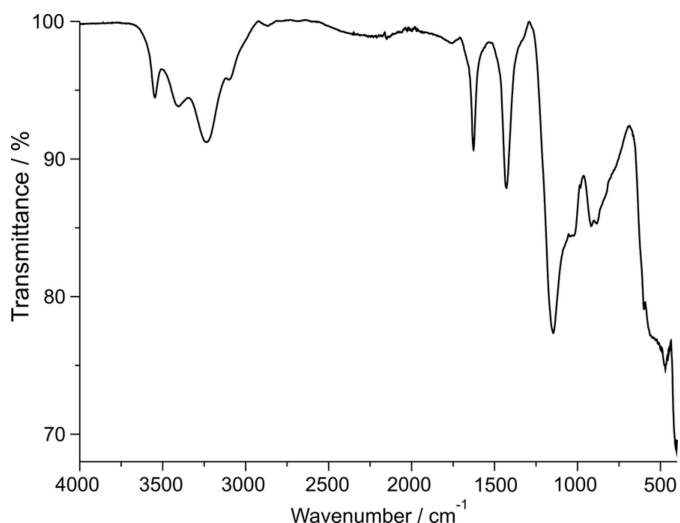


Figure 5
IR spectrum of $(\text{NH}_4)\text{MgH}(\text{SO}_4)_2(\text{H}_2\text{O})_2$ at room temperature.

Table 6
Hydrogen-bond geometry (Å, °) for $(\text{NH}_4)\text{MgH}(\text{SO}_4)_2(\text{H}_2\text{O})_2$ at 296 K.

| $D-H \cdots A$ | $D-H$ | $H \cdots A$ | $D \cdots A$ | $D-H \cdots A$ |
|---|----------|--------------|--------------|----------------|
| O5—H5B ⁱⁱ ··O2 ⁱⁱ | 0.81 (2) | 2.52 (2) | 3.0010 (9) | 118.9 (19) |
| O5—H5A ⁱⁱⁱ ··O3 ⁱⁱⁱ | 0.78 (2) | 2.05 (2) | 2.7684 (9) | 154.0 (19) |
| O5—H5B ⁱⁱⁱ ··O3 ⁱⁱⁱ | 0.81 (2) | 2.42 (2) | 3.1678 (10) | 152 (2) |
| N1—H1A ^{iv} ··O3 ^{iv} | 0.90 (5) | 2.03 (5) | 2.9260 (6) | 175 (5) |
| N1—H1C ^v ··O1 | 0.89 (6) | 2.28 (6) | 3.1536 (7) | 164 (4) |
| N1—H1B ^v ··O1 ^v | 0.83 (5) | 2.30 (5) | 3.1122 (8) | 164 (5) |
| N1—H1D ^v ··O4 ^v | 0.82 (5) | 2.08 (5) | 2.9042 (6) | 173 (5) |
| O1—H1O ^{iv} ··O1 ^{iv} | 0.84 (3) | 1.64 (3) | 2.4790 (12) | 176 (4) |

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

shows the $hk1$ plane of reciprocal space of $(\text{NH}_4)\text{MgH}(\text{SO}_4)_2(\text{H}_2\text{O})_2$ without noticeable superstructure reflections for the room-temperature data set. Investigations of the exact ordering temperatures for this reversible phase transition upon cooling and heating, as well as a systematic study of other $(\text{NH}_4)\text{M}^{\text{II}}(\text{HSO}_4)(\text{SO}_4)(\text{H}_2\text{O})_2 \rightleftharpoons (\text{NH}_4)\text{M}^{\text{II}}\text{H}(\text{SO}_4)_2(\text{H}_2\text{O})_2$ (M = first-row transition metals) phases, are underway.

3.1.3. IR spectroscopy. The IR spectrum of $(\text{NH}_4)\text{MgH}(\text{SO}_4)_2(\text{H}_2\text{O})_2$ shows similarities to that of synthetic bousingaultite (Jayakumar *et al.*, 1988) and is displayed in Fig. 5. Wavenumbers/ cm^{-1} : 3547 (*w*), 3403 (*br*), 3235 (*br*), 3100 (*w*), 2866 (*vw*), 1753 (*vw*), 1627 (*m*), 1429 (*m*), 1146 (*s*), 1044 (*sh*), 918 (*m*), 884 (*m*), ≈ 600 (*sh*) (*br* = broad; *m* = medium; *s* = strong; *sh* = shoulder; *w* = weak; *vw* = very weak).

In the wavenumber range $3700\text{--}2500 \text{ cm}^{-1}$, bands due to O—H stretching vibrations of the H_2O groups overlap with various bands of the NH_4 group. The bands at 3547 and 3403 cm^{-1} are assigned to the O—H stretching vibrations, while the band at 2335 cm^{-1} is tentatively assigned to the $\nu_3(\text{NH}_4)$ stretching vibration, and the shoulder at 3100 cm^{-1} to the $\nu_1(\text{NH}_4)$ stretch, the shoulder possibly also to an additional combination band $\nu_2 + \nu_4(\text{NH}_4)$. The very small band at 2866 cm^{-1} is probably caused by a combination band $2\nu_4(\text{NH}_4)$. The very strong hydrogen bonding involving the protonated $\{\text{H}(\text{SO}_4)_2\}$ group is reflected by an extremely

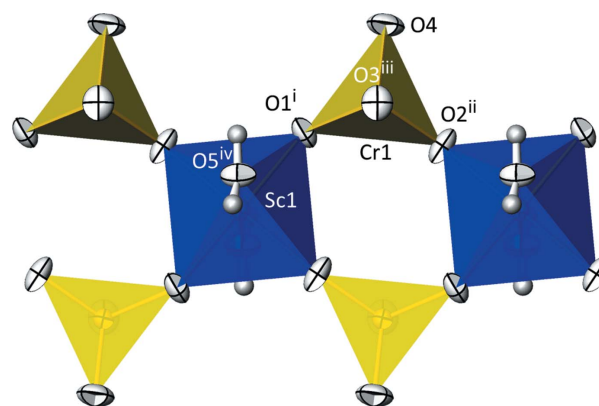


Figure 6
The kröhnkite-type chain in the crystal structure of $\text{NaSc}(\text{CrO}_4)_2(\text{H}_2\text{O})_2$. $[\text{ScO}_4(\text{OH}_2)_2]$ octahedra are blue and CrO_4 tetrahedra are yellow. Displacement ellipsoids are drawn at the 74% probability level and H atoms are given as grey spheres of arbitrary radius. [Symmetry codes: (i) $x, -y + 1, z + \frac{1}{2}$; (ii) $x, y + 1, z$; (iii) $x, -y + 1, z - \frac{1}{2}$; (iv) $-x + \frac{1}{2}, -y + \frac{1}{2}, -z$.]

Table 7
 Selected bond lengths (Å) for NaSc(CrO₄)₂(H₂O)₂.

| | | | |
|----------------------|-------------|---------------------|-------------|
| Na—O3 ⁱ | 2.5201 (15) | Sc—O5 | 2.1222 (14) |
| Na—O2 ⁱ | 2.5358 (15) | Cr—O4 | 1.6045 (14) |
| Na—O1 ⁱⁱ | 2.7207 (17) | Cr—O3 ^{iv} | 1.6204 (14) |
| Na—O4 ⁱ | 2.9360 (18) | Cr—O1 ^{iv} | 1.6829 (12) |
| Sc—O1 ⁱⁱⁱ | 2.0747 (12) | Cr—O2 ^v | 1.6960 (11) |
| Sc—O2 | 2.0772 (12) | | |

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

broad band in the range between roughly 1200 and 1000 cm⁻¹ (Beran & Libowitzky, 1999; Libowitzky, 1999), which appears ‘hidden’ in the background. The wavenumber range between 1800 and 1250 cm⁻¹ contains bands due to the ν₂(NH₄) bending vibration (1627 cm⁻¹, possibly also the very small band at 1753 cm⁻¹) and the ν₄(NH₄) bending vibration (1429 cm⁻¹). The range 1250–700 cm⁻¹ shows bands due to vibrations of the SO₄/HSO₄ groups. The band at 1146 cm⁻¹ is due to the ν₃(SO₄) stretching vibration, while the bands at 1044, 918 and 884 cm⁻¹ are assigned to the ν₁(SO₄) stretching vibration. The shoulder at ~600 cm⁻¹ is probably due to the ν₄(SO₄) vibration. The ν₂(SO₄) bending vibration will cause bands <500 cm⁻¹, where the spectrum is cut off and where bands due to vibrations of the MgO₆ octahedron, the librational modes of the NH₄ group and lattice modes are expected. Note that the presence of ‘forbidden’ SO₄ and NH₄ vibrations in the IR spectrum is in agreement with the presence of distorted shapes for these two building units.

3.2. NaSc(CrO₄)₂(H₂O)₂

3.2.1. Structure analysis. NaSc(CrO₄)₂(H₂O)₂ adopts type **F** (subtype **F1**) of the classification scheme for structures with kröhnkite-type chains (Table 1). Subtype **F1** (space group *C2/c*, *Z* = 2) can be considered as a superstructure of subtype **F2** (space group *C2/m*, *Z* = 1) that has a halved unit-cell volume relative to **F1** [transformation matrix **F1**→**F2** is (0 0 1, 0 1 0, $\frac{1}{2}$ 0 $\frac{1}{2}$)]. The group–subgroup relationship between subtypes **F2** and **F1** is klassengleich with index 2 (k2) (Müller, 2013). In the crystal structure of NaSc(CrO₄)₂(H₂O)₂, [ScO₄(OH₂)₂] octahedra (point-group symmetry $\bar{1}$) are linked by CrO₄ tetrahedra into chains running parallel to [010] (Fig. 6). The Na^I cations (site symmetry 2) connect adjacent chains into a three-dimensional framework that is stabilized by hydrogen bonds between water molecules and sulfate O atoms (Fig. 7).

In the [ScO₄(OH₂)₂] octahedron, the longest bond [2.1222 (14) Å] is that to the axially bound O5 atom of the water molecule, whereas the equatorial O atoms (O3 and O4), which are also part of a CrO₄ tetrahedron, have shorter Sc—O bonds, with a mean of 2.076 Å (Table 7). The overall mean value for the Sc—O bond lengths is 2.091 Å, which matches very well the literature values of 2.10 (7) and 2.098 (41) Å given by Serezhkin *et al.* (2003) and Gagné & Hawthorne (2020), respectively. In the CrO₄ tetrahedron, the longest Cr—O bonds (≈1.69 Å) are realized for O1 and O2, which are part of the kröhnkite chains. The other two O atoms (O3 and

Table 8
 Hydrogen-bond geometry (Å, °) for NaSc(CrO₄)₂(H₂O)₂.

| <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> |
|--------------------------|-------------|---------------|-----------------------|-------------------------|
| O5—H1···O4 ^{vi} | 0.82 (3) | 1.86 (3) | 2.682 (2) | 179 (3) |
| O5—H2···O3 | 0.82 (2) | 1.97 (3) | 2.765 (2) | 164 (3) |

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

O4) have considerably shorter Cr—O bonds (≈1.62 Å) and are the acceptor atoms for two nearly linear hydrogen bonds of medium–strong nature involving both water H atoms (Table 8). Again, the mean Cr—O bond length of 1.651 Å is in very good agreement with the literature value of 1.65 (6) Å (Gagné & Hawthorne, 2020). The Na^I cation shows a [6 + 2] coordination with the six closer O atoms defining a distorted octahedron (O1, O2 and their symmetry-related counterparts in equatorial sites, and O3 and its symmetry-related counterpart in axial sites), with the two remote O4 atoms capping two faces of the octahedron (Table 7). Notably, the water molecule is not part of the coordination sphere of Na. The mean Na—O bond length is 2.678 Å, somewhat longer than that of the literature value of 2.60 (19) Å for eightfold-coordinated Na^I (Gagné & Hawthorne, 2016). This elongation is also reflected in the slight underbonding of Na1 in the structure (BVS = 0.83 v.u.), with a deviation of 17% from the expected value of +1. Sc^{III} and Cr^{VI}, on the other hand, have BVS values of 3.12 and 5.92 v.u., respectively, and deviate much less (by about 4 and 2%) from the expected values.

NaSc(CrO₄)₂(H₂O)₂ is isotypic with NaAl(CrO₄)₂(H₂O)₂ (Cudennec & Riou, 1977) and NaFe(CrO₄)₂(H₂O)₂ (Hardy & Gravereau, 1970), the only other members of structure type **F1** in the classification of structures with kröhnkite-type chains. The title scandium compound is the first of this series for which the H atoms have been localized, thus making an unambiguous assignment of the hydrogen-bonding scheme possible (see above). For a quantitative structural comparison of the three isotypic Na*M*(CrO₄)₂(H₂O)₂ (*M* = Sc, Al or Fe) structures, the program *compstru* (de la Flor *et al.*, 2016),

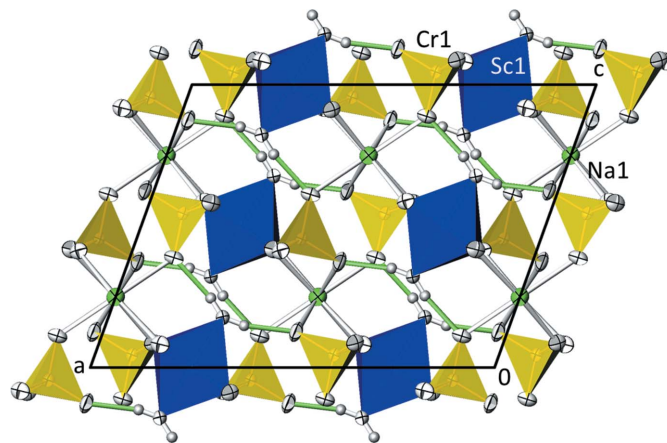


Figure 7
 The crystal structure of NaSc(CrO₄)₂(H₂O)₂ in a projection along [010]. Colour codes and the probability level of displacement ellipsoids are as in Fig. 3. Hydrogen bonds are indicated as green lines.

Table 9

Absolute atomic displacements (\AA), arithmetic mean (d_{av} , \AA), degree of lattice distortion (S) and measure of similarity (Δ) in the isotypic $\text{NaM}(\text{CrO}_4)_2(\text{H}_2\text{O})_2$ ($M = \text{Al}$ and Fe) structures relative to $\text{NaSc}(\text{CrO}_4)_2(\text{H}_2\text{O})_2$.

| | $M = \text{Al}^b$ | $M = \text{Fe}^c$ |
|-----------------|-------------------|-------------------|
| Na1 | 0.0595 | 0.0773 |
| M1 | 0 | 0 |
| Cr1 | 0.0839 | 0.0536 |
| O1 | 0.1477 | 0.1608 |
| O2 | 0.1227 | 0.1436 |
| O3 | 0.0595 | 0.0395 |
| O4 | 0.0395 | 0.0520 |
| O5 | 0.1916 | 0.0751 |
| d_{av} | 0.0964 | 0.0805 |
| Δ | 0.023 | 0.019 |
| S | 0.0186 | 0.0107 |

Notes: (a) H atoms were omitted from comparison because in the $M = \text{Al}$ and Fe structures, H atoms were not localized. (b) Unit-cell parameters $a = 14.080$ (10), $b = 5.338$ (3), $c = 10.655$ (6) \AA and $\beta = 110.33$ (5) $^\circ$ (Cudennec & Riou, 1977). (c) Unit-cell parameters $a = 14.247$ (2), $b = 5.425$ (5), $c = 10.689$ (2) \AA and $\beta = 109.30$ (1) $^\circ$ (Hardy & Gravereau, 1970).

available at the Bilbao Crystallographic Server (Aroyo *et al.*, 2006), was used. With $\text{NaSc}(\text{CrO}_4)_2(\text{H}_2\text{O})_2$ as the reference structure, Table 9 compiles the absolute distances between paired atoms and numerical values regarding the arithmetic mean of the distance between paired atoms, the degree of lattice distortion (Δ) and the measure of similarity (S). There is no clear trend as to the largest displacement of an atom pair in the three crystal structures. Whereas the water O atom (O5) in the $M = \text{Al}$ structure shows the largest displacement, it is O1 in the $M = \text{Fe}$ structure. In general, the rather low values of S indicate high similarities between $\text{NaSc}(\text{CrO}_4)_2(\text{H}_2\text{O})_2$ and the two $\text{NaM}(\text{CrO}_4)_2(\text{H}_2\text{O})_2$ ($M = \text{Al}$ and Fe) structures, whereby the $M = \text{Fe}$ structure has a higher absolute similarity to $\text{NaSc}(\text{CrO}_4)_2(\text{H}_2\text{O})_2$. Most likely, this behaviour is related to the ionic radii (Shannon, 1976) of the three M^{III} cations. For coordination number 6, the ionic radius of Sc^{III} (0.745 \AA) is closer to that of Fe^{III} (0.645 \AA , assuming a high-spin state) than to that of Al^{III} (0.535 \AA).

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supporting information

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(NH₄)Mg(HSO₄)(SO₄)(H₂O)₂ and NaSc(CrO₄)₂(H₂O)₂, two crystal structures comprising kröhnkite-type chains, and the temperature-induced phase transition (NH₄)Mg(HSO₄)(SO₄)(H₂O)₂ ⇌ (NH₄)MgH(SO₄)₂(H₂O)₂

Matthias Weil and Uwe Kolitsch

Computing details

Data collection: *APEX2* (Bruker, 2016) for NH₄MgHSO₄SO₄H₂O₂_100K, NH₄MgHSO₄2H₂O_296K; *COLLECT* (Nonius, 2003) for NaScCrO₄2H₂O₂. Cell refinement: *APEX2* (Bruker, 2016) for NH₄MgHSO₄SO₄H₂O₂_100K, NH₄MgHSO₄2H₂O_296K; *SCALEPACK* (Otwinowski *et al.*, 2003) for NaScCrO₄2H₂O₂. Data reduction: *APEX2* (Bruker, 2016) for NH₄MgHSO₄SO₄H₂O₂_100K, NH₄MgHSO₄2H₂O_296K; *DENZO* and *SCALEPACK* (Otwinowski *et al.*, 2003) for NaScCrO₄2H₂O₂. Program(s) used to solve structure: *SHELXT* (Sheldrick, 2015a) for NH₄MgHSO₄SO₄H₂O₂_100K, NH₄MgHSO₄2H₂O_296K; *SHELXS97* (Sheldrick, 2008) for NaScCrO₄2H₂O₂. For all structures, program(s) used to refine structure: *SHELXL2018* (Sheldrick, 2015b); molecular graphics: *ATOMS* (Dowty, 2006); software used to prepare material for publication: *pubCIF* (Westrip, 2010).

Ammonium magnesium hydrogen sulfate sulfate dihydrate (NH₄MgHSO₄SO₄H₂O₂_100K)

Crystal data

| | |
|--|---|
| (NH ₄)Mg(HSO ₄)(SO ₄)(H ₂ O) ₂ | <i>Z</i> = 2 |
| <i>M_r</i> = 271.51 | <i>F</i> (000) = 280 |
| Triclinic, <i>P</i> 1 | <i>D_x</i> = 2.127 Mg m ⁻³ |
| <i>a</i> = 7.0631 (7) Å | Mo <i>K</i> α radiation, λ = 0.71073 Å |
| <i>b</i> = 7.7065 (7) Å | Cell parameters from 5976 reflections |
| <i>c</i> = 8.3372 (8) Å | θ = 2.6–36.8° |
| α = 84.603 (3)° | μ = 0.75 mm ⁻¹ |
| β = 73.339 (3)° | <i>T</i> = 100 K |
| γ = 77.387 (3)° | Pinacoid, colourless |
| <i>V</i> = 424.03 (7) Å ³ | 0.12 × 0.09 × 0.02 mm |

Data collection

| | |
|--|---|
| Bruker APEXII CCD diffractometer | 4431 independent reflections |
| ω- and φ-scans | 3120 reflections with <i>I</i> > 2σ(<i>I</i>) |
| Absorption correction: multi-scan (<i>SADABS</i> ; Krause <i>et al.</i> , 2015) | <i>R_{int}</i> = 0.051 |
| <i>T_{min}</i> = 0.708, <i>T_{max}</i> = 0.747 | θ _{max} = 37.9°, θ _{min} = 2.6° |
| 30998 measured reflections | <i>h</i> = -12→12 |
| | <i>k</i> = -13→13 |
| | <i>l</i> = -14→14 |

Refinement

| | |
|-------------------------------------|---|
| Refinement on <i>F</i> ² | <i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)] = 0.033 |
| Least-squares matrix: full | <i>wR</i> (<i>F</i> ²) = 0.081 |

$S = 1.02$
 4431 reflections
 163 parameters
 0 restraints
 Hydrogen site location: difference Fourier map
 All H-atom parameters refined

$$w = 1/[\sigma^2(F_o^2) + (0.0361P)^2 + 0.0561P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.52 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.54 \text{ e } \text{\AA}^{-3}$

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|---------------|---------------|--------------|----------------------------------|
| Mg1 | 0.25213 (6) | 0.25317 (5) | 0.49234 (5) | 0.00567 (7) |
| S1 | -0.19941 (4) | 0.38328 (3) | 0.73690 (3) | 0.00502 (6) |
| S2 | 0.70853 (4) | 0.10199 (3) | 0.26420 (3) | 0.00530 (6) |
| O1 | -0.39760 (12) | 0.34252 (11) | 0.75373 (10) | 0.00958 (15) |
| O2 | -0.05079 (12) | 0.28210 (10) | 0.59864 (10) | 0.00822 (14) |
| O3 | -0.20153 (13) | 0.57529 (10) | 0.70321 (10) | 0.00806 (14) |
| O4 | -0.14301 (13) | 0.33542 (11) | 0.89683 (10) | 0.00882 (15) |
| O5 | 0.90861 (12) | 0.13643 (11) | 0.23946 (11) | 0.01002 (15) |
| O6 | 0.68946 (12) | -0.08145 (10) | 0.31322 (10) | 0.00829 (15) |
| O7 | 0.55714 (12) | 0.22644 (10) | 0.38042 (10) | 0.00864 (15) |
| O8 | 0.66111 (13) | 0.12937 (11) | 0.09245 (10) | 0.00890 (15) |
| O9 | 0.26987 (14) | 0.45448 (11) | 0.63118 (11) | 0.00911 (15) |
| O10 | 0.23916 (13) | 0.04746 (11) | 0.35662 (11) | 0.00886 (15) |
| N1 | 0.75582 (17) | 0.73990 (14) | 0.00652 (14) | 0.00946 (16) |
| H1W | 0.369 (3) | 0.456 (3) | 0.660 (3) | 0.030 (5)* |
| H2W | 0.220 (4) | 0.554 (3) | 0.621 (3) | 0.052 (7)* |
| H3W | 0.289 (4) | -0.051 (3) | 0.354 (3) | 0.038 (6)* |
| H4W | 0.143 (3) | 0.048 (2) | 0.324 (2) | 0.020 (5)* |
| H1O | 0.735 (4) | 0.204 (4) | 0.024 (3) | 0.080 (9)* |
| H1N | 0.863 (3) | 0.707 (2) | 0.047 (2) | 0.018 (4)* |
| H2N | 0.650 (3) | 0.717 (2) | 0.078 (2) | 0.025 (5)* |
| H3N | 0.784 (3) | 0.688 (2) | -0.088 (2) | 0.020 (4)* |
| H4N | 0.726 (3) | 0.855 (3) | -0.002 (2) | 0.022 (5)* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|--------------|--------------|--------------|---------------|---------------|--------------|
| Mg1 | 0.00613 (15) | 0.00512 (15) | 0.00574 (16) | -0.00112 (12) | -0.00169 (12) | 0.00014 (12) |
| S1 | 0.00499 (11) | 0.00546 (10) | 0.00472 (11) | -0.00155 (8) | -0.00140 (8) | 0.00075 (8) |
| S2 | 0.00537 (11) | 0.00583 (11) | 0.00482 (11) | -0.00165 (8) | -0.00150 (8) | 0.00079 (8) |
| O1 | 0.0057 (3) | 0.0144 (4) | 0.0092 (4) | -0.0041 (3) | -0.0018 (3) | 0.0010 (3) |
| O2 | 0.0072 (3) | 0.0083 (3) | 0.0085 (3) | -0.0008 (3) | -0.0011 (3) | -0.0020 (3) |
| O3 | 0.0121 (4) | 0.0046 (3) | 0.0078 (3) | -0.0014 (3) | -0.0038 (3) | 0.0006 (3) |

| | | | | | | |
|-----|------------|------------|------------|-------------|-------------|-------------|
| O4 | 0.0106 (4) | 0.0106 (3) | 0.0068 (3) | -0.0046 (3) | -0.0046 (3) | 0.0046 (3) |
| O5 | 0.0059 (3) | 0.0143 (4) | 0.0106 (4) | -0.0042 (3) | -0.0026 (3) | 0.0021 (3) |
| O6 | 0.0115 (4) | 0.0059 (3) | 0.0079 (3) | -0.0018 (3) | -0.0039 (3) | 0.0018 (3) |
| O7 | 0.0074 (3) | 0.0075 (3) | 0.0104 (4) | -0.0016 (3) | -0.0009 (3) | -0.0020 (3) |
| O8 | 0.0118 (4) | 0.0110 (3) | 0.0056 (3) | -0.0054 (3) | -0.0039 (3) | 0.0035 (3) |
| O9 | 0.0098 (4) | 0.0076 (3) | 0.0114 (4) | -0.0016 (3) | -0.0053 (3) | -0.0007 (3) |
| O10 | 0.0081 (4) | 0.0069 (3) | 0.0128 (4) | -0.0007 (3) | -0.0049 (3) | -0.0020 (3) |
| N1 | 0.0102 (4) | 0.0101 (4) | 0.0082 (4) | -0.0035 (3) | -0.0015 (3) | -0.0010 (3) |

Geometric parameters (Å, °)

| | | | |
|---------------------------------------|-------------|-------------------------|------------|
| Mg1—O2 | 2.0382 (9) | S2—O7 | 1.4611 (8) |
| Mg1—O7 | 2.0601 (9) | S2—O8 | 1.5474 (9) |
| Mg1—O3 ⁱ | 2.0630 (9) | O8—H1O | 0.91 (3) |
| Mg1—O10 | 2.0645 (9) | O9—H1W | 0.81 (2) |
| Mg1—O9 | 2.0660 (10) | O9—H2W | 0.78 (3) |
| Mg1—O6 ⁱⁱ | 2.0715 (9) | O10—H3W | 0.77 (2) |
| S1—O1 | 1.4659 (9) | O10—H4W | 0.80 (2) |
| S1—O2 | 1.4685 (8) | N1—H1N | 0.896 (19) |
| S1—O3 | 1.4775 (8) | N1—H2N | 0.85 (2) |
| S1—O4 | 1.4901 (9) | N1—H3N | 0.868 (19) |
| S2—O5 | 1.4480 (9) | N1—H4N | 0.870 (19) |
| S2—O6 | 1.4583 (8) | | |
| O2—Mg1—O7 | 178.82 (4) | O5—S2—O7 | 111.08 (5) |
| O2—Mg1—O3 ⁱ | 90.48 (4) | O6—S2—O7 | 111.15 (5) |
| O7—Mg1—O3 ⁱ | 88.37 (4) | O5—S2—O8 | 107.57 (5) |
| O2—Mg1—O10 | 88.76 (4) | O6—S2—O8 | 103.50 (5) |
| O7—Mg1—O10 | 90.94 (4) | O7—S2—O8 | 108.31 (5) |
| O3 ⁱ —Mg1—O10 | 88.11 (4) | S1—O2—Mg1 | 135.68 (5) |
| O2—Mg1—O9 | 91.90 (4) | S1—O3—Mg1 ⁱ | 132.86 (5) |
| O7—Mg1—O9 | 88.42 (4) | S2—O6—Mg1 ⁱⁱ | 134.27 (5) |
| O3 ⁱ —Mg1—O9 | 93.19 (4) | S2—O7—Mg1 | 134.89 (5) |
| O10—Mg1—O9 | 178.53 (4) | S2—O8—H1O | 110.9 (17) |
| O2—Mg1—O6 ⁱⁱ | 90.93 (4) | Mg1—O9—H1W | 122.6 (15) |
| O7—Mg1—O6 ⁱⁱ | 90.23 (4) | Mg1—O9—H2W | 124.0 (19) |
| O3 ⁱ —Mg1—O6 ⁱⁱ | 178.55 (4) | H1W—O9—H2W | 105 (2) |
| O10—Mg1—O6 ⁱⁱ | 92.25 (4) | Mg1—O10—H3W | 132.2 (17) |
| O9—Mg1—O6 ⁱⁱ | 86.43 (4) | Mg1—O10—H4W | 121.5 (14) |
| O1—S1—O2 | 108.73 (5) | H3W—O10—H4W | 102 (2) |
| O1—S1—O3 | 111.52 (5) | H1N—N1—H2N | 111.1 (17) |
| O2—S1—O3 | 109.47 (5) | H1N—N1—H3N | 107.9 (16) |
| O1—S1—O4 | 109.60 (5) | H2N—N1—H3N | 113.2 (19) |
| O2—S1—O4 | 110.65 (5) | H1N—N1—H4N | 109.1 (17) |
| O3—S1—O4 | 106.87 (5) | H2N—N1—H4N | 101.8 (17) |
| O5—S2—O6 | 114.71 (5) | H3N—N1—H4N | 113.6 (17) |

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

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|------------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| O8—H1O \cdots O4 ⁱⁱⁱ | 0.91 (3) | 1.59 (3) | 2.5048 (12) | 177 (3) |
| O9—H1W \cdots O1 ^{iv} | 0.81 (2) | 2.02 (2) | 2.7623 (13) | 154 (2) |
| O9—H2W \cdots O2 ⁱ | 0.78 (3) | 2.56 (3) | 3.1278 (12) | 132 (2) |
| O9—H2W \cdots O5 ^v | 0.78 (3) | 2.62 (3) | 3.2887 (12) | 146 (2) |
| O9—H2W \cdots O7 ^v | 0.78 (3) | 2.55 (3) | 2.9578 (12) | 115 (2) |
| O10—H3W \cdots O1 ^{vi} | 0.77 (2) | 2.38 (2) | 3.0970 (12) | 156 (2) |
| O10—H3W \cdots O2 ^{vi} | 0.77 (2) | 2.64 (2) | 3.0615 (12) | 117 (2) |
| O10—H4W \cdots O5 ^{vii} | 0.80 (2) | 1.95 (2) | 2.7125 (13) | 160.5 (19) |
| N1—H1N \cdots O4 ^v | 0.896 (19) | 2.108 (19) | 2.9913 (14) | 168.2 (15) |
| N1—H2N \cdots O1 ⁱ | 0.85 (2) | 2.04 (2) | 2.8920 (14) | 179.2 (18) |
| N1—H3N \cdots O3 ⁱⁱⁱ | 0.868 (19) | 1.98 (2) | 2.8451 (14) | 170.5 (18) |
| N1—H4N \cdots O8 ^{viii} | 0.870 (19) | 2.229 (19) | 3.0368 (14) | 154.4 (16) |

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

Ammonium magnesium hydrogen sulfate sulfate dihydrate (NH₄MgHSO₄2H₂O_296K)

Crystal data

(NH₄)Mg(HSO₄)(SO₄)(H₂O)₂

$M_r = 271.51$

Triclinic, $P\bar{1}$

$a = 4.6771$ (1) Å

$b = 5.7697$ (1) Å

$c = 8.3697$ (2) Å

$\alpha = 104.208$ (1)°

$\beta = 98.189$ (1)°

$\gamma = 94.508$ (1)°

$V = 215.20$ (1) Å³

$Z = 1$

$F(000) = 140$

$D_x = 2.095$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 7912 reflections

$\theta = 2.6$ – 35.3 °

$\mu = 0.73$ mm⁻¹

$T = 296$ K

Plate, colourless

$0.12 \times 0.09 \times 0.01$ mm

Data collection

Bruker APEXII CCD

diffractometer

ω - and ϕ -scans

Absorption correction: multi-scan

(*SADABS*; Krause *et al.*, 2015)

$T_{\min} = 0.699$, $T_{\max} = 0.747$

11147 measured reflections

1940 independent reflections

1843 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.024$

$\theta_{\max} = 35.3$ °, $\theta_{\min} = 3.7$ °

$h = -7 \rightarrow 7$

$k = -9 \rightarrow 9$

$l = -13 \rightarrow 13$

Refinement

Refinement on F^2

Least-squares matrix: full

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

$wR(F^2) = 0.054$

$S = 1.18$

1940 reflections

95 parameters

0 restraints

Hydrogen site location: difference Fourier map

All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0253P)^2 + 0.0476P]$

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

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

$\Delta\rho_{\max} = 0.30$ e Å⁻³

$\Delta\rho_{\min} = -0.47$ e Å⁻³

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ | Occ. (<1) |
|-----|---------------|---------------|--------------|----------------------------------|-----------|
| Mg1 | 0.000000 | 0.000000 | 0.000000 | 0.01247 (7) | |
| S1 | −0.59079 (4) | −0.31416 (3) | −0.23679 (2) | 0.01173 (5) | |
| O1 | −0.51060 (17) | −0.30087 (12) | −0.40386 (8) | 0.02378 (13) | |
| O2 | −0.32610 (14) | −0.27473 (11) | −0.11297 (9) | 0.02162 (12) | |
| O3 | −0.74294 (16) | −0.55094 (11) | −0.25509 (9) | 0.02480 (13) | |
| O4 | −0.76973 (14) | −0.11827 (12) | −0.19257 (8) | 0.02018 (11) | |
| O5 | 0.18095 (15) | −0.22150 (12) | 0.13791 (9) | 0.02120 (12) | |
| H5A | 0.087 (4) | −0.321 (3) | 0.162 (2) | 0.049 (5)* | |
| H5B | 0.333 (5) | −0.281 (4) | 0.133 (3) | 0.060 (6)* | |
| N1 | 0.000000 | 0.000000 | −0.500000 | 0.0263 (2) | |
| H1A | −0.069 (11) | −0.139 (9) | −0.578 (6) | 0.063 (12)* | 0.5 |
| H1B | −0.132 (11) | 0.088 (9) | −0.505 (7) | 0.061 (13)* | 0.5 |
| H1C | −0.139 (12) | −0.062 (9) | −0.454 (7) | 0.069 (13)* | 0.5 |
| H1D | −0.076 (12) | 0.023 (10) | −0.589 (6) | 0.060 (13)* | 0.5 |
| H1O | −0.510 (7) | −0.438 (5) | −0.467 (4) | 0.029 (7)* | 0.5 |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|--------------|--------------|--------------|--------------|--------------|--------------|
| Mg1 | 0.01185 (14) | 0.01281 (15) | 0.01307 (15) | 0.00322 (11) | 0.00293 (11) | 0.00293 (11) |
| S1 | 0.01341 (7) | 0.01017 (7) | 0.01104 (7) | 0.00074 (5) | 0.00381 (5) | 0.00085 (5) |
| O1 | 0.0404 (4) | 0.0175 (3) | 0.0157 (2) | 0.0039 (2) | 0.0159 (2) | 0.0022 (2) |
| O2 | 0.0170 (2) | 0.0185 (3) | 0.0266 (3) | 0.00044 (19) | −0.0042 (2) | 0.0056 (2) |
| O3 | 0.0303 (3) | 0.0159 (2) | 0.0246 (3) | −0.0093 (2) | 0.0071 (2) | 0.0010 (2) |
| O4 | 0.0218 (3) | 0.0237 (3) | 0.0188 (2) | 0.0125 (2) | 0.0094 (2) | 0.0061 (2) |
| O5 | 0.0202 (3) | 0.0193 (3) | 0.0282 (3) | 0.0060 (2) | 0.0048 (2) | 0.0123 (2) |
| N1 | 0.0342 (6) | 0.0223 (5) | 0.0204 (5) | 0.0016 (4) | −0.0022 (4) | 0.0059 (4) |

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

| | | | |
|-----------------------|------------|----------------------|----------|
| Mg1—O2 | 2.0509 (6) | O5—H5A | 0.78 (2) |
| Mg1—O2 ⁱ | 2.0509 (6) | O5—H5B | 0.81 (2) |
| Mg1—O4 ⁱⁱ | 2.0720 (6) | N1—H1A | 0.90 (5) |
| Mg1—O4 ⁱⁱⁱ | 2.0720 (6) | N1—H1B | 0.83 (5) |
| Mg1—O5 | 2.0731 (6) | N1—H1C | 0.89 (6) |
| Mg1—O5 ⁱ | 2.0731 (6) | N1—H1D | 0.82 (5) |
| S1—O3 | 1.4525 (6) | N1—H1A ^{iv} | 0.90 (5) |
| S1—O2 | 1.4604 (6) | N1—H1B ^{iv} | 0.83 (5) |
| S1—O4 | 1.4651 (6) | N1—H1C ^{iv} | 0.89 (6) |

| | | | |
|---|------------|---|--------------|
| S1—O1 | 1.5164 (6) | N1—H1D ^{iv} | 0.82 (5) |
| O1—H1O | 0.84 (3) | | |
| O2—Mg1—O2 ⁱ | 180.0 | H1A—N1—H1C | 78 (4) |
| O2—Mg1—O4 ⁱⁱ | 90.33 (3) | H1B—N1—H1C | 75 (4) |
| O2 ⁱ —Mg1—O4 ⁱⁱ | 89.67 (3) | H1A—N1—H1D | 67 (4) |
| O2—Mg1—O4 ⁱⁱⁱ | 89.67 (3) | H1B—N1—H1D | 58 (4) |
| O2 ⁱ —Mg1—O4 ⁱⁱⁱ | 90.33 (3) | H1C—N1—H1D | 108 (4) |
| O4 ⁱⁱ —Mg1—O4 ⁱⁱⁱ | 180.0 | H1A—N1—H1A ^{iv} | 179.999 (12) |
| O2—Mg1—O5 | 88.48 (3) | H1B—N1—H1A ^{iv} | 75 (4) |
| O2 ⁱ —Mg1—O5 | 91.52 (3) | H1C—N1—H1A ^{iv} | 102 (4) |
| O4 ⁱⁱ —Mg1—O5 | 87.09 (3) | H1D—N1—H1A ^{iv} | 113 (4) |
| O4 ⁱⁱⁱ —Mg1—O5 | 92.91 (3) | H1A—N1—H1B ^{iv} | 75 (4) |
| O2—Mg1—O5 ⁱ | 91.52 (3) | H1B—N1—H1B ^{iv} | 180.00 (3) |
| O2 ⁱ —Mg1—O5 ⁱ | 88.48 (3) | H1C—N1—H1B ^{iv} | 105 (4) |
| O4 ⁱⁱ —Mg1—O5 ⁱ | 92.91 (3) | H1D—N1—H1B ^{iv} | 122 (4) |
| O4 ⁱⁱⁱ —Mg1—O5 ⁱ | 87.09 (3) | H1A ^{iv} —N1—H1B ^{iv} | 105 (4) |
| O5—Mg1—O5 ⁱ | 180.00 (2) | H1A—N1—H1C ^{iv} | 102 (4) |
| O3—S1—O2 | 109.75 (4) | H1B—N1—H1C ^{iv} | 105 (4) |
| O3—S1—O4 | 113.42 (4) | H1C—N1—H1C ^{iv} | 179.999 (12) |
| O2—S1—O4 | 110.24 (4) | H1D—N1—H1C ^{iv} | 72 (4) |
| O3—S1—O1 | 108.87 (4) | H1A ^{iv} —N1—H1C ^{iv} | 78 (4) |
| O2—S1—O1 | 109.29 (4) | H1B ^{iv} —N1—H1C ^{iv} | 75 (4) |
| O4—S1—O1 | 105.10 (4) | H1A—N1—H1D ^{iv} | 113 (4) |
| S1—O1—H1O | 112 (2) | H1B—N1—H1D ^{iv} | 122 (4) |
| S1—O2—Mg | 137.25 (4) | H1C—N1—H1D ^{iv} | 72 (4) |
| S1—O4—Mg ^v | 135.08 (4) | H1D—N1—H1D ^{iv} | 179.999 (17) |
| Mg—O5—H5A | 122.3 (15) | H1A ^{iv} —N1—H1D ^{iv} | 67 (4) |
| Mg—O5—H5B | 129.2 (15) | H1B ^{iv} —N1—H1D ^{iv} | 58 (4) |
| H5A—O5—H5B | 100 (2) | H1C ^{iv} —N1—H1D ^{iv} | 108 (4) |
| H1A—N1—H1B | 105 (4) | | |

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

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

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|------------------------------------|----------|-------------|-------------|---------------|
| O5—H5B \cdots O2 ^{vi} | 0.81 (2) | 2.52 (2) | 3.0010 (9) | 118.9 (19) |
| O5—H5A \cdots O3 ^{vii} | 0.78 (2) | 2.05 (2) | 2.7684 (9) | 154.0 (19) |
| O5—H5B \cdots O3 ^{vi} | 0.81 (2) | 2.42 (2) | 3.1678 (10) | 152 (2) |
| N1—H1A \cdots O3 ^{viii} | 0.90 (5) | 2.03 (5) | 2.9260 (6) | 175 (5) |
| N1—H1C \cdots O1 | 0.89 (6) | 2.28 (6) | 3.1536 (7) | 164 (4) |
| N1—H1B \cdots O1 ^{ix} | 0.83 (5) | 2.30 (5) | 3.1122 (8) | 164 (5) |
| N1—H1D \cdots O4 ^{ix} | 0.82 (5) | 2.08 (5) | 2.9042 (6) | 173 (5) |
| O1—H1O \cdots O1 ^{viii} | 0.84 (3) | 1.64 (3) | 2.4790 (12) | 176 (4) |

Symmetry codes: (vi) $-x, -y-1, -z$; (vii) $-x-1, -y-1, -z$; (viii) $-x-1, -y-1, -z-1$; (ix) $-x-1, -y, -z-1$.

Sodium scandium bis(chromate) dihydrate (NaScCrO₄2H₂O₂)

Crystal data

NaSc(CrO₄)₂(H₂O)₂
M_r = 335.98
 Monoclinic, *C*2/*c*
a = 14.505 (3) Å
b = 5.563 (1) Å
c = 10.763 (2) Å
 β = 109.82 (3)°
V = 817.0 (3) Å³
Z = 4

F(000) = 656
D_x = 2.731 Mg m⁻³
 Mo *K*α radiation, λ = 0.71073 Å
 Cell parameters from 1933 reflections
 θ = 3.0–34.9°
 μ = 3.51 mm⁻¹
T = 293 K
 Plate, orange
 0.17 × 0.10 × 0.03 mm

Data collection

Nonius KappaCCD
 diffractometer
 φ and ω scans
 Absorption correction: multi-scan
 (SCALEPACK; Otwinowski *et al.*, 2003)
T_{min} = 0.755, *T_{max}* = 0.949
 3418 measured reflections

1783 independent reflections
 1372 reflections with *I* > 2σ(*I*)
R_{int} = 0.017
 θ_{\max} = 34.9°, θ_{\min} = 3.0°
h = -23→23
k = -8→8
l = -17→17

Refinement

Refinement on *F*²
 Least-squares matrix: full
R[*F*² > 2σ(*F*²)] = 0.027
wR(*F*²) = 0.081
S = 1.08
 1783 reflections
 75 parameters
 2 restraints
 Hydrogen site location: difference Fourier map
 All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.039P)^2 + 0.8547P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 (Δ/σ)_{max} < 0.001
 Δρ_{max} = 1.06 e Å⁻³
 Δρ_{min} = -0.69 e Å⁻³
 Extinction correction: SHELXL2018
 (Sheldrick, 2015b),
 $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.0016 (4)

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.

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

| | <i>x</i> | <i>y</i> | <i>z</i> | <i>U_{iso}</i> */ <i>U_{eq}</i> |
|----|--------------|-------------|--------------|---|
| Na | 0.000000 | 0.7736 (2) | 0.250000 | 0.0316 (3) |
| Sc | 0.250000 | 0.250000 | 0.000000 | 0.01231 (10) |
| Cr | 0.40774 (2) | 0.75898 (4) | 0.04494 (2) | 0.01252 (9) |
| O1 | 0.34782 (10) | 0.4739 (2) | 0.58227 (12) | 0.0244 (3) |
| O2 | 0.36740 (9) | 0.0218 (2) | 0.08791 (11) | 0.0212 (2) |
| O3 | 0.39085 (11) | 0.2399 (2) | 0.38820 (13) | 0.0241 (3) |
| O4 | 0.52248 (9) | 0.7300 (2) | 0.12625 (15) | 0.0279 (3) |
| O5 | 0.21670 (10) | 0.2416 (2) | 0.17735 (13) | 0.0217 (3) |
| H1 | 0.1575 (19) | 0.237 (5) | 0.162 (4) | 0.075 (12)* |

| | | | | |
|----|-----------|-----------|-----------|-------------|
| H2 | 0.262 (2) | 0.224 (5) | 0.247 (3) | 0.070 (12)* |
|----|-----------|-----------|-----------|-------------|

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|----|--------------|--------------|--------------|---------------|--------------|---------------|
| Na | 0.0255 (6) | 0.0418 (7) | 0.0261 (6) | 0.000 | 0.0068 (4) | 0.000 |
| Sc | 0.01293 (17) | 0.00985 (16) | 0.01428 (17) | -0.00036 (11) | 0.00479 (13) | -0.00011 (11) |
| Cr | 0.01109 (13) | 0.01064 (12) | 0.01520 (13) | -0.00011 (7) | 0.00361 (8) | -0.00014 (7) |
| O1 | 0.0299 (6) | 0.0191 (5) | 0.0244 (6) | 0.0129 (5) | 0.0096 (5) | 0.0030 (4) |
| O2 | 0.0232 (5) | 0.0163 (5) | 0.0219 (6) | 0.0074 (4) | 0.0049 (4) | -0.0009 (4) |
| O3 | 0.0316 (7) | 0.0247 (6) | 0.0172 (5) | 0.0000 (4) | 0.0100 (5) | 0.0001 (4) |
| O4 | 0.0126 (5) | 0.0356 (7) | 0.0322 (7) | 0.0034 (4) | 0.0035 (5) | -0.0004 (5) |
| O5 | 0.0157 (5) | 0.0326 (7) | 0.0175 (5) | -0.0002 (4) | 0.0062 (4) | 0.0017 (4) |

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

| | | | |
|--|-------------|---|-------------|
| Na—O3 ⁱ | 2.5201 (16) | Sc—O2 ^{vii} | 2.0772 (12) |
| Na—O3 ⁱⁱ | 2.5201 (15) | Sc—O2 | 2.0772 (12) |
| Na—O2 ⁱ | 2.5358 (15) | Sc—O5 | 2.1222 (14) |
| Na—O2 ⁱⁱ | 2.5358 (15) | Sc—O5 ^{vii} | 2.1222 (14) |
| Na—O1 ⁱⁱⁱ | 2.7207 (17) | Cr—O4 | 1.6045 (14) |
| Na—O1 ^{iv} | 2.7207 (17) | Cr—O3 ^v | 1.6204 (14) |
| Na—O4 ⁱⁱ | 2.9360 (18) | Cr—O1 ^v | 1.6829 (12) |
| Na—O4 ⁱ | 2.9360 (18) | Cr—O2 ^{viii} | 1.6960 (11) |
| Sc—O1 ^v | 2.0747 (12) | O5—H1 | 0.82 (3) |
| Sc—O1 ^{vi} | 2.0747 (12) | O5—H2 | 0.82 (2) |
| O3 ⁱ —Na—O3 ⁱⁱ | 171.47 (7) | O1 ^{vi} —Sc—O2 ^{vii} | 85.51 (6) |
| O3 ⁱ —Na—O2 ⁱ | 83.30 (5) | O1 ^v —Sc—O2 | 85.51 (6) |
| O3 ⁱⁱ —Na—O2 ⁱ | 91.98 (5) | O1 ^{vi} —Sc—O2 | 94.49 (6) |
| O3 ⁱ —Na—O2 ⁱⁱ | 91.98 (5) | O2 ^{vii} —Sc—O2 | 180.0 |
| O3 ⁱⁱ —Na—O2 ⁱⁱ | 83.30 (5) | O1 ^v —Sc—O5 | 87.91 (5) |
| O2 ⁱ —Na—O2 ⁱⁱ | 112.92 (7) | O1 ^{vi} —Sc—O5 | 92.09 (5) |
| O3 ⁱ —Na—O1 ⁱⁱⁱ | 100.22 (5) | O2 ^{vii} —Sc—O5 | 91.24 (5) |
| O3 ⁱⁱ —Na—O1 ⁱⁱⁱ | 84.22 (4) | O2—Sc—O5 | 88.76 (5) |
| O2 ⁱ —Na—O1 ⁱⁱⁱ | 175.72 (4) | O1 ^v —Sc—O5 ^{vii} | 92.09 (5) |
| O2 ⁱⁱ —Na—O1 ⁱⁱⁱ | 64.74 (4) | O1 ^{vi} —Sc—O5 ^{vii} | 87.91 (5) |
| O3 ⁱ —Na—O1 ^{iv} | 84.22 (4) | O2 ^{vii} —Sc—O5 ^{vii} | 88.76 (5) |
| O3 ⁱⁱ —Na—O1 ^{iv} | 100.22 (5) | O2—Sc—O5 ^{vii} | 91.24 (5) |
| O2 ⁱ —Na—O1 ^{iv} | 64.74 (4) | O5—Sc—O5 ^{vii} | 180.0 |
| O2 ⁱⁱ —Na—O1 ^{iv} | 175.72 (4) | O4—Cr—O3 ^v | 109.32 (8) |
| O1 ⁱⁱⁱ —Na—O1 ^{iv} | 117.83 (7) | O4—Cr—O1 ^v | 108.45 (7) |
| O3 ⁱ —Na—O4 ⁱⁱ | 66.92 (4) | O3 ^v —Cr—O1 ^v | 110.09 (6) |
| O3 ⁱⁱ —Na—O4 ⁱⁱ | 121.38 (5) | O4—Cr—O2 ^{viii} | 109.14 (6) |
| O2 ⁱ —Na—O4 ⁱⁱ | 127.94 (4) | O3 ^v —Cr—O2 ^{viii} | 109.65 (6) |
| O2 ⁱⁱ —Na—O4 ⁱⁱ | 109.92 (4) | O1 ^v —Cr—O2 ^{viii} | 110.16 (7) |
| O1 ⁱⁱⁱ —Na—O4 ⁱⁱ | 56.12 (4) | Cr ^{ix} —O1—Sc ⁱⁱ | 143.31 (7) |
| O1 ^{iv} —Na—O4 ⁱⁱ | 70.41 (5) | Cr ^{ix} —O1—Na ⁱⁱⁱ | 100.80 (6) |

| | | | |
|---------------------------------------|-------------|--|------------|
| O3 ⁱ —Na—O4 ⁱ | 121.38 (5) | Sc ⁱⁱ —O1—Na ⁱⁱⁱ | 100.64 (5) |
| O3 ⁱⁱ —Na—O4 ⁱ | 66.92 (4) | Cr ^x —O2—Sc | 135.07 (7) |
| O2 ⁱ —Na—O4 ⁱ | 109.92 (4) | Cr ^x —O2—Na ^{xi} | 114.36 (6) |
| O2 ⁱⁱ —Na—O4 ⁱ | 127.94 (4) | Sc—O2—Na ^{xi} | 106.80 (5) |
| O1 ⁱⁱⁱ —Na—O4 ⁱ | 70.41 (5) | Cr ^{ix} —O3—Na ^{xi} | 135.29 (8) |
| O1 ^{iv} —Na—O4 ⁱ | 56.12 (4) | Cr—O4—Na ^{xi} | 94.60 (6) |
| O4 ⁱⁱ —Na—O4 ⁱ | 60.30 (6) | Sc—O5—H1 | 111 (3) |
| O1 ^v —Sc—O1 ^{vi} | 180.00 (11) | Sc—O5—H2 | 118 (3) |
| O1 ^v —Sc—O2 ^{vii} | 94.49 (6) | H1—O5—H2 | 130 (4) |

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

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|----------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| O5—H1 \cdots O4 ^{xii} | 0.82 (3) | 1.86 (3) | 2.682 (2) | 179 (3) |
| O5—H2 \cdots O3 | 0.82 (2) | 1.97 (3) | 2.765 (2) | 164 (3) |

Symmetry code: (xii) $x-1/2, y-1/2, z$.