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$(NH_4)Mg(HSO_4)(SO_4)(H_2O)_2$ and $NaSc(CrO_4)_2$-$(H_2O)_2$, two crystal structures comprising kröhnkite-type chains, and the temperature-induced phase transition $(NH_4)Mg(HSO_4)(SO_4)(H_2O)_2 \rightleftharpoons (NH_4)MgH(SO_4)_2(H_2O)_2$

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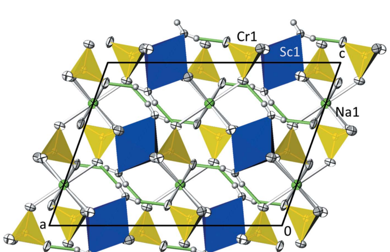
The crystal structure of the mineral kröhnkite, $Na_2Cu(SO_4)_2(H_2O)_2$, contains infinite chains composed of $[CuO_4(OH_2)_2]$ octahedra corner-linked with SO_4 tetrahedra. Such or similar tetrahedral–octahedral 'kröhnkite-type' chains are present in the crystal structures of numerous compounds with the composition $A_nM(XO_4)_2(H_2O)_2$. The title compounds, $(NH_4)Mg(HSO_4)(SO_4)(H_2O)_2$, ammonium magnesium hydrogen sulfate sulfate dihydrate, and $NaSc(CrO_4)_2$-$(H_2O)_2$, sodium scandium bis(chromate) dihydrate, are members of the large family with such kröhnkite-type chains. At 100 K, $(NH_4)Mg(HSO_4)(SO_4)$-$(H_2O)_2$ has an unprecedented triclinic crystal structure and contains $[MgO_4(OH_2)_2]$ octahedra linked by $SO_3(OH)$ and SO_4 tetrahedra into chains extending parallel to $[\overline{1}10]$. Adjacent chains are linked by very strong hydrogen bonds between $SO_3(OH)$ 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. $(NH_4)Mg(HSO_4)(SO_4)(H_2O)_2$ 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 $NaSc(CrO_4)_2(H_2O)_2$ adopts structure type **F1** in the classification scheme for structures with kröhnkite-type chains. Here, $[ScO_4(OH_2)_2]$ octahedra (point group symmetry $\overline{1}$) 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 $NaSc(CrO_4)_2(H_2O)_2$ and its isotypic $NaM(CrO_4)_2(H_2O)_2$ (M = 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-$(XO_4)_2(H_2O)_2$ (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 $A_nM(XO_4)_2(H_2O)_2$ compounds frequently comprise infinite chains composed of more or less distorted $[MO_4(OH_2)_2]$

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	$M1^{II}{}_2M2^{II}(TO_4)_2(H_2O)_2$: $M1$ = Ca, Sr; M^{II} = Mg, Mn, Fe, Co, Ni, Zn; T = P, As. $M1^{I}{}_2M2^{II}(TO_4)_2(H_2O)_2$: $M1$ = Na, K, NH_4; M^{II} = Mg, Fe, Co, Ni, Cu, Zn; T = S, Se, Cr, Mo, W	$P\bar{1}$	1	$a \simeq 5.7$–7.1, $b \simeq 6.7$–7.9, $c \simeq 5.3$–6.0, $\alpha \simeq 93$–105, $\beta \simeq 105$–112, $\gamma \simeq 103$–111	32
B	$M1^{II}{}_2M2^{II}(TO_4)_2(H_2O)_2$: $M1$ = Ca; $M2$ = Mn, Fe; T = P	$P\bar{1}$	1	$a \simeq 5.8$–6.0, $b \simeq 6.5$–6.6, $c \simeq 5.5$, $\alpha \simeq 102$–103, $\beta \simeq 108$–109, $\gamma \simeq 90$–91	2
C	$M1^{I}{}_2M2^{II}(TO_4)_2(H_2O)_2$: $M1$ = K, Rb; $M2$ = Mn, Cd; T = S, Se, Cr	$P\bar{1}$	2	$a \simeq 6.6$–6.9, $b \simeq 7.3$–7.7, $c \simeq 10.7$–11.4, $\alpha \simeq 72$–73, $\beta \simeq 74$–75, $\gamma \simeq 70$	4
C1	$K_2Fe(SO_4)_2(H_2O)_2$	$P1^a$	2	$a \simeq 6.6$, $b \simeq 7.3$, $c \simeq 10.7$, $\alpha \simeq 73$, $\beta \simeq 74$, $\gamma \simeq 70$	1
D	$M1^{II}{}_2M2^{II}(TO_4)_2(H_2O)_2$: $M1$ = Ca; $M2$ = Mg, Mn, Co, Cu, Zn; T = As. $M1^{I}{}_2M2^{II}(TO_4)_2(H_2O)_2$: $M1$ = Na, NH_4, Rb; $M2$ = Cr, Mn, Fe, Cu, Cd; T = S, Se, Cr, Mo	$P2_1/c$	2	$a \simeq 5.8$–6.8, $b \simeq 12.8$–14.3, $c \simeq 5.4$–5.9, $\beta \simeq 106$–111	14
E	$M1^{I}M2^{II}H(TO_4)_2(H_2O)_2$: $M1$ = K, NH_4, Cs; $M2$ = Mg, Mn, Fe, Co, Zn; T = S, Se	$P\bar{1}$	1	$a \simeq 4.6$–4.8, $b \simeq 5.7$–5.9, $c \simeq 8.1$–8.6, $\alpha \simeq 103$–104, $\beta \simeq 96$–100, $\gamma \simeq 94$–97	8
E1^b	$(NH_4)Mg(HSO_4)(SO_4)(H_2O)_2$ (at 100 K)	$P\bar{1}$	2	$a \simeq 7.1$, $b \simeq 7.7$, $c \simeq 8.3$, $\alpha \simeq 84.6$, $\beta \simeq 73.3$, $\gamma \simeq 77.4$	1
F1^c	$M1^{I}M2^{III}(TO_4)_2(H_2O)_2$: $M1$ = Na; $M2$ = Al, Sc, Fe; T = Cr	$C2/c$	4	$a \simeq 14.1$–14.5, $b \simeq 5.3$–5.6, $c \simeq 10.7$–10.8, $\beta \simeq 109$–110	3
F2^c	$M1^{I}M2^{III}(TO_4)_2(H_2O)_2$: $M1$ = Na, K, NH_4, Tl; $M2$ = Al, Fe, In; T = Cr	$C2/m$	2	$a \simeq 10.7$–11.0, $b \simeq 5.4$–5.6, $c \simeq 7.5$–7.6, $\beta \simeq 114$	5
G	$AgSc(CrO_4)_2(H_2O)_2$	$P\bar{1}$	1	$a \simeq 5.6$, $b \simeq 6.1$, $c \simeq 7.4$, $\alpha \simeq 111$, $\beta \simeq 90$, $\gamma \simeq 117$	1
H^d	$K_2Zn(CrO_4)_2(H_2O)_2$	$C2/c$	4	$a \simeq 15.0$, $b \simeq 5.7$, $c \simeq 12.3$, $\beta \simeq 117$	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 [$Na_2Cu(SO_4)_2$-$(H_2O)_2$; Dahlman, 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 occurence 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.*, 2002*a*; 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

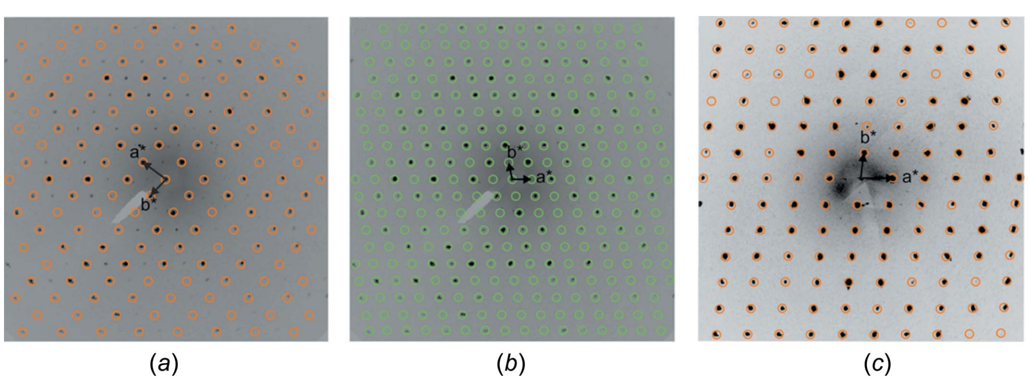


Figure 1
The *hk*1 plane of reciprocal space of $(NH_4)Mg(HSO_4)(SO_4)(H_2O)_2$ [100 K data in parts (*a*)/(*b*)] and of $(NH_4)MgH(SO_4)_2(H_2O)_2$ [296 K data in part (*c*)] reconstructed from CCD data. (*a*) The small cell with matching reflections is marked with orange circles; no superstructure 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.

	$(NH_4)Mg(HSO_4)(SO_4)(H_2O)_2$ at 100 K	$(NH_4)MgH(SO_4)_2(H_2O)_2$ at 296 K	$NaSc(CrO_4)_2(H_2O)_2$
Crystal data			
Chemical formula	$(NH_4)Mg(HSO_4)(SO_4)(H_2O)_2$	$(NH_4)Mg(HSO_4)(SO_4)(H_2O)_2$	$NaSc(CrO_4)_2(H_2O)_2$
M_r	271.51	271.51	335.98
Crystal system, space group	Triclinic, $P\overline{1}$	Triclinic, $P\overline{1}$	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 (*SADABS*; Krause *et al.*, 2015)	Multi-scan (*SADABS*; Krause *et al.*, 2015)	Multi-scan (*SCALEPACK*; Otwinowski *et al.*, 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
$(\sin \theta/\lambda)_{max}$ (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
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (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, 2015*a*), *SHELXS97* (Sheldrick, 2008), *SHELXL2018* (Sheldrick, 2015*b*), *ATOMS* (Dowty, 2006) and *publCIF* (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 ≃ 0.1 mm). Powder X-ray diffraction (PXRD) of the bulk revealed spiroffite-type $Mg_2Te_3O_8$ (Lin *et al.*, 2013) as the main product, and $MgTe_2O_5$ (Weil, 2005) as a minor product. The grown crystals correspond to the title compound. Structure refinement showed NH_4^+ 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 $(NH_4)Mg(HSO_4)(SO_4)(H_2O)_2$, equimolar aqueous solutions of NH_4HSO_4 and $MgSO_4(H_2O)_7$ were mixed at room temperature and stirred for homogeneity. The used NH_4HSO_4 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 NH_4HSO_4 and $MgSO_4$ solutions were evaporated to dryness at 353 K in a drying oven and also much more slowly at room temperature. Semi-quantitaive phase analysis using the Rietveld method with *HighScore Plus* (Degen *et al.*, 2014) revealed a phase mixture of $(NH_4)MgH(SO_4)_2(H_2O)_2$ and synthetic boussingaultite [$(NH_4)_2Mg(SO_4)_2(H_2O)_6$] in a ratio of ≃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 $(NH_4)MgH(SO_4)_2(H_2O)_2$ 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. $NaSc(CrO_4)_2(H_2O)_2$. 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 Na_2CO_3 (Merck), Sc_2O_3 (99.99%, alphametall, Germany) and reagent-grade CrO_3 (Merck). The crystals were often arranged in radiating clusters. They were associated with pale orange–yellow blade-shaped crystals of $Na_2Cr_2O_7{\cdot}2H_2O$ (Casari *et al.*, 2007).

2.2. Refinement

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

For the refinement of $(NH_4)Mg(HSO_4)(SO_4)(H_2O)_2$ (100 K data), all H atoms were located from difference Fourier maps and were refined freely. Reflections $\overline{2}03$, 102, 100 and 230 were obstructed by the beam stop and were therefore omitted from the refinement. For the structure analysis of $(NH_4)MgH(SO_4)_2(H_2O)_2$ (296 K data), a different crystal was measured (resulting in a different orienting matrix; see Fig. 1). For

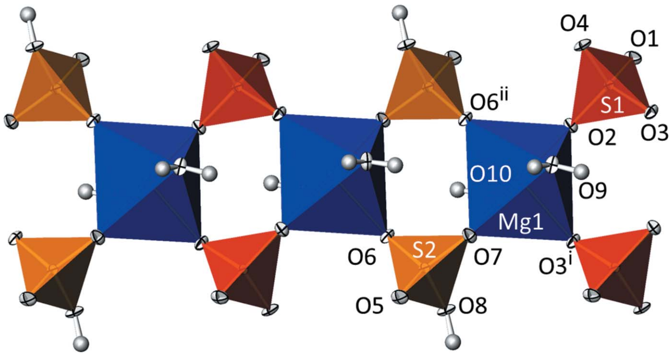


Figure 2
The kröhnkite-type chain in the crystal structure of $(NH_4)Mg(HSO_4)$-$(SO_4)(H_2O)_2$ at 100 K. $[MgO_4(OH_2)_2]$ octahedra are blue, $SO_3(OH)$ tetrahedra are orange and SO_4 tetrahedra are red. Displacement ellipsoids are drawn at the 74% probability level and H atoms are given as grey spheres 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 isotypic Fe compound (Heinicke *et al.*, 2004). In this structure, the H atoms (H1*A*–H1*D*) 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 $NaSc(CrO_4)_2(H_2O)_2$, the coordinates and labelling of atoms were taken from isotypic $NaFe(CrO_4)_2$-$(H_2O)_2$ (Hardy & Gravereau, 1970). The H atoms of the water molecule were located from a difference Fourier map and were refined with a constraint of O—H = 0.90 ± 0.03 Å.

3. Results and discussion

3.1. $(NH_4)Mg(HSO_4)(SO_4)(H_2O)_2$

3.1.1. Structure analysis. $(NH_4)Mg(HSO_4)(SO_4)(H_2O)_2$ was obtained serendipitously from a hydrothermal synthesis intended to crystallize a compound in the system Mg–S^{VI}–Te^{IV}–O–H (Weil & Shirkhanlou, 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 $MgSO_4$.

$(NH_4)Mg(HSO_4)(SO_4)(H_2O)_2$ is the fourth compound in the NH_3–MgO–SO_3–H_2O system. The three other known members are the two minerals boussingaultite, *i.e.* $(NH_4)_2Mg$-$(SO_4)_2(H_2O)_6$ (Maslen *et al.*, 1988), and efremovite, *i.e.* $(NH_4)_2Mg_2(SO_4)_3$ (Shcherbakova & Bazhenova, 1989), and synthetic $(NH_4)_2Mg_3(OH)_2(SO_4)_3(H_2O)_2$ (Marri *et al.*, 2017). Monoclinic boussingaultite is a representative of the picromerite group and crystallizes isotypically with many other synthetic $A^I_2M^{II}(XO_4)_2(H_2O)_6$ compounds (A^I = NH_4, K, Rb, Cs or Tl; M^{II} = Mg, V, Cr, Mn, Fe, Co, Ni, Cu, Zn or Cd; X = S, 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 $(NH_4)_2Mg_3(OH)_2(SO_4)_3(H_2O)_2$ is isotypic with its cadmium analogue $(NH_4)_2Cd_3(OH)_2(SO_4)_3(H_2O)_2$ (Yin, 2011).

$(NH_4)Mg(HSO_4)(SO_4)(H_2O)_2$ 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.*, 2002*a*; Table 1). All atoms in the triclinic structure are situated on general positions. $[MgO_4(OH_2)]$ octahedra are corner-linked by $SO_3(OH)$ 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 Mg—O bond lengths in the $[MgO_4(OH_2)_2]$ 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 Mg—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 S—O distance is 1.473 Å, with minimum and maximum S—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

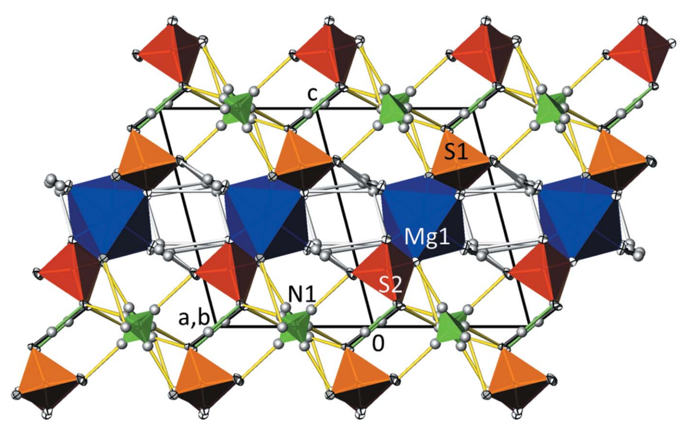


Figure 3
The crystal structure of $(NH_4)Mg(HSO_4)(SO_4)(H_2O)_2$ at 100 K in a projection along $[\bar{1}10]$. $[MgO_4(OH_2)_2]$ octahedra are blue, SO_4 tetrahedra are red, $SO_3(OH)$ tetrahedra are orange and ammonium groups are green. The strong hydrogen bond between the $SO_3(OH)$ and SO_4 tetrahedra 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 $(NH_4)Mg(HSO_4)(SO_4)(H_2O)_2$ 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) $-x+1, -y, -z+1$.

Table 4
Hydrogen-bond geometry (Å, °) for $(NH_4)Mg(HSO_4)(SO_4)(H_2O)_2$ at 100 K.

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O8—H1O$\cdots$O4^{iii}	0.91 (3)	1.59 (3)	2.5048 (12)	177 (3)
O9—H1$W\cdots$O1^{iv}	0.81 (2)	2.02 (2)	2.7623 (13)	154 (2)
O9—H2$W\cdots$O2^{i}	0.78 (3)	2.56 (3)	3.1278 (12)	132 (2)
O9—H2$W\cdots$O5^{v}	0.78 (3)	2.62 (3)	3.2887 (12)	146 (2)
O9—H2$W\cdots$O7^{v}	0.78 (3)	2.55 (3)	2.9578 (12)	115 (2)
O10—H3$W\cdots$O1^{vi}	0.77 (2)	2.38 (2)	3.0970 (12)	156 (2)
O10—H3$W\cdots$O2^{vi}	0.77 (2)	2.64 (2)	3.0615 (12)	117 (2)
O10—H4$W\cdots$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) $x, y+1, z$.

sulfate group. The corresponding $S2O_3(OH)$ 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.* $Mg(HSO_4)_2(H_2O)$ (Worzala *et al.*, 1991) or $Th(HSO_4)_2(SO_4)$ (Betke & Wickleder, 2012). In the magnesium compound, with its two independent $SO_3(OH)$ 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 $SO_3(OH)$ tetrahedra; the SO_4 group in the thorium compound has a mean S—O bond length of 1.467 Å.

In the crystal structure of $(NH_4)Mg(HSO_4)(SO_4)(H_2O)_2$, the short hydrogen bond between the $S2O_3(OH)$ and the $S1O_4$ tetrahedra [O8$\cdots$O4^{iii} = 2.5048 (12) Å; Table 4] is linear [177 (3)°] and considered as strong (Jeffrey, 1997). In comparison, the other types of O—H$\cdots$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 O2^{i}, O5^{v} and O7^{v}; numerical values of these interactions, as well as

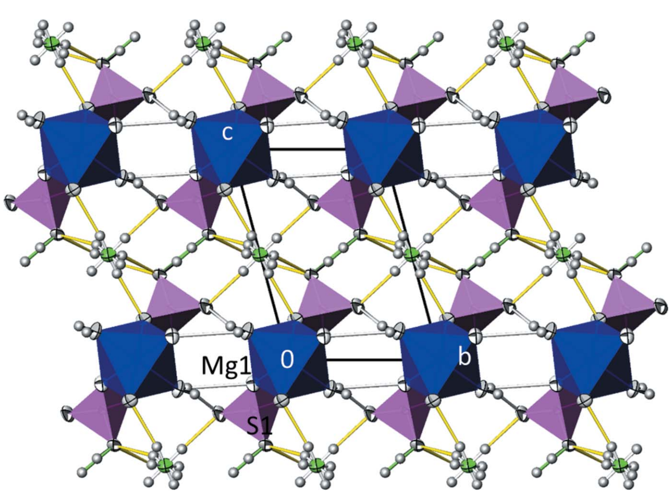


Figure 4
The crystal structure of $(NH_4)MgH(SO_4)_2(H_2O)_2$ at 296 K in a projection along [100]. Displacement ellipsoids and colour codes are as in Fig. 2, except for the SO_4 tetrahedra which are lilac. The disordered ammonium group and the H1O atom disordered between two sulfate tetrahedra are shown.

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$\cdots$O angles range from 168.2 (15) to 179.2 (18)°] by the O atoms of the sulfate group (O4^{v}, O1^{i} and O3^{iii}) 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'Keeffe (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, $(NH_4)Mg(HSO_4)(SO_4)(H_2O)_2$ 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 $P\overline{1}$, $Z = 1$) in the classification of compounds with kröhnkite-type chains (Table 1). Next to the six isotypic sulfates $KFeH(SO_4)_2(H_2O)_2$ (Fleck *et al.*, 2002*b*), $KMgH(SO_4)_2(H_2O)_2$ (Macíček *et al.*, 1994), $KZnH(SO_4)_2(H_2O)_2$, $KMnH(SO_4)_2(H_2O)_2$, $CsMnH(SO_4)_2(H_2O)_2$ (Troyanov *et al.*, 2002) and $NH_4FeH(SO_4)_2(H_2O)_2$ (Heinicke *et al.*, 2004), and the selenate $KMgH(SeO_4)_2(H_2O)_2$ (Troyanov *et al.*, 2002), $(NH_4)Mg(HSO_4)(SO_4)(H_2O)_2$, or more precisely $(NH_4)MgH(SO_4)_2(H_2O)_2$ at this temperature, is the eighth member of this structure type. The $[M^{II}O_4(OH_2)_2]$ octahedron in these structures (Fig. 4 and Table 5) is located on an inversion centre, just like the A cation (for A = NH_4; 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$\cdots$O contacts around 2.5 Å (Table 6). In comparison, in the crystal structure of $(NH_4)Mg(HSO_4)(SO_4)(H_2O)_2$ at 100 K, the H atom is ordered between two sulfate tetrahedra, defining distinct SO_3OH and SO_4 groups. This

Table 5
Selected bond lengths (Å) for $(NH_4)MgH(SO_4)_2(H_2O)_2$ 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 O1···O1^{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 $(NH_4)MgH(SO_4)_2(H_2O)_2$.

The crystal structure of $(NH_4)Mg(HSO_4)(SO_4)(H_2O)_2$ at 100 K represents a twofold superstructure with ordered H atoms for the ammonium and hydrogen sulfate groups relative to the crystal structure of $(NH_4)MgH(SO_4)_2(H_2O)_2$ at 296 K with a halved unit-cell volume. The subcell of the latter is related to the doubled cell of the $(NH_4)Mg(HSO_4)(SO_4)$-$(H_2O)_2$ 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 *hk*1 plane of reciprocal space of $(NH_4)Mg(HSO_4)(SO_4)(H_2O)_2$ and the relation of the subcell (Fig. 1*a*) and the cell of the actual superstructure (Fig. 1*b*); the missing reflections for the subcell clearly indicate that the doubled cell is correct at this temperature. Fig. 1(*c*)

Table 6
Hydrogen-bond geometry (Å, °) for $(NH_4)MgH(SO_4)_2(H_2O)_2$ at 296 K.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5*B*···O2^{ii}	0.81 (2)	2.52 (2)	3.0010 (9)	118.9 (19)
O5—H5*A*···O3^{iii}	0.78 (2)	2.05 (2)	2.7684 (9)	154.0 (19)
O5—H5*B*···O3^{ii}	0.81 (2)	2.42 (2)	3.1678 (10)	152 (2)
N1—H1*A*···O3^{iv}	0.90 (5)	2.03 (5)	2.9260 (6)	175 (5)
N1—H1*C*···O1	0.89 (6)	2.28 (6)	3.1536 (7)	164 (4)
N1—H1*B*···O1^{v}	0.83 (5)	2.30 (5)	3.1122 (8)	164 (5)
N1—H1*D*···O4^{v}	0.82 (5)	2.08 (5)	2.9042 (6)	173 (5)
O1—H1O···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 *hk*1 plane of reciprocal space of $(NH_4)MgH$-$(SO_4)_2(H_2O)_2$ 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 $(NH_4)M^{II}(HSO_4)(SO_4)(H_2O)_2 \rightleftharpoons (NH_4)M^{II}H(SO_4)_2$-$(H_2O)_2$ (*M* = first-row transition metals) phases, are underway.

3.1.3. IR spectroscopy. The IR spectrum of $(NH_4)Mg$-$H(SO_4)_2(H_2O)_2$ shows similarities to that of synthetic boussingaultite (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 3700–2500 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 $\nu_3(NH_4)$ stretching vibration, and the shoulder at 3100 cm^{-1} to the $\nu_1(NH_4)$ stretch, the shoulder possibly also to an additional combination band $\nu_2 + \nu_4(NH_4)$. The very small band at 2866 cm^{-1} is probably caused by a combination band $2\nu_4(NH_4)$. The very strong hydrogen bonding involving the protonated $\{H(SO_4)_2\}$ group is reflected by an extremely

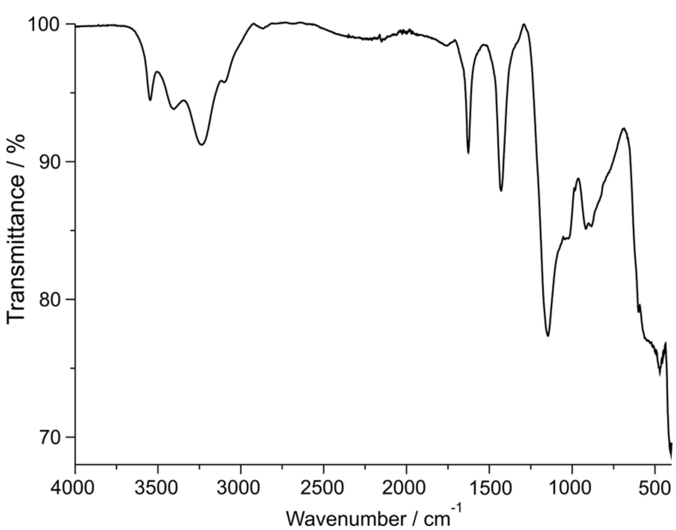


Figure 5
IR spectrum of $(NH_4)MgH(SO_4)_2(H_2O)_2$ at room temperature.

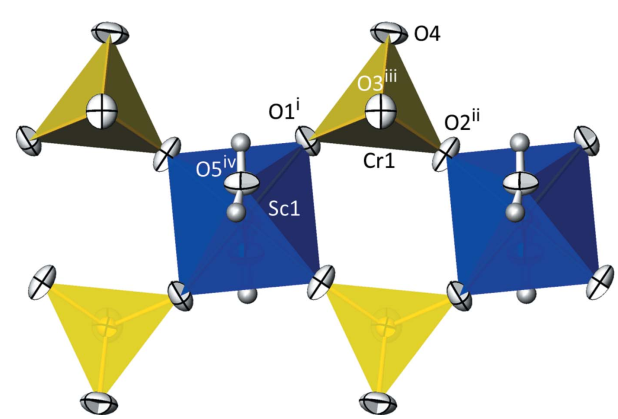


Figure 6
The kröhnkite-type chain in the crystal structure of $NaSc(CrO_4)_2(H_2O)_2$. $[ScO_4(OH_2)_2]$ octahedra are blue and CrO_4 tetrahedra are yellow. Displacement ellipsoids are drawn at the 74% probability level and H atoms are given as grey spheres of arbitrary radius. [Symmetry codes: (i) $x, -y + 1, z + \frac{1}{2}$; (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 $NaSc(CrO_4)_2(H_2O)_2$.

$Na-O3^{i}$	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) $x, y+1, z$.

Table 8
Hydrogen-bond geometry (Å, °) for $NaSc(CrO_4)_2(H_2O)_2$.

D—H···A	D—H	H···A	D···A	D—H···A
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) $x-\frac{1}{2}, y-\frac{1}{2}, z$.

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 $\nu_2(NH_4)$ bending vibration (1627 cm^{-1}, possibly also the very small band at 1753 cm^{-1}) and the $\nu_4(NH_4)$ bending vibration (1429 cm^{-1}). The range 1250–700 cm^{-1} shows bands due to vibrations of the SO_4/HSO_4 groups. The band at 1146 cm^{-1} is due to the $\nu_3(SO_4)$ stretching vibration, while the bands at 1044, 918 and 884 cm^{-1} are assigned to the $\nu_1(SO_4)$ stretching vibration. The shoulder at ~600 cm^{-1} is problably due to the $\nu_4(SO_4)$ vibration. The $\nu_2(SO_4)$ bending vibration will cause bands <500 cm^{-1}, where the spectrum is cut off and where bands due to vibrations of the MgO_6 octahedron, the librational modes of the NH_4 group and lattice modes are expected. Note that the presence of 'forbidden' SO_4 and NH_4 vibrations in the IR spectrum is in agreement with the presence of distorted shapes for these two building units.

3.2. $NaSc(CrO_4)_2(H_2O)_2$

3.2.1. Structure analysis. $NaSc(CrO_4)_2(H_2O)_2$ 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, $\bar{\frac{1}{2}}$ 0 $\bar{\frac{1}{2}}$)]. The group–subgroup relationship between subtypes **F2** and **F1** is klassengleich with index 2 (k2) (Müller, 2013). In the crystal structure of $NaSc(CrO_4)_2(H_2O)_2$, $[ScO_4(OH_2)_2]$ octahedra (point-group symmetry $\bar{1}$) are linked by CrO_4 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 $[ScO_4(OH_2)_2]$ 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 CrO_4 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 CrO_4 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 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 +I. 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.

$NaSc(CrO_4)_2(H_2O)_2$ is isotypic with $NaAl(CrO_4)_2(H_2O)_2$ (Cudennec & Riou, 1977) and $NaFe(CrO_4)_2(H_2O)_2$ (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 $NaM(CrO_4)_2(H_2O)_2$ (M = Sc, Al or Fe) structures, the program *compstru* (de la Flor *et al.*, 2016),

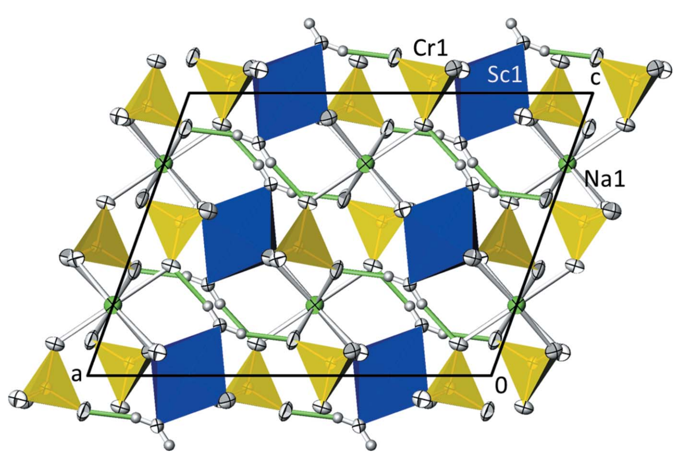


Figure 7
The crystal structure of $NaSc(CrO_4)_2(H_2O)_2$ in a projection along [010]. Colour codes and the probability level of displacement ellipsoids are as in Fig. 3. Hydrogen bonds are indicated as green lines.

Table 9
Absolute atomic displacements (Å), arithmetic mean (d_{av}, Å), degree of lattice distortion (S) and measure of similarity (Δ) in the isotypic NaM(CrO$_4$)(H$_2$O)$_2$ (M = Al and Fe) structures relative to NaSc(CrO$_4$)$_2$(H$_2$O)$_2$[a].

	M = Al[b]	M = 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 = 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) Å and β = 110.33 (5)° (Cudennec & Riou, 1977). (*c*) Unit-cell parameters a = 14.247 (2), b = 5.425 (5), c = 10.689 (2) Å and β = 109.30 (1)° (Hardy & Gravereau, 1970).

available at the Bilbao Crystallographic Server (Aroyo *et al.*, 2006), was used. With NaSc(CrO$_4$)$_2$(H$_2$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 = Al structure shows the largest displacement, it is O1 in the M = Fe structure. In general, the rather low values of S indicate high similarities between NaSc(CrO$_4$)$_2$(H$_2$O)$_2$ and the two NaM(CrO$_4$)$_2$(H$_2$O)$_2$ (M = Al and Fe) structures, whereby the M = Fe structure has a higher absolute similarity to NaSc(CrO$_4$)$_2$(H$_2$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 Å) is closer to that of Fe^{III} (0.645 Å, assuming a high-spin state) than to that of Al^{III} (0.535 Å).

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supporting information

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$(NH_4)Mg(HSO_4)(SO_4)(H_2O)_2$ and $NaSc(CrO_4)_2(H_2O)_2$, two crystal structures comprising kröhnkite-type chains, and the temperature-induced phase transition $(NH_4)Mg(HSO_4)(SO_4)(H_2O)_2 \rightleftharpoons (NH_4)MgH(SO_4)_2(H_2O)_2$

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: *publCIF* (Westrip, 2010).

Ammonium magnesium hydrogen sulfate sulfate dihydrate (NH4MgHSO4SO4H2O2_100K)

Crystal data

$(NH_4)Mg(HSO_4)(SO_4)(H_2O)_2$	$Z = 2$
$M_r = 271.51$	$F(000) = 280$
Triclinic, $P\overline{1}$	$D_x = 2.127$ Mg m^{-3}
$a = 7.0631$ (7) Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$b = 7.7065$ (7) Å	Cell parameters from 5976 reflections
$c = 8.3372$ (8) Å	$\theta = 2.6$–$36.8°$
$\alpha = 84.603$ (3)°	$\mu = 0.75$ mm^{-1}
$\beta = 73.339$ (3)°	$T = 100$ K
$\gamma = 77.387$ (3)°	Pinacoid, colourless
$V = 424.03$ (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 $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Krause *et al.*, 2015)	$R_{int} = 0.051$
$T_{min} = 0.708$, $T_{max} = 0.747$	$\theta_{max} = 37.9°$, $\theta_{min} = 2.6°$
30998 measured reflections	$h = -12 \rightarrow 12$
	$k = -13 \rightarrow 13$
	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	$R[F^2 > 2\sigma(F^2)] = 0.033$
Least-squares matrix: full	$wR(F^2) = 0.081$

$S = 1.02$
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 $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.52$ e Å^{-3}
$\Delta\rho_{min} = -0.54$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{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 (Å^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, º)

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, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O8—H1*O*···O4^{iii}	0.91 (3)	1.59 (3)	2.5048 (12)	177 (3)
O9—H1*W*···O1^{iv}	0.81 (2)	2.02 (2)	2.7623 (13)	154 (2)
O9—H2*W*···O2^{i}	0.78 (3)	2.56 (3)	3.1278 (12)	132 (2)
O9—H2*W*···O5^{v}	0.78 (3)	2.62 (3)	3.2887 (12)	146 (2)
O9—H2*W*···O7^{v}	0.78 (3)	2.55 (3)	2.9578 (12)	115 (2)
O10—H3*W*···O1^{vi}	0.77 (2)	2.38 (2)	3.0970 (12)	156 (2)
O10—H3*W*···O2^{vi}	0.77 (2)	2.64 (2)	3.0615 (12)	117 (2)
O10—H4*W*···O5^{vii}	0.80 (2)	1.95 (2)	2.7125 (13)	160.5 (19)
N1—H1*N*···O4^{v}	0.896 (19)	2.108 (19)	2.9913 (14)	168.2 (15)
N1—H2*N*···O1^{i}	0.85 (2)	2.04 (2)	2.8920 (14)	179.2 (18)
N1—H3*N*···O3^{iii}	0.868 (19)	1.98 (2)	2.8451 (14)	170.5 (18)
N1—H4*N*···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

$(NH_4)Mg(HSO_4)(SO_4)(H_2O)_2$
M_r = 271.51
Triclinic, $P\bar{1}$
a = 4.6771 (1) Å
b = 5.7697 (1) Å
c = 8.3697 (2) Å
α = 104.208 (1)°
β = 98.189 (1)°
γ = 94.508 (1)°
V = 215.20 (1) Å^3
Z = 1
$F(000)$ = 140
D_x = 2.095 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 7912 reflections
θ = 2.6–35.3°
μ = 0.73 mm^{-1}
T = 296 K
Plate, colourless
0.12 × 0.09 × 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_{int} = 0.024
θ_{max} = 35.3°, θ_{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 (Å²)

	x	y	z	U_{iso}*/U_{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 (Å²)

	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 (Å, º)

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 (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5*B*···O2^{vi}	0.81 (2)	2.52 (2)	3.0010 (9)	118.9 (19)
O5—H5*A*···O3^{vii}	0.78 (2)	2.05 (2)	2.7684 (9)	154.0 (19)
O5—H5*B*···O3^{vi}	0.81 (2)	2.42 (2)	3.1678 (10)	152 (2)
N1—H1*A*···O3^{viii}	0.90 (5)	2.03 (5)	2.9260 (6)	175 (5)
N1—H1*C*···O1	0.89 (6)	2.28 (6)	3.1536 (7)	164 (4)
N1—H1*B*···O1^{ix}	0.83 (5)	2.30 (5)	3.1122 (8)	164 (5)
N1—H1*D*···O4^{ix}	0.82 (5)	2.08 (5)	2.9042 (6)	173 (5)
O1—H1*O*···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

$NaSc(CrO_4)_2(H_2O)_2$	$F(000) = 656$
$M_r = 335.98$	$D_x = 2.731$ Mg m^{-3}
Monoclinic, $C2/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 14.505$ (3) Å	Cell parameters from 1933 reflections
$b = 5.563$ (1) Å	$\theta = 3.0$–$34.9°$
$c = 10.763$ (2) Å	$\mu = 3.51$ mm^{-1}
$\beta = 109.82$ (3)°	$T = 293$ K
$V = 817.0$ (3) Å^3	Plate, orange
$Z = 4$	0.17 × 0.10 × 0.03 mm

Data collection

Nonius KappaCCD diffractometer	1783 independent reflections
φ and ω scans	1372 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski *et al.*, 2003)	$R_{int} = 0.017$
$T_{min} = 0.755$, $T_{max} = 0.949$	$\theta_{max} = 34.9°$, $\theta_{min} = 3.0°$
3418 measured reflections	$h = -23 \rightarrow 23$
	$k = -8 \rightarrow 8$
	$l = -17 \rightarrow 17$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.039P)^2 + 0.8547P]$ where $P = (F_o^2 + 2F_c^2)/3$
Least-squares matrix: full	$(\Delta/\sigma)_{max} < 0.001$
$R[F^2 > 2\sigma(F^2)] = 0.027$	$\Delta\rho_{max} = 1.06$ e Å^{-3}
$wR(F^2) = 0.081$	$\Delta\rho_{min} = -0.69$ e Å^{-3}
$S = 1.08$	Extinction correction: SHELXL2018 (Sheldrick, 2015b), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
1783 reflections	Extinction coefficient: 0.0016 (4)
75 parameters	
2 restraints	
Hydrogen site location: difference Fourier map	
All H-atom parameters refined	

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 (Å^2)

	x	y	z	$U_{iso}*/U_{eq}$
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 (Å^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 (Å, º)

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, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H1···$O4^{xii}$	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: (xii) *x*−1/2, *y*−1/2, *z*.