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Isotypism and phase transitions of $(NH_4)M(HSO_4)(SO_4)(H_2O)_2$ (M = Fe, Co and Ni) compounds

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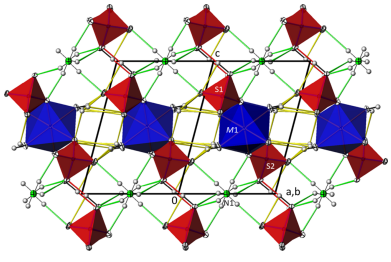
A systematic crystallization study of $\{(NH_4)[M(HSO_4)(SO_4)(H_2O)_2]\}_n$ compounds, *catena*-poly[ammonium [[diaquametal(II)]-μ-(hydrogen sulfato)-μ-sulfato]], revealed that crystals of the M = Fe, Co and Ni members could be grown by evaporation of mixed equimolar aqueous solutions of NH_4HSO_4 and the corresponding MSO_4 sulfate, whereas for M = Mn, Cu, Zn different products were obtained under these conditions. The $(NH_4)M(HSO_4)(SO_4)(H_2O)_2$ compounds (M = Fe, Co, Ni) show a similar behavior to the magnesium analogue, *i.e.* a reversible structural phase transition from an ordered triclinic crystal structure at T = 100 K (Z = 2) to a disordered triclinic structure at T = 296 K (Z = 1). The symmetry relationship between the structure at 296 K and the superstructure at 100 K is of the isomorphic type with index 2. At 100 K, the $[MO_4(OH_2)_2]$ octahedra are linked by distinct $[SO_3(OH)]$ and $[SO_4]$ tetrahedra into chains. Adjacent chains are linked by very strong hydrogen bonds ($O\cdots O \simeq$ 2.5 Å) between the two types of sulfate tetrahedra into layers. These layers are held together by hydrogen-bonding interactions of medium-to-weak strength between the ammonium cations and water molecules. At 296 K, the H atoms of the ammonium tetrahedron and the H atom between two symmetry-related sulfate groups are disordered. Quantitative structural comparisons are made between the isotypic $(NH_4)M(HSO_4)(SO_4)(H_2O)_2$ structures (M = Mg, Fe, Co, Ni) at 296 K and 100 K, respectively.

1. Chemical context

In an earlier study on $(NH_4)Mg(HSO_4)(SO_4)(H_2O)_2$, it was found that this phase can be crystallized in high yields from equimolar aqueous solutions of NH_4HSO_4 and $MgSO_4$ by slow evaporation to dryness (Weil & Kolitsch, 2021). (NH_4)-$Mg(HSO_4)(SO_4)(H_2O)_2$ is dimorphic and the corresponding crystal structures were determined and refined on basis of single crystal X-ray data sets recorded at 296 K and 100 K. The crystal structure of the corresponding iron compound (NH_4)-$Fe(HSO_4)(SO_4)(H_2O)_2$ was already known previously from a 100 K data set (Heinicke *et al.*, 2004) and interestingly shows isotypism with the crystal structure of the magnesium compound at 296 K.

The aim of the present study is to determine to what extent the corresponding first-row transition-metal compounds $(NH_4)M(HSO_4)(SO_4)(H_2O)_2$ (M^{II} = Mn, Fe, Co, Ni, Cu, Zn) can be crystallized from aqueous solutions in a similar way to the magnesium compound, and if so, whether they are also dimorphic.

While under these conditions other crystallization products were obtained for M = Mn, Cu and Zn, the corresponding $(NH_4)M(HSO_4)(SO_4)(H_2O)_2$ compounds could be crystallized for M = Fe, Co and Ni, all of which are dimorphic and



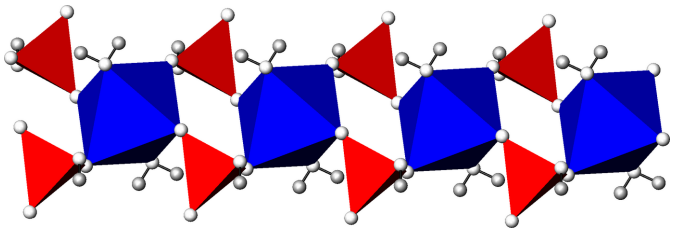


Figure 1
The kröhnkite-type chains in the crystal structures of $(NH_4)M(HSO_4)(SO_4)(H_2O)_2$ compounds at 296 K in polyhedral representation. The chains are composed of $[MO_4(OH_2)_2]$ octahedra (blue) corner-linked by sulfate/hydrogen sulfate tetrahedra (red).

crystallize isotypically with the corresponding Mg structures at 296 K and 100 K, respectively. The results of these structural investigations are reported in the present article.

2. Structural commentary

The title sulfates are new representatives of compounds with kröhnkite-type chains, which are composed of $[MO_4(OH_2)_2]$ octahedra corner-linked by XO_4 tetrahedra (Fig. 1). The widespread occurrence of this motif is related to flexible variations of the octahedral-tetrahedral building units within a chain. Compounds comprising kröhnkite-type chains have been classified into different structure types (Fleck *et al.*, 2002).

2.1. The disordered $(NH_4)M(HSO_4)(SO_4)(H_2O)_2$ crystal structure at 296 K

At 296 K, the three isotypic $(NH_4)M(HSO_4)(SO_4)(H_2O)_2$ (M = Fe, Co, Ni) compounds adopt a triclinic structure (space group $P\overline{1}$, Z = 1) corresponding to type E in the classification of compounds with kröhnkite-type chains (Fleck *et al.*, 2002). As explained below, it is more accurate to describe these compounds at this temperature by the general formula $(NH_4)MH(SO_4)_2(H_2O)_2$.

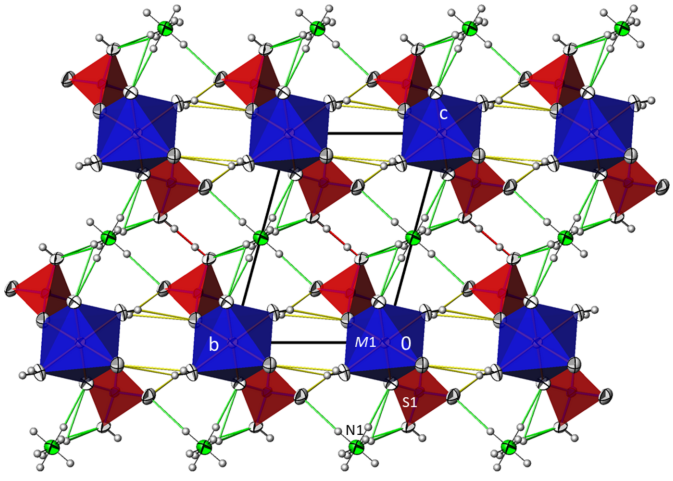


Figure 2
The crystal structure of $(NH_4)M(HSO_4)(SO_4)(H_2O)_2$ compounds at 296 K (data from M = Co) in a projection along [$\overline{1}$00]. Very strong hydrogen bonds between disordered sulfate/hydrogen sulfate groups are given in red, medium-strong to weak hydrogen bonds involving the water molecules in yellow and those involving the disordered ammonium cations in green. Displacement ellipsoids are drawn at the 74% probability level; H atoms are displayed with arbitrary radius.

Table 1
Hydrogen-bond geometry (Å, °) for M = Fe at 296 K.

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1O···O1^{i}	0.80 (1)	1.70 (1)	2.4840 (13)	167 (3)
O5—H5B···O2^{ii}	0.79 (2)	2.51 (3)	2.9951 (10)	121 (2)
O5—H5B···O3^{ii}	0.79 (2)	2.52 (2)	3.2141 (11)	148 (2)
N1—H1A···O3^{i}	0.90 (1)	2.02 (1)	2.9218 (7)	175 (6)
N1—H1B···O1^{iii}	0.90 (1)	2.29 (3)	3.1712 (8)	166 (9)
N1—H1C···O1	0.90 (1)	2.31 (3)	3.1222 (7)	150 (5)
N1—H1B···O1^{iii}	0.90 (1)	2.29 (3)	3.1712 (8)	166 (9)
N1—H1D···O4^{iii}	0.90 (1)	1.98 (1)	2.8736 (7)	174 (7)

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

Table 2
Hydrogen-bond geometry (Å, °) for M = Co at 296 K.

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1O···O1^{i}	0.70 (4)	1.80 (4)	2.4810 (16)	165 (5)
O5—H5A···O2^{ii}	0.74 (3)	2.57 (3)	2.9923 (11)	118 (2)
O5—H5B···O3^{ii}	0.77 (2)	2.51 (2)	3.2020 (13)	149 (2)
N1—H1A···O3^{i}	0.98 (7)	1.96 (7)	2.9298 (8)	169 (7)
N1—H1D···O1^{iii}	0.84 (7)	2.33 (7)	3.1657 (10)	175 (7)
N1—H1C···O1	0.87 (6)	2.29 (6)	3.1098 (8)	158 (6)
N1—H1D···O1^{iii}	0.84 (7)	2.33 (7)	3.1657 (10)	175 (7)
N1—H1B···O4^{iii}	0.91 (9)	1.97 (9)	2.8773 (7)	176 (8)

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

Table 3
Hydrogen-bond geometry (Å, °) for M = Ni at 296 K.

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1O···O1^{i}	0.85 (1)	1.65 (2)	2.484 (3)	167 (6)
O5—H5A···O2^{ii}	0.84 (1)	2.50 (4)	3.005 (2)	119 (3)
O5—H5B···O3^{ii}	0.84 (1)	2.43 (2)	3.177 (3)	147 (4)
N1—H1A···O3^{i}	0.90 (1)	2.05 (3)	2.9360 (17)	166 (9)
N1—H1D···O1^{iii}	0.90 (1)	2.29 (4)	3.152 (2)	159 (9)
N1—H1C···O1	0.90 (1)	2.23 (2)	3.0898 (18)	160 (6)
N1—H1D···O1^{iii}	0.90 (1)	2.29 (4)	3.152 (2)	159 (9)
N1—H1B···O4^{iii}	0.90 (1)	2.07 (7)	2.8761 (16)	149 (11)

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

The kröhnkite-type chains run along [100] and are joined into sheets parallel to (001) by O—H···O hydrogen bonds involving the water molecule (O5) as the donor group. These sheets are held together along [001] through an asymmetric hydrogen bond between two SO_4 tetrahedra of two adjacent chains. This hydrogen bond involves the disordered H1O atom. The corresponding O1···O1^{i} [symmetry code: (i) $-x-1, -y-1, -x-1$] distance of about 2.48 Å indicates a very strong hydrogen bond (Jeffrey, 1997). The cohesion within the structure is completed by multiple N—H···O hydrogen bonds arising from the ammonium cations, which are located between the sheets (Fig. 2). The N atom of the ammonium cation is situated at an inversion centre and consequently its hydrogen atoms are equally disordered over two sets of sites. Numerical details of the hydrogen-bonding

interactions for the 296 K structures are compiled in Tables 1–3 for the three phases.

The $[M^{II}O_4(OH_2)_2]$ octahedra in the 296 K structures exhibit point group symmetry $\bar{1}$ with mean bond lengths of 2.118 Å for M = Fe, 2.091 Å for Co, and 2.059 Å for Ni, in good agreement with grand mean values of 2.147 (89), 2.108 (62) and 2.070 (54) Å, respectively, reported in literature (Gagné & Hawthorne, 2020). The S—O1(H10) bond (average 1.515 Å for the three structures) is the longest in the sulfate tetrahedron and is about 0.05 Å longer than the S—O bonds to the other O atoms (average 1.462 Å for the three structures). The S—O bond lengths are in 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).

2.2. The ordered $(NH_4)M(HSO_4)(SO_4)(H_2O)_2$ crystal structure at 100 K

At 100 K, the three $(NH_4)M(HSO_4)(SO_4)(H_2O)_2$ compounds also adopt a triclinic structure (space group $P\bar{1}$, Z = 2) corresponding to type E1 (Weil & Kolitsch, 2021) in the classification of compounds with kröhnkite-type chains. A search in the current version of the Inorganic Crystal Structure Database (ICSD, data release 2024-1; Zagorac *et al.*, 2019), revealed that, apart from the Mg analogue (Weil & Kolitsch, 2021) and the title compounds, there are no other members that adopt this structure type.

The crystal structures of the $(NH_4)M(HSO_4)(SO_4)(H_2O)_2$ (M = Fe, Co, Ni) compounds at 100 K represent a twofold superstructure with ordered hydrogen atoms for the ammonium group and of the hydrogen sulfate group relative to the crystal structure of $(NH_4)MH(SO_4)_2(H_2O)_2$ at 296 K. The unit cells of the latter compounds are related to the doubled unit cells of the $(NH_4)M(HSO_4)(SO_4)(H_2O)_2$ superstructures at 100 K by the transformation **–a–b**, **a–b**, **c**. The symmetry relationship (Müller & de la Flor, 2024) between the substructure at 296 K and the superstructure at 100 K is of isomorphic type with index 2 (i2). All atoms in the superstructure are situated in general positions. $[MO_4(OH_2)]$ octahedra are corner-linked by distinct $[SO_3(OH)]$ and $[SO_4]$ tetrahedra into chains running parallel to $[\bar{1}10]$. Adjacent chains are joined by O—H···O hydrogen bonds between the hydrogen sulfate and sulfate tetrahedra into sheets extending parallel to (111). The ammonium cations, situated in-between the sheets, and water molecules are also involved in hydrogen-bonding and consolidate the crystal packing (Fig. 3). In comparison, the bond lengths of all principal building units in

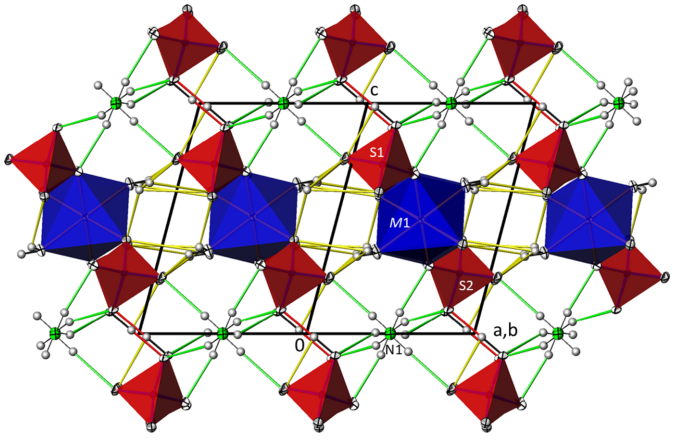


Figure 3
The crystal structure of $(NH_4)M(HSO_4)(SO_4)(H_2O)_2$ compounds at 100 K (data from M = Co) in a projection along $[\bar{1}10]$. Color code and displacement ellipsoids are as in Fig. 2.

Table 4
Hydrogen-bond geometry (Å, °) for M = Fe at 100 K.

D—H···A	D—H	H···A	D···A	D—H···A
O8—H1O···O4^{i}	1.040 (16)	1.467 (16)	2.5067 (8)	178.6 (12)
O9—H1W···O1^{ii}	0.816 (17)	2.030 (19)	2.7537 (7)	147.5 (18)
O9—H2W···O2^{iii}	0.875 (17)	2.447 (17)	3.0785 (7)	129.5 (13)
O9—H2W···O5^{iv}	0.875 (17)	2.599 (16)	3.3272 (8)	141.3 (13)
O9—H2W···O7^{iv}	0.875 (17)	2.439 (17)	2.9556 (7)	118.3 (14)
O10—H3W···O1^{v}	0.849 (19)	2.389 (18)	3.1467 (8)	149.0 (15)
O10—H3W···O2^{v}	0.849 (19)	2.541 (19)	3.0541 (7)	119.9 (16)
O10—H4W···O5^{vi}	0.840 (17)	1.946 (18)	2.7115 (7)	151.2 (18)
N1—H3N···O3^{i}	0.873 (12)	1.967 (12)	2.8334 (7)	171.5 (11)
N1—H1N···O4^{iv}	0.871 (12)	2.138 (12)	2.9931 (7)	167.2 (11)
N1—H2N···O1^{iii}	0.857 (13)	2.040 (13)	2.8965 (7)	178.3 (12)
N1—H4N···O8^{vii}	0.890 (14)	2.266 (14)	3.0869 (8)	153.2 (11)

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

Table 5
Hydrogen-bond geometry (Å, °) for M = Co at 100 K.

D—H···A	D—H	H···A	D···A	D—H···A
O8—H1O···O4^{i}	0.99 (2)	1.51 (2)	2.5007 (11)	177.4 (17)
O9—H1W···O1^{ii}	0.77 (2)	2.08 (2)	2.7566 (10)	147 (2)
O9—H2W···O2^{iii}	0.82 (2)	2.468 (19)	3.0700 (10)	130.8 (16)
O9—H2W···O5^{iv}	0.82 (2)	2.59 (2)	3.2918 (10)	144.5 (16)
O9—H1W···O7^{iv}	0.77 (2)	2.51 (2)	2.9549 (10)	118.1 (19)
O10—H3W···O1^{v}	0.79 (2)	2.42 (2)	3.1378 (10)	151.0 (18)
O10—H3W···O2^{v}	0.79 (2)	2.58 (2)	3.0492 (10)	119.8 (19)
O10—H4W···O5^{vi}	0.80 (2)	1.98 (2)	2.7127 (10)	152 (2)
N1—H3N···O3^{i}	0.887 (15)	1.955 (15)	2.8328 (10)	170.1 (15)
N1—H1N···O4^{iv}	0.897 (16)	2.114 (16)	2.9890 (11)	164.7 (14)
N1—H2N···O1^{iii}	0.864 (17)	2.038 (17)	2.9017 (11)	178.9 (16)
N1—H4N···O8^{vii}	0.894 (18)	2.252 (18)	3.0777 (11)	153.4 (15)

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

Table 6
Hydrogen-bond geometry (Å, °) for M = Ni at 100 K.

D—H···A	D—H	H···A	D···A	D—H···A
O8—H1O···O4^{i}	0.88 (4)	1.62 (4)	2.499 (2)	173 (4)
O9—H1W···O1^{ii}	0.80 (5)	2.08 (5)	2.747 (3)	140 (5)
O9—H2W···O2^{iii}	0.65 (4)	2.55 (4)	3.061 (3)	138 (4)
O9—H2W···O5^{iv}	0.65 (4)	2.69 (4)	3.232 (2)	143 (4)
O9—H1W···O7^{iv}	0.80 (5)	2.44 (5)	2.963 (2)	124 (5)
O10—H3W···O1^{v}	0.80 (3)	2.41 (3)	3.115 (2)	148 (3)
O10—H3W···O2^{v}	0.80 (3)	2.59 (4)	3.051 (2)	119 (3)
O10—H4W···O5^{vi}	0.78 (3)	2.00 (3)	2.719 (3)	155 (3)
N1—H3N···O3^{i}	0.87 (3)	1.97 (3)	2.834 (3)	172 (3)
N1—H1N···O4^{iv}	0.93 (3)	2.07 (3)	2.980 (3)	169 (3)
N1—H2N···O1^{iii}	0.91 (3)	2.00 (3)	2.903 (3)	174 (3)
N1—H4N···O8^{vii}	0.79 (6)	2.31 (6)	3.068 (3)	159 (5)

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

Table 7
Structural comparison of isotypic $(NH_4)M(HSO_4)(SO_4)(H_2O)_2$ structures with the M = Mg members as references.

	Co (100 K)	Fe (100 K)	Ni (100 K)	Co (296 K)	Fe (296 K)	Ni (296 K)
S	0.0047	0.0062	0.0074	0.0055	0.0062	0.0078
d_{max} (atom)	0.0860 (O1)	0.0893 (O5)	0.1031 (O8)	0.0905 (O3)	0.1003 (O3)	0.0944 (O2)
d_{av}	0.0469	0.0485	0.0545	0.0521	0.0525	0.0574
δ	0.012	0.021	0.017	0.048	0.051	0.062

S is the degree of lattice distortion, d_{max} (Å) is the maximum difference between two atomic positions, d_{av} (Å) is the arithmetic mean of the differences between two atomic positions and δ is the measure of similarity.

the ordered 100 K structure are similar to those in the disordered 296 K structure.

The mean M—O bond lengths in the $[MO_4(OH_2)]$ octahedra (2.116 Å for M = Fe, 2.087 Å for Co, and 2.053 Å for Ni) hardly differ from those of the 296 K structures. The ordering of the hydrogen atom (H1*O*) between two sulfate tetrahedra defines distinct $S1O_4$ and $S2O_3(OH)$ groups. The longest bond in the S1O4 tetrahedron is the bond to O4 (1.491 Å on average for all three structures in contrast to 1.476 Å on average for all other S—O bonds). O4 serves as the acceptor atom for the hydrogen bond with the OH group of the hydrogen sulfate group as donor group. The corresponding $[S2O_3(OH)]$ tetrahedron shows the typical distribution of S—O bond lengths in a hydrogen sulfate group, whereby the bond to the OH group (O8) is significantly longer by about 0.09 Å than the remaining three S—O bonds (average for all structures 1.459 Å). The hydrogen bond between the $[S2O_3(OH)]$ and $[S1O_4]$ tetrahedra (O8···O4 is on average 2.502 Å) is almost linear [178.6 (12)° for Fe, 177.4 (17)° for Co and 173 (4)° for Ni]. Like the 296 K structures, the other types of O—H···O hydrogen-bonding interactions are much weaker and involve the water molecules. One of them (O9) shows pairs of bifurcated (M = Co, Ni) or trifurcated (M = Fe) medium-strong to weak hydrogen bonds. The other water molecule (O10) is involved in one medium-strong and a weak bifurcated hydrogen bond. All H atoms of the ammonium cation are engaged in almost linear hydrogen-bonding interactions to the O atoms of the sulfate group as acceptor atoms. Numerical details of hydrogen-bonding interactions are compiled in Tables 4–6 for the three 100 K structures.

As already mentioned in the Introduction, the crystal structure of the Fe compound (Heinicke *et al.*, 2004) was determined in a previous measurement at 100 K in structure type E, *i.e.* in the disordered variant with Z = 1, which occurs for all other $(NH_4)M(HSO_4)(SO_4)(H_2O)_2$ representatives at 296 K. However, the 100 K data obtained in the present study originate from a slowly cooled $(NH_4)Fe(HSO_4)(SO_4)(H_2O)_2$ crystal and clearly show the ordered variant with Z = 2. Whether this difference is possibly due to a different temperature treatment cannot be conclusively clarified, as no details were given in the original publication (Heinicke *et al.*, 2004). The investigation of the exact ordering temperatures for this and all other $(NH_4)M(HSO_4)(SO_4)(H_2O)_2$ representatives, *e.g.* with temperature-dependent powder X-ray diffraction and/or differential scanning calorimetry (DSC) methods, still has to be carried out, but is outside the scope of the present structural study.

3. Structural comparison

For a quantitative structural comparison of the $(NH_4)M(HSO_4)(SO_4)(H_2O)_2$ (M = Mg, Fe, Co, Ni) structures at 296 K and 100 K, respectively, the program *compstru* (de la Flor *et al.*, 2016) available at the Bilbao Crystallographic Server (Aroyo *et al.*, 2006) was used. With M = Mg as the reference structure, Table 7 compiles the maximum distance (d_{max}) between paired atoms and numerical values regarding the arithmetic mean (d_{av}) of the distance between paired atoms, the degree of lattice distortion (δ), and the measure of similarity (S).

As expected for isotypic structures, the comparison between the individual structures (M = Fe, Co, Ni) and the reference structure (M = Mg) shows very similar numerical values. A clearly recognizable trend cannot be identified, however it may be noted that the numerical parameters for the smallest deviations are always connected with the M = Co structure.

4. Synthesis and crystallization

Equimolar aqueous solutions of NH_4HSO_4 and the corresponding MSO_4 sulfate (M = Mn, Fe, Co, Ni, Cu, Zn) were mixed at room temperature and stirred for homogeneity. The mixed solutions were then slowly evaporated to dryness for several days at room temperature. Semi-quantitative phase analysis of the obtained bulk using the HighScorePlus program (Degen *et al.*, 2014) revealed the title compounds $(NH_4)M(HSO_4)(SO_4)(H_2O)_2$ (M = Fe, Co, Ni) as the main products (> 90%$_{wt}$) and $[(NH_4)_2M(SO_4)_2(H_2O)_6]$ phases as the minor products. For batches with M = Cu and Zn, $(NH_4)_2M(SO_4)_2(H_2O)_6$ phases were the main products and $CuSO_4(H_2O)_5$ and $ZnSO_4(H_2O)_6$ the minor products, for both with an approximate phase ratio of 3:1. For the batch with M = Mn, langbeinite-type $(NH_4)_2Mn_2(SO_4)_3$ was the only phase obtained.

The $(NH_4)M(HSO_4)(SO_4)(H_2O)_2$ single crystals (M = Fe, Co, Ni) used for the diffraction studies were broken from larger specimens.

5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 8. For the low-temperature measurements, the crystals were cooled from 296 K to 100 K within two h. For refinement, coordinates and labeling of

Table 8
Experimental details.

	M = Fe at 296 K	M = Co at 296 K	M = Ni at 296 K
Crystal data			
Chemical formula	$(NH_4)[Fe(HSO_4)(SO_4)(H_2O)_2]$	$(NH_4)[Co(HSO_4)(SO_4)(H_2O)_2]$	$(NH_4)[Ni(HSO_4)(SO_4)(H_2O)_2]$
M_r	303.05	306.13	305.91
Crystal system, space group	Triclinic, $P\overline{1}$	Triclinic, $P\overline{1}$	Triclinic, $P\overline{1}$
Temperature (K)	296	296	296
a, b, c (Å)	4.6369 (6), 5.8481 (8), 8.4135 (11)	4.6182 (2), 5.8243 (3), 8.3576 (4)	4.5746 (16), 5.7944 (19), 8.347 (2)
α, β, γ (°)	104.010 (2), 98.145 (2), 95.077 (3)	104.2261 (11), 98.1916 (13), 94.7068 (11)	104.049 (11), 98.133 (12), 94.420 (12)
V (Å^3)	217.33 (5)	214.07 (2)	211.04 (12)
Z	1	1	1
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	2.26	2.53	2.84
Crystal size (mm)	0.21 × 0.12 × 0.10	0.13 × 0.05 × 0.02	0.12 × 0.06 × 0.01
Data collection			
Diffractometer	Bruker APEXII CCD	Bruker APEXII CCD	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)	Multi-scan (*SADABS*; Krause *et al.*, 2015)	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.616, 0.748	0.677, 0.747	0.598, 0.747
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	8735, 2967, 2634	4115, 1920, 1739	3532, 1591, 1307
R_{int}	0.028	0.016	0.027
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.939	0.812	0.769
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.022, 0.060, 1.05	0.019, 0.053, 1.05	0.031, 0.077, 1.10
No. of reflections	2967	1920	1591
No. of parameters	96	96	95
No. of restraints	7	0	7
H-atom treatment	All H-atom parameters refined	All H-atom parameters refined	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.53, −0.45	0.43, −0.40	0.47, −0.59

	M = Fe at 100 K	M = Co at 100 K	M = Ni at 100 K
Crystal data			
Chemical formula	$(NH_4)[Fe(HSO_4)(SO_4)(H_2O)_2]$	$(NH_4)[Co(HSO_4)(SO_4)(H_2O)_2]$	$(NH_4)[Ni(HSO_4)(SO_4)(H_2O)_2]$
M_r	303.05	306.13	305.91
Crystal system, space group	Triclinic, $P\overline{1}$	Triclinic, $P\overline{1}$	Triclinic, $P\overline{1}$
Temperature (K)	100	100	100
a, b, c (Å)	7.0847 (6), 7.7956 (7), 8.3868 (7)	7.0775 (4), 7.7268 (4), 8.3281 (5)	7.0437 (9), 7.6405 (9), 8.3097 (10)
α, β, γ (°)	84.5953 (15), 73.2990 (15), 76.2281 (17)	84.5548 (11), 73.1705 (15), 76.3233 (11)	84.571 (3), 73.376 (3), 76.056 (3)
V (Å^3)	430.74 (6)	423.40 (4)	415.73 (9)
Z	2	2	2
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	2.28	2.56	2.88
Crystal size (mm)	0.12 × 0.09 × 0.02	0.12 × 0.09 × 0.02	0.12 × 0.09 × 0.02
Data collection			
Diffractometer	Bruker APEXII CCD	Bruker APEXII CCD	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)	Multi-scan (*SADABS*; Krause *et al.*, 2015)	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.619, 0.748	0.669, 0.747	0.609, 0.747
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	17276, 5854, 5143	8668, 4184, 3507	7657, 3609, 2253
R_{int}	0.027	0.019	0.031
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.937	0.854	0.827
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.022, 0.062, 1.05	0.023, 0.062, 1.04	0.038, 0.092, 1.02
No. of reflections	5854	4184	3609
No. of parameters	164	164	163
No. of restraints	0	0	0
H-atom treatment	All H-atom parameters refined	All H-atom parameters refined	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.78, −0.76	0.54, −0.53	0.86, −0.95

Computer programs: *APEX4* and *SAINT* (Bruker, 2020), *SHELXL* (Sheldrick, 2015), *ATOMS* (Dowty, 2006) and *publCIF* (Westrip, 2010).

atoms of all $(NH_4)M(HSO_4)(SO_4)(H_2O)_2$ structures were taken from the isotypic $(NH_4)Mg(HSO_4)(SO_4)(H_2O)_2$ structures for the 296 K and the 100 K data sets (Weil & Kolitsch, 2021). For all data (296 and 100 K), hydrogen atoms were

discernible in difference-Fourier maps and were refined freely. For all 296 K structures, the four ammonium hydrogen atoms (H1*A*–H1*D*) and the H1*O* atom located between two symmetry-related sulfate tetrahedra are all equally disordered across a centre of symmetry and thus were refined with half-occupancy.

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Isotypism and phase transitions of $(NH_4)M(HSO_4)(SO_4)(H_2O)_2$ (M = Fe, Co and Ni) compounds

Matthias Weil

Computing details

***catena*-Poly[ammonium [[diaquairon(II)]-μ-(hydrogen sulfato)-μ-sulfato]] (Fe_296K)**

Crystal data

$(NH_4)[Fe(HSO_4)(SO_4)(H_2O)_2]$
M_r = 303.05
Triclinic, $P\overline{1}$
a = 4.6369 (6) Å
b = 5.8481 (8) Å
c = 8.4135 (11) Å
α = 104.010 (2)°
β = 98.145 (2)°
γ = 95.077 (3)°
V = 217.33 (5) Å^3
Z = 1
$F(000)$ = 154
D_x = 2.315 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5383 reflections
θ = 3.6–41.7°
μ = 2.26 mm^{-1}
T = 296 K
Fragment, light green
0.21 × 0.12 × 0.10 mm

Data collection

Bruker APEXII CCD diffractometer
ω– and φ–scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.616, T_{max} = 0.748
8735 measured reflections
2967 independent reflections
2634 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 41.9°, θ_{min} = 3.9°
h = −8→8
k = −10→10
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.022
$wR(F^2)$ = 0.060
S = 1.05
2967 reflections
96 parameters
7 restraints
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0316P)^2 + 0.0223P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 e Å^{-3}
Extinction correction: SHELXL (Sheldrick, 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.216 (10)

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)
Fe1	0.000000	0.000000	0.000000	0.01282 (4)	
S1	−0.59664 (3)	−0.31685 (3)	−0.23953 (2)	0.01243 (4)	
O1	−0.50160 (18)	−0.30338 (11)	−0.40240 (8)	0.02469 (12)	
O2	−0.33634 (14)	−0.28331 (11)	−0.11117 (8)	0.02220 (11)	
O3	−0.76173 (16)	−0.54770 (11)	−0.26110 (8)	0.02589 (13)	
O4	−0.77081 (14)	−0.11909 (11)	−0.19883 (7)	0.02041 (10)	
O5	0.19351 (15)	−0.21979 (12)	0.14262 (9)	0.02261 (11)	
H5A	0.107 (5)	−0.329 (4)	0.156 (3)	0.063 (6)*	
H5B	0.338 (5)	−0.281 (5)	0.131 (3)	0.055 (6)*	
N1	0.000000	0.000000	−0.500000	0.0274 (2)	
H1A	−0.080 (14)	−0.134 (7)	−0.578 (6)	0.093 (18)*	0.5
H1B	−0.129 (15)	0.104 (12)	−0.510 (14)	0.12 (3)*	0.5
H1C	−0.169 (7)	−0.034 (10)	−0.463 (7)	0.071 (14)*	0.5
H1D	−0.078 (14)	0.045 (13)	−0.590 (6)	0.081 (19)*	0.5
H1O	−0.528 (7)	−0.431 (3)	−0.468 (4)	0.038 (9)*	0.5

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.01286 (6)	0.01288 (6)	0.01309 (6)	0.00327 (4)	0.00294 (4)	0.00303 (4)
S1	0.01381 (7)	0.01094 (6)	0.01176 (7)	0.00064 (4)	0.00384 (5)	0.00085 (4)
O1	0.0402 (4)	0.0180 (2)	0.0176 (2)	0.0022 (2)	0.0168 (2)	0.00169 (18)
O2	0.0181 (2)	0.0191 (2)	0.0269 (3)	0.00131 (18)	−0.00379 (19)	0.0059 (2)
O3	0.0308 (3)	0.0175 (2)	0.0253 (3)	−0.0097 (2)	0.0077 (2)	0.0006 (2)
O4	0.0220 (2)	0.0238 (2)	0.0188 (2)	0.0119 (2)	0.00839 (19)	0.00588 (19)
O5	0.0210 (3)	0.0206 (2)	0.0311 (3)	0.00657 (19)	0.0057 (2)	0.0139 (2)
N1	0.0354 (6)	0.0237 (4)	0.0217 (4)	0.0013 (4)	−0.0013 (4)	0.0074 (4)

Geometric parameters (Å, º)

Fe1—O2	2.1051 (6)	O5—H5A	0.76 (2)
Fe1—O2^{i}	2.1051 (6)	O5—H5B	0.79 (2)
Fe1—O4^{ii}	2.1174 (6)	N1—H1A	0.901 (10)
Fe1—O4^{iii}	2.1174 (6)	N1—H1B	0.900 (10)
Fe1—O5^{i}	2.1314 (6)	N1—H1C	0.904 (10)
Fe1—O5	2.1314 (6)	N1—H1D	0.901 (10)
S1—O3	1.4493 (6)	N1—H1A^{iv}	0.901 (10)
S1—O2	1.4655 (6)	N1—H1B^{iv}	0.900 (10)
S1—O4	1.4694 (6)	N1—H1C^{iv}	0.904 (10)

S1—O1	1.5151 (6)	N1—H1D^{iv}	0.901 (10)
O1—H1O	0.798 (6)		
O2—Fe1—O2^{i}	180.0	H1A—N1—H1C	79 (5)
O2—Fe1—O4^{ii}	90.82 (3)	H1B—N1—H1C	67 (5)
O2^{i}—Fe1—O4^{ii}	89.19 (3)	H1A—N1—H1D	73 (5)
O2—Fe1—O4^{iii}	89.18 (3)	H1B—N1—H1D	50 (6)
O2^{i}—Fe1—O4^{iii}	90.81 (3)	H1C—N1—H1D	98 (5)
O4^{ii}—Fe1—O4^{iii}	180.0	H1A—N1—H1A^{iv}	180 (4)
O2—Fe1—O5^{i}	91.74 (3)	H1B—N1—H1A^{iv}	76 (6)
O2^{i}—Fe1—O5^{i}	88.26 (3)	H1C—N1—H1A^{iv}	101 (5)
O4^{ii}—Fe1—O5^{i}	93.41 (3)	H1D—N1—H1A^{iv}	107 (5)
O4^{iii}—Fe1—O5^{i}	86.59 (3)	H1A—N1—H1B^{iv}	76 (6)
O2—Fe1—O5	88.26 (3)	H1B—N1—H1B^{iv}	180.00 (5)
O2^{i}—Fe1—O5	91.74 (3)	H1C—N1—H1B^{iv}	113 (5)
O4^{ii}—Fe1—O5	86.59 (3)	H1D—N1—H1B^{iv}	130 (6)
O4^{iii}—Fe1—O5	93.41 (3)	H1A^{iv}—N1—H1B^{iv}	104 (6)
O5^{i}—Fe1—O5	180.0	H1A—N1—H1C^{iv}	101 (5)
O3—S1—O2	109.74 (4)	H1B—N1—H1C^{iv}	113 (5)
O3—S1—O4	113.36 (4)	H1C—N1—H1C^{iv}	179.998 (15)
O2—S1—O4	110.13 (4)	H1D—N1—H1C^{iv}	82 (5)
O3—S1—O1	109.11 (4)	H1A^{iv}—N1—H1C^{iv}	79 (5)
O2—S1—O1	109.39 (4)	H1B^{iv}—N1—H1C^{iv}	67 (5)
O4—S1—O1	104.96 (4)	H1A—N1—H1D^{iv}	107 (5)
S1—O1—H1O	111 (3)	H1B—N1—H1D^{iv}	130 (6)
S1—O2—Fe1	133.85 (4)	H1C—N1—H1D^{iv}	82 (5)
S1—O4—Fe1^{v}	132.75 (4)	H1D—N1—H1D^{iv}	180 (8)
Fe1—O5—H5A	122.5 (18)	H1A^{iv}—N1—H1D^{iv}	73 (5)
Fe1—O5—H5B	127.2 (16)	H1B^{iv}—N1—H1D^{iv}	50 (6)
H5A—O5—H5B	95 (2)	H1C^{iv}—N1—H1D^{iv}	98 (5)
H1A—N1—H1B	104 (6)		

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*
O1—H1*O*···O1^{vi}	0.80 (1)	1.70 (1)	2.4840 (13)	167 (3)
O5—H5*B*···O2^{vii}	0.79 (2)	2.51 (3)	2.9951 (10)	121 (2)
O5—H5*B*···O3^{vii}	0.79 (2)	2.52 (2)	3.2141 (11)	148 (2)
N1—H1*A*···O3^{vi}	0.90 (1)	2.02 (1)	2.9218 (7)	175 (6)
N1—H1*B*···O1^{viii}	0.90 (1)	2.29 (3)	3.1712 (8)	166 (9)
N1—H1*C*···O1	0.90 (1)	2.31 (3)	3.1222 (7)	150 (5)
N1—H1*B*···O1^{viii}	0.90 (1)	2.29 (3)	3.1712 (8)	166 (9)
N1—H1*D*···O4^{viii}	0.90 (1)	1.98 (1)	2.8736 (7)	174 (7)

Symmetry codes: (vi) −*x*−1, −*y*−1, −*z*−1; (vii) −*x*, −*y*−1, −*z*; (viii) −*x*−1, −*y*, −*z*−1.

catena-Poly[ammonium [[diaquacobalt(II)]-μ-(hydrogen sulfato)-μ-sulfato]] (Co_296K)

Crystal data

$(NH_4)[Co(HSO_4)(SO_4)(H_2O)_2]$
M_r = 306.13
Triclinic, $P\overline{1}$
a = 4.6182 (2) Å
b = 5.8243 (3) Å
c = 8.3576 (4) Å
α = 104.2261 (11)°
β = 98.1916 (13)°
γ = 94.7068 (11)°
V = 214.07 (2) Å^3
Z = 1
$F(000)$ = 155
D_x = 2.375 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2718 reflections
θ = 2.5–35.3°
μ = 2.53 mm^{-1}
T = 296 K
Fragment, violet
0.13 × 0.05 × 0.02 mm

Data collection

Bruker APEXII CCD diffractometer
ω– and φ–scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.677, T_{max} = 0.747
4115 measured reflections
1920 independent reflections
1739 reflections with $I > 2\sigma(I)$
R_{int} = 0.016
θ_{max} = 35.3°, θ_{min} = 3.6°
h = −7→7
k = −9→9
l = −12→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.019
$wR(F^2)$ = 0.053
S = 1.05
1920 reflections
96 parameters
0 restraints
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0286P)^2 + 0.0382P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}
Extinction correction: SHELXL (Sheldrick, 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.091 (6)

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}	Occ. (<1)
Co1	0.000000	0.000000	0.000000	0.01220 (6)	
S1	−0.59237 (5)	−0.31481 (4)	−0.23633 (2)	0.01192 (6)	
O1	−0.4974 (2)	−0.30250 (15)	−0.40060 (10)	0.02333 (16)	
O2	−0.33063 (17)	−0.28375 (14)	−0.10752 (10)	0.02082 (15)	
O3	−0.7600 (2)	−0.54530 (14)	−0.25781 (10)	0.02425 (16)	
O4	−0.76453 (17)	−0.11339 (14)	−0.19483 (9)	0.01918 (14)	
O5	0.19361 (19)	−0.21456 (15)	0.14362 (11)	0.02058 (14)	
H5A	0.108 (6)	−0.320 (4)	0.157 (3)	0.056 (7)*	
H5B	0.338 (5)	−0.271 (5)	0.134 (3)	0.051 (6)*	

N1	0.000000	0.000000	−0.500000	0.0263 (3)	
H1A	−0.103 (16)	−0.149 (12)	−0.576 (9)	0.09 (2)*	0.5
H1B	−0.07 (2)	0.041 (16)	−0.596 (10)	0.09 (3)*	0.5
H1C	−0.155 (13)	−0.046 (11)	−0.462 (8)	0.066 (16)*	0.5
H1D	−0.126 (15)	0.084 (12)	−0.530 (10)	0.064 (18)*	0.5
H1O	−0.528 (9)	−0.416 (7)	−0.456 (5)	0.033 (10)*	0.5

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co1	0.01195 (8)	0.01226 (8)	0.01283 (8)	0.00288 (5)	0.00301 (5)	0.00313 (6)
S1	0.01354 (10)	0.01038 (9)	0.01119 (9)	0.00054 (7)	0.00395 (7)	0.00082 (7)
O1	0.0382 (4)	0.0170 (3)	0.0162 (3)	0.0016 (3)	0.0156 (3)	0.0011 (3)
O2	0.0171 (3)	0.0176 (3)	0.0252 (3)	0.0004 (2)	−0.0035 (3)	0.0056 (3)
O3	0.0280 (4)	0.0167 (3)	0.0244 (4)	−0.0087 (3)	0.0068 (3)	0.0011 (3)
O4	0.0208 (3)	0.0216 (3)	0.0183 (3)	0.0103 (3)	0.0086 (3)	0.0058 (3)
O5	0.0198 (3)	0.0186 (3)	0.0274 (4)	0.0056 (3)	0.0058 (3)	0.0115 (3)
N1	0.0331 (8)	0.0233 (6)	0.0211 (6)	0.0014 (6)	−0.0008 (5)	0.0070 (5)

Geometric parameters (Å, º)

Co1—O2^{i}	2.0821 (8)	O1—H1O	0.70 (4)
Co1—O2	2.0821 (8)	O5—H5A	0.74 (3)
Co1—O4^{ii}	2.0890 (7)	O5—H5B	0.77 (2)
Co1—O4^{iii}	2.0890 (7)	N1—H1A	0.98 (7)
Co1—O5	2.1016 (8)	N1—H1B	0.91 (9)
Co1—O5^{i}	2.1016 (8)	N1—H1C	0.87 (6)
S1—O3	1.4508 (8)	N1—H1D	0.84 (7)
S1—O2	1.4664 (8)	N1—H1B^{iv}	0.91 (9)
S1—O4	1.4699 (8)	N1—H1C^{iv}	0.87 (6)
S1—O1	1.5155 (8)	N1—H1D^{iv}	0.84 (7)
O2^{i}—Co1—O2	180.0	Co1—O5—H5A	122 (2)
O2^{i}—Co1—O4^{ii}	90.83 (3)	Co1—O5—H5B	126.8 (18)
O2—Co1—O4^{ii}	89.17 (3)	H5A—O5—H5B	97 (3)
O2^{i}—Co1—O4^{iii}	89.17 (3)	H1A—N1—H1B	76 (6)
O2—Co1—O4^{iii}	90.83 (3)	H1A—N1—H1C	70 (5)
O4^{ii}—Co1—O4^{iii}	180.00 (3)	H1B—N1—H1C	107 (6)
O2^{i}—Co1—O5	92.27 (3)	H1A—N1—H1D	94 (5)
O2—Co1—O5	87.73 (3)	H1B—N1—H1D	44 (6)
O4^{ii}—Co1—O5	86.33 (3)	H1C—N1—H1D	77 (5)
O4^{iii}—Co1—O5	93.67 (3)	H1A—N1—H1B^{iv}	104 (6)
O2^{i}—Co1—O5^{i}	87.73 (3)	H1B—N1—H1B^{iv}	180.00 (2)
O2—Co1—O5^{i}	92.27 (3)	H1C—N1—H1B^{iv}	73 (5)
O4^{ii}—Co1—O5^{i}	93.67 (3)	H1D—N1—H1B^{iv}	136 (6)
O4^{iii}—Co1—O5^{i}	86.33 (3)	H1A—N1—H1C^{iv}	110 (5)
O5—Co1—O5^{i}	180.0	H1B—N1—H1C^{iv}	73 (5)
O3—S1—O2	109.62 (5)	H1C—N1—H1C^{iv}	179.999 (15)

O3—S1—O4	113.51 (5)	H1D—N1—H1C^{iv}	103 (5)
O2—S1—O4	110.18 (5)	H1B^{iv}—N1—H1C^{iv}	107 (6)
O3—S1—O1	109.29 (5)	H1A—N1—H1D^{iv}	86 (5)
O2—S1—O1	109.38 (5)	H1B—N1—H1D^{iv}	136 (6)
O4—S1—O1	104.71 (5)	H1C—N1—H1D^{iv}	103 (5)
S1—O1—H1O	109 (3)	H1D—N1—H1D^{iv}	180 (7)
S1—O2—Co1	132.41 (5)	H1B^{iv}—N1—H1D^{iv}	44 (6)
S1—O4—Co1^{v}	132.25 (5)	H1C^{iv}—N1—H1D^{iv}	77 (5)

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*
O1—H1*O*···O1^{vi}	0.70 (4)	1.80 (4)	2.4810 (16)	165 (5)
O5—H5*A*···O2^{vii}	0.74 (3)	2.57 (3)	2.9923 (11)	118 (2)
O5—H5*B*···O3^{vii}	0.77 (2)	2.51 (2)	3.2020 (13)	149 (2)
N1—H1*A*···O3^{vi}	0.98 (7)	1.96 (7)	2.9298 (8)	169 (7)
N1—H1*D*···O1^{viii}	0.84 (7)	2.33 (7)	3.1657 (10)	175 (7)
N1—H1*C*···O1	0.87 (6)	2.29 (6)	3.1098 (8)	158 (6)
N1—H1*D*···O1^{viii}	0.84 (7)	2.33 (7)	3.1657 (10)	175 (7)
N1—H1*B*···O4^{viii}	0.91 (9)	1.97 (9)	2.8773 (7)	176 (8)

Symmetry codes: (vi) −*x*−1, −*y*−1, −*z*−1; (vii) −*x*, −*y*−1, −*z*; (viii) −*x*−1, −*y*, −*z*−1.

catena-Poly[ammonium [[diaquanickel(II)]-μ-(hydrogen sulfato)-μ-sulfato]] (Ni_296K)

Crystal data

$(NH_4)[Ni(HSO_4)(SO_4)(H_2O)_2]$
M_r = 305.91
Triclinic, $P\overline{1}$
a = 4.5746 (16) Å
b = 5.7944 (19) Å
c = 8.347 (2) Å
α = 104.049 (11)°
β = 98.133 (12)°
γ = 94.420 (12)°
V = 211.04 (12) Å^3

Z = 1
$F(000)$ = 156
D_x = 2.407 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1458 reflections
θ = 2.6–32.9°
μ = 2.84 mm^{-1}
T = 296 K
Plate, green
0.12 × 0.06 × 0.01 mm

Data collection

Bruker APEXII CCD diffractometer
ω– and φ–scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.598, T_{max} = 0.747
3532 measured reflections

1591 independent reflections
1307 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 33.2°, θ_{min} = 3.7°
h = −7→6
k = −8→8
l = −12→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.077

S = 1.10
1591 reflections
95 parameters
7 restraints

Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.034P)^2 + 0.0522P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.47$ e Å^{-3}
$\Delta\rho_{min} = -0.59$ 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)
Ni1	0.000000	0.000000	0.000000	0.01194 (10)	
S1	−0.58959 (10)	−0.31438 (8)	−0.23407 (5)	0.01127 (11)	
O1	−0.4942 (4)	−0.3026 (3)	−0.39878 (19)	0.0221 (3)	
O2	−0.3263 (3)	−0.2831 (3)	−0.1046 (2)	0.0189 (3)	
O3	−0.7571 (4)	−0.5462 (3)	−0.2553 (2)	0.0221 (3)	
O4	−0.7659 (3)	−0.1127 (3)	−0.19328 (18)	0.0170 (3)	
O5	0.1932 (4)	−0.2084 (3)	0.1425 (2)	0.0182 (3)	
H5A	0.105 (8)	−0.329 (5)	0.161 (5)	0.057 (12)*	
H5B	0.352 (6)	−0.271 (7)	0.131 (5)	0.068 (14)*	
N1	0.000000	0.000000	−0.500000	0.0247 (6)	
H1A	−0.077 (19)	−0.149 (7)	−0.560 (11)	0.05 (2)*	0.5
H1B	−0.07 (2)	−0.02 (2)	−0.610 (3)	0.07 (3)*	0.5
H1C	−0.167 (8)	−0.054 (11)	−0.469 (8)	0.023 (15)*	0.5
H1D	−0.11 (2)	0.104 (17)	−0.540 (15)	0.09 (4)*	0.5
H1O	−0.475 (14)	−0.439 (5)	−0.459 (6)	0.020 (15)*	0.5

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.01190 (18)	0.01163 (16)	0.01267 (17)	0.00243 (12)	0.00245 (12)	0.00331 (12)
S1	0.0131 (2)	0.00957 (19)	0.0104 (2)	0.00016 (16)	0.00340 (16)	0.00082 (16)
O1	0.0367 (10)	0.0162 (7)	0.0149 (7)	0.0010 (7)	0.0147 (7)	0.0012 (6)
O2	0.0159 (7)	0.0152 (6)	0.0238 (7)	0.0005 (5)	−0.0023 (6)	0.0053 (6)
O3	0.0259 (9)	0.0142 (7)	0.0234 (8)	−0.0080 (6)	0.0058 (6)	0.0017 (6)
O4	0.0180 (7)	0.0195 (7)	0.0165 (6)	0.0090 (6)	0.0074 (6)	0.0057 (6)
O5	0.0179 (8)	0.0162 (7)	0.0238 (7)	0.0041 (6)	0.0042 (6)	0.0101 (6)
N1	0.0324 (17)	0.0203 (13)	0.0194 (13)	0.0000 (12)	−0.0024 (12)	0.0059 (11)

Geometric parameters (Å, º)

Ni1—O5^{i}	2.0539 (16)	O5—H5A	0.838 (10)
Ni1—O5	2.0539 (16)	O5—H5B	0.843 (10)
Ni1—O2^{i}	2.0592 (16)	N1—H1A	0.901 (10)
Ni1—O2	2.0592 (16)	N1—H1B	0.898 (10)
Ni1—O4^{ii}	2.0642 (15)	N1—H1C	0.899 (10)

Ni1—O4^{iii}	2.0642 (15)	N1—H1D	0.899 (10)
S1—O3	1.4525 (15)	N1—H1A^{iv}	0.901 (10)
S1—O2	1.4678 (16)	N1—H1B^{iv}	0.898 (10)
S1—O4	1.4703 (16)	N1—H1C^{iv}	0.899 (10)
S1—O1	1.5153 (16)	N1—H1D^{iv}	0.899 (10)
O1—H1O	0.845 (10)		

O5^{i}—Ni1—O5	180.00 (6)	H1A—N1—H1C	66 (6)
O5^{i}—Ni1—O2^{i}	87.64 (7)	H1B—N1—H1C	99 (7)
O5—Ni1—O2^{i}	92.36 (7)	H1A—N1—H1D	108 (7)
O5^{i}—Ni1—O2	92.36 (7)	H1B—N1—H1D	58 (8)
O5—Ni1—O2	87.64 (7)	H1C—N1—H1D	87 (7)
O2^{i}—Ni1—O2	180.0	H1A—N1—H1A^{iv}	179.998 (12)
O5^{i}—Ni1—O4^{ii}	85.92 (7)	H1B—N1—H1A^{iv}	118 (7)
O5—Ni1—O4^{ii}	94.08 (7)	H1C—N1—H1A^{iv}	114 (6)
O2^{i}—Ni1—O4^{ii}	89.36 (7)	H1D—N1—H1A^{iv}	72 (7)
O2—Ni1—O4^{ii}	90.64 (7)	H1A—N1—H1B^{iv}	118 (7)
O5^{i}—Ni1—O4^{iii}	94.08 (7)	H1B—N1—H1B^{iv}	180.00 (3)
O5—Ni1—O4^{iii}	85.92 (7)	H1C—N1—H1B^{iv}	81 (7)
O2^{i}—Ni1—O4^{iii}	90.64 (7)	H1D—N1—H1B^{iv}	122 (8)
O2—Ni1—O4^{iii}	89.36 (7)	H1A^{iv}—N1—H1B^{iv}	62 (7)
O4^{ii}—Ni1—O4^{iii}	180.00 (11)	H1A—N1—H1C^{iv}	114 (6)
O3—S1—O2	109.59 (10)	H1B—N1—H1C^{iv}	81 (7)
O3—S1—O4	113.40 (10)	H1C—N1—H1C^{iv}	179.999 (12)
O2—S1—O4	110.21 (9)	H1D—N1—H1C^{iv}	93 (7)
O3—S1—O1	109.00 (9)	H1A^{iv}—N1—H1C^{iv}	66 (6)
O2—S1—O1	109.71 (10)	H1B^{iv}—N1—H1C^{iv}	99 (7)
O4—S1—O1	104.79 (9)	H1A—N1—H1D^{iv}	72 (7)
S1—O1—H1O	112 (4)	H1B—N1—H1D^{iv}	122 (8)
S1—O2—Ni1	131.92 (10)	H1C—N1—H1D^{iv}	93 (7)
S1—O4—Ni1^{v}	132.17 (9)	H1D—N1—H1D^{iv}	180.00 (6)
Ni1—O5—H5A	124 (3)	H1A^{iv}—N1—H1D^{iv}	108 (7)
Ni1—O5—H5B	126 (3)	H1B^{iv}—N1—H1D^{iv}	58 (8)
H5A—O5—H5B	94 (4)	H1C^{iv}—N1—H1D^{iv}	87 (7)
H1A—N1—H1B	62 (7)		

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*
O1—H1*O*···O1^{vi}	0.85 (1)	1.65 (2)	2.484 (3)	167 (6)
O5—H5*A*···O2^{vii}	0.84 (1)	2.50 (4)	3.005 (2)	119 (3)
O5—H5*B*···O3^{vii}	0.84 (1)	2.43 (2)	3.177 (3)	147 (4)
N1—H1*A*···O3^{vi}	0.90 (1)	2.05 (3)	2.9360 (17)	166 (9)
N1—H1*D*···O1^{viii}	0.90 (1)	2.29 (4)	3.152 (2)	159 (9)
N1—H1*C*···O1	0.90 (1)	2.23 (2)	3.0898 (18)	160 (6)

N1—H1*D*···O1^{viii}	0.90 (1)	2.29 (4)	3.152 (2)	159 (9)
N1—H1*B*···O4^{viii}	0.90 (1)	2.07 (7)	2.8761 (16)	149 (11)

Symmetry codes: (vi) −*x*−1, −*y*−1, −*z*−1; (vii) −*x*, −*y*−1, −*z*; (viii) −*x*−1, −*y*, −*z*−1.

catena-Poly[ammonium [[diaquairon(II)]-μ-(hydrogen sulfato)-μ-sulfato]] (Fe_100K)

Crystal data

$(NH_4)[Fe(HSO_4)(SO_4)(H_2O)_2]$
M_r = 303.05
Triclinic, $P\overline{1}$
a = 7.0847 (6) Å
b = 7.7956 (7) Å
c = 8.3868 (7) Å
α = 84.5953 (15)°
β = 73.2990 (15)°
γ = 76.2281 (17)°
V = 430.74 (6) Å^3
Z = 2
$F(000)$ = 308
D_x = 2.337 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 8457 reflections
θ = 3.6–41.7°
μ = 2.28 mm^{-1}
T = 100 K
Fragment, light green
0.12 × 0.09 × 0.02 mm

Data collection

Bruker APEXII CCD diffractometer
ω– and φ–scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.619, T_{max} = 0.748
17276 measured reflections
5854 independent reflections
5143 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 41.8°, θ_{min} = 3.4°
h = −13→13
k = −13→14
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.022
$wR(F^2)$ = 0.062
S = 1.05
5854 reflections
164 parameters
0 restraints
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0297P)^2 + 0.0364P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.78 e Å^{-3}
$\Delta\rho_{min}$ = −0.76 e Å^{-3}
Extinction correction: SHELXL (Sheldrick, 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0254 (17)

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}
Fe1	0.25151 (2)	0.25374 (2)	0.49272 (2)	0.00495 (3)
S1	−0.20455 (2)	0.38437 (2)	0.73962 (2)	0.00486 (3)
S2	0.71243 (2)	0.10045 (2)	0.26107 (2)	0.00509 (3)
O1	−0.40545 (7)	0.35213 (7)	0.75846 (6)	0.00970 (8)
O2	−0.05984 (7)	0.28137 (6)	0.59849 (6)	0.00805 (7)

O3	−0.20277 (8)	0.57470 (6)	0.70817 (6)	0.00806 (7)
O4	−0.14330 (7)	0.33145 (7)	0.89603 (6)	0.00878 (8)
O5	0.91507 (7)	0.12707 (7)	0.23313 (6)	0.01001 (8)
O6	0.69127 (7)	−0.08135 (6)	0.30682 (6)	0.00790 (7)
O7	0.56594 (7)	0.22548 (7)	0.38158 (6)	0.00866 (7)
O8	0.65788 (8)	0.13443 (7)	0.09282 (6)	0.00920 (8)
O9	0.26255 (8)	0.46053 (7)	0.63591 (6)	0.00918 (8)
O10	0.24415 (8)	0.04282 (7)	0.35229 (6)	0.00923 (8)
N1	0.75683 (8)	0.74104 (7)	0.00607 (6)	0.00962 (9)
H1W	0.366 (3)	0.468 (3)	0.656 (2)	0.040 (5)*
H2W	0.219 (3)	0.573 (2)	0.6169 (19)	0.034 (4)*
H3W	0.291 (3)	−0.067 (2)	0.362 (2)	0.043 (5)*
H4W	0.136 (3)	0.036 (2)	0.334 (2)	0.036 (4)*
H1O	0.741 (2)	0.217 (2)	0.0126 (16)	0.060 (6)*
H1N	0.8604 (18)	0.7089 (16)	0.0468 (14)	0.019 (3)*
H2N	0.6513 (19)	0.7147 (17)	0.0744 (15)	0.023 (3)*
H3N	0.7821 (18)	0.6913 (16)	−0.0890 (15)	0.020 (3)*
H4N	0.7343 (19)	0.8583 (19)	−0.0057 (16)	0.027 (3)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.00537 (4)	0.00455 (4)	0.00480 (4)	−0.00135 (3)	−0.00113 (3)	0.00033 (3)
S1	0.00463 (6)	0.00543 (6)	0.00448 (5)	−0.00159 (4)	−0.00111 (4)	0.00087 (4)
S2	0.00486 (6)	0.00560 (6)	0.00485 (5)	−0.00169 (4)	−0.00126 (4)	0.00085 (4)
O1	0.00568 (17)	0.0146 (2)	0.00951 (18)	−0.00435 (15)	−0.00205 (13)	0.00177 (15)
O2	0.00666 (16)	0.00801 (17)	0.00835 (17)	−0.00108 (13)	−0.00005 (13)	−0.00243 (13)
O3	0.01220 (19)	0.00485 (16)	0.00735 (16)	−0.00189 (14)	−0.00339 (14)	0.00101 (13)
O4	0.00992 (18)	0.01136 (19)	0.00653 (16)	−0.00446 (15)	−0.00414 (13)	0.00401 (13)
O5	0.00555 (17)	0.0147 (2)	0.01045 (18)	−0.00411 (15)	−0.00241 (14)	0.00212 (15)
O6	0.01117 (18)	0.00526 (16)	0.00762 (16)	−0.00207 (14)	−0.00348 (14)	0.00165 (13)
O7	0.00748 (17)	0.00758 (17)	0.00980 (17)	−0.00150 (14)	−0.00002 (13)	−0.00261 (13)
O8	0.01154 (19)	0.01164 (19)	0.00667 (16)	−0.00573 (15)	−0.00466 (14)	0.00377 (14)
O9	0.01044 (18)	0.00697 (17)	0.01178 (18)	−0.00233 (14)	−0.00507 (15)	−0.00103 (14)
O10	0.00890 (18)	0.00700 (17)	0.01307 (19)	−0.00151 (14)	−0.00468 (15)	−0.00192 (14)
N1	0.0103 (2)	0.0110 (2)	0.0080 (2)	−0.00397 (18)	−0.00154 (17)	−0.00116 (17)

Geometric parameters (Å, º)

Fe1—O2	2.0939 (5)	S2—O7	1.4662 (5)
Fe1—O3^{i}	2.1063 (5)	S2—O8	1.5508 (5)
Fe1—O7	2.1192 (5)	O8—H1O	1.040 (16)
Fe1—O6^{ii}	2.1222 (5)	O9—H1W	0.816 (17)
Fe1—O9	2.1273 (5)	O9—H2W	0.875 (17)
Fe1—O10	2.1294 (5)	O10—H3W	0.849 (19)
S1—O1	1.4654 (5)	O10—H4W	0.840 (17)
S1—O2	1.4786 (5)	N1—H1N	0.871 (12)
S1—O3	1.4848 (5)	N1—H2N	0.857 (13)

S1—O4	1.4897 (5)	N1—H3N	0.873 (12)
S2—O5	1.4486 (5)	N1—H4N	0.890 (14)
S2—O6	1.4642 (5)		
O2—Fe1—O3^{i}	91.39 (2)	O5—S2—O7	110.90 (3)
O2—Fe1—O7	179.016 (16)	O6—S2—O7	111.31 (3)
O3^{i}—Fe1—O7	87.88 (2)	O5—S2—O8	107.81 (3)
O2—Fe1—O6^{ii}	90.08 (2)	O6—S2—O8	103.28 (3)
O3^{i}—Fe1—O6^{ii}	178.490 (16)	O7—S2—O8	108.40 (3)
O7—Fe1—O6^{ii}	90.65 (2)	S1—O2—Fe1	132.45 (3)
O2—Fe1—O9	92.376 (19)	S1—O3—Fe1^{i}	131.15 (3)
O3^{i}—Fe1—O9	93.37 (2)	S2—O6—Fe1^{ii}	132.50 (3)
O7—Fe1—O9	88.326 (19)	S2—O7—Fe1	132.00 (3)
O6^{ii}—Fe1—O9	86.24 (2)	S2—O8—H1O	112.0 (7)
O2—Fe1—O10	88.086 (19)	Fe1—O9—H1W	122.3 (13)
O3^{i}—Fe1—O10	87.74 (2)	Fe1—O9—H2W	124.8 (10)
O7—Fe1—O10	91.225 (19)	H1W—O9—H2W	97.4 (17)
O6^{ii}—Fe1—O10	92.63 (2)	Fe1—O10—H3W	129.4 (11)
O9—Fe1—O10	178.780 (17)	Fe1—O10—H4W	120.2 (12)
O1—S1—O2	108.49 (3)	H3W—O10—H4W	97.2 (17)
O1—S1—O3	111.55 (3)	H1N—N1—H2N	110.8 (12)
O2—S1—O3	109.53 (3)	H1N—N1—H3N	110.5 (12)
O1—S1—O4	110.01 (3)	H2N—N1—H3N	110.0 (12)
O2—S1—O4	110.58 (3)	H1N—N1—H4N	106.1 (12)
O3—S1—O4	106.69 (3)	H2N—N1—H4N	107.8 (13)
O5—S2—O6	114.63 (3)	H3N—N1—H4N	111.5 (13)

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}	1.040 (16)	1.467 (16)	2.5067 (8)	178.6 (12)
O9—H1*W*···O1^{iv}	0.816 (17)	2.030 (19)	2.7537 (7)	147.5 (18)
O9—H2*W*···O2^{i}	0.875 (17)	2.447 (17)	3.0785 (7)	129.5 (13)
O9—H2*W*···O5^{v}	0.875 (17)	2.599 (16)	3.3272 (8)	141.3 (13)
O9—H2*W*···O7^{v}	0.875 (17)	2.439 (17)	2.9556 (7)	118.3 (14)
O10—H3*W*···O1^{vi}	0.849 (19)	2.389 (18)	3.1467 (8)	149.0 (15)
O10—H3*W*···O2^{vi}	0.849 (19)	2.541 (19)	3.0541 (7)	119.9 (16)
O10—H4*W*···O5^{vii}	0.840 (17)	1.946 (18)	2.7115 (7)	151.2 (18)
N1—H3*N*···O3^{iii}	0.873 (12)	1.967 (12)	2.8334 (7)	171.5 (11)
N1—H1*N*···O4^{v}	0.871 (12)	2.138 (12)	2.9931 (7)	167.2 (11)
N1—H2*N*···O1^{i}	0.857 (13)	2.040 (13)	2.8965 (7)	178.3 (12)
N1—H4*N*···O8^{viii}	0.890 (14)	2.266 (14)	3.0869 (8)	153.2 (11)

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*.

catena-Poly[ammonium [[diaquacobalt(II)]-μ-(hydrogen sulfato)-μ-sulfato]] (Co_100K)

Crystal data

$(NH_4)[Co(HSO_4)(SO_4)(H_2O)_2]$
M_r = 306.13
Triclinic, $P\overline{1}$
a = 7.0775 (4) Å
b = 7.7268 (4) Å
c = 8.3281 (5) Å
α = 84.5548 (11)°
β = 73.1705 (15)°
γ = 76.3233 (11)°
V = 423.40 (4) Å^3
Z = 2
$F(000)$ = 310
D_x = 2.401 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3880 reflections
θ = 2.5–37.9°
μ = 2.56 mm^{-1}
T = 100 K
Fragment, violet
0.12 × 0.09 × 0.02 mm

Data collection

Bruker APEXII CCD diffractometer
ω– and φ–scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.669, T_{max} = 0.747
8668 measured reflections
4184 independent reflections
3507 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 37.4°, θ_{min} = 2.7°
h = −11→11
k = −12→13
l = −12→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.062
S = 1.04
4184 reflections
164 parameters
0 restraints
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0279P)^2 + 0.0625P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.54 e Å^{-3}
$\Delta\rho_{min}$ = −0.53 e Å^{-3}
Extinction correction: SHELXL (Sheldrick, 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0088 (12)

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}
Co1	0.25150 (2)	0.25333 (2)	0.49323 (2)	0.00461 (4)
S1	−0.20148 (3)	0.38406 (3)	0.73639 (3)	0.00453 (5)
S2	0.70903 (3)	0.10225 (3)	0.26461 (3)	0.00474 (5)
O1	−0.40347 (10)	0.35343 (10)	0.75566 (9)	0.00908 (12)
O2	−0.05715 (10)	0.27901 (9)	0.59451 (8)	0.00768 (12)
O3	−0.19659 (11)	0.57524 (9)	0.70390 (8)	0.00763 (12)
O4	−0.14110 (11)	0.33068 (9)	0.89441 (8)	0.00829 (12)
O5	0.91201 (10)	0.12872 (10)	0.23703 (9)	0.00941 (12)
O6	0.68627 (11)	−0.08057 (9)	0.31075 (8)	0.00761 (12)

O7	0.56238 (10)	0.22842 (9)	0.38606 (9)	0.00817 (12)
O8	0.65547 (11)	0.13697 (10)	0.09487 (8)	0.00857 (12)
O9	0.25915 (11)	0.45756 (10)	0.63827 (9)	0.00840 (12)
O10	0.24613 (11)	0.04547 (10)	0.35051 (9)	0.00849 (12)
N1	0.75577 (13)	0.74180 (11)	0.00616 (10)	0.00941 (14)
H1W	0.358 (3)	0.468 (3)	0.655 (3)	0.033 (5)*
H2W	0.212 (3)	0.564 (3)	0.624 (2)	0.034 (5)*
H3W	0.291 (3)	−0.058 (3)	0.359 (2)	0.037 (5)*
H4W	0.142 (3)	0.038 (3)	0.335 (2)	0.031 (5)*
H1O	0.739 (3)	0.211 (3)	0.015 (2)	0.050 (6)*
H1N	0.860 (2)	0.708 (2)	0.0516 (19)	0.021 (4)*
H2N	0.652 (3)	0.713 (2)	0.078 (2)	0.026 (4)*
H3N	0.786 (2)	0.692 (2)	−0.0922 (19)	0.018 (4)*
H4N	0.730 (3)	0.861 (2)	−0.004 (2)	0.030 (4)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co1	0.00482 (6)	0.00409 (6)	0.00483 (6)	−0.00083 (4)	−0.00140 (4)	0.00022 (4)
S1	0.00434 (9)	0.00492 (9)	0.00429 (8)	−0.00129 (7)	−0.00126 (6)	0.00089 (6)
S2	0.00451 (9)	0.00505 (9)	0.00468 (8)	−0.00134 (7)	−0.00135 (6)	0.00081 (6)
O1	0.0053 (3)	0.0131 (3)	0.0095 (3)	−0.0040 (2)	−0.0020 (2)	0.0014 (2)
O2	0.0062 (3)	0.0077 (3)	0.0083 (3)	−0.0009 (2)	−0.0004 (2)	−0.0023 (2)
O3	0.0111 (3)	0.0044 (3)	0.0077 (3)	−0.0014 (2)	−0.0037 (2)	0.0008 (2)
O4	0.0095 (3)	0.0106 (3)	0.0063 (3)	−0.0043 (2)	−0.0041 (2)	0.0041 (2)
O5	0.0049 (3)	0.0135 (3)	0.0102 (3)	−0.0034 (2)	−0.0022 (2)	0.0018 (2)
O6	0.0106 (3)	0.0052 (3)	0.0074 (3)	−0.0019 (2)	−0.0035 (2)	0.0017 (2)
O7	0.0069 (3)	0.0071 (3)	0.0094 (3)	−0.0014 (2)	0.0001 (2)	−0.0025 (2)
O8	0.0106 (3)	0.0114 (3)	0.0059 (3)	−0.0054 (2)	−0.0045 (2)	0.0037 (2)
O9	0.0087 (3)	0.0067 (3)	0.0113 (3)	−0.0017 (2)	−0.0049 (2)	−0.0009 (2)
O10	0.0075 (3)	0.0065 (3)	0.0124 (3)	−0.0008 (2)	−0.0044 (2)	−0.0018 (2)
N1	0.0102 (4)	0.0109 (4)	0.0072 (3)	−0.0036 (3)	−0.0014 (3)	−0.0007 (3)

Geometric parameters (Å, º)

Co1—O2	2.0695 (7)	S2—O7	1.4668 (7)
Co1—O3^{i}	2.0771 (7)	S2—O8	1.5506 (7)
Co1—O6^{ii}	2.0895 (7)	O8—H1O	0.99 (2)
Co1—O7	2.0905 (7)	O9—H1W	0.77 (2)
Co1—O9	2.0957 (7)	O9—H2W	0.82 (2)
Co1—O10	2.1003 (7)	O10—H3W	0.79 (2)
S1—O1	1.4647 (7)	O10—H4W	0.80 (2)
S1—O2	1.4797 (7)	N1—H1N	0.897 (16)
S1—O3	1.4831 (7)	N1—H2N	0.864 (17)
S1—O4	1.4912 (7)	N1—H3N	0.887 (15)
S2—O5	1.4482 (7)	N1—H4N	0.894 (18)
S2—O6	1.4621 (7)		

O2—Co1—O3^{i}	89.43 (3)	O5—S2—O7	110.82 (4)
O2—Co1—O6^{ii}	91.87 (3)	O6—S2—O7	111.17 (4)
O3^{i}—Co1—O6^{ii}	178.71 (3)	O5—S2—O8	107.85 (4)
O2—Co1—O7	178.83 (2)	O6—S2—O8	103.30 (4)
O3^{i}—Co1—O7	89.54 (3)	O7—S2—O8	108.44 (4)
O6^{ii}—Co1—O7	89.16 (3)	S1—O2—Co1	131.08 (4)
O2—Co1—O9	92.65 (3)	S1—O3—Co1^{i}	130.79 (4)
O3^{i}—Co1—O9	93.92 (3)	S2—O6—Co1^{ii}	132.17 (4)
O6^{ii}—Co1—O9	85.97 (3)	S2—O7—Co1	130.91 (4)
O7—Co1—O9	87.96 (3)	S2—O8—H1O	112.7 (10)
O2—Co1—O10	87.61 (3)	Co1—O9—H1W	122.4 (15)
O3^{i}—Co1—O10	87.02 (3)	Co1—O9—H2W	124.7 (13)
O6^{ii}—Co1—O10	93.08 (3)	H1W—O9—H2W	97 (2)
O7—Co1—O10	91.79 (3)	Co1—O10—H3W	129.1 (14)
O9—Co1—O10	179.03 (3)	Co1—O10—H4W	119.8 (14)
O1—S1—O2	108.56 (4)	H3W—O10—H4W	97 (2)
O1—S1—O3	111.65 (4)	H1N—N1—H2N	106.8 (15)
O2—S1—O3	109.44 (4)	H1N—N1—H3N	110.4 (15)
O1—S1—O4	110.01 (4)	H2N—N1—H3N	112.7 (16)
O2—S1—O4	110.63 (4)	H1N—N1—H4N	107.8 (16)
O3—S1—O4	106.55 (4)	H2N—N1—H4N	107.4 (16)
O5—S2—O6	114.78 (4)	H3N—N1—H4N	111.4 (16)

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}	0.99 (2)	1.51 (2)	2.5007 (11)	177.4 (17)
O9—H1*W*···O1^{iv}	0.77 (2)	2.08 (2)	2.7566 (10)	147 (2)
O9—H2*W*···O2^{i}	0.82 (2)	2.468 (19)	3.0700 (10)	130.8 (16)
O9—H2*W*···O5^{v}	0.82 (2)	2.59 (2)	3.2918 (10)	144.5 (16)
O9—H1*W*···O7^{v}	0.77 (2)	2.51 (2)	2.9549 (10)	118.1 (19)
O10—H3*W*···O1^{vi}	0.79 (2)	2.42 (2)	3.1378 (10)	151.0 (18)
O10—H3*W*···O2^{vi}	0.79 (2)	2.58 (2)	3.0492 (10)	119.8 (19)
O10—H4*W*···O5^{vii}	0.80 (2)	1.98 (2)	2.7127 (10)	152 (2)
N1—H3*N*···O3^{iii}	0.887 (15)	1.955 (15)	2.8328 (10)	170.1 (15)
N1—H1*N*···O4^{v}	0.897 (16)	2.114 (16)	2.9890 (11)	164.7 (14)
N1—H2*N*···O1^{i}	0.864 (17)	2.038 (17)	2.9017 (11)	178.9 (16)
N1—H4*N*···O8^{viii}	0.894 (18)	2.252 (18)	3.0777 (11)	153.4 (15)

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*.

catena-Poly[ammonium [[diaquanickel(II)]-μ-(hydrogen sulfato)-μ-sulfato]] (Ni_100K)

Crystal data

$(NH_4)[Ni(HSO_4)(SO_4)(H_2O)_2]$
M_r = 305.91
Triclinic, $P\overline{1}$
a = 7.0437 (9) Å
b = 7.6405 (9) Å
c = 8.3097 (10) Å
α = 84.571 (3)°
β = 73.376 (3)°

γ = 76.056 (3)°
V = 415.73 (9) Å^3
Z = 2
F(000) = 312
D_x = 2.444 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å

Cell parameters from 1834 reflections
θ = 2.6–35.4°
μ = 2.88 mm^{-1}
T = 100 K
Plate, green
0.12 × 0.09 × 0.02 mm

Data collection

Bruker APEXII CCD diffractometer
ω– and φ–scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.609, T_{max} = 0.747
7657 measured reflections

3609 independent reflections
2253 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 36.0°, θ_{min} = 2.8°
h = −11→11
k = −12→10
l = −13→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.092
S = 1.01
3609 reflections
163 parameters
0 restraints

Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0362P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.86 e Å^{-3}
$\Delta\rho_{min}$ = −0.95 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}
Ni1	0.25124 (5)	0.25282 (4)	0.49418 (4)	0.00539 (7)
S1	−0.20008 (8)	0.38376 (7)	0.73454 (6)	0.00486 (11)
S2	0.70693 (8)	0.10508 (7)	0.26711 (6)	0.00487 (11)
O1	−0.4025 (3)	0.3526 (2)	0.75282 (19)	0.0092 (3)
O2	−0.0549 (2)	0.2761 (2)	0.59314 (19)	0.0075 (3)
O3	−0.1951 (3)	0.5767 (2)	0.70128 (19)	0.0071 (3)
O4	−0.1400 (3)	0.3306 (2)	0.89363 (19)	0.0083 (3)
O5	0.9098 (2)	0.1337 (2)	0.24048 (19)	0.0084 (3)
O6	0.6869 (3)	−0.0809 (2)	0.31174 (19)	0.0083 (3)
O7	0.5585 (2)	0.2307 (2)	0.39011 (19)	0.0077 (3)
O8	0.6523 (3)	0.1411 (2)	0.09759 (19)	0.0084 (3)
O9	0.2564 (3)	0.4542 (2)	0.6385 (2)	0.0078 (3)
O10	0.2491 (3)	0.0488 (2)	0.3518 (2)	0.0082 (3)
N1	0.7538 (4)	0.7431 (3)	0.0062 (3)	0.0094 (3)
H1W	0.360 (8)	0.473 (7)	0.648 (6)	0.082 (19)*
H2W	0.209 (7)	0.536 (5)	0.628 (5)	0.042 (14)*

H3W	0.294 (6)	−0.056 (5)	0.364 (4)	0.026 (10)*
H4W	0.146 (4)	0.046 (4)	0.337 (3)	0.001 (7)*
H1O	0.724 (6)	0.203 (5)	0.019 (4)	0.053 (12)*
H1N	0.864 (5)	0.713 (4)	0.051 (4)	0.017 (8)*
H2N	0.641 (6)	0.712 (4)	0.076 (4)	0.029 (9)*
H3N	0.782 (5)	0.694 (4)	−0.091 (4)	0.017 (8)*
H4N	0.730 (9)	0.850 (8)	0.004 (6)	0.10 (2)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.00552 (12)	0.00502 (12)	0.00545 (12)	−0.00102 (9)	−0.00143 (9)	0.00004 (8)
S1	0.0051 (3)	0.0053 (2)	0.0042 (2)	−0.00159 (19)	−0.00140 (19)	0.00091 (17)
S2	0.0044 (3)	0.0058 (2)	0.0046 (2)	−0.00155 (19)	−0.00146 (18)	0.00088 (17)
O1	0.0066 (8)	0.0123 (8)	0.0092 (7)	−0.0039 (6)	−0.0021 (6)	0.0020 (6)
O2	0.0060 (8)	0.0074 (7)	0.0079 (6)	0.0005 (6)	−0.0009 (6)	−0.0023 (5)
O3	0.0098 (8)	0.0045 (7)	0.0076 (6)	−0.0008 (6)	−0.0042 (6)	0.0008 (5)
O4	0.0093 (8)	0.0118 (8)	0.0052 (6)	−0.0045 (6)	−0.0037 (6)	0.0040 (5)
O5	0.0043 (8)	0.0139 (8)	0.0089 (7)	−0.0047 (6)	−0.0034 (6)	0.0024 (6)
O6	0.0121 (9)	0.0051 (7)	0.0083 (6)	−0.0025 (6)	−0.0038 (6)	0.0022 (5)
O7	0.0067 (8)	0.0066 (7)	0.0089 (7)	−0.0007 (6)	−0.0009 (6)	−0.0021 (5)
O8	0.0113 (8)	0.0116 (8)	0.0050 (6)	−0.0059 (6)	−0.0045 (6)	0.0032 (6)
O9	0.0081 (9)	0.0067 (8)	0.0094 (7)	−0.0014 (6)	−0.0035 (6)	−0.0008 (6)
O10	0.0067 (9)	0.0077 (8)	0.0115 (7)	−0.0007 (6)	−0.0050 (6)	−0.0009 (6)
N1	0.0108 (8)	0.0107 (9)	0.0072 (8)	−0.0043 (7)	−0.0013 (7)	−0.0009 (6)

Geometric parameters (Å, º)

Ni1—O2	2.0457 (17)	S2—O7	1.4660 (16)
Ni1—O10	2.0491 (18)	S2—O8	1.5472 (17)
Ni1—O9	2.0513 (18)	O8—H1O	0.88 (4)
Ni1—O3^{i}	2.0519 (16)	O9—H1W	0.80 (5)
Ni1—O7	2.0594 (16)	O9—H2W	0.65 (4)
Ni1—O6^{ii}	2.0595 (17)	O10—H3W	0.80 (3)
S1—O1	1.4645 (17)	O10—H4W	0.78 (3)
S1—O3	1.4806 (16)	N1—H1N	0.93 (3)
S1—O2	1.4808 (16)	N1—H2N	0.91 (3)
S1—O4	1.4924 (17)	N1—H3N	0.87 (3)
S2—O5	1.4487 (17)	N1—H4N	0.79 (6)
S2—O6	1.4639 (16)		
O2—Ni1—O10	87.59 (7)	O5—S2—O7	110.67 (10)
O2—Ni1—O9	92.83 (7)	O6—S2—O7	110.96 (9)
O10—Ni1—O9	179.11 (9)	O5—S2—O8	107.92 (9)
O2—Ni1—O3^{i}	89.49 (7)	O6—S2—O8	103.32 (10)
O10—Ni1—O3^{i}	86.25 (7)	O7—S2—O8	108.63 (10)
O9—Ni1—O3^{i}	94.54 (7)	S1—O2—Ni1	130.45 (10)
O2—Ni1—O7	178.91 (7)	S1—O3—Ni1^{i}	130.68 (10)

O10—Ni1—O7	91.91 (7)	S2—O6—Ni1^{ii}	131.99 (11)
O9—Ni1—O7	87.68 (7)	S2—O7—Ni1	130.71 (10)
O3^{i}—Ni1—O7	89.52 (7)	S2—O8—H1O	117 (3)
O2—Ni1—O6^{ii}	91.53 (7)	Ni1—O9—H1W	123 (4)
O10—Ni1—O6^{ii}	93.68 (7)	Ni1—O9—H2W	120 (4)
O9—Ni1—O6^{ii}	85.52 (7)	H1W—O9—H2W	99 (5)
O3^{i}—Ni1—O6^{ii}	178.97 (8)	Ni1—O10—H3W	127 (2)
O7—Ni1—O6^{ii}	89.46 (7)	Ni1—O10—H4W	117 (2)
O1—S1—O3	111.91 (10)	H3W—O10—H4W	100 (3)
O1—S1—O2	108.25 (10)	H1N—N1—H2N	113 (3)
O3—S1—O2	109.45 (9)	H1N—N1—H3N	111 (3)
O1—S1—O4	109.98 (9)	H2N—N1—H3N	109 (3)
O3—S1—O4	106.50 (10)	H1N—N1—H4N	102 (4)
O2—S1—O4	110.77 (10)	H2N—N1—H4N	106 (4)
O5—S2—O6	114.88 (10)	H3N—N1—H4N	115 (4)

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}	0.88 (4)	1.62 (4)	2.499 (2)	173 (4)
O9—H1*W*···O1^{iv}	0.80 (5)	2.08 (5)	2.747 (3)	140 (5)
O9—H2*W*···O2^{i}	0.65 (4)	2.55 (4)	3.061 (3)	138 (4)
O9—H2*W*···O5^{v}	0.65 (4)	2.69 (4)	3.232 (2)	143 (4)
O9—H1*W*···O7^{v}	0.80 (5)	2.44 (5)	2.963 (2)	124 (5)
O10—H3*W*···O1^{vi}	0.80 (3)	2.41 (3)	3.115 (2)	148 (3)
O10—H3*W*···O2^{vi}	0.80 (3)	2.59 (4)	3.051 (2)	119 (3)
O10—H4*W*···O5^{vii}	0.78 (3)	2.00 (3)	2.719 (3)	155 (3)
N1—H3*N*···O3^{iii}	0.87 (3)	1.97 (3)	2.834 (3)	172 (3)
N1—H1*N*···O4^{v}	0.93 (3)	2.07 (3)	2.980 (3)	169 (3)
N1—H2*N*···O1^{i}	0.91 (3)	2.00 (3)	2.903 (3)	174 (3)
N1—H4*N*···O8^{viii}	0.79 (6)	2.31 (6)	3.068 (3)	159 (5)

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*.