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Article

Kristjánite, KNa$_2$H(SO$_4$)$_2$, a new fumarolic mineral from Iceland containing the [SO$_4$–H–SO$_4$]$^{3-}$ anion in the crystal structure

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Abstract

Kristjánite, KNa$_2$H(SO$_4$)$_2$, is a new mineral (IMA2022–131) found in a high-temperature fumarole on Fimmvörðuháls, Iceland. It is monoclinic, $P2_1/n$, a = 6.9625(1), b = 9.9953(1), c = 11.0928(2) (Å), β = 105.637(2)° and V = 743.40(2) Å^3. Kristjánite forms colourless transparent crystals up to ∼100 μm in size in compact aggregates with metathénardite, belomarinaite, aphthitalite, ivsite and an unknown mineral with tentative composition K$_2$NaH(SO$_4$)$_2$, or grows in white globules of smaller grains on their surface. The eight strongest maxima in a powder X-ray diffraction diagram are [d, Å (Intensity)] 4.37 (59), 3.65 (32), 3.48 (78), 3.36 (100), 3.18 (76), 2.83 (43), 2.73 (38) and 2.405 (23). The mineral represents a novel crystal structure type. In it, K is coordinated by eight O atoms, and two symmetrically independent Na atoms by seven O atoms. Two symmetrically independent S atoms are in tetrahedral coordination by O atoms. A close to linear, very short hydrogen bond (2.44 Å), connects the two to a SO$_4$–H–SO$_4$ dimer. K and Na coordination polyhedra share vertices, edges and even faces forming with sulfate tetrahedra a tight structure with narrow [100] channels lined on two opposite sides by hydrogen bonds.

Introduction

An eruption on Fimmvörðuháls, the pass that lies between Mýrdalsjökul and Eyjafjallajökul glaciers in south Iceland, began in March 2010. In June, July and September of 2010, Dr. Kristján Jónasson from the Icelandic Institute of Natural History collected a number of fumarolic samples, which formed on the fresh lava at Fimmvörðuháls and sent some fragments to the X-ray Diffraction Laboratory of the Department of Geosciences and Natural Resource Management of the Copenhagen University for analyses. We recognised an unknown powder diffraction pattern in the sample collected on 28th June from a new crater called Magni. Dr. Kristján Jónasson measured the temperature at the collection spot to be 670°C.

The new mineral was found in the part of the sample consisting of a seemingly glassy, greenish mass of metathénardite mixed with the new mineral and small amounts of belomarinaite (KNaSO$_4$), aphthitalite [K$_3$Na(SO$_4$)$_2$], ivsite [Na$_3$H(SO$_4$)$_2$], thénardite and kröhnkite (the last two being most probably products of a transformation of metathénardite at low temperatures after collection), with several unattributed diffraction lines. Blades of belomarinaite and white globules made of tiny crystals of the new mineral overgrow the 'glassy' mass (Fig. 1).

We named the new mineral after Kristján Jónasson. The type material is deposited in the mineral collection of the Icelandic Institute of Natural History, Urriðaholtsstræti 6–8, 210 Garðabær, Iceland, catalogue number 24468. The Commission on New Minerals, Nomenclature and Classification of the International Mineralogical Association (IMA–CNMNC) approved the mineral and its name (symbol Kjn) as IMA2022–131 (Balić-Žunić *et al.*, 2023).

Experimental methods

Chemical analyses were performed using a Scanning Electron Microscope with Energy Dispersive Spectrometer (SEM-EDS) Quanta FEG 200 ESEM equipped with a detector from Oxford Instruments, SDD x-max 80 mm^2, at the Nanocenter, Danish Technical University. The instrument has a superb resolution and is the best possible for this type of sensitive material. Conditions were: acceleration voltage = 15 kV; beam current = 0.5 nA; and beam diameter = 1.5 μm.

Powder X-ray diffraction (PXRD) measurements were carried out using a Bruker-AXS D8 Advance Powder Diffractometer, operating in the Bragg-Brentano reflection geometry. The instrument is equipped with a primary Ge111 monochromator producing a Cu*K*α1 (1.54059 Å) radiation and a silicon-strip LynxEye detector. A small quantity of powdered material was spread on the zero-background quartz plate covered by a thin film of silica fat (to prevent the sample from gliding off the plate during measurement) embedded in a variable height sample holder. The
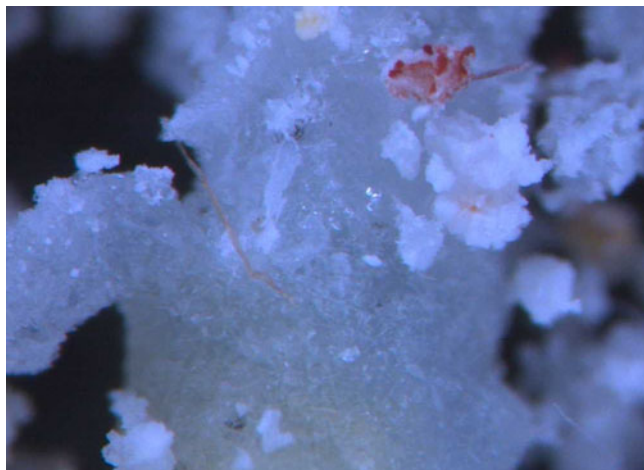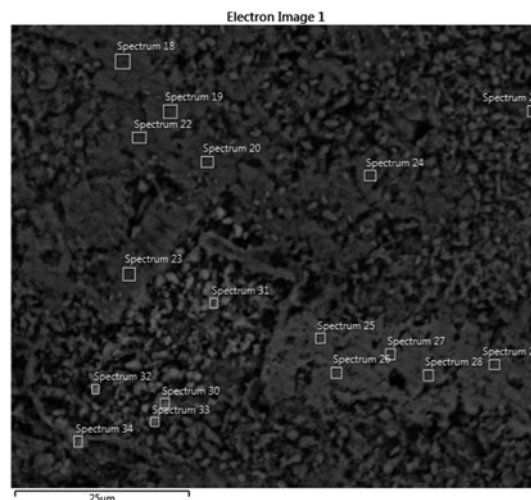
Figure 1. White globules of kristjánite on the surface of a blade of belomarinaite, specimen 24468. The width of the photograph is 1 mm.

Figure 2. Back-scattered electron image of the sample where chemical composition of kristjánite was determined. Spots 18–20 + 22–24: kristjánite. Spots 25–29: mineral with tentative composition $K_2NaH(SO_4)_2$.

mounted sample was gently pressed by a glass slide and adjusted on the linear focus of the instrument. The measurements were made on rotating sample in steps of 0.02°2θ with measuring time of four seconds per step, over a 2θ range of 5 to 90°.

Among the grains found in debris fallen from the sample, one hemimorphic grain showed clear X-ray diffraction and gave unit cell parameters consistent with those of the new mineral phase observed by PXRD. Single-crystal X-ray diffraction (SCXRD)

was performed using a Supernova diffractometer (Rigaku Oxford Diffraction) equipped with a micro (Mo) X-ray source (wavelength 0.71073 Å) and a Pilatus Dectris 200K area detector. Details of the crystal structure, measurement and refinement are in Table 1. The *CrysAlisPro* 1.171.40.55a (Rigaku Oxford Diffraction, 2019) program was used for data collection, data reduction, unit cell refinement and absorption correction. Empirical absorption correction was performed using spherical harmonics, implemented in *SCALE3 ABSPACK* scaling algorithm. The program *JANA* (Petříček *et al.*, 2023), including the charge-flipping method *SUPERFLIP* program (Oszlányi and Sütõ, 2004; Palatinus and Chapuis, 2007) was used for structure solution and refinement.

## Results

Chemical composition

The chemical measurements were done on six spots on a polished, largely porous sample embedded in epoxy containing kristjánite and a mineral with tentative composition $K_2NaH(SO_4)_2$ (Fig. 2). The results of the chemical analysis on kristjánite are given in Table 2. Besides the elements cited in the table, we also measured Mg, Al, Si, Ca, Ti, Fe and Cu. Their quantities were under the detection limits in kristjánite.

The empirical formula for kristjánite, calculated by the charge balance, is $K_{0.88}Na_{1.65}HS_2O_{7.76}$ (according to structural analysis, the mineral contains 1 H per formula unit). The ideal formula

Table 1. Details of the crystal, SCXRD measurement and crystal structure refinement.

Crystal data	
Crystal formula	$KNa_2H(SO_4)_2$
Crystal system, space group	Monoclinic, $P2_1/n$
a, b, c (Å)	6.9625(1), 9.9953(1), 11.0928(2)
β	105.637(2)°
V (Å³)	743.40(2)
Reflections for unit cell refinement	14232, 2.773–31.849°2θ
Z	4
Calculated density (g cm⁻¹)	2.4856
μ (mm⁻¹)	1.403
Crystal description	Colourless columnar
Crystal dimensions (mm)	0.150 × 0.100 × 0.075
Data collection	
Temperature (K)	293
Radiation type and wavelength (Å)	X-rays, 0.71073
Absorption correction	Multi-scan
T_{min}, T_{max}	0.88896, 1.00000
$θ_{min}$, $θ_{max}$ (°)	2.79, 31.91
Indices range	$-10 \leq h \leq 10$
	$-14 \leq k \leq 14$
	$-16 \leq l \leq 15$
Measured fraction	0.98
R_{int}	0.0163
Refinement	
Reflections	2427
Observed ($I > 3\sigma_I$)	2258
R_1 ($I > 3\sigma_I$)	1.87%
R_1 (all)	2.04%
wR (all)	3.68%
GoF	1.878
Weighting function	$w = 1/\sqrt{[(\sigma^2)F_o+(0.016F_o)^2]}$
Parameters	122
Residual ρ_e (+, −)	0.37, −0.30

Table 2. Chemical data (in wt.%) for kristjánite.

Constituent	Mean	Range	S.D. (σ)	Reference material
Na	14.51	13.81–15.28	0.61	Blödite
K	13.11	11.43–15.07	1.18	Leonite
S	24.48	24.26–24.77	0.20	Blödite
O	43.75	41.36–47.83	2.38	Blödite
Total	95.87	92.95–99.02	2.29	

S.D. = standard deviation
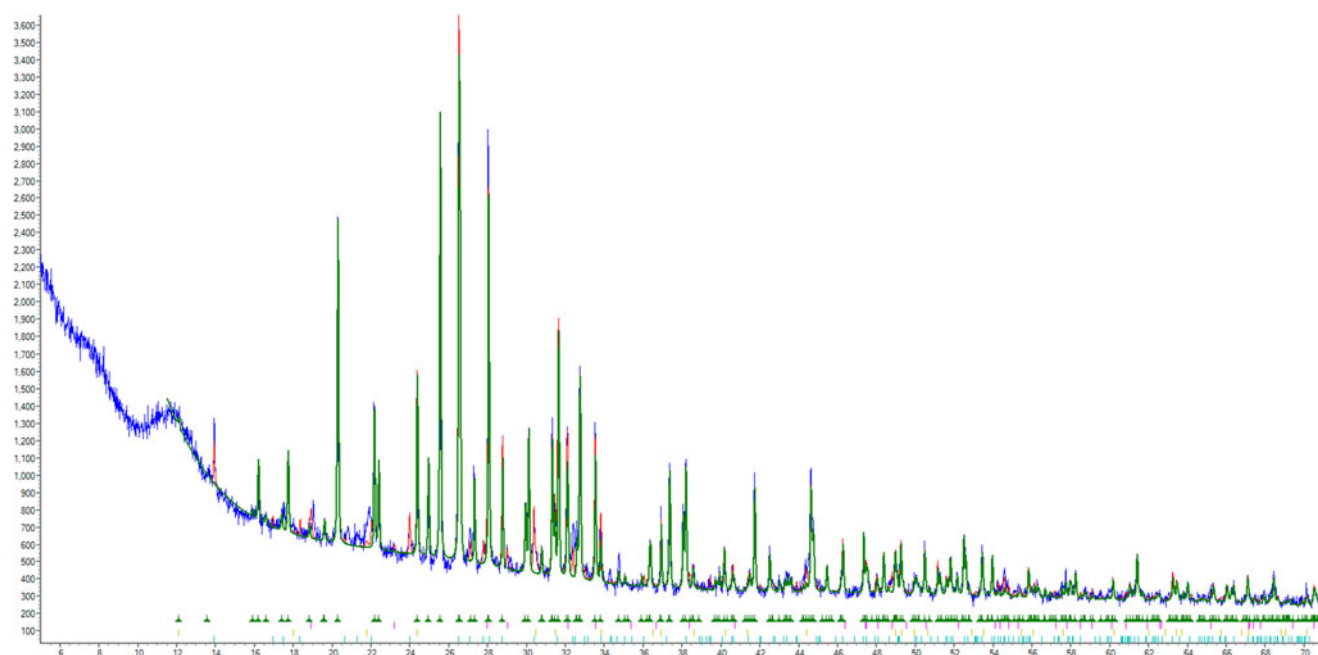
Figure 3. Powder X-ray diffraction diagram of a sample containing 86 wt.% kristjánite with small amounts of kröhnkite and thénardite, probably formed from metathénardite on cooling and aphthitalite (Rietveld analysis by program *Topas V.6*, Bruker-AXS). Green: calculated pattern of kristjánite; blue: experimental pattern; red: theoretical pattern.

Table 3. Powder X-ray diffraction data (*d* in Å) for kristjánite.*

I_{meas}	d_{meas}	d_{calc}	hkl	I_{meas}	d_{meas}	d_{calc}	hkl	I_{meas}	d_{meas}	d_{calc}	hkl
9	5.46	5.46	1 1 $\bar{1}$	6	2.58	2.58	0 1 4	6	1.912	1.914	2 4 1
5	5.06	5.09	1 0 1	3	2.50	2.50	0 4 0			1.911	1 5 $\bar{1}$
13	5.00	5.00	0 2 0	9	2.469	2.471	1 2 3	4	1.893	1.893	1 0 5
4	4.71	4.71	0 1 2			2.468	2 1 2	7	1.880	1.881	1 3 4
4	4.52	4.53	0 2 1	15	2.432	2.433	0 4 1			1.880	2 4 $\bar{3}$
59	**4.37**	4.37	1 1 $\bar{2}$				2 0 $\bar{4}$	8	1.858	1.859	2 0 4
27	4.00	4.01	1 2 0	**23**	**2.405**	2.406	2 3 $\bar{1}$			1.856	3 1 2
16	3.97	3.97	1 2 $\bar{1}$	15	2.362	2.364	2 1 $\bar{4}$	9	1.849	1.849	1 4 $\bar{4}$
32	**3.65**	3.65	0 2 2			2.363	2 3 0	3	1.821	1.825	0 4 4
18	3.57	3.57	1 0 $\bar{3}$	23	2.354	2.355	0 2 4			1.822	3 3 $\bar{3}$
			1 2 1	5	2.332	2.334	1 4 $\bar{1}$			1.818	1 1 $\bar{6}$
78	**3.48**	3.48	1 1 2	4	2.256	2.257	3 1 $\bar{1}$			1.816	2 3 3
100	**3.36**	3.36	1 1 $\bar{3}$	6	2.243	2.243	1 4 1	10	1.806	1.807	3 0 $\bar{5}$
		3.35	0 1 3	5	2.219	2.223	1 4 $\bar{2}$	5	1.783	1.784	2 0 $\bar{6}$
			2 0 0			2.222	1 1 4	4	1.780	1.780	0 0 6
19	3.26	3.26	2 0 $\bar{2}$			2.220	2 3 1	7	1.763	1.763	3 3 1
76	**3.18**	3.18	0 3 1			2.216	1 0 $\bar{5}$	4	1.752	1.753	0 1 6
			2 1 0	4	2.175	2.176	3 0 $\bar{3}$	11	1.742	1.742	2 2 4
23	3.10	3.10	2 1 $\bar{2}$	21	2.162	2.163	1 1 $\bar{5}$	8	1.739	1.739	4 0 $\bar{2}$
13	2.98	2.98	1 3 0				1 3 3	9	1.713	1.715	3 3 $\bar{4}$
			1 2 2	8	2.125	2.126	3 1 3			1.713	4 1 $\bar{2}$
24	2.97	2.97	1 3 $\bar{1}$	3	2.101	2.102	3 2 $\bar{1}$	7	1.698	1.699	3 4 $\bar{1}$
5	2.90	2.90	0 2 3	3	2.082	2.089	0 1 5			1.698	3 0 3
28	2.85	2.85	2 2 $\bar{1}$			2.084	0 3 4	3	1.689	1.690	3 4 $\bar{2}$
			2 1 1			2.078	3 0 1	5	1.645	1.646	0 6 1
13	2.84	2.84	1 0 3			2.074	1 4 2	5	1.595	1.595	1 1 6
43	**2.83**	2.83	0 3 2	5	2.040	2.040	3 2 0	3	1.589	1.590	0 6 2
27	2.79	2.79	1 3 1	22	2.028	2.029	2 4 $\bar{1}$			1.589	4 2 0
		2.78	2 2 0	13	2.023	2.026	1 2 $\bar{5}$			1.588	3 3 $\bar{5}$
9	2.75	2.75	2 1 $\bar{3}$			2.024	2 3 2	5	1.583	1.583	1 6 1
38	**2.73**	2.73	1 1 3	5	1.994	1.995	3 2 $\bar{3}$	2	1.571	1.573	2 5 2
			2 2 $\bar{2}$	10	1.960	1.961	3 1 $\bar{4}$			1.570	0 3 6
27	2.67	2.67	0 0 4	10	1.918	1.919	3 2 1	4	1.537	1.537	2 2 5
10	2.65	2.65	1 1 $\bar{4}$								

*Intensities of the eight strongest diffraction maxima are in bold.

Table 4. Fractional atomic coordinates and equivalent isotropic displacement parameters or isotropic displacement parameter (for H) (Å^2).

| Site	x	y	z	U_{eq}/*U_{iso}
K1	0.83569(4)	0.77976(3)	0.03181(3)	0.02503(9)
S1	0.77920(4)	0.49878(2)	0.83958(2)	0.01453(8)
S2	0.27181(3)	0.88062(2)	0.85599(2)	0.01350(8)
Na1	0.30316(7)	0.56670(5)	0.87861(4)	0.02187(14)
Na2	0.40630(7)	0.81654(5)	0.15175(4)	0.02063(14)
O1	0.41776(13)	0.79390(9)	−0.06059(8)	0.0239(2)
O2	0.65292(13)	0.53007(9)	−0.07806(8)	0.0238(3)
O3	0.69272(13)	0.68159(10)	0.23334(8)	0.0261(3)
O4	0.95573(11)	0.58344(8)	0.86794(8)	0.0210(2)
O5	0.16436(13)	0.95943(9)	0.20573(8)	0.0245(3)
O6	0.10310(13)	0.89839(9)	−0.08331(8)	0.0232(2)
O7	0.17851(13)	0.64306(8)	0.16189(9)	0.0258(3)
O8	0.65071(14)	0.98685(8)	0.15899(9)	0.0261(3)
H1	0.010(4)	0.036(3)	0.138(3)	0.093(9)*

is $KNa_2H(SO_4)_2$, which requires K 14.05 wt.%, Na 16.53 wt.%, H 0.36 wt.%, S 23.05 wt.% and O 46.01 wt.%.

Powder X-ray diffraction

The best PXRD was obtained from the white globules from the surface of the sample. According to Rietveld quantitative calculations (using the *Topas V.6* program from Bruker-AXS with fundamental parameters used to define profile shapes) it contained 86 wt.% kristjánite with 7 wt.% kröhnkite, 4 wt.% thénardite and 3 wt.% aphthitalite (Fig. 3). We suppose that kröhnkite and thénardite, which are low temperature phases, are products of the decomposition of metastable metathénardite after sample collection and preparation. The numerical PXRD data for kristjánite are in Table 3. The unit cell parameters obtained through PXRD analysis are: $a = 6.9632(3)$ Å, $b = 9.9950(4)$ Å, $c = 11.0939(4)$ Å and $\beta = 105.638(2)°$.

Single crystal X-ray diffraction

The structure solution (using the program *SUPERFLIP* based on the charge-flipping method) revealed the positions of all non-
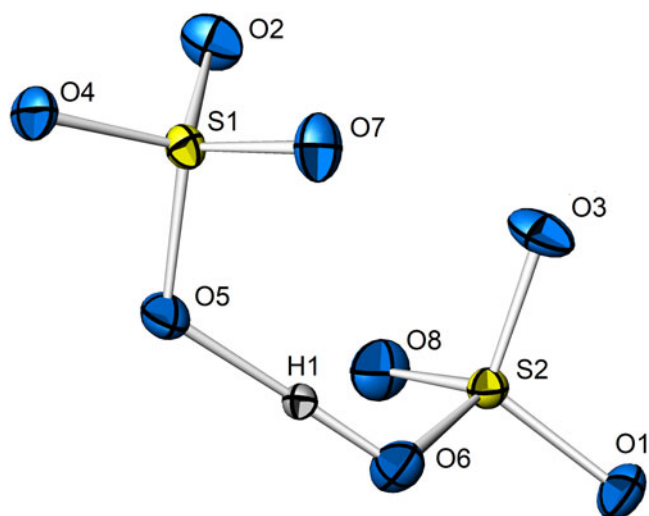
Figure 4. SO$_4$–H–SO$_4$ dimer. Atomic displacement ellipsoids drawn at the 50% probability level.

Table 5. The parameters of the coordination polyhedra calculated with the program *IVTON* (Balić Žunić and Vicković, 1996).

Cation	CN	<d> (Å)	BVS	Vp (Å^3)	vd	asp	ecc
K1	8	2.904	1.029	37.94(3)	0.1479	0.0368	0.0260
S1	4	1.477	5.981	1.647(2)	0.0012	0	0.0455
S2	4	1.473	6.020	1.637(2)	0.0008	0	0.0281
Na1	7	2.538	1.096	22.35(2)	0.1241	0.0795	0.0997
Na2	7	2.495	1.201	22.46(2)	0.0733	0.0741	0.0978

Notes: CN = coordination number; <d> = average bond length; BVS = bond valence sum, calculated using the exponential function of Brown and Altermatt (1985) with the parameters of Brese and O'Keeffe (1991); Vp = polyhedral volume; vd = volume distortion; asp = asphericity; ecc = eccentricity.

Table 6. Bond valence sums (BVS) for oxygen atoms. The oxygens that participate in the hydrogen bond are marked with a *.

Oxygen	BVS	Oxygen	BVS
O1	2.09	O5*	1.83
O2	2.10	O6*	2.26
O3	2.01	O7	1.99
O4	2.03	O8	2.05

hydrogen atoms. The position of hydrogen was obtained from a difference-Fourier map after one isotropic refinement of the model obtained by the structure solution. The last refinement was made with anisotropic displacement parameters for all atomic sites except the H site that was kept isotropic. Fractional atomic coordinates and displacement parameters are in Table 4, other details of structure refinement are in Table 1 and in the

Table 7. The bond lengths in the coordination polyhedra of cations, hydrogen bond excluded (can be found in the text).

Cation	Oxygen	Symmetry operator, oxygen	Bond length (Å)
K1	O6	1+x, y, z	2.790(1)
	O7	1+x, y, z	2.791(1)
	O1	x, y, z	2.816(1)
	O3	x, y, z	2.855(1)
	O2	x, y, z	2.915(1)
	O4	x, y, −1+z	2.945(1)
	O8	x, y, z	2.986(1)
	O5	1+x, y, z	3.131(1)
S1	O7	1−x, 1−y, 1−z	1.449(1)
	O4	x, y, z	1.455(1)
	O2	x, y, 1+z	1.462(1)
	O5	½+x, ½+1−y, ½+z	1.542(1)
S2	O8	1−x, 2−y, 1−z	1.457(1)
	O1	x, y, 1+z	1.461(1)
	O3	−½+x, ½+1−y, ½+z	1.462(1)
	O6	x, y, 1+z	1.514(1)
Na1	O2	1−x, 1−y, 1−z	2.358(1)
	O2	x, y, 1+z	2.380(1)
	O4	−1+x, y, z	2.396(1)
	O8	−½+x, ½+1−y, ½+z	2.440(1)
	O1	x, y, 1+z	2.441(1)
	O3	1−x, 1−y, 1−z	2.779(1)
	O3	−½+x, ½+1−y, ½+z	2.975(1)
Na2	O7	x, y, z	2.373(1)
	O3	x, y, z	2.375(1)
	O1	x, y, z	2.389(1)
	O8	x, y, z	2.393(1)
	O5	x, y, z	2.404(1)
	O4	−½+x, ½+1−y, ½+z	2.535(1)
	O6	x, y, z	2.993(1)
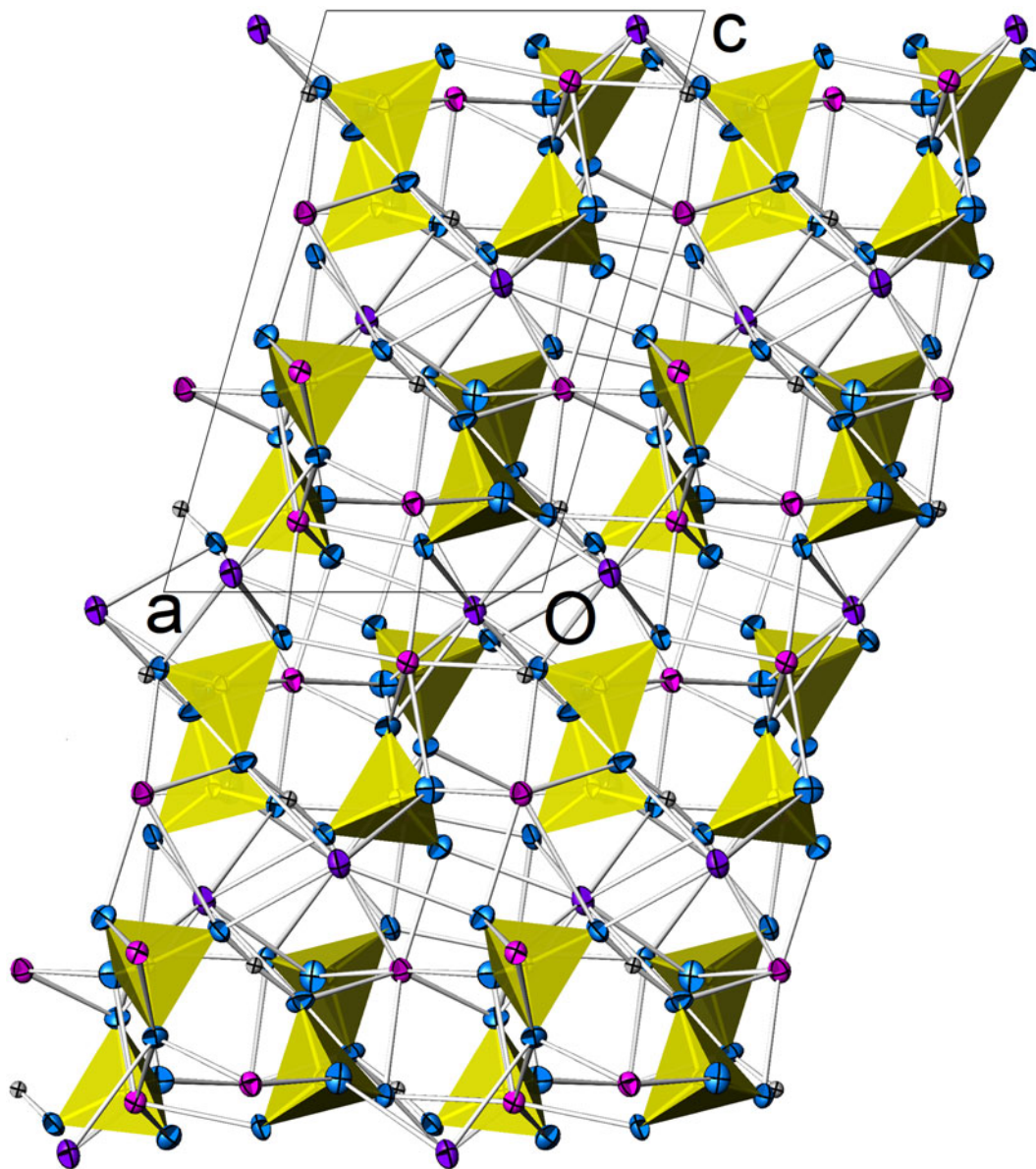
Figure 5. Crystal structure viewed along [010]. Dark violet = K atoms; magenta = Na atoms; yellow = S coordinations; blue = O atoms; grey = H atoms. One unit cell indicated. Atomic displacement ellipsoids drawn at the 50% probability level. Drawn using *Atoms* (www.shapesoftware.com).

crystallographic information file which has been deposited with the Principal Editor of *Mineralogical Magazine* and is available as Supplementary material (see below).

Sulfur atoms at the S1 and S2 sites are in tetrahedral coordination with three bond lengths of 1.46 Å and the fourth over 1.5 Å, which is the distance to the O atom (O5 in S1 or O6 in S2 coordination) involved in the hydrogen bond. H sits approximately on a straight line between them with distances 1.08(3) Å to O6 and 1.37(3) Å to O5, bond angle 176(3)°. Its total valence is 1.103 (from the bond valence sum). The SO_4–H–SO_4 dimer is shown on Fig. 4.

The potassium atom at the K1 site is in a distorted bis-disphenoid [coordination number (CN) 8 with bonds between 2.8 and 3.1 Å].

The sodium atoms at the Na1 and Na2 sites are in a CN 7 coordination best described as a split octahedron (Na1–O bond distances of 2.36 to 2.97 Å, Na2–O 2.37 to 2.99 Å).

Parameters of the coordination polyhedra are in Table 5, bond valence sums for oxygens are in Table 6 and all bond lengths in Table 7 except those in hydrogen bonds, described earlier.

The general appearance of the crystal structure is the following: sulfate tetrahedra and alkali atoms are arranged in (100) parallel layers; the layers are interconnected through K–O, Na–O and hydrogen bonds; and sulfate tetrahedra and alkali atoms also form two-sulfate-tetrahedra thick slabs parallel to (001), interconnected by K–O and Na–O bonds (Fig. 5).

The K1 and Na2 coordinations share two triangular faces and thus form relatively straight chains along [100]. Na2 coordinations additionally share a vertex with one of the neighbouring chains and thus connect chains in (010) wrapped sheets. SO_4 tetrahedra interconnect the sheets. Na1 coordinations that, taken alone, form [010] zigzag chains by mutually sharing two of the vertices, additionally connect K–Na2 sheets and SO_4 groups through vertex, edge and face sharing. The 3D arrangement of K,
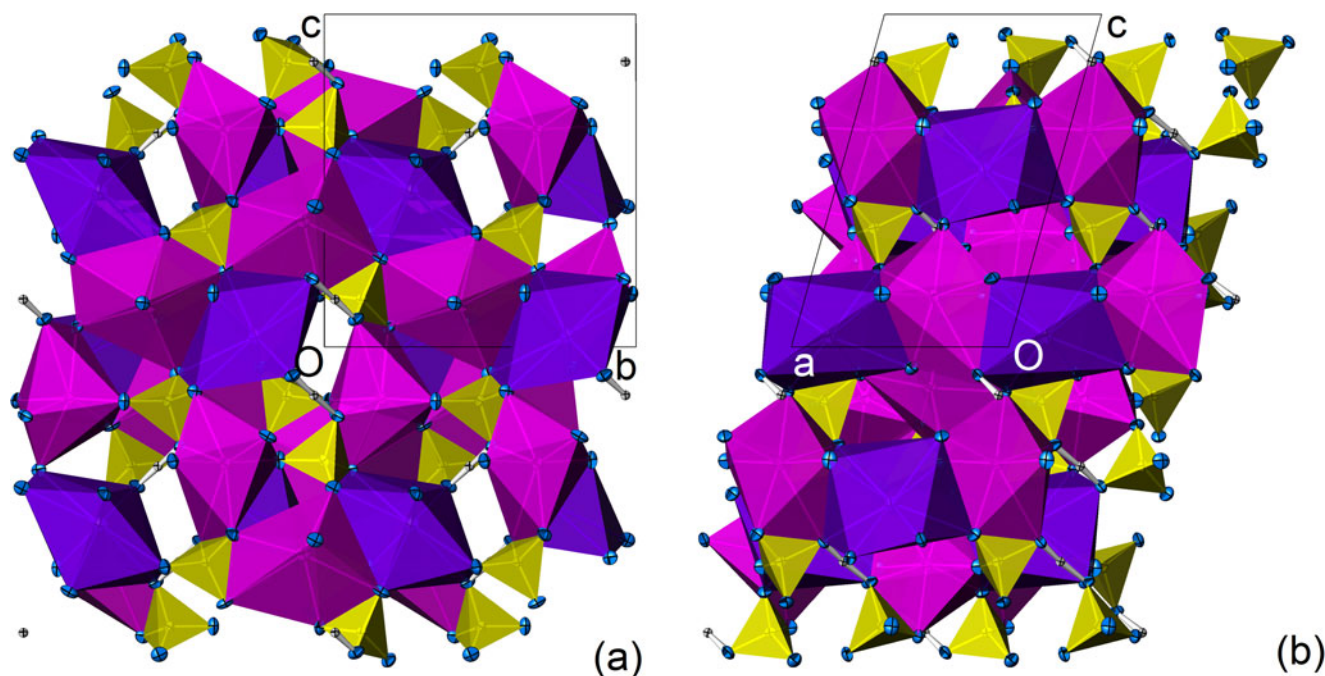
Figure 6. Crystal structure along [100]. Dark violet = K coordinations; magenta = Na coordinations; yellow = S coordinations; blue = O atoms; grey = H atoms. One unit cell indicated. Atomic displacement ellipsoids drawn at the 50% probability level.

Na and S coordination polyhedra forms a tight structure that features narrow [100] channels around inversion centres at $(x,0,0)$ and $(x,\frac{1}{2},\frac{1}{2})$, lined along the two narrow opposite sides by hydrogen bonds, and along the two longer sides by K and Na2 coordination polyhedra (Fig. 6).

Discussion

Kristjánite belongs to the mineral crystal structure group 7AD: anhydrous sulfates with only large cations (Strunz and Nickel, 2001). It moreover contains SO_4–H–SO_4 dimers where a very short hydrogen bond (<2.5 Å) connects two SO_4 groups. In letovicite, $(NH_4)_3H(SO_4)_2$ (Leclaire *et al.*, 1985), $Rb_3H(SO_4)_2$ (Swain and Row, 2007) and $K_3H(SO_4)_2$ (Oh *et al.*, 2019) the hydrogen is placed symmetrically, whereas in ivsite, $Na_3H(SO_4)_2$ (Joswig *et al.*, 1982) and kristjánite it is asymmetrical but with a small difference between the donor and acceptor bonds (1.16, 1.28 Å in $Na_3H(SO_4)_2$ and 1.08, 1.37 Å in kristjánite). The differences in crystal structures between kristjánite and other structures with (SO_4)–H–(SO_4) dimers are profound and it thus represents a novel structure type.

Conclusions

The new mineral, kristjánite, was found in association with metathénardite, belomarinaite, aphthitalite and ivsite at a high-temperature fumarole (≥600°C) on Fimmvörðuháls, south Iceland. Its chemical formula is $KNa_2H(SO_4)_2$. It is most probably formed through a reaction between lava minerals (sanidine and augite) and SO_2 and probably H_2O from volcanic gas. It has a unique crystal structure for which we could not find a closely related counterpart. It contains the SO_4–H–SO_4 dimers featuring an almost straight linear and very short (<2.5 Å) hydrogen bond also observed in some other $A_3H(SO_4)_2$ compounds (where A stands for large monovalent cations).

References

Balić-Žunić T., Nestola F. and Pamato M. (2023) Kristjánite, IMA 2022-131. CNMNC Newsletter 73; *Mineralogical Magazine*, **87**, https://doi.org/10.1180/mgm.2023.44.

Balić Žunić T. and Vicković I. (1996) IVTON – Program for the calculation of geometrical aspects of crystal structures and some crystal chemical applications. *Journal of Applied Crystallography*, **29**, 305–306.

Brese N.E. and O'Keeffe M. (1991) Bond-valence parameters for solids. *Acta Crystallographica*, **B47**, 192–197.

Brown I.D. and Altermatt D. (1985) Bond-valence parameters obtained from a systematic analysis of the Inorganic Crystal Structure Database. *Acta Crystallographica*, **B41**, 244–247.

Joswig W., Fuess H. and Ferraris G. (1982) Neutron diffraction study of the hydrogen bond in trisodium hydrogen bissulfate and a survey of very short O-H··O bonds. *Acta Crystallographica*, **B38**, 2798–2801.

Leclaire A., Ledesert M., Monier J., Daoud A. and Damak M. (1985) Structure du disulfate acide de triammonium. Une redetermination. Relations des chaines de liaisons hydrogene avec la morphologie et la conductivite eletrique. *Acta Crystallographica*, **B41**, 209–213.

Oh I-H., Sohn Y.J., Meven M. and Heger G. (2019) Symmetrical Hydrogen Bond in SO_4–H–SO_4 Dimer in $K_3H(SO_4)_2$ by Neutron Diffraction. *Journal of the Physical Society of Japan*, **88**, 124601.

Oszlányi G. and Sütő A. (2004) *Ab initio* structure solution by charge flipping. *Acta Crystallographica*, **A60**, 134–141.

Palatinus L. and Chapuis G. (2007) SUPERFLIP – a computer program for the solution of crystal structures by charge flipping in arbitrary dimensions. *Journal of Applied Crystallography*, **40**, 786–790.

Petříček V., Palatinus L., Plášil J. and Dušek M. (2023) Jana2020 – a new version of the crystallographic computing system Jana. *Zeitschrift für Kristallographie - Crystalline Materials*, **238**, 271–282. https://doi.org/10.1515/zkri-2023-0005

Rigaku Oxford Diffraction (2019) *CrysAlis PRO*. Rigaku Oxford Diffraction, Yarnton, England.

Strunz H. and Nickel E.H. (2001) *Strunz Mineralogical Tables. 9th edition*. Schwaizerbart, Stuttgart, Germany, 870 pp.

Swain D. and Row T.N.G. (2007) Analysis of phase transition pathways in $X_3H(SO_4)_2$ (X = Rb, NH_4, K, Na): Variable temperature single-crystal X-ray diffraction studies. *Inorganic Chemistry*, **46**, 4411–4421.