

Oskarssonite, AlF_3 , a new fumarolic mineral from Eldfell volcano, Heimaey, Iceland

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ABSTRACT

The new mineral oskarssonite (IMA2012-088), with ideal formula AlF_3 , was found in August 2009 at the surface of fumaroles on the Eldfell volcano, Heimaey Island, Iceland (GPS coordinates 63°25'58.9"N 20°14'50.3"W). It occurs as sub-micron-sized crystals forming a white powder in association with anhydrite, bassanite, gypsum, jarosite, anatase, hematite, opal, ralstonite, jakobssonite and meniaylovite. Chemical analyses by energy-dispersive spectrometry with a scanning electron-microscope produced the following mean elemental composition: Al, 31.70; F, 58.41; O, 9.22; total 99.33 wt.%. The empirical chemical formula is $\text{AlF}_{2.6}(\text{OH})_{0.5}$ which suggests partial substitution of F by OH. Oskarssonite is rhombohedral, space group $R\bar{3}c$, with $a_h = 4.9817(4)$ Å, $c = 12.387(1)$ Å, $V_{uc} = 266.23(5)$ Å³, $Z = 6$. The five strongest lines in the powder diffraction diagram [d in Å (I) (hkl)] are as follows: 3.54 (100) (012), 2.131 (13) (113), 1.771 (20) (024), 1.59 (15) (116), 1.574 (10) (122). Rietveld refinement confirms the identity of oskarssonite with the synthetic rhombohedral form of AlF_3 . Its structure can be described as a rhombohedral deformation of the idealized cubic perovskite-type octahedral framework of corner-sharing AlF_6 groups. Oskarssonite appears in the surface part of the fumaroles where fluorides are abundant. At greater depths (below 10 cm) sulfates dominate among the fumarolic minerals. In accordance with its occurrence, we surmise that oskarssonite forms in the later stages of the fumarolic activity in an environment poor in alkalies and Mg. Ralstonite ($\text{Na}_x\text{Mg}_{1-x}\text{Al}_{1-x}\text{F}_3(\text{H}_2\text{O})_y$), which, unlike oskarssonite, contains Na and Mg as important constituents, dominated in the first-formed fumaroles, but now, 41 years after the eruption of Eldfell, is only a minor phase. The new mineral is named after the Icelandic volcanologist Niels Oskarsson.

KEYWORDS: oskarssonite, new mineral, fumaroles, sublimates, fluoride, Eldfell, Iceland.

Introduction

IN the summer of 2009 our team undertook an expedition to the Eldfell volcano on the Heimaey

Island in the Vestmannaeyjar archipelago, Iceland, in order to investigate the fumaroles, which at that time had been active for 36 years after the eruption of the volcano. A previous investigation in the years closely following the eruption revealed rich mineralogy with several new minerals in fumaroles developed on hawaiite scoria host rock (Jakobsson *et al.*, 2008). The descriptions of three of them have already been published (Balić-Žunić *et al.*, 2009, 2012; Mitolo *et al.*, 2013) and more are under preparation. Our

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purpose was to record the long-term development of the fumaroles. Although the extent of the fumarolic field and the peak temperatures decreased with time, the existing fumaroles proved to be still active, with temperatures at a depth of ~50–60 cm exceeding 400°C. Surprisingly, we observed that the relative abundances of the previously recorded minerals changed significantly and even noted some new ones. This work describes one of those. It is the low-temperature form of AlF_3 which so far has not been fully characterized from natural occurrences. To the best of our knowledge, the only records of its probable occurrence are from Hekla after the 1970 eruption (Oskarsson, 1981) and from Mont Erebus, Antarctica (Rosenberg, 1988).

The new mineral and its name have been approved by the IMA Commission on New Minerals Nomenclature and Classification (CNMNC) (No. 2012-088). The mineral is named after Niels Oskarsson (1944–present), an Icelandic volcanologist, in acknowledgement of his pioneering work on Icelandic fumaroles.

The holotype is kept in the mineral collection of the Icelandic Institute of Natural History, Gardabaer, Iceland, under the sample number NI 24489. The sample is temporarily on loan at the Natural History Museum of Denmark in Copenhagen due to on-going analysis on several other new minerals. Other samples, in which oskarssonite has been identified, are kept in the mineral collection of the Natural History Museum of Denmark and have the following numbers: 2012.112, 2012.113 and 2012.114.

Mineral paragenesis and physical properties

The samples with the new mineral were collected on August 7, 2009 at GPS coordinates 63°25'58.9"N 20°14'50.3"W. Oskarssonite occurs as a primary phase in recently deposited encrustations at a measured temperature of 90°C. It forms a white to greyish-yellow fine powder close to the surface of fumarole vents exhausting through hawaiite scoria, mixed with anhydrite, bassanite, gypsum, jarosite, anatase, hematite, opal, ralstonite, jakobssonite, meniaylovite, and four other fluorides which also appear to be new minerals and are presently being investigated by our group. Macroscopically, it appears as a white powder (Fig. 1). Investigation by scanning electron microscopy (SEM) reveals massive aggregates of sub-micron crystals

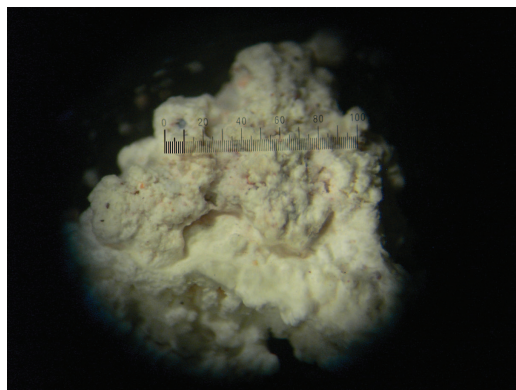


FIG. 1. A photograph of almost pure oskarssonite. The divisions on the scale correspond to 0.1 mm.

(Fig. 2). Oskarssonite and other fluoride minerals have been collected only from the upper 10 cm at this locality. At greater depths, a completely different association of fumarolic minerals occurs, dominated by sulfates.

Due to the minute size of the crystals, it was not possible to quantify the basic mineral properties like hardness, indices of refraction, etc. Synthetic AlF_3 is uniaxial positive with $n_o = 1.3765$ and $n_E = 1.3770$ (5893 Å) (Staritzky and Asprey, 1957). The mean index of refraction is reported to increase with the OH content (Rosenberg, 2006). The density, calculated from the X-ray diffraction (XRD) data and the empirical chemical formula is 3.184 g cm^{-3} .

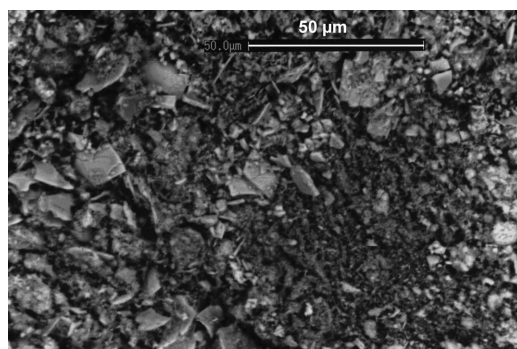


FIG. 2. SEM image of a sample which contains abundant oskarssonite. Oskarssonite forms the darker background of sub-micron crystals on which some brighter platy crystals of a Ca-Al fluoride-hydroxide hydrate are scattered.

Chemical composition

Five point chemical analyses were obtained by SEM energy-dispersive spectroscopy. The instrument used was an S 360 Cambridge SEM coupled with an Oxford-Link Ge ISIS energy-dispersive spectrometer equipped with a Super Atmosphere thin window as this allows better detection of light elements. An energy-dispersive spectrometer was chosen for quantitative analyses of the material in preference to a wavelength-dispersive spectrometer, due to the better specimen stability provided by lower beam currents. The investigated sample consisted of an aggregate of sub-micron crystals (Fig. 2). As we had to measure uneven surfaces, a “non-critical” working distance was utilized (Ruste, 1979; Acquafredda and Paglionico, 2004). The sample was sputtered with a 30 nm thick carbon film before analysis. The operating conditions were 15 kV accelerating potential, 500 pA probe current, 3 µm beam diameter, 2500 counts per second as the average count rate across the whole spectrum and typical counting times of 100 s. The X-ray intensities were converted to wt.% elements by *ZAF4/FLS* quantitative analysis software provided by Oxford-Link Analytical (UK). The procedure allows the sample peak heights and areas to be compared with standards, and it takes the relative contribution of the matrix and partial or complete overlaps of peaks produced by different elements into account. Analytical data are given in Table 1. All the measured amounts of O have been attributed to OH.

The empirical formula (based on Al = 1 a.p.f.u.) is: $\text{AlF}_{2.62}(\text{OH})_{0.49}$. The ideal pure fluoride, AlF_3 , requires: Al = 32.13 wt.%, F = 67.87 wt.%.

In a study of the system $\text{AlF}_3\text{--Al}_2\text{O}_3\text{--H}_2\text{O--HF}$ (Rosenberg, 2006) $\text{Al}(\text{F,OH})_3$, corresponding to oskarssonite, was synthesized as one of the phases at 450–700°C and 2 kbar. It showed compositions

reaching $\text{AlF}_{2.45}(\text{OH})_{0.55}$. This is quite close to the empirical composition obtained in the present investigation. Unfortunately, Rosenberg has chosen an unusual way of reporting the XRD data for this phase: as a difference in angle for the *024* reflection (in Rosenberg’s notation *220*, because he uses the rhombohedral assignment of crystal axes) and a non-specified reflection of the standard used. It is therefore not possible for us to compare our diffraction data with his to check which hydration grade they suggest. It also seems that his and our data are contradictory, because we obtained a larger d_{024} than observed for pure AlF_3 , whereas Rosenberg reports decreasing values with increasing hydration. In any case, the conclusion from the latter work is that the hydration grade increases with decreasing temperature in the $\text{AlF}_3\text{--Al}_2\text{O}_3\text{--SiO}_2\text{--H}_2\text{O}$ system, whereas it increases with increasing temperature in the $\text{AlF}_3\text{--Al}_2\text{O}_3\text{--H}_2\text{O--HF}$ system. The reason for the former behaviour is that HF, released through hydration, is consumed by the formation of gaseous SiF_4 (Rosenberg, 1992). As the fumarolic system at Eldfell hosts abundant SiO_2 , we can expect similar conditions, and the occurrence of oskarssonite at the coldest upper part of fumaroles suggests the maximum possible hydration, again in accordance with the chemical analysis.

Diffraction properties and crystal structure

X-ray powder diffraction data (Table 2, Fig. 3) were collected on a Bruker-AXS D8 diffractometer in Bragg-Brentano geometry with a primary-beam $\text{Ge}(111)$ monochromator, using $\text{CuK}\alpha_1$ radiation ($\lambda = 1.5406 \text{ \AA}$) and a fixed divergence slit. The diagram matches the data of synthetic rhombohedral AlF_3 (Hoppe and Kissel, 1984) well. The purest powder sample, also used for the Rietveld analysis, contained small amounts of opal, bassanite, anatase and hematite. The

TABLE 1. Analytical data for oskarssonite.

Constituent	Wt.%	Range	Standard deviation	Standard used
Al	31.70	29.71–33.49	1.75	Corundum
F	58.41	56.49–59.34	1.12	Synthetic LiF
O	9.22	7.94–10.57	1.12	Periclase
Total	99.33			

TABLE 2. X-ray powder diffraction data of oskarssonite compared with the experimental diagram of synthetic AlF_3 (Hoppe and Kissel, 1984). The five strongest oskarssonite and synthetic AlF_3 reflections are listed in bold. The reflections of anatase (*A*), bassanite (*B*), hematite (*H*) and still unidentified phase(s) (?) are listed in italics.

Oskarssonite				– Synthetic AlF_3^* –	
<i>I/I</i> ₀ (%)	<i>d</i> _{meas.} (Å)	<i>d</i> _{calc.} (Å)	<i>hkl</i>	<i>I/I</i> ₀ (%)	<i>d</i> _{meas.} (Å)
18	6.02		<i>B</i>		
14	3.67		<i>H</i>		
100	3.54	3.54	0 1 2 + <i>A</i>	100	3.52
9	3.08		?		
11	3.02		<i>B</i>		
15	2.70		<i>H</i>		
12	2.52	2.52	1 0 4 + <i>H</i>	5	2.51
7	2.43		<i>A</i>		
8	2.385		<i>A</i>		
7	2.338		<i>A</i>		
9	2.205		<i>H</i>		
13	2.131	2.133	1 1 3	20	2.119
8	2.066	2.065	0 0 6	30	2.074
8	2.044	2.037	2 0 2	10	2.018
7	2.009		?		
7	1.919		<i>B</i>		
9	1.897		<i>A</i>		
11	1.840		<i>H</i>		
10	1.789		?		
20	1.771	1.770	0 2 4	50	1.760
7	1.742		<i>B</i>		
7	1.707		<i>A</i>		
11	1.695		<i>H</i>		
8	1.672		<i>A</i>		
8	1.613	1.617	2 1 1	20	1.599
15	1.590	1.590	1 1 6	35	1.586
10	1.574	1.577	1 2 2	40	1.561
7	1.540		?		
9	1.486		<i>A</i> + <i>H</i>		
8	1.453		<i>H</i>		
7	1.440	1.443	2 1 4	25	1.432
		1.438	0 3 0	35	1.422
7	1.363	1.362	1 2 5		
6	1.342		<i>A</i>		
6	1.311		<i>H</i>		
9	1.259	1.258	2 0 8		
7	1.249	1.245	2 2 0		
7	1.192	1.191	1 3 1 + 1 0 10		
7	1.176	1.175	3 1 2		

* data from Guinier de Wolf camera.

former three are admixed with oskarssonite as fine powders, whereas the latter occurs as relatively large black metallic crystals up to mm size dispersed through the sample. For the data in Table 2, the positions and intensities of individual diffraction maxima were determined using the Bruker-AXS program *EVA*. Attribution of the

maxima and indexing were done in accordance with the results of the Rietveld refinement (program *Topas 4.1*, Bruker-AXS product). Unit-cell data are as follows: crystal system – rhombohedral (hexagonal setting); space group – $R\bar{3}c$, $a_h = 4.9817(4)$ Å, $c = 12.387(1)$ Å, $V = 266.23(5)$ Å³, $Z = 6$.

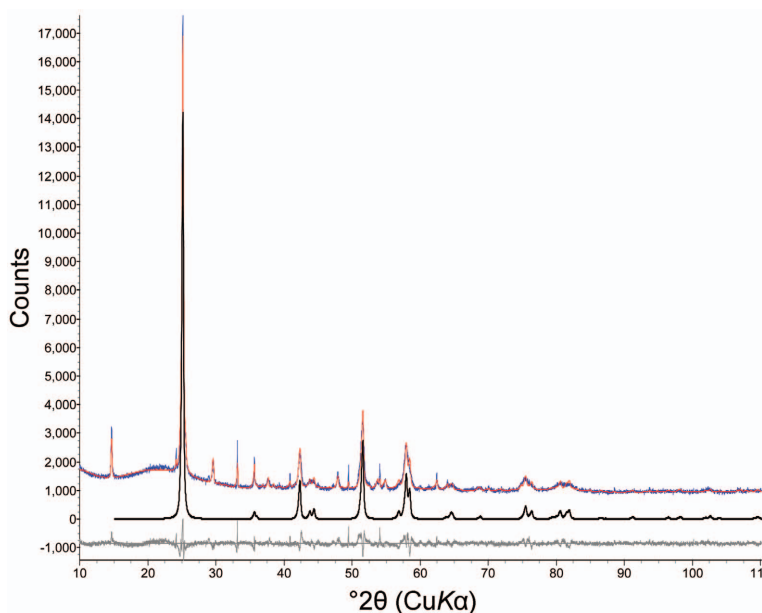


FIG. 3. Powder XRD pattern of the sample of oskarssonite with minor opal, bassanite, anatase and hematite. The experimental (blue) and the Rietveld fitted data (red) are shown together with the calculated theoretical diagram of oskarssonite (black) and the Rietveld difference curve (grey). The amorphous opal, visible as the very broad maximum at $\sim 22^\circ 2\theta$, was treated as background during the Rietveld refinement.

Rietveld refinement data are given in Table 3. Refinement confirms the identity of oskarssonite with the rhombohedral form of AlF_3 (Daniel *et al.*, 1990). The structure can be described as a

rhombohedral deformation of the idealized cubic perovskite-type octahedral framework of corner-sharing AlF_6 groups. The Al coordination octahedra are very regular and the deformation

TABLE 3. Rietveld refinement details.

2 θ range ($^\circ$)	10–110
Step scan ($^\circ 2\theta$)	0.02
Profile fitting function	fundamental parameters
Fixed divergence slit ($^\circ$)	0.45
Background and amorphous modelling	Chebyshev polynomials
R_p	3.96%
R_{wp}	5.37%
R_E	2.79%
Oskarssonite parameters	
R_B	1.32%
Space group	$R\bar{3}cH$
Unit-cell parameters	see Table 4.
Al site (multiplicity, Wyckoff)	6 b
F site (multiplicity, Wyckoff)	18 e
F x coordinate	0.4456(4)
F B (\AA^2)	2.79(9)
Al B (\AA^2)	2.06(8)
Average crystallite size (nm)	55(1)

may be described as their rigid-body rotations around one of the $\langle 111 \rangle$ directions of the idealized cubic structure, decreasing the Al–F–Al bond angles from 180° to around 160° . The unit cell contains only two independent sites, Al (Wyckoff position $6b$, coordinates 0, 0, 0) and F (Wyckoff position $18e$, coordinates x , 0, $1/4$). Results of the oskarssonite chemical analyses suggest partial substitution of OH for F in the structure. It has larger a_b , smaller c , and a larger unit-cell volume than the pure synthetic AlF_3 (Table 4) but the difference in bond length Al–F(OH) is marginal, being $1.797(3)$ Å in synthetic AlF_3 (Daniel *et al.*, 1990) compared to $1.791(1)$ Å in oskarssonite.

Relation to other minerals

Oskarssonite corresponds to the stable low-temperature form of AlF_3 . At 450°C it transforms reversibly to the cubic ReO_3 structure type (Daniel *et al.*, 1990). This is a displacive phase transition where both phases contain a perovskite-like framework of octahedra which in the low-temperature phase is rhombohedrally distorted from the ideal cubic arrangement. Through rotation of octahedra around one of the $\langle 111 \rangle$ sets of cubic directions, it becomes the c axis in the hexagonal setting of oskarssonite. Several other metastable synthetic forms of AlF_3 have been reported and structurally confirmed. The orthorhombic $Cmcm$ polymorph with a slightly distorted hexagonal tungsten bronze (HTB) structure type (Le Bail *et al.*, 1988); the tetragonal $P4/nmm$ modification with a complex octahedral framework (Le Bail *et al.*, 1992; Herron *et al.*, 1995); plus a cubic $Fd3m$ form with a pyrochlore-type framework of octahedra (Herron *et al.*, 1995) are known. Furthermore, a possible tetragonal $P4/mbm$ poly-

morph was discovered by Herron *et al.* (1995), having a tetragonal tungsten bronze (TTB) framework (Le Bail and Calvayrac, 2006) but still lacking a completely satisfactory structure confirmation. All metastable synthetic forms crystallize under specific conditions and from specific precursors, transforming irreversibly to the high- or low-temperature polymorphs with perovskite-related octahedral frameworks on heating (Herron *et al.*, 1995). It is therefore not very likely that they will be found as natural products. Also, due to the fast reversible transition, it is not expected that the stable high-temperature cubic form will be found as a mineral and therefore it seems that oskarssonite is the only probable mineral form of AlF_3 , at least under a low-pressure regime.

The relationship between oskarssonite and ralstonite is quite interesting. The crystal structure of ralstonite is based on the pyrochlore-type framework of AlF_6 octahedra where some Al is substituted by Mg, the valence-balance being compensated by additional Na atoms in the framework cavities and additional H_2O molecules that coordinate Na (Effenberger and Kluger, 1984). Thus, the ralstonite octahedral framework seems to be stabilized by other cations. Although $\text{Al}(\text{OH},\text{F})_3$ phases and even practically pure AlF_3 with the latter framework have been synthesized (Herron *et al.*, 1995), the pure unsubstituted form seems to be metastable (see above). One can expect that ralstonite forms readily in fumarolic environments rich in additional elements (Mg, Na) and possibly also at a higher H_2O activity, whereas the formation of oskarssonite needs a different chemistry with Al dominant over other possible constituents of ralstonite. This is supported by the geological evidence at Eldfell where ralstonite is dominant in the initial fumarolic stage and in the “fresh fumaroles”

TABLE 4. Comparison of the unit-cell parameters of oskarssonite and synthetic AlF_3 .

	Oskarssonite	Synthetic AlF_3	
	This paper	Hoppe and Kissel (1984)	Daniel <i>et al.</i> (1990)
Space group	$R\bar{3}c$	$R\bar{3}$	$R\bar{3}c$
a (Å)	4.9817(4)	4.9254(7)	4.9305(6)
c (Å)	12.387(1)	12.4472(52)	12.4462(7)
V (Å ³)	266.23(5)	261.51(8)	262.03(4)
Al–F	1.791(1)	1.794(2), 1.801(4)*	1.797(3)

* inaccurate due to incorrect space group.

(Jakobsson *et al.*, 2008). During recent sampling of old fumaroles ralstonite occurred as a minor fluoride phase whereas a conspicuous concentration of oskarssonite was found at the very top of the fumaroles. The presence of opal, cristobalite, anatase and corundum in present-day fumaroles also suggests a 'simple' chemistry, represented by phases which can be regarded as final products after leaching of the majority of Na, K, Mg, Ca and Fe in the host rock.

Among the known hydrated phases of AlF_3 there is so far only one accepted mineral, rosenbergite, $\text{AlF}_{2.5}(\text{OH})_{0.5}(\text{H}_2\text{O})_3$. However, another hydrated form with ralstonite structural characteristics has been reported in a number of papers (Rosenberg, 2006 and references therein) but so far not approved as a valid mineral species. Its composition is given as $\text{AlF}_{3-y}(\text{OH})_y(\text{H}_2\text{O})_n$ with y varying between 1.3 and 1.9 and n between 0.9 and 1 according to the synthesis study of Rosenberg (2006). The latter phase in association with ralstonite and jakobssonite has also been indicated by an XRD diagram of a fumarolic sample formed after the Hekla 1992 eruption. This suspected new mineral was labelled HS by Jakobsson *et al.* (2008), and was later given the CNMNC number UM2008-II-?[10] (Smith and Nickel, 2007). It has so far not been observed in samples collected on Eldfell. Rosenbergite, however, is observed in the samples collected on Eldfell in 2009 at the very top of the fumaroles (determined by XRD). It forms rare, small stalagmitic aggregates, which suggests it forms due to a reaction with atmospheric humidity.

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