

Möhnite, $(\text{NH}_4)\text{K}_2\text{Na}(\text{SO}_4)_2$, a new guano mineral from Pabellón de Pica, Chile

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Abstract The new mineral möhnite, ideally $(\text{NH}_4)\text{K}_2\text{Na}(\text{SO}_4)_2$, the ammonium analogue of apthitalite, is found in a guano deposit on the Pabellón de Pica mountain, near Chanabaya, Iquique Province, Tarapacá Region, Chile. It is associated with salammoniac, halite, joanneumite, natroxalate, nitratine, chanabayaita, and a clay mineral. Möhnite occurs as random aggregates and clusters of brown imperfect bipyramidal or spindle-shaped crystals. The mineral is brittle, with Mohs' hardness of 3; D_{meas} is 2.4(1) g/cm³ and D_{calc} is 2.461 g/cm³. The IR spectrum shows the presence of NH_4^+ cations (the bands at 1431, 3076 and 3240 cm⁻¹). Möhnite is almost isotropic, optically neutral; $\varepsilon = \omega = 1.505(2)$. The chemical composition (electron-microprobe data, N determined by gas chromatography of products of ignition, H calculated by stoichiometry, wt%) is: $(\text{NH}_4)_2\text{O}$ 7.99, Na_2O 9.49, K_2O 32.34, SO_3 51.32, total 101.14. The empirical formula is $(\text{NH}_4)_{0.95}\text{Na}_{0.95}\text{K}_{2.14}\text{S}_{1.99}\text{O}_8$. The crystal structure was solved and refined to $R=0.049$ based on 241 unique reflections with $I > 2\sigma(I)$. Möhnite is trigonal, space group $P\bar{3}$

$m1$, $a=5.7402(3)$ Å, $c=7.435(1)$ Å, $V=212.16(4)$ Å³, $Z=1$. The strongest reflections of the powder X-ray diffraction pattern [d , Å (I ,%) (hkl)] are: 4.955 (27) (100), 4.122 (37) (101, 011), 3.708 (29) (002), 2.969 (74) (102, 012), 2.861 (100) (110), 2.474 (20) (003), 2.060 (33) (022). The mineral is named in honour of the German amateur mineralogist Gerhard Möhn (born 1959).

Introduction

Among multiple guano occurrences of the Tarapacá region, Chile, the deposit located on the lower part of the steep northern slope of the Pabellón de Pica mountain, 1.5 km south of the village of Chanabaya, Iquique Province (20°55'S, 70°08'W), is of particular interest from the viewpoint of the behaviour of copper in lithospheric processes. Pabellón de Pica is the type locality of several N-bearing and organic copper minerals: ammineite, $\text{CuCl}_2(\text{NH}_3)_2$ (Bojar et al. 2010), joanneumite, $\text{Cu}(\text{C}_3\text{N}_3\text{O}_3\text{H}_2)_2(\text{NH}_3)_2$ (Bojar and Walter 2012), shilovite, $\text{Cu}(\text{NH}_3)_4(\text{NO}_3)_2$ (Chukanov et al. 2015a), chanabayaita, $\text{Cu}_4(\text{N}_3\text{C}_2\text{H}_2)_4(\text{NH}_3)_4\text{Cl}_2(\text{Cl},\text{OH})_2 \cdot \text{H}_2\text{O}$ (Chukanov et al. 2015b), and antipinite, $\text{KNa}_3\text{Cu}_2(\text{C}_2\text{O}_4)_4$ (Chukanov et al. 2015c). Altered accessory chalcocopyrite of the host rock (gabbro) served as a source of Cu for these minerals.

The ammonium potassium sodium sulphate möhnite described in this paper is the first copper-free new mineral species discovered at this locality. The mineral is named in honour of the prominent German amateur mineralogist and mineral collector Gerhard Möhn (born 1959). He has provided many samples from Chilean and German localities to professional mineralogists for studies after his own very careful investigations (mainly chemical tests). G. Möhn is a coauthor of descriptions of several new mineral species (vendidaite,

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lahnsteinite, okruschite, chanabayaite, shilovite and antipinite), as well as of popular articles on the mineralogy of the abandoned Friedrichsseen mine, Germany. The specimens containing möhnite were collected by him.

The mineral and its name were approved by the IMA CNMNC (IMA2014-101). The type specimen is deposited in the collection of the Fersman Mineralogical Museum of the Russian Academy of Sciences, Moscow, Russia, with the registration number 4638/1.

Occurrence, general appearance and physical properties

Numerous bird guano deposits are distributed along the northern coast of Chile. Some details of the geological setting and the history of guano deposits development in the Tarapacá region are published elsewhere (Ericksen 1981; Pankhurst and Herve 2007; Appleton and Nothold 2002; Bojar et al. 2010).

Möhnite is a supergene mineral formed at the contact between a deeply altered guano deposit and chalcopyrite-bearing gabbro. Guano was the source of chemically fixed nitrogen. Both guano and oxidized chalcopyrite could be sources of sulfate ions. Minerals associated with möhnite are salammoniac, halite, joanneumite, natroxalate, nitratine, chanabayaite, and a clay mineral. Möhnite crystals, as well as aggregates of partially altered joanneumite, overgrow crystalline crusts of salammoniac and encrust cavities in salammoniac aggregates. Halite forms late, white, fine-grained aggregates on the surfaces of salammoniac crusts. Natroxalate occurs as light brown, fibrous aggregates intergrown with salammoniac. Nitratine is present as an interspersed trace admixture. Chanabayaite occurs in association with möhnite sporadically.

The new mineral forms random aggregates and clusters (up to 1 mm across), as well as crusts consisting of imperfect bipyramidal, light brown to brown spindle-shaped crystals up to $0.07 \times 0.07 \times 0.15$ mm in size (Fig. 1). Möhnite is brittle, with Mohs' hardness of 3. Cleavage is not observed. Density measured by flotation in heavy liquids (mixtures of bromoform with acetone) is $2.4(1)$ g/cm³. Density calculated from the empirical formula is 2.461 g/cm³. Möhnite is non-fluorescent under the UV radiation.

Under plain polarized light möhnite is light brownish-yellow and non-pleochroic. In crossed nicols it looks isotropic. The mineral is optically neutral, with $\varepsilon = \omega = 1.505(2)$.

Infrared spectroscopy

Powdered sample of the mineral was mixed with KBr, pelletized, and analyzed using an ALPHA FTIR spectrometer



Fig. 1 Aggregate of slightly split, spindel-like möhnite crystals on salammoniac. Field width 0.4 mm. Photograph: M. Burkard

(Bruker Optics) at the resolution of 4 cm⁻¹ (average of 16 scans). An IR spectrum of an analogous pellet of pure KBr was used as a reference.

The infrared spectrum of möhnite (Fig. 2) is similar to that of aphthitalite (Chukanov 2014), but contains additional bands due to the presence of NH₄⁺ ions. Absorption bands and their assignments are (cm⁻¹; s – strong band, m – medium-intensity band; w – weak band): 3240 m, 3076 m [$\nu_3(F_2)$ asymmetric N–H stretching vibrations of NH₄⁺ cations forming rather strong hydrogen bonds], 2150w, 2078w (overtone and combination mode corresponding to S–O stretching vibrations), 1431 [$\nu_4(F_2)$ – bending vibrations of NH₄⁺ cations], 1165 (shoulder, *i. e.* inflection point indicating a poor-resolved band) and 1111s [$\nu_3(F_2)$ asymmetric

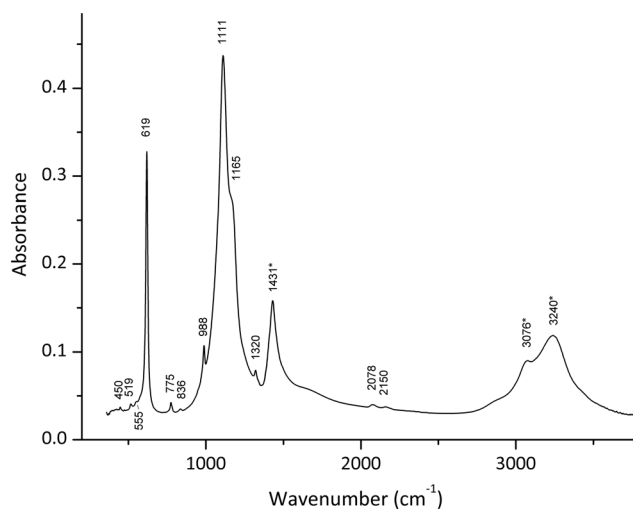


Fig. 2 Powder IR spectrum of möhnite

stretching vibrations of SO_4^{2-} ions], 988 m [$\nu_1(A_1)$ symmetric stretching vibrations of SO_4^{2-} ions], 836w, 775w (NH_4^+ librational modes), 619 s [$\nu_4(F_2)$ – bending vibrations of SO_4^{2-} ions], 555w, 519w, 450w (possibly, symmetric bending $\nu_2(E)$ -mode of SO_4 and/or overtones of Na–O and K–O stretching modes). The band assignment was made in accordance with Nakamoto (2008, 2009).

Assignment of the weak band at 1320 cm^{-1} is ambiguous. One can suppose that it corresponds to a minor amount of NH_4^+ cations that are present at the K site (see below) and do not form strong hydrogen bonds, or to traces of NO_3^- anions substituting SO_4^{2-} .

A rather low intensity of the non-degenerate $\nu_1(A_1)$ band at 988 cm^{-1} , as well as low intensity of $\nu_2(E)$ and moderate splitting of the degenerate $\nu_3(F_2)$ band ($1165+1111\text{ cm}^{-1}$) reflect a low degree of distortion of the SO_4 tetrahedron (see description of the crystal structure).

Bands in the range from 1500 to 1700 cm^{-1} that could belong to H_2O molecules are absent in the IR spectrum of möhnite.

Chemical data

Five chemical analyses for Na, K and S were carried out using a digital scanning electron microscope Tescan VEGA-II XMU with energy-dispersive spectrometer INCA Energy 450, at the accelerating voltage of 20 kV, beam current of 600 pA and beam diameter 0.16–0.18 μm . The beam was rasterized on an area $8\text{ }\mu\text{m}\times 8\text{ }\mu\text{m}$ avoiding damage of an unstable mineral. Contents of other elements with atomic numbers less than 8 are below detection limits. Gas chromatography of products of ignition at $1200\text{ }^\circ\text{C}$ was used to determine N. The H content could not be measured because of insufficient amount of substance available. Analytical data are given in Table 1.

The empirical formula (based on 8 O *apfu*) is $(\text{NH}_4)_{0.95}\text{Na}_{0.95}\text{K}_{2.14}\text{S}_{1.99}\text{O}_8$. The idealized formula is $(\text{NH}_4)\text{K}_2\text{Na}(\text{SO}_4)_2$, which requires $(\text{NH}_4)_2\text{O}$ 8.36, Na_2O 9.95, K_2O 30.26, SO_3 51.43, total 100.00 wt%.

Möhnite dissolves in water but is stable in dry air. Heating in a sealed tube gives a white sublimate, possibly ammonium

Table 1 Chemical composition of möhnite

Constituent	Mean content, wt%	Range, wt%	Standard deviation, wt%	Probe Standard
$(\text{NH}_4)_2\text{O}^a$	7.99			
Na_2O	9.49	8.98–9.94	0.38	Albite
K_2O	32.34	31.89–32.79	0.39	Microcline
SO_3	51.32	50.76–51.92	0.41	BaSO_4
Total	101.14			

^a 7.99 wt% $(\text{NH}_4)_2\text{O}$ is calculated from 4.3 wt% N determined by gas chromatography

Table 2 Powder X-ray diffraction data for möhnite

I_{obs}	d_{obs}	I_{calc}^*	d_{calc}^{**}	$h\ k\ l$
10	7.44	12	7.435	001
27	4.955	29	4.971	100
37	4.122	9, 16	4.132, 4.132	101, 011
29	3.708	31	3.717	002
74	2.969	53, 8	2.977, 2.977	102, 012
100	2.861	100	2.870	110
20	2.474	14	2.478	003
9	2.350	4, 8	2.357, 2.357	021, 201
2	2.265	4	2.272	112
4	2.212	6	2.218	103
33	2.060	46	2.066	022
3	1.871	4	1.876	113
3	1.853	3	1.859	004
1	1.819	0.5	1.822	211
3	1.752	4, 2	1.755, 1.755	203, 023
8	1.675	6	1.677	212
8	1.655	12	1.657	300
1	1.616	0.5, 0.5	1.617, 1.617	301, 031
7	1.559	8	1.560	114
2	1.512	1	1.513	032
5	1.487	3, 4	1.489, 1.489	204, 024
11	1.433	14	1.435	220
3	1.423	2, 1	1.425, 1.425	105, 015
1	1.377	0.5, 0.5	1.379, 1.379	130, 310
3	1.337	3	1.339	222
2	1.320	1, 1	1.321, 1.321	124, 214
6	1.291	4, 4	1.293, 1.293	132, 312
3	1.274	3, 0.5	1.276, 1.276	025, 205
2	1.240	0.5, 3	1.243, 1.242	400, 223
3	1.236	3, 1	1.237, 1.237	034, 304
2	1.202	2	1.202	106
3	1.177	4	1.179	402
2	1.164	2, 2	1.166, 1.166	125, 215
2	1.134	3	1.136	224
2	1.107	0.5, 2	1.109, 1.109	206, 026

*For the calculated pattern, only reflections with intensities ≥ 0.5 are given

**For the unit cell parameters calculated from single-crystal data

Table 3 Crystal data, data collection information and structure refinement details for möhnlite

Crystal-chemical formula	$[(\text{NH}_4)_{0.85}\text{K}_{0.15}]\text{K}_2\text{Na}(\text{SO}_4)_2$
a (Å)	5.7402(3)
c (Å)	7.435(1)
V (Å ³)	212.16(4)
Crystal system; sp.gr.	Trigonal, $Pm\bar{1}$
Z	1
μ (mm ⁻¹)	1.751
F (000)	154
Crystal size (mm)	0.11×0.13×0.15
Diffractometer	SMART APEX2 CCD
Radiation; λ	MoK α ; 0.71069
Index ranges	-8<h<8, -8<k<8, -10<l<10
R_{int} (%)	5.72
Number of reflections collected	3612
Number of unique reflections _i	283
Number of observed reflections with $F_{\text{obs}} > 4\sigma F$	241
Refinement method	Full-matrix least squares on F Weight scheme: $1/(\sigma^2 F + 0.004225 F^2)$
Number of refinement parameters	20
Final R_1 index (%) for $[F_{\text{obs}} > 4\sigma F]$	4.92
wR_2 index for all data	8.33
GOF	1.07
$\Delta\rho_{\text{min}}/\Delta\rho_{\text{max}}$ (e Å ⁻³)	-0.52/0.87
Program	Jana2006; AREN

sulfate. Tests with hexanitrocobaltate(III) demonstrate the presence of K⁺ and/or NH₄⁺. Tests with an acid solution of hexacyanoferrate(II) show the absence of Cu²⁺ and Fe³⁺. A test with HNO₃/AgNO₃ shows the absence of Cl. No gas evolution is observed in 20 % HCl, showing the absence of CO₃²⁻ groups. Möhnlite gives a positive reaction for SO₄²⁻ with HCl/BaCl₂ solution (white precipitate).

The Gladstone-Dale compatibility index is: $1 - (\text{Kp}/\text{Kc}) = 0.016$, superior (Mandarino 1981).

Table 4 Fractional atom coordinates, site multiplicities (Q) and equivalent displacement parameters (U_{eq} , Å²) for möhnlite

Site	x/a	y/b	z/c	Q	U_{eq}
A ^a	0	0	0	1	0.0108(13)
Na	0	0	0.5	1	0.0135(10)
K	0.3333	0.6667	0.6900(3)	2	0.0179(5)
S	0.6667	0.3333	0.2418(2)	2	0.0092(5)
O1	0.6667	0.3333	0.0443(11)	2	0.045(2)
O2	0.8070(7)	0.1930(3)	0.3080(5)	6	0.0261(12)

^a The refined composition of the A site is (NH₄)_{0.85}K_{0.15}

Table 5 Anisotropic displacement parameters (Å²) for möhnlite

Site	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
A	0.015(2)	0.015(1)	0.002(2)	0.0076(8)	0	0
Na	0.012(1)	0.012(1)	0.016(2)	0.0061(6)	0	0
K	0.0161(6)	0.0161(6)	0.0214(9)	0.0081(3)	0	0
S	0.0073(6)	0.0073(6)	0.0130(9)	0.0036(3)	0	0
O1	0.014(2)	0.019(1)	0.044(2)	0.0069(7)	0.015(1)	0.008(1)
O2	0.059(3)	0.059(3)	0.016(3)	0.0295(15)	0	0

X-ray diffraction data and crystal structure

Powder X-ray diffraction data (Table 2) were collected using a Rigaku R-AXIS Rapid II diffractometer (curved image plate detector, Debye-Scherrer geometry, $d=127.4$ mm, CoK α , 40 kV, 15 mA). The unit cell dimensions refined from the powder data are: $a=5.732(1)$, $c=7.425(1)$ Å, $V=211.3(1)$ Å³.

The single-crystal X-ray data were collected at room temperature with a Bruker SMART APEX2 CCD diffractometer with graphite-monochromatized MoK α radiation ($\lambda=0.71073$ Å) and a CCD detector using the ω scanning mode. The following hexagonal (trigonal) unit-cell parameters have been obtained: $a=5.7402(3)$ Å, $c=7.435(1)$ Å, $V=212.16(4)$ Å³, $Z=1$; space group $P\bar{3}m1$. A total of 3612 reflections within the sphere limited by $\theta=30.63^\circ$ were measured. Raw data were integrated by using the program SAINT and then scaled, merged, and corrected for Lorentz-polarization effects using the SADABS package. The experimental details of the data collection and refinement results are listed in Table 3. The initial model for the structure refinement was based on coordinates of the apthitalite structure (Okada and Ossaka 1980) and refined up to $R=4.92\%$ [$283 I > 2\sigma(I)$] in anisotropic approximation using the crystallographic program Jana2006 (Petricek et al. 2006). The structural data on möhnlite are given in Tables 4, 5 and 6.

Möhnlite belongs to the apthitalite group and is isostructural with apthitalite, K₃Na(SO₄)₂ (Moore 1981; Egorov-Tismenko et al. 1984; Lazoryak 1996; Nikolova and Kostov-Kytin 2013). The fundamental building block of the apthitalite structure is a layer consisting of vertex-connected NaO₆ octahedra and SO₄ tetrahedra (Fig. 3). In the möhnlite

Table 6 Interatomic distances (Å) in the crystal structure of möhnlite

K-O2	2.634(9)	Na-O1	2.392(4)x6
K-O1	2.882(2)x6	Mean	<2.392>
K-O1	3.164(4)x3	N-O1	2.988(4)x6
Mean	<2.942>	N-O2	3.330(1)x6
S-O2	1.468(8)	Mean	<3.159>
S-O1	1.480(4)x3		
Mean	<1.477>		

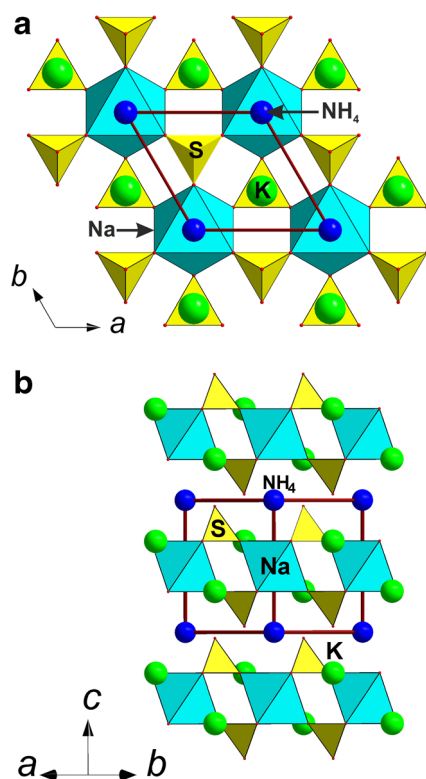


Fig. 3 The crystal structure of möhnite: (001) (a) and (110) (b) projections. The unit cell is outlined

structure Na forms regular octahedron with distances Na–O = 2.39 Å. The larger cations (K^+ and NH_4^+) are ordered in two sites, one of which (with multiplicity $Q=2$) is attached to the layer; the other (with $Q=1$) is situated between the layers. They form 10-vertex and 12-vertex polyhedra with the mean cation-oxygen distances 2.94 and 3.16 Å, respectively.

The main structural and chemical feature of möhnite is the predominance of NH_4^+ (with minor K) in the A site. Thus, it is the first NH_4^+ -dominant member of the aphthitalite group. The crystal chemical formula of möhnite obtained as a result of the crystal structure refinement is $[(NH_4)_{0.85}K_{0.15}]K_2Na(SO_4)_2$, where square brackets denote the composition of two sites between the layers. The idealized formula of möhnite can be written as $(NH_4)K_2Na(SO_4)_2$.

Discussion

In aphthitalite, both sites which are analogous to the sites A and K in möhnite are predominantly occupied by K, and the mean K–O distances in corresponding K-centered polyhedra are 2.900 and 3.098 Å, respectively (Okada and Ossaka 1980). In möhnite, ammonium cations are mainly concentrated in the larger polyhedron corresponding to the A site with the multiplicity $Q=1$. As indicated by the IR spectrum, the ions NH_4^+ form hydrogen bonds. Hydrogen bonding seems to be an additional cause for the ordering of K^+ and NH_4^+ in

Table 7 Comparative data for möhnite and aphthitalite

Mineral	Möhnite	Aphthitalite
Formula	$(NH_4)K_2Na(SO_4)_2$	$K_3Na(SO_4)_2 = KK_2Na(SO_4)_2$
Crystal system	Trigonal	Trigonal
Space group	$P\bar{3}m1$	$P\bar{3}m1$
Unit cell data		
a , Å	5.7402	5.66–5.68
c , Å	7.435	7.30–7.33
V , Å ³	212.16	202.5–204.7
Z	1	1
Strongest reflections of the powder X-ray diffraction pattern: d , Å (I , %)	4.955 (27) 4.122 (37) 3.708 (29) 2.969 (74) 2.861 (100) 2.474 (20) 2.060 (33)	4.09 (30) 3.67 (20) 2.940 (75) 2.839 (100) 2.443 (16) 2.330 (14) 2.042 (45)
Density, g cm ⁻³	2.461 (calc.) 2.4 (meas.)	2.697 (calc.) 2.66–2.71 (meas.)
Optical data		
ω	1.505	1.487–1.494
ϵ	1.505	1.492–1.503
Optical sign	Not determined	(+)
Strong and characteristic bands in the IR spectrum	3240 3076 1431 1165 1111 988 619	1188 1103 990 617
References	This work	Palache et al. (1951); Moore (1973); Okada and Ossaka (1980); Feklichev (1989).

möhnite. A similar situation takes place in another anhydrous sulfate therasiaite, $[(NH_4)_{2.25}K_{0.75}]KNa_2Fe_2(SO_4)_3Cl_5$ (Demartin et al. 2014) in which potassium and ammonium cations are substantially ordered and NH_4^+ forms hydrogen bonds similar to those in möhnite.

A mineral with 5.68 wt% $(NH_4)_2O$ and 33.87 wt% K_2O from the Guañape Islands, Peru, was described by Frondel (1950) as an ammonium variety of aphthitalite. The crystal structure of this mineral was not investigated, but according to chemical data it may be an ammonium-deficient variety of möhnite. Comparative data for möhnite and aphthitalite are given in Table 7.

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