Schöllhornite, Na_{0.3}(H_2O)_{1}[CrS_2], a new mineral in the Norton County enstatite achondrite

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Abstract

Schöllhornite, ideally Na_{0.3}(H_2O)_{1}[CrS_2], occurs in the Norton County enstatite achondrite as thin bands a few µm wide in caswellsilverite, NaCrS_2, and as individual grains up to 250 µm in size, adjacent to caswellsilverite. Other associated phases are daubreelite, titanoan troilite, ferromagnesian alabandite, oldhamite, kamacite and perryite. In reflected light, schöllhornite is gray in air and bluish gray in oil. It has distinct reflection pleochroism: in air, brownish gray (brighter) and bluish gray (darker); in oil, gray with an extremely faint yellowish tint (brighter) and bluish gray (darker). Anisotropism is strong and the phase is uniaxial (-). Reflectance at λ = 546 nm is R_δ = 19.3 and R_p = 16.0%. Electron microprobe and ion microprobe analyses show the mineral to be (ideally) Na_{0.3}(H_2O)_{1}[CrS_2], with only minor Ti and Mn (avg. 0.17 wt.% each). Synthetic and natural schöllhornite have similar X-ray powder diffraction patterns with the following lines (in Å): 8.85 (vs) (003); 4.43 (w) (006); 2.80 (m) (102); 2.53 (m) (105); 2.21 (vw) (0012); 1.67 (m) (110). Because of the small amount of natural schöllhornite available for X-ray studies, the weak (006) and very, very weak (0012) lines could not be reliably measured. Lattice constants are a = 3.32 Å and c = 26.6 Å, based on a hexagonal setting. Possible space groups are R3m, R3m and R32. The measured density of synthetic schöllhornite is 2.70 g/cm³ and the calculated density is 2.74 g/cm³. Experimental work by us and others shows that sodium chromium sulfide hydrate (bilayered molecular water form) is readily produced by the partial hydration of caswellsilverite (NaCrS_2) in aqueous solution at room temperature. The monolayered molecular water form, schöllhornite, forms by subsequent partial dehydration of the bilayered phase. Based on its intergrowth with caswellsilverite, its composition, water content and structure and the lack of indigenous water in the highly reduced Norton County enstatite achondrite, we propose that schöllhornite is the terrestrial weathering product of caswellsilverite. We conclude that caswellsilverite was altered to the bilayered sodium chromium sulfide hydrate through reaction with moisture while the meteorite specimens rested in the soil of Nebraska. During subse-
quent storage and drying in the arid atmosphere of New Mexico, the bilayered phase converted into the monolayered sodium chromium sulfide hydrate, i.e., into schöllhornite. We have named this new mineral after Robert Schöllhorn, in honor of his contributions to the study of synthetic hydrated layered chalcogenides.

Introduction

During a comprehensive mineralogical and petrological study of the Norton County (Kansas–Nebraska) enstatite achondrite (Okada et al., 1980), a new mineral, caswellsilverite (NaCrS₂), was discovered, and the occurrence of another new (the "dark-gray") phase, sodium chromium sulfide hydrate, was noted (Okada and Keil, 1982). This new mineral is described here. We have named the mineral schöllhornite, in honor of Professor Dr. Robert Schöllhorn, Anorganisch-Chemisches Institut der Universität Münster, Federal Republic of Germany, for his contributions to the study of hydrated layered chalcogenides. The mineral and mineral name have been approved by the Commission on New Minerals and Mineral Names of the International Mineralogical Association. The reference material is retained in the Institute of Meteoritics and Department of Geology, University of New Mexico, Albuquerque, New Mexico 87131, USA.

Occurrence and physical properties

Schöllhornite is a rare mineral in the Norton County enstatite achondrite and occurs as thin bands, often a few micrometers wide, within caswellsilverite and as individual grains, up to 0.25 mm in size, adjacent to caswellsilverite. The latter is usually associated with daubreelite, titanoan troilite, ferromagnesian alabandite, oldhamite, kamacite and perryite (Fig. 1). Schöllhornite forms strips, branching replacement veinlets, and straight-sided patches in caswellsilverite and contains minute, irregular relics of caswellsilverite, too small to be resolved in Figure 1. In spite of its softness, schöllhornite polishes to a nearly scratch-free surface, but larger areas retain coarse fractures and almost submicroscopic pits. The polishing hardness of schöllhornite is close to that of caswellsilverite and less than that of ferromagnesian alabandite. In reflected light, schöllhornite looks gray—in air, nearly as bright as ferromagnesian alabandite but without the pinkish tint shown by ferromagnesian alabandite in this association, and without the uniformly smooth surface of ferromagnesian alabandite. Against caswellsilverite, schöllhornite looks gray, considerably darker than caswellsilverite, and untinted relative to the yellowish or greenish tint of caswellsilverite. In oil, schöllhornite looks dark bluish gray, and its contrast with the adjacent minerals is considerably enhanced. One's first impression of the grayness of schöllhornite is dispelled by careful examination. The mineral has distinct reflection pleochroism: in air, brownish gray (brighter) to bluish gray (darker); in oil, gray with an extremely faint yellowish tint (brighter) to distinctly bluish gray (darker). Anisotropism seen with polars slightly uncrossed is strong in air and in oil, but color effects (orange-pinkish gray to bluish gray) are not conspicuous. Internal reflection, not detected in air, is orange in oil, visible only locally, but strong enough to mask anisotropism at some sites. Doubly tapering lamellae, at most a few micrometers wide, pass from caswellsilverite into schöllhornite. In caswellsilverite, each lamella seems to be one domain, but in schöllhornite some lamellae consist of many individuals, almost submicroscopic in size and variably oriented with respect to the edges of the lamellae. This suggests that the gross form of the lamellae in schöllhornite was inherited from the form of the shock-induced twin lamellae in caswellsilverite, minute particles of schöllhornite then having grown by replacement in the terrestrial environment without everywhere mimicking the crystallographic orientation of domains of the pre-existing caswellsilverite. Fine lamellation, small particle size, and close spacing of caswellsilverite relics preclude the accurate measurement of the reflectance and microindentation hardness of schöllhornite. Approximate values for R in air at 470, 546, 589, and 650 nm are 19.6, 20.8, 19.8, and 18.1%.

Physical properties of synthetic schöllhornite

Synthetic schöllhornite was made by immersing NaCrS₂ crystals in distilled water for 2-4 days at room temperature and by subsequent drying in a desiccator for more than two weeks. It forms hexagonal platelets up to 5 mm across and 0.5 mm thick. In obliquely incident daylight, the platelets are black with a submetallic luster. Platelets mounted with the c-axis horizontal can be polished exceedingly well, but many of them come apart across the perfect basal cleavage. The trace of the basal cleavage is conspicuous, even in unsevered platelets, presenting hairlines that are spaced only a few micrometers apart. A few platelets bend during polishing. Platelets mounted with the c-axis vertical cannot

![Fig. 1. Photomicrograph of schöllhornite (Sh), coexisting with caswellsilverite (Cw), ferromagnesian alabandite (Ab) and daubreelite (Db). Scale bar is 0.08 mm long. Reflected, plane polarized light.](image)
be polished satisfactorily; they develop wavy or step-sheared surfaces that retain an abundance of closely spaced pits. In some of these platelets, the pits are concentrically zoned: most abundant in the interior, least abundant near the edge. We attribute the apparent zoning to mechanical growth defects retained during polishing, rather than to compositional change, because zoning is not detectable in platelets mounted with the c-axis horizontal.

In reflected light, synthetic schöllhornite looks very light gray—almost white—in air and somewhat darker in oil. Reflection pleochroism in air is strong: OI([0001]) white with a yellowish tint (brighter); E\(\perp\{0001\}\) light gray with a bluish or greenish tint (darker). In oil, the bireflectance is increased, but the tints are suppressed. Anisotropism is very strong. In air, with polars slightly uncrossed, the polarization colors are grayish yellow to greenish gray; in oil, the greenish tint is enhanced, the yellow suppressed. In the diagonal position, giving maximum illumination between perfectly crossed polars, the phase is grayish yellow in air, light gray in oil. Internal reflection, not detected with assurance in air, is orange in oil but visible only in the thinnest platelets. Cleavage \{0001\} is perfect and conspicuous. The trace of an \(\{001\}\) (?) parting is visible in some disrupted platelets. Twinning is absent.

The reflectance of synthetic schöllhornite was measured with a Zeiss MPM microphotometer fitted with a Smith vertical illuminator and a Veril type-S running interference filter whose half-width at half-height is \(\sim 10\) nm. Objective 16 \(\times\) Pol, N.A. 0.35, and Zeiss-calibrated SiC standard no. 052 were used. The results are given in Table 1. The assignment of \(R_1\) to \(R_0\) and \(R_1\) to \(R_E\) is based on qualitative observations of brightness and color in relation to morphology and cleavage. The customary check of optical orientation by direct measurement of reflectance on \{0001\} sections could not be made, because the sections would not polish. The data of Table 1 show that synthetic schöllhornite is uniaxial (\(\sim\)) throughout the spectral range of 420 to 700 nm, that bireflectance increases with increasing wavelength, and that the dispersion of \(R_\alpha\) is almost nil from 460 to 660 nm. The quantitative color designation calculated according to weighted ordinates of Atkin and Harvey (1979, Table 1) is given in Table 2.

The microindentation hardness of synthetic schöllhornite varies with crystallographic orientation and has a wide range of values. At 15-gram-force load, four indentations on \{001\} sections give VHN 41.8-90.8, five indentations on sections mounted with the c-axis horizontal give VHN 54.5-155, and the mean of nine indentations is 80.3 ± 38.3. Most indentations are perfect, but the shape of the indentations varies with orientation. Indentations on \{001\} sections are equant; the sides are (1) all concave, or (2) varied—two opposite sides straight, one side concave, opposite side convex. Indentations made on sections mounted with the c-axis horizontal and addressed with one diagonal of the Vickers pyramid perpendicular to the trace of the \{001\} cleavage are elongate parallel to the cleavage trace; the sides are concave. Other indentations made in this orientation are defective, because the pyramid skids, leaving a track perpendicular to the cleavage trace. A single, perfect indentation made with pyramid diagonals at 45° to the cleavage trace is equant, concave, giving VHN 59.1 and thus resembling in shape and size some of the indentations made on \{001\} sections.

The determinable physical properties and the compositions determined by electron microprobe analysis of natural and synthetic schöllhornite are nearly the same; thus, the synthetic substance is a close analog of natural schöllhornite. The density of synthetic schöllhornite, measured by suspension in tetrabromoethane-tetrachloro-carbon mixtures, is 2.70 g/cm³.

### Chemical composition

Schöllhornite and its synthetic analog were analyzed using an automated ARL EMX-SM electron-microprobe X-ray analyzer. Compositionally homogeneous and chemically well-analyzed crystals of albite, orthoclase, wollastonite, enstatite, chromite, troilite, Mn-Fe garnet, and TiO\(_{0.99}\) were used as standards for quantitative analysis of Na, K, Ca, Mg, Cr, Fe, S, Mn, and Ti. Synthetic crystals of NaCrS\(_2\) were also used as a secondary standard. The wavelength dispersive analyses were done at an acceleration voltage of 15 kV and a sample current of about 0.02 µA. In order to prevent loss of volatile components during prolonged electron bombardment, the electron beam diameter was somewhat defocussed and counting times were only 5 seconds. Corrections for differential matrix effects were made by the ZAF method. The correction factor for the apparent manganese content due to the overlap of the CrK\(\beta\) line with the MnK\(\alpha\) line was obtained by measuring Mn in the Mn-free NaCrS\(_2\) standard. Semiquantitative analyses of the oxygen contents of natural and synthetic schöllhornite were done at Sandia National Laboratories, courtesy of P. F. Hlava and W. F. Chambers, using a Cameca electron probe microanalyzer. The abundances of hydrogen and other light (Z < 8) elements in schöllhornite

<table>
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<th>(\lambda (\text{nm}))</th>
<th>(R_0 (%)</th>
<th>(R_E (%)</th>
<th>(R_\alpha (%)</th>
<th>(R_\beta (%)</th>
<th>(\alpha (\text{nm}))</th>
<th>(\beta (\text{nm}))</th>
<th>(\gamma (%)</th>
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Table 2. Quantitative color designation of synthetic schöllhornite referred to I.C.I. illuminant C

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<th>(R_0 (%)</th>
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<th>(\beta (\text{nm}))</th>
<th>(\gamma (%)</th>
<th>(F (%)</th>
<th>(\phi (%)</th>
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and the synthetic analog were determined with the University of Chicago AEI ion microprobe, following procedures described by Banner and Stimpson (1975) and Steele et al. (1977). Secondary ions were generated by bombardment of the samples with a mass-analyzed, 20 keV $^{16}$O$^-$ primary ion beam of 5 nA current focussed into a 10 µm diameter spot. The secondary ion signals corresponding to $^{1}$H$^+$, $^{7}$Li$^+$, $^{9}$Be$^+$, $^{11}$B$^+$, $^{12}$C$^+$, and $^{54}$Cr$^+$ were measured in coexisting caswellisiterite and natural and synthetic schöllhornite, following the analytical procedures described in Steele et al. (1981). Secondary ion intensities were recorded using 10 sec counting intervals in the sequence $^{54}$Cr$^-$-$^{12}$C$^-$-$^{11}$B-$^{9}$Be-$^{7}$Li-$^{1}$H-$^{54}$Cr. Initial studies showed very low intensities of $^{9}$Be$^+$, $^{11}$B$^+$ and $^{12}$C$^+$ and no significant differences between schöllhornite and caswellisiterite; the majority of the data were subsequently collected using the abbreviated sequence $^{54}$Cr$^-$-$^{12}$C$^-$-$^{1}$H$^-$-$^{7}$Li-$^{54}$Cr. The variation in $^{54}$Cr$^+$ intensity during a sequence was typically ≤ 1%. Elemental abundances in schöllhornite were determined by comparing the secondary ion signals measured in natural and synthetic schöllhornite, after normalizing to the respective $^{54}$Cr$^+$ signals to correct for differences in total ion yield. The raw $^{1}$H$^+$ intensities were additionally corrected for background, monitored by measuring the $^{1}$H$^+$ intensity in synthetic schöllhornite, after normalizing to the respective $^{54}$Cr$^+$ signals to correct for differences in total ion yield. The raw $^{1}$H$^+$ intensities were additionally corrected for background, monitored by measuring the $^{1}$H$^+$ intensity in synthetic schöllhornite, after normalizing to the respective $^{54}$Cr$^+$ signals to correct for differences in total ion yield. The raw $^{1}$H$^+$ intensities were additionally corrected for background, monitored by measuring the $^{1}$H$^+$ intensity in synthetic schöllhornite, after normalizing to the respective $^{54}$Cr$^+$ signals to correct for differences in total ion yield.

The ion probe data also yield another interesting result, namely that schöllhornite contains 5 to 10 times more $^7$Li than coexisting caswellisiterite, although the absolute abundances are quite low (< 0.1 ppm).

The analytical results are shown in Table 3. Fe, Ni, Co, Cu, Zn, Mg and K were below detection limits. Near the cracks and the edge adjacent to the weathered area, minor amounts of Ca (0.09–1.4 wt.%) were detected, but this could be a contaminant, because Ca content was below the detection limit in most exposed areas of schöllhornite. By comparison with caswellisiterite, schöllhornite has a considerably lower Na content (about 5 vs. 16 wt.%; Okada and Keil, 1982). Instead, schöllhornite contains about 14 wt.% H$_2$O. The oxygen content of 12.6 wt.%, as obtained by semiquantitative electron probe microanalysis, and the hydrogen content of 1.6 wt.%, as determined by ion microprobe analysis, give an atomic ratio of H/O = 2.0, in good agreement with that of the water molecule. Schöllhornite also contains minor amounts of Ti and Mn (Table 3). These elements are also minor constituents of caswellisiterite and are considered to be present in the Cr position in the structure (Okada and Keil, 1982). The atomic ratio of S/Cr is nearly 2, equivalent to that of caswellisiterite, and the molecular formula of schöllhornite is therefore Na$_x$(H$_2$O)$_y$[CrS$_2$] (x ~ 0.3 and y ~ 1.1) or, ideally, Na$_{0.3}$(H$_2$O)$_{1.1}$[CrS$_2$].

In a neutral aqueous solution, synthetic NaCrS$_2$ transforms easily into the nonstoichiometric hydrated compound, Na$_x$(H$_2$O)$_y$[CrS$_2$] (0.3 < x < 0.8; y ~ 2 at maximum), with bilayers of water molecules (Schöllhorn et al., 1979; Schöllhorn, 1981, pers. comm.). In this hydrate, the Na content is dependent upon the degree of oxidation, and the H$_2$O content is dependent upon the ambient water vapor pressure. In addition, a partially dehydrated compound (y ~ 0.6) with monolayers of water molecules is formed in vacuo (25°C and 10$^{-7}$ Torr), and only very small amounts of residual water are left after extended evacuation at higher temperatures of 50–150°C (Schöllhorn, 1981, pers. comm.). We synthesized the partially dehydrated phase in order to compare its physical and chemical properties with those of schöllhornite. NaCrS$_2$ single crystals were immersed in distilled water for 2–4 days at room temperature. After filtration and washing with fresh distilled water, the grains were dried in the desiccator for more than two weeks. During the immersion in water, the NaCrS$_2$ crystals turned black, lost their metallic luster, and seemed to have developed cleavages parallel to the basal plane. The partially dehydrated Na$_x$(H$_2$O)$_y$[CrS$_2$] obtained after drying is a very soft and friable material which is black to the naked eye and peels very easily; it has an X-ray powder pattern similar to that of schöllhornite (see below). The chemical composition of the synthetic material obtained by treating synthetic NaCrS$_2$ with water for 40 hours and subsequent drying in a desiccator is shown in Table 3. Its composition is essentially identical to that of natural schöllhornite and the molecular formula is calculated to be Na$_{0.3}$(H$_2$O)$_{1.1}$[CrS$_2$].

X-ray diffractions study

Schöllhornite was scraped off a polished thin section of the Norton County enstatite achondrite with a sharp microtool and was mounted on the top of a thin glass rod with an organic adhesive. X-ray powder diffraction pat-
Table 4. X-ray powder diffraction patterns of natural and synthetic schöllhornite

<table>
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<th>Synthetic schöllhornite</th>
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<td>vs, B</td>
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<tr>
<td>110</td>
<td>1.67</td>
<td>m, B</td>
</tr>
</tbody>
</table>

* Indexed by hexagonal setting.
** Visually estimated (vs: very strong; m: medium; w: weak; vW: very, very weak; B: broad). Because of the very small amounts of natural schöllhornite available for x-ray diffraction, it was difficult to measure reliably its weak (006) and very, very weak (00,12) lines.

The crystal of schöllhornite was examined using Weissenberg and precession cameras with CuKα radiation. Both natural and synthetic schöllhornite show similar X-ray powder diffraction patterns, consisting of diffuse lines (Table 4). A weak (006) and very, very weak (00,12) line of natural schöllhornite could not be reliably measured, due to the small amount of the phase available for X-ray diffraction studies. A synthetic crystal of schöllhornite was examined using Weissenberg and precession cameras with CuKα radiation. The reflections, indexed based on a hexagonal setting, indicate that hkl with h + k + l ≠ 3n and 00l with l ≠ 3n are missing. Laue symmetry is 3m. The possible space groups are R3m, R3m and R32. The lattice parameters are a = 3.32(1)Å and c = 26.6(1)Å, and Z equals 3. The calculated density, 2.74 g/cm³, agrees well with the measured value of 2.70 g/cm³.

The layer-structured sulfide, NaCr₃S₂ (space group: R3m), consists of hexagonal Na-, Cr- and S-layers, in which the Na-layer is sandwiched between two CrS₂-layers (Boon and MacGillavry, 1942; Rüdorff and Stegemann, 1943). By partial oxidation in distilled water containing oxygen, NaCr₃S₂ alters to Na₉(H₂O)₃[CrS₄]₂, and the hexagonal lattice dimensions change from a = 3.55 and c = 19.35Å to a = 3.38 and c = 34.41Å. The increase of the c-dimension may be explained by the presence of bilayered water molecules between CrS₂-layers in the hydrated sulfide, because the interlayer space height between CrS₂-layers of the hydrated sulfide, 5.5Å, is twice the van der Waals thickness of the water molecule (ca. 2.8Å) (Schöllhorn et al., 1979).

In the fully hydrated alkaline and alkaline earth titanium sulfides, Li, Na, Mg, Ca and Sr compounds are bilayered, the dimension (c/3) is 11.36-11.49Å and the interlayer space height is 5.4-5.5Å, whereas K, Rb and Cs compounds are monolayered, c/3 is 8.56-8.83Å, and the interlayer spacing is 2.6-2.8Å (Schöllhorn et al., 1979). Sodium chromium sulfide hydrate having monolayered water molecules is obtained by the partial dehydration of the bilayered form. The chemical composition, X-ray powder pattern and optical properties indicate that schöllhornite is analogous to the monolayered form. From c/3 (~ 8.9Å) for schöllhornite and the van der Waals thickness (6Å) for CrS₂, the inter-spacing height of schöllhornite in Norton County is ~ 2.9Å, equivalent to the thickness of monolayered water molecules.

**Discussion**

Schöllhornite is a hydrated alkal-alkali-bearing transition metal chalcogenide and is only one of few occurrences of such compounds in nature (e.g., Czamanske et al., 1980; Erd and Czamanske, 1983). On the basis of its intergrowth with caswellsilverite, its composition, water content and structure, and the lack of indigenous water in the highly reduced Norton County enstatite achondrite, we propose that schöllhornite is the terrestrial weathering product of caswellsilverite. As is indicated by the experimental work of Schöllhorn et al. (1979), Schöllhorn (1981, pers. comm.) and by us, sodium chromium sulfide hydrate (bilayered form) is readily produced by the partial hydration of NaCrS₃ in aqueous solution at room temperature. The monolayered form (i.e., schöllhornite) grows by subsequent partial dehydration of the bilayered phase. We suggest that the same process occurred after the fall of the Norton County enstatite achondrite. This meteorite fell on Feb. 18, 1948 near the Nebraska–Kansas border and the first specimen was recovered April 6, 1948 (LaPaz, 1949), with most specimens found several weeks or months after this date (Beck and LaPaz, 1951); thus, the meteorite specimens were exposed to moisture in the soil for that time. During transportation to Albuquerque, New Mexico, after recovery, acid soluble material (apparently magnesium carbonate hydrate) came out with water from the interior of the meteorite and deposited as fibrous crystals on the surface (Beck and LaPaz, 1951). The occurrence of various other minerals of terrestrial weathering origin, i.e., portlandite, vaterite, calcite and bassanite, indicates that they were produced by the primary reaction between oldhamite and meteoric water (Okada et al., 1981). Thus, we conclude that caswellsilverite altered to the bilayered sodium chromium sulfide hydrate through reaction with moisture while the meteorite rested in the soil of Nebraska. This compound then converted to schöllhornite, i.e., the monolayered sodium chromium sulfide hydrate, during subsequent storage and desiccation in the dry atmosphere of New Mexico. Finally, it should be noted that in enstatite achondrites and chondrites, Ramdohr (1963, 1964) noted the occurrence of new layer-structured opaque minerals that he designated I, II, III, A and B, although he did not define their compositions and structures. From microscopic appearance and occurrence, schöllhornite, caswellsilverite and an as yet unnamed calcium chromium sulfide mineral (probably hydrated), discovered by us in the Norton County meteorite seem to correspond to some of Ramdohr's (1963, 1964) layered opaque minerals.

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