Empressite and "Stuetzite"

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Abstract

Empressite, from Empress Josephine mine, Colorado (type specimen), stuetzite, the original crystal described by Schrauf (1878), a homogeneous fusion with composition Ag₅Te₃ and hydrosynthetic crystals (Ag-Te) give identical x-ray powder patterns.

The crystal of stuetzite and the synthetic crystals are hexagonal with probably space group C6/mmm, a = 13.46 c = 8.46 kX. The x-ray powder pattern is readily indexed with these dimensions. Analyses indicate the composition AgTe or Ag₇Te₅ for natural empressite. Fusions of the elements clearly indicate a structural formula 7[Ag₅Te₃], close to 3[Ag₄Te₃] (Koern, 1939). A possible general structural formula would be 18[Ag₉₋ₓTe₁₄₊ₓ] where x may have values 0.1 to 0.5 with corresponding calculated specific gravities 7.73 to 7.90 in fair agreement with measured values 7.99 (Ag₅Te₃ fusion) and 7.61 (empressite).

This joint work is the result of studies which commenced in 1944 when Thompson (1946) made a broad study of the descriptive mineralogy and occurrence of telluride minerals. A short description of empressite with x-ray powder data was published by Thompson (1949). Further studies were made possible by the availability of the original crystal of stuetzite kindly loaned by Professor Machatschki from the Vienna Museum. In May and June of 1950, Professor Peacock remeasured the stuetzite crystal and also obtained single crystal x-ray measurements on it. This was the last laboratory work done by Professor Peacock. During 1948–49 in the course of a study of hydrosynthesis of silver and gold tellurides, Rowland (1950) obtained crystals which gave an x-ray powder pattern identical with the pattern of empressite. The present account, prepared by Berry with the help of Thompson, has been largely extracted from the unpublished theses of Thompson (1946) and Rowland (1950) and from Professor Peacock's notes.

Previous Observations

Empressite, a new silver telluride, from the Empress Josephine Mine, Kerber Creek District, Colorado, was discovered and named by Professor R. D. George of the University of Colorado, and later described by Bradley (1914, p. 63) with two analyses in good agreement with the formula AgTe. The mineral was described as massive to finely granular with no indication of crystal form; brittle to friable with a finely con-

1 Graduate student, (1948–49) Queen's University and holder of a bursary from the National Research Council (Canada) with whose permission this paper is published.
choidal to uneven fracture. Hardness between 3 and 3½; specific gravity 7.510; lustre metallic and colour pale bronze.

The blowpipe reactions as given by Bradley are: in the oxidizing flame on charcoal it fuses at F = 1 giving a heavy white coating of tellurium dioxide and a black globule, which if placed in the reducing flame gives on cooling white dendritic points of silver on its surface. Prolonged heating in the oxidizing flame gives a globule of silver. In the open tube a faint white sublimate of tellurium dioxide is formed which if strongly ignited fuses to colourless globules. The mineral is readily soluble in hot dilute nitric acid.

The mineral is not described by Short (1940), but Mathewson (in Bradley, 1914) stated that empressite is structurally homogeneous with large irregular light and dark polygonal grains and no interstitial matter. There is no published x-ray information on the mineral.

Stuetzite, was described by Schrauf (1878) in highly modified hexagonal or pseudohexagonal crystals from a single specimen in the mineral collections of Vienna University; the locality was probably Nagyág, Transylvania. It is associated with gold and hessite on quartz. The composition was given as Ag₄Te; the silver was determined approximately with the blowpipe.

The mineral was described as lead-grey with reddish tinge, lustre metallic, fracture uneven to subconchoidal. It is easily fusible to a dark bead from which a silver globule is obtained by reduction with soda and yields tellurium dioxide in the open tube. Stuetzite is not described by Short (1940).

**Material Studied**

1. Empressite, Empress Josephine Mine, Kerber Creek District, Colorado (USNM, R 7243, type specimen). A compact mass of empressite partially covered with a coating of a clay-like material. On one corner is a small area of galena showing cubic cleavage. Small amounts of yellow-brown sphalerite and finely crystalline pyrite are disseminated throughout the clay-like material.

2. Empressite, Red Cloud Mine, Boulder County, Colorado (mislabelled petzite). Empressite and altaite sparsely disseminated throughout rock.

3. Stuetzite, probably from Nagyág, Transylvania (Mineralogical Museum of Vienna University, 5808). Original crystal described by Schrauf (1878).

4. "Empressite," probably part of original material collected by Dr. George from Empress Josephine mine, Colorado. Obtained from University of Colorado collections through E. M. Gunnell, Denver.

**Physical and Microscopical Observations**

In materials 1 and 2 empressite has a heavy metallic lustre and an almost jet black colour like petzite but with a slight bronzy cast. It has no cleavage, and is quite brittle, with a finely conchoidal to uneven fracture.
The hardness of 3½ and the specific gravity measured on several fragments is 7.61 ± 0.01, as compared to 7.51 (Bradley). Empressite polishes to a smooth surface which shows intense reflection pleochroism, light gray-mauve to creamy-white, depending on the orientation (Fig. 1). The anisotropism is likewise very strong with polarization colours white, yellowish-green, russet-brown, brown, dark blue. Twinning and evidence of cleavage are absent. The mineral makes a solid moderate coarse grained mosaic (Fig. 2).

Etch-reactions: HNO₃ slow effervescence, slowly stains iridescent which may be removed by hard buffing; HCl negative; KCN negative; FeCl₃ stains iridescent and remains on buffing; KOH negative; HgCl₂ quickly stains iridescent and remains on buffing. The hardness estimated with a needle is C.

The sections examined consisted essentially of empressite with small amounts of altaite and pyrite which are present as inclusions in the empressite (Fig. 1). These inclusions were identified by an x-ray powder photograph on material 1. Altaite forms ill-defined stringers with small blebs of chalcopyrite and sphalerite which cut the empressite in the type material. A few small crystals of pyrite and marcasite are completely surrounded and corroded by the empressite. Galena, noted on the hand specimen but not observed in polished section, was checked with an x-ray powder photograph on material 1. It gave a positive test for Te with sulphuric acid.

An analysis of 250 mg. of the type material, carefully selected under the binocular microscope was made by R. N. Williams with the following result: Ag 54.77, Te 45.26, total 100.03. This differs substantially from the previous analyses and corresponds to Ag₅Te₈ (Ag 53.0, Te 47.0) or Ag₇Te₈ (Ag 54.2, Te 45.8). In reporting these results the analyst wrote: "The impurities which consist of gold, lead, iron and copper, do not in my opinion exceed 0.10 per cent." The new analysis indicates distinctly more silver than that required by the original formula AgTe but not as much as is required by the composition Ag₅Te₈ suggested by the fusion experiments described later.

Polished section observations could not be made on the crystal of stuetzite. A small fragment of the crystal, which became detached in transit, yielded the same x-ray powder pattern as materials 1 and 2.

Material 4, consisting of a few fragments, showed physical properties similar to the other empressite specimens. The specific gravity measured on several pieces is 7.30 ± .04. In polished section the mineral is similar to empressite and associated with altaite. A spectrographic analysis showed mainly silver and tellurium with minor lead and traces of iron, copper and silicon. This material gave an x-ray powder pattern (table 5)
Fig. 1, 2.—Empressite, Empress Josephine mine, Kerber Creek District, Colorado (material 1). Polished section; X135. Fig. 1. Showing intense reflection pleochroism with a white inclusion of altaite (one nicol). Fig. 2. Coarse mosaic showing strong anisotropism (crossed nicols).

Figs. 3-5.—Ag-Te fusion products in polished section. Fig. 3. Ag:Te=1:1, laths of empressite (gray) in an eutectic groundmass of empressite and tellurium (one nicol, X290). Fig. 5. Ag:Te=3:2, empressite (gray) with elongate streaks of tellurium (white) (one nicol, X290). Fig. 4. Ag:Te=5:3, homogeneous mosaic with strong anisotropism identical with empressite (crossed nicols, X135). Fig. 6. Ag:Te=7:4, interlocking crystals of empressite (black and white) with interstitial patches of twinned hessite (crossed nicols, X290).

Unlike empressite or any other known telluride minerals. A few small fragments were fused in vacuum; the fusion product gave the powder pattern of empressite plus the strong lines of clausenthalite.
SvNRnnsts ol Eupnrssrre

following compositions were prepared in an empressite:

Pyrosyntheses.

Charges with the following compositions were prepared in an endeavour to synthesize empressite:

Table 1. Ag-Te Fusions

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<th>Composition</th>
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<th>Te %</th>
<th>Products</th>
<th>S.G. (meas.)</th>
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<td>54.77</td>
<td>45.26</td>
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<td>44.1</td>
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<tr>
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<td></td>
<td></td>
<td></td>
<td>tellurium*</td>
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</tr>
<tr>
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<td>41.5</td>
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<td>40.8</td>
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<td>8.04 ± 0.03</td>
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<td></td>
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<td></td>
<td></td>
<td>hessite*</td>
<td></td>
</tr>
<tr>
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<td>7 Ag:4 Te</td>
<td>59.6</td>
<td>40.4</td>
<td>empressite*</td>
<td>8.02 ± 0.01</td>
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<tr>
<td></td>
<td></td>
<td></td>
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<td>hessite*</td>
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</tr>
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</table>

* Identified by x-ray powder patterns.

The charge Ag:Te = 1:1 fused fairly readily and produced on cooling a stony, black, brittle regulus. A polished section showed a non-homogeneous product consisting of two phases: large laths of a gray phase with polarization colours like those of empressite, in a groundmass consisting of an eutectic between the gray phase and a white phase resembling tellurium (Fig. 3). An x-ray powder photograph identified these two phases as empressite and tellurium.

The charge with the composition corresponding to Williams’ analysis, Ag 54.77, Te 45.26, fused fairly readily with slight green fluorescence to a black, splendent, brittle, steel-gray, somewhat pitted regulus but with indication of crystallinity on the lower side. A polished section showed
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an inhomogeneous product consisting of an intimate intergrowth of three phases. The main component occurs in lath-like gray crystals with polarization colours similar to those of empressite. A creamy-gray phase with low polarization colours similar to those of hessite contains pinkish-white streaks of a third phase which resembles native tellurium. In places there is a delicate eutectic between the gray and creamy-gray phases. An x-ray powder photograph of a random fragment gave only the empressite pattern.

The charge Ag:Te = 3:2 fused at a fairly high temperature and produced on cooling a black, stony, brittle regulus which developed a sprout and several small spherical globules on its surface. A polished section showed an inhomogeneous product which consisted of two phases. The major phase, a gray-white substance encloses many elongated stringers of a pinkish white phase (Fig. 5). Under crossed nicols the major phase shows irregular lath-like crystals. An x-ray powder photograph of a fragment from the main regulus gave the empressite pattern. An x-ray powder photograph of the main sprout on the regulus gave the empressite pattern plus a few lines of tellurium.

The charge Ag:Te = 5:3 fused readily with slight green fluorescence and produced a cooling in gray, brittle regulus. On a fresh fracture the colour is lustrous black, the most "empressite looking" of all the fusions. This regulus as well as all previous ones shows no cleavage. It has however, unlike the previous fusion products, a finely conchoidal fracture similar to that of empressite. A polished section revealed a homogeneous product, gray-white in colour with very weak pleochroism. With crossed nicols it makes a moderate, approximately equidimensional mosaic with polarization colours like those of empressite (Fig. 4). An x-ray powder photograph gave only the empressite pattern.

The charge Ag:Te = 7:4 fused fairly readily and produced a lustrous, black, stony, brittle regulus. A polished section showed an intimate intergrowth of two phases which were identified by an x-ray powder photograph as empressite and hessite (Ag₂Te). The empressite occurs in elongated grains with small interstitial areas of hessite which show confused lamellar twinning (Fig. 6).

On heating several fragments of empressite in an evacuated silica glass tube, a black vapour, presumably tellurium was observed creeping up the
sides of the tube. The black somewhat sectile regulus was too small for a polished section study but an x-ray powder photograph of a fragment gave the hessite \((\text{Ag}_2\text{Te})\) pattern. Apparently the empressite lost some of its tellurium and formed hessite.

The results of this brief study of the silver-tellurium system are not in full accord with the results of previous workers (Hansen, 1936, p. 65) who reported two compounds: one with the composition \(\text{Ag}_2\text{Te}\) corresponding to the mineral hessite, which was produced synthetically and the other with a doubtful composition, \(\text{Ag}_4\text{Te}_9\), or \(\text{AgTe}\), presumably corresponding to empressite. Our results indicate a homogeneous product similar to empressite with the composition \(\text{Ag}_5\text{Te}_a\) while Chikashige & Saito (1916, p. 368) state that \(\text{Ag}_2\text{Te}\) reacts with the melt at 443° and forms another compound \(\text{Ag}_7\text{Te}_4\) which changes at 403° to the \(\beta\) form. Koern (1939) reports, in abstract, finding two compounds \(\text{Ag}_2\text{Te}\) and \(\text{Ag}_1\text{Te}_7\) both in two modifications.

Hydrosynthesis. In the course of study of the hydrosynthesis of gold and silver tellurides (Rowland, 1950), artificial crystals of hessite, calaverite and empressite were formed. The synthesis were performed in the type of graphite lined steel bomb developed by Dr. F. G. Smith at the University of Toronto and used in synthesis of ore minerals by Béland (1948), Robinson (1948) and others. Empressite was recognized in the products of two bomb runs designated VI & VII; the composition of the charges and the conditions are shown in table 2.

<table>
<thead>
<tr>
<th>Table 2. Empressite: Composition of Bomb Charges</th>
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<tr>
<td>No.</td>
</tr>
<tr>
<td>------</td>
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<tr>
<td>VI</td>
</tr>
<tr>
<td>VII</td>
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</table>

The crystals of empressite produced in run VI were blocky, hexagonal and commonly in crystalline aggregates displaying parallel growth and purple tarnish. Those produced by run VII were untarnished silvery and blocky, usually as single crystals with well defined prismatic zone and poor terminal faces. The crystals were rather poorly formed and from 0.1 to 0.25 mm. in greatest dimension.

Crystallography

The x-ray powder pattern of empressite was established on type material (1) by Thompson (1946, 1949). The same pattern was also given
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by material 2 and a homogeneous fusion of composition Ag₆Te₄. The pattern was relatively complex and no attempt was made to index it. In 1949, Rowland (1950) obtained synthetic empressite crystals giving an x-ray powder pattern identical with the pattern of natural empressite.

**Synthetic crystals.** Fifteen crystals of empressite from two bomb runs (VI and VII) were measured on the two-circle optical goniometer and the measured $\phi$ and $\rho$ angles plotted in gnomonic projection. The projections clearly indicate hexagonal symmetry and normal indexing of the projections leads to the axial ratio from the $\phi$ and $\rho$ measurements.

$$a:c=1:0.6323$$

The forms observed on nine measured crystals are listed in Table 3 with measured and calculated $\phi$ and $\rho$ angles.

A single crystal, adjusted to rotate about the c-axis on the optical goniometer, yielded sharp rotation, zero and first layer Weissenberg films. The reciprocal lattice projections clearly indicate a hexagonal lattice with unit cell dimensions:

$$a=13.46, \quad c=8.45 \text{ kX}; \quad a:c=1:0.628$$

The observed diffractions (000l not observed) showing no systematic extinctions, are characteristic of the space group $C6mmn$ of the crystals belong to the holohedral class.

**Original crystal of stuetzite.** This crystal (about $\frac{3}{4}$ mm across), first measured by Schrauf (1878), was remeasured by Peacock. In his original notes he recorded measurements for 27 faces on one end and 28 faces on the other end (Fig. 7). He noted that "the projection of the measured crystal suggests Schrauf's (1012) as the better unit form" and deduced, from the measurements, the hexagonal axial ratio in close agreement with Schrauf's value with $c$ divided by two.

$$a:c=1:0.6287 \quad (M.A.P.)$$

$$a:c/2=1:0.6265 \quad (\text{Schrauf}, 1878)$$

Peacock also noted that there seems to be no reason for not placing the mineral in the hexagonal system. Schrauf had suggested that it was pseudohexagonal, probably monoclinic.

In table 3 the measured $\phi$ and $\rho$ angles for artificial empressite crystals and the original stuetzite crystal are given with angles calculated from the axial ratio. In Schrauf's setting all $l$ indices are doubled.

Peacock obtained rotation and zero layer Weissenberg films from the stuetzite crystal. The rotation film about $c$ is poor due to the large size of the crystal and did not yield sharp measurements. The zero layer Weis-

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1 Using wavelength CuKα 1.5374.
 senberg shows distinct hexagonal symmetry and the two films lead to the lattice dimensions:

\[ a = 13.4, \quad c = 8.5 \pm 0.1 \text{kX}. \]
Koern (1939) gives hexagonal lattice dimensions for $\alpha$-$\text{Ag}_{12}\text{Te}_7$ which compare closely with our measurements for empressite and stuetzite.

\begin{align*}
\text{c:} & 8.4508 \text{ kX Koern (1939)} \\
\text{c:} & 8.5 \text{ kX Stuetzite (M.A.P.)} \\
\text{c:} & 8.45 \text{ kX Artificial crystals (J.F.R.)} \\
\text{c:} & 8.46 \text{ kX Empressite (powder data)}
\end{align*}

**X-ray powder pattern.** Empressite, materials 1 and 2, stuetzite, the homogeneous fusion $\text{Ag}_{12}\text{Te}_3$ and the hydrosynthetic crystals of $\text{Ag}-\text{Te}$ all give an identical x-ray powder pattern. The observed intensities, measured $\theta$ values and interplanar spacings for type empressite from the

[Image: Fig. 7.—Stuetzite, original crystal described by Schrauf (1878), idealized drawing (L. G. B.) from sketches by M. A. Peacock. The crystal actually shows two ($h0\bar{h}l$) and two ($h\bar{h}2l$) zones complete from (0001) to (0001), the forms shown are $c(0001), m(10\bar{1}0), a(11\bar{2}0), b(21\bar{3}0), d(10\bar{1}2), f(10\bar{1}1), g(20\bar{2}1), s(30\bar{3}1), u(1\bar{1}23), z(1\bar{1}22), y(1\bar{1}21), x(2\bar{2}41), i(2131), a(31\bar{4}1).$]

Empress Josephine Mine, Kerber Creek District, Colorado (Thompson, 1949) are given in Table 4 together with the indices and calculated spacings for all the observed lines. There is a good agreement between each measured spacing and one or more spacings calculated from the lattice dimensions.

Empressite? (material 4) said to be part of the original material collected from the Empress Josephine mine gave a different x-ray powder pattern. The observed intensities, measured $\theta$ values and interplanar spacing from this pattern are given in Table 5. There is little hope of determining lattice dimensions from the material at present available.
Could this material really represent the original empressite with composition AgTe indicated by the early analyses? Or might it be the natural counterpart of the second modification of Ag$_{12}$Te$_7$ mentioned by Koern (1939) without any descriptive details? The pattern is recorded here in the hope that it might be recognized elsewhere.

Table 4. Empressite: Ag$_{2-x}$Te: X-Ray Powder Pattern (R.M.T.)

Hexagonal, C6/mmm; \(a = 13.46\), \(c = 8.46\) kX; \(Z = 21\)

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</table>

**Composition and Cell Content**

The reported composition of the materials we find to be structurally identical are shown in Table 6 together with the measured specific gravity.

The volume of the unit cell determined for empressite is 1327.7 kX$^3$. Combined with the measured specific gravity, 7.61 we get \(M = 6122\). M
values and calculated specific gravities for some of the above compositions are given below.

\[
\begin{align*}
&\text{26}[\text{AgTe}] & \text{M}=6123.0 & \text{G(calc.)}=7.61 \\
&\text{4}[\text{Ag}_2\text{Te}_3] & \text{M}=5374.0 & \text{G(calc.)}=6.68 \\
&\text{5}[\text{Ag}_2\text{Te}_3] & \text{M}=5967.5 & \text{G(calc.)}=8.66 \\
&\text{7}[\text{Ag}_2\text{Te}_3] & \text{M}=6455.6 & \text{G(calc.)}=8.03 \\
&\text{3}[\text{Ag}_2\text{Te}_3] & \text{M}=6563.5 & \text{G(calc.)}=8.16
\end{align*}
\]

The early analyses of empressite (Bradley 1914, 1915) and the measured specific gravity clearly lead to a unit cell content of 26[AgTe] with good agreement between measured and calculated specific gravities. Our observations on pyrosyntheses indicate a cell content of 7 [Ag$_2$Te$_3$] for which the measured and calculated specific gravities are in substantial agreement. This cell content is very similar to 3[Ag$_2$Te$_3$] found by Koern (1939), which requires a higher specific gravity. If the composition
were written \( \text{Ag}_{2-x} \text{Te} \) the artificial materials would have a cell content of 21[\( \text{Ag}_{2-x} \text{Te} \)] where \( x \) is about 0.3 but for the natural materials with \( x = 1 \) or \( \frac{5}{6} \), \( Z = 21 \) would be much too low. Or, the formula might be expressed as \( \text{Ag}_{2-x} \text{Te}_{1+x} \) and the cell contents indicated by the observed compositions would then be:

\[
\begin{align*}
\text{AgTe} & \quad 18[\text{Ag}_{2.3} \text{Te}_{1.3}] \quad M = 6358.5 \quad G(\text{Calc.}) = 7.90 \\
\text{Ag}_{2/3} \text{Te}_{5/6} & \quad 18[\text{Ag}_{2.5} \text{Te}_{1.5}] \quad M = 6259.4 \quad G(\text{Calc.}) = 7.78 \\
\text{Ag}_{2/3} \text{Te}_{1} & \quad 18[\text{Ag}_{2.12} \text{Te}_{1.12}] \quad M = 6223.1 \quad G(\text{Calc.}) = 7.75 \\
\text{Ag}_{2/3} \text{Te}_{7/6} & \quad 18[\text{Ag}_{2.9} \text{Te}_{1.9}] \quad M = 6216.1 \quad G(\text{Calc.}) = 7.73
\end{align*}
\]

This structural formula, 18[\( \text{Ag}_{2-x} \text{Te}_{1+x} \)], appears to reconcile the differences in composition between natural and artificial materials except that the calculated specific gravity is lower than the measured value for the artificial fusion material with higher silver content. A complete structure determination must be undertaken however before this formula can be accepted for empressite.

Since the composition of this mineral, although still in some doubt, is certainly close to that originally ascribed to empressite, and the composition given for stuetzite is quite different and based on very uncertain chemical methods, it is proposed that the name empressite be retained and the name stuetzite be dropped.

References


