New data on and discreditation of "texasite," "albrittonite," "cuproartinite," "cuprohydromagnesite," and "yttromicrolite," with corrected data on nickelbischofite, rowlandite, and yttrocrasite

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Abstract

Data are presented which show that "texasite" and "albrittonite" exist only as synthetic phases and that "cuproartinite" and "cuprohydromagnesite" are not known to exist in any form. "Yttromicrolite" is a heterogeneous mixture of yttrian microlite and tantalite. These five phases have therefore been discredited as minerals. Major parts of the descriptions of the minerals rowlandite, yttrocrasite, and nickelbischofite from Llano County, Texas should also be revised. We conclude, among other things, that the formula of rowlandite is still in question, that yttrocrasite has not yet been definitively shown to be a member of the euxenite group and that nickelbischofite does not occur at the Oxford Quarry, Llano County, Texas. It is suggested that procedures for accreditation of a new mineral should include deposition of a type or cotype specimen in matrix to a suitable institution in sufficient quantity for X-ray diffraction or chemical analysis, coupled with the complete manuscript to be published at a later time.

Introduction

We have critically re-examined "texasite" (Crook, 1977a), "albrittonite" (Crook and Marcotty, 1978), nickelbischofite (Crook and Jambor, 1979), rowlandite (Crook et al., 1978), yttrocrasite (Crook, 1977b), "yttromicrolite" (Crook, 1979), "cuprohydromagnesite" and "cuproartinite" (Os- wald and Crook, 1979). We find that aspects of all of these papers are subject to question. The results of our studies as presented as part of this paper led us to recommend that the minerals "texasite", "albrittonite", "cuproartinite", "cuprohydromagnesite", and "yttromicrolite" should be discredited. This recommendation has been approved by the I.M.A. Commission on New Minerals and Mineral Names (Kato, written communication, 1981).

Data on specific minerals

"Tasasite"

Crook (1977a) states that "texasite" (defined as $Pr_2O_3SO_4$) is a "supergene alteration product of primary rare-earth minerals" occurring in pegmatites of the Llano region of Texas. The primary parent minerals contain typical undifferentiated suites of rare-earth elements. That "texasite" should have such an origin, and that Pr should be so highly concentrated as to represent >99.9% of the rare earth elements present is geochemically unrea-
sonable. We believe that the explanation for this is simply that synthetic Pr$_2$O$_2$SO$_4$ has been described as though it were a natural material.

Dr. John Haschke, a former faculty member of the Department of Chemistry, University of Michigan, synthesized a number of rare-earth compounds and donated samples to Peacor prior to Crook’s time of residence at The University of Michigan. Among these was a container of crystals of Pr$_2$O$_2$SO$_4$ (identical in appearance to crystals of “texasite”) which served as the source of material for X-ray crystallographic studies (Peacor, unpubl. data). This container of crystals could not be found subsequent to the publication of the description of “texasite” by Crook, although other samples of Haschke’s synthetic rare-earth compounds were still present in Peacor’s laboratory. This demonstrates that crystals of this extremely rare compound were available in the laboratory where the “texasite” was studied. This is at first sight a remarkable coincidence, but it is compatible with our other direct evidence showing the equivalence of synthetic and natural materials.

We submitted samples of “texasite” (so identified originally as such by Crook) for neutron activation analysis, along with samples of synthetic Pr$_2$O$_2$SO$_4$ obtained from John Haschke and said by him (written communication, 1979) to have been synthesized with the same starting source materials and as part of the same experiments as the Pr$_2$O$_2$SO$_4$ originally given to Peacor. The analyses were carried out by Mr. John Jones of the Michigan Memorial Phoenix Project, The University of Michigan. Results are presented in part in Table 1. These results show that there are striking similarities between Crook’s “texasite” and synthetic Pr$_2$O$_2$SO$_4$. In the following discussion we emphasize: (1) general similarities, (2) Nd/Pr ratios, and (3) Au and Ag values.

First, there are clear, general similarities between the analyses of Crook’s type “texasite” and Haschke’s synthetic phase. Where rare earth elements are relatively high in one they are relatively high in the other. There are some exceptions to this rule, but since the analyses were obtained on exceptionally small samples, the differences are not unreasonable. Where the differences are highest (for Pr/Eu and Pr/Sm) “texasite” has a lower concentration of a given element relative to praseodymium than the sample obtained from Haschke. It is important to recognize that the absolute values of ratios such as Pr/Tb or Pr/Eu are on the order of 1,000 to

<table>
<thead>
<tr>
<th>Rare earth activity ratios</th>
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<tr>
<td>Synthetic</td>
</tr>
<tr>
<td>Pr/Tb</td>
</tr>
<tr>
<td>Pr/Dy</td>
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<tr>
<td>Pr/Sm</td>
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<tr>
<td>Pr/Sm</td>
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<td>Pr/Sm</td>
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<tr>
<td>Pr/Sm</td>
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<tr>
<td>Eu/Dy</td>
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<tr>
<td>Sm/Dy</td>
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<tr>
<td>Ce/Dy</td>
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<tr>
<td>Dy/Tb</td>
</tr>
<tr>
<td>Sm/Tb</td>
</tr>
<tr>
<td>Eu/Tb</td>
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<tr>
<th>Estimated absolute concentrations (ppm)</th>
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<tbody>
<tr>
<td>Au</td>
</tr>
<tr>
<td>Ag</td>
</tr>
<tr>
<td>Tb</td>
</tr>
<tr>
<td>Nd</td>
</tr>
</tbody>
</table>

Second, the fact that Nd was not detected in either sample (and thus is present only at levels less than 200 ppm, see Table 1) is especially noteworthy. Haschke notes (written communication, 1979) that “the probability of finding a texasite sample
with the same Nd/Pr ratio as my synthetic sample. . . is so incredible that it must be excluded. The separation of ‘didymium’ (mixture of Nd and Pr) into the rather pure elements (neodymium and praseodymium) is one of the interesting historical events of lanthanide chemistry.” This difficult separation is accomplished using ion exchange resins. Repeated exchange of progressively more differentiated fractions is necessary in order to ultimately achieve high resolution. Haschke therefore concludes that “attainment of the same resolution of Nd and Pr in a natural system and in a chemical processing facility is beyond belief.” We further emphasize that not only are Nd levels the same in both samples, but Nd is not detected in the “texasite” sample. Such differentiation of Nd relative to Pr is not credible in a natural mineral.

Third, of even greater interest is the fact that gold was detected in both the “texasite” and synthetic Pr2O2SO4 samples (Table 1). Such levels of gold in a rare-earth differentiated pegmatite mineral are unreasonable. Haschke synthesized his Pr2O2SO4 under hydrothermal conditions in gold capsules containing HNO3 at 15,000 psi pressure and 500°C (written communication, 1979). Such conditions could be expected to yield low but variable concentrations of gold as contaminants in the run products. These data are similar in every respect to those for the synthetic Pr2O2SO4 present in Peacor’s laboratory and synthesized by Haschke. We also note that although the gold contents are different, the magnitude of the difference is not unusual for a contaminated in specimens synthesized under the conditions noted by Haschke. It is the presence of ppm levels of gold in the so-called natural phase which is not credible. In addition, Jones (written communication, 1979) has analyzed (using NAA) a number of pegmatite samples from the Llano region and, indeed, no gold was detectable.

The chemical analyses on “texasite” given by Crook (1977a) show average values of: Pr2O3, 80.61%; La2O3, 0.08%; Ce2O3, 0.03%; Nd2O3, 0.02%; SO3, 19.52%. Crook states that he used synthetic Pr2O2SO4 as a standard for his Pr analysis but galena for S. The use of galena as a standard for S in “texasite” instead of Pr2O2SO4 is surprising in that the synthetic sulfate of presumed identical composition to “texasite” was used as a Pr standard. Galena is a poor standard for S and Pr2O2SO4 is an excellent one in this case considering that the average atomic numbers of the two compounds are very different. Furthermore, Crook’s average value for SO3 (19.52%) is remarkably close to the expected stoichiometric value (19.53%) considering that galena was used as a standard for S, and considering the accuracy of normal probe data and correction procedures at The University of Michigan. The quoted weight percents for La2O3, Ce2O3, and Nd2O3 (all less than 0.10%) are below minimum detectable limits, especially considering X-ray interferences. This is further indicated by the fact that Nd was not detected in “texasite” by neutron activation analysis, as emphasized above. In short, these data show that Crook’s reported analytical results are almost certainly erroneous.

Dr. Haschke states (written communication, 1979) that: “The lattice parameters and d spacings, reported on page 1007 of American Mineralogist, Vol. 62 are my data for Pr2O2SO4. However the I/I0 values are not.” Haschke further emphasizes that: “I have never given Mr. Crook permission to publish any of my data or to use any statements attributed to me.” Dr. Haschke also asserts that other statements attributed to him by Crook are not correct. For example Haschke says that (written communication, 1979, in contrast to Crook’s statement on page 1008 of American Mineralogist): “. . . I do not (and never have) attributed the existence of a pure praseodymium mineral to crystalline stability. The structures of Nd2O2SO4 and Pr2O2SO4 are identical, and in my opinion, segregation of Pr could never be attributed to a unique structural stability of the praseodymium compound.”

In addition to the report of “texasite” from the Clear Creek and Rode Ranch pegmatites in Texas, Crook (1978a) also reported it from the Luster pegmatite in Colorado. The Colorado “texasite” is reported to have somewhat higher levels of La, Ce, and Nd, but otherwise is rather similar to the Texas occurrence. While we have not been able to analyze the Colorado “texasite” for rare earth and other elements, most of the objections raised above for the type “texasite” also apply to this occurrence.

The discreditation of “texasite” has been approved by the I.M.A. Commission on New Minerals and Mineral Names (Kato, written communication, 1981).

“Albrittonite”

“Albrittonite” was described by Crook and Marcotty (1978).1 We have found discrepancies in the

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1Marcotty (written communication, 1978) has informed us that her part in the research was to obtain powder X-ray patterns and this did not extend to their interpretation. Our comments are addressed towards the research attributable to Crook.
crystal drawings and related data, in the inherent
unlikelihood of the reported occurrence given the
high solubility in H₂O and the miscibility with
nickelbischofite, and in inconsistencies in the anal-
ysis.

We reproduce Figure 1 of the “albrittonite”
(CoCl₂ · 6H₂O) description as part of our Figure 1.
Beside it we have placed direct reproductions of
certain figures from Vol. II of The System of
Mineralogy, Seventh Edition (Palache et al., 1951).
These two figures represent crystals of liroconite
(p. 921 of The System of Mineralogy) and para-
laurionite (p. 65). Detailed comparison shows that the
“albrittonite” crystal drawings and those of para-
laurionite and liroconite are identical, except for the
style of lettering on the faces. We therefore con-
clude that the “albrittonite” crystal drawings have
been copied from The System of Mineralogy. Fur-
thermore, an early version of the albrittonite paper
on record with the Smithsonian Institution with
albrittonite specimen accession papers, contains
another drawing entitled “typical morphological
development of albrittonite.” This drawing is enti-
tirely different than those pictured as Figure 1. It is
clear that the morphology of “albrittonite” crystals
has been misrepresented.

J. L. Jambor (written communication, 1980) pro-
vided us with a copy of a letter from Crook to
Jambor dated July 5, 1978. In this letter Crook
states: “Now, my diagram of albrittonite. I agree
that the drawing does not look like it has a (001) face
but the measured data are:

\[
\begin{align*}
\phi & = 9000' \\
\rho & = 2712' \\
\phi_2 & = 6248' \\
\rho_2 & = 9000' \\
A & = 6247'
\end{align*}
\]

This fits the indices for (001) well—perhaps the
drawing is just a poor projection.” The diagram to
which Crook is referring is the one which is identi-
cal to the one shown with the description of the
mineral paralaurionite in The System of Mineral-
ogy, Volume II (1951). On page 64 of Volume II of
The System of Mineralogy (1951) also under the

**LIROCOrite**
(From The System of Mineralogy, Vol.II, p. 921)

![Liroconite Crystal Diagram](image)

**PARALAUROiONITE**
(From The System of Mineralogy, Vol.II, p. 65)

![Paralaurionite Crystal Diagram](image)

Fig. 1. Copy of Fig. 1 from Crook and Marcotty (1978), said to be “typical morphological development of albrittonite” (on the left), together with copies of figures (on the right) of crystals of liroconite and paralaurionite from Vol. II of The System of Mineralogy.
description of paralaurionite, is a table of data dealing with the morphology of paralaurionite crystals. The first line reads:

\[
\begin{align*}
\phi & \quad \rho & \quad \phi_2 \\
001 & \ 90^\circ00' & \ 27^\circ12'1/2 & \ 62^\circ471/2' \\
p_\phi & \quad B & \quad C & \quad A \\
90^\circ00' & \ 62^\circ471/2' & \\
\end{align*}
\]

These angles are essentially identical with those given above, angles which Crook states he measured for “albrittonite.” This indicates that the interfacial angles for paralaurionite have been substituted for those of “albrittonite” and casts further doubt upon the crystal drawings of “albrittonite.”

Crook (written communication, 1980) has prepared hand-drawn crystal drawings which are similar to those which he figured in his paper on “albrittonite,” and which he states are his notes relating to measurements of “albrittonite” crystals. On his drawings are morphologic data which he states he measured from “albrittonite” crystals using an optical goniometer. Using the values on Crook’s drawing, which resemble the System figure of liroconite, we calculate the following ratios:

\[
b/(a \sin \beta) = 0.58 \\
(c \sin \beta/b) = 1.31
\]

For comparison, we calculate from the published crystallographic data on “albrittonite” and from equivalent values for liroconite that:

<table>
<thead>
<tr>
<th></th>
<th>Liroconite</th>
<th>&quot;albrittonite&quot;</th>
</tr>
</thead>
<tbody>
<tr>
<td>(b/(a \sin \beta))</td>
<td>0.60</td>
<td>0.60</td>
</tr>
<tr>
<td>((c \sin \beta/b))</td>
<td>1.31</td>
<td>0.93</td>
</tr>
</tbody>
</table>

This shows that the measurements (Crook’s, written communication, 1980) could not possibly be obtained for “albrittonite.” However Crook’s interfacial angles predict axial ratios in agreement with those of liroconite.

A second drawing by Crook (written communication, 1980) resembles the figure in The System of Mineralogy, Volume II (1951) of a paralaurionite crystal. It also has numbers measured by Crook from an “albrittonite” crystal. Comparisons similar to those given above are:

<table>
<thead>
<tr>
<th></th>
<th>Calculated from values on Crook’s drawing</th>
<th>Calculated from data on paralaurionite</th>
<th>Calculated from data on &quot;albrittonite&quot;</th>
</tr>
</thead>
<tbody>
<tr>
<td>(b/(a \sin \beta))</td>
<td>0.40</td>
<td>0.41</td>
<td>0.80</td>
</tr>
<tr>
<td>(b)</td>
<td>117.03°</td>
<td>117.22°</td>
<td>97.25°</td>
</tr>
</tbody>
</table>

Here again the correspondence between Crook’s interfacial angles and those reported in The System of Mineralogy for another unrelated mineral is clear. On the other hand, Crook’s values as given in his notes dealing with “albrittonite” are incompatible with his own unit cell data on “albrittonite.” It therefore appears as if interaxial angles for liroconite and paralaurionite have been used, changed slightly, and reported as values for “albrittonite.”

“Albrittonite” is approximately twice as soluble in water as NaCl (and nickelbischofite, which Crook and Marcotty (1978) state to be associated with “albrittonite,” is seven times as soluble according to Weast, 1980). These minerals are said to occur due to the “influx of water through ground water, rain, etc. causes albrittonite to dissolve readily and to be reprecipitated upon the resumption of arid conditions.” Dr. R. L. Freed (written communication, 1980) of Trinity University, San Antonio, Texas, questions this statement and states that “The Llano region is really not all that arid, receiving approximately 28 inches of rain per year.” The extreme solubility of CoCl₂ · 6H₂O makes the Llano occurrence unlikely although not impossible. However, Dr. Freed notes that he has visited the serpentine quarry reported to be the source of “albrittonite” (and nickelbischofite) in an attempt to collect it and “to search for the vein of cobaltite, linnaeite, siegenite, and nickelene mentioned in Crook and Marcotty (1978). The Llano region is unusual in its lack of sulfide mineralization; the report of primary sulfide and arsenide minerals is noteworthy in its own right.” The latter minerals were reported by Crook and Marcotty (1978) to serve as the primary source of the secondary “albrittonite” and nickelbischofite. Freed (written communication, 1980) notes that “The minerals and the vein were reported from the north side of the quarry and I spent approximately three hours searching this north side. Two other scientists, a mineralogist and geologist, were with me during the search, and they also were unable to find either the primary vein or the new minerals.” Freed also notes that “The Oxford serpentine quarry has been a field trip stop for students and professional geologists for many years. It is amazing that minerals of such distinctive colors have not been reported either prior to 1976, or after 1976.” Crook and Marcotty (1978) reported the occurrence of zaratite, erythrite, and annabergite with "albrittonite" and nickelbischofite and it is to those colorful minerals that Freed refers. They are not readily watersoluble and once formed would not be easily dis-
solved. These relations cast doubt on the validity of the natural occurrence of “albrittonite” (CoCl₂·6H₂O) and nickelbischofite (NiCl₂·6H₂O) at the Oxford Quarry, Llano County, Texas. We note that a quantity of material provided to his co-author, Marcotty (personal communication), is identical in appearance to synthetic CoCl₂·6H₂O and, considering their statements regarding its origin, remarkably free of any inclusions or contaminants. We conclude that the above evidence suggests that the “albrittonite” and nickelbischofite stated to be naturally occurring at Oxford, Texas are instead synthetic material.

Because CoCl₂·6H₂O (“albrittonite”) and NiCl₂·6H₂O (nickelbischofite) are isostructural they may be expected to exhibit mutual solid solution. Crook and Marcotty (1978) and Crook and Jambor (1979) report that they occur together and were derived from the same vein of primary sulfides at the Oxford Quarry. Because his analyses of “albrittonite” average only 0.3% Ni (and nickelbischofite is reported to have only 0.5% Co) it appeared that our prediction of solid solution was at variance with Crook’s analytical data. We therefore prepared solutions of approximately equal amounts of both compounds and allowed the water to slowly evaporate so that crystals were formed. Well-developed, single-phase crystals grew. They were shown to be of an intermediate composition by X-ray diffraction. This demonstrates that extensive mutual solid solution exists between CoCl₂·6H₂O and NiCl₂·6H₂O. The coexistence of nearly pure “albrittonite” and nickelbischofite under the conditions reported is highly unlikely.

The analytical data for “albrittonite” (and nickelbischofite) are themselves subject to question. Because they are of remarkably high accuracy, we have considerable difficulty understanding how this could have been accomplished. First, “albrittonite” is so soluble that during the polishing process it partially dissolves even in alcohol and kerosene. No mention of this difficulty was noted. Marcotty (written communication, 1980) noted a similar difficulty in preparing smears of CoCl₂·6H₂O for X-ray diffraction studies. We obtained a mediocre polish with great difficulty only after dry grinding and polishing. More significant, however, is the fact that in our attempts to duplicate Crook’s microprobe analyses, these samples were found to be highly unstable under the electron beam. Under the conditions stated by them (12 kV, 150 na), we observed it to “boil” and exhibit signs of extensive beam damage the instant the beam is exposed to the sample regardless of the beam width employed in analysis. Accurate quantitative microprobe analysis is simply not possible under these conditions.

Another serious analytical problem concerns the analyses of “albrittonite” for H₂O. The two reported values (45.61 and 45.16 percent) are surprisingly close to the ideal value of 45.43 percent. Crook and Marcotty (1978) state that “water was measured by measuring total weight loss on ignition minus the chlorine content obtained from the probe analysis” (Crook and Marcotty, 1978). This is unlikely according to M. H. Hey, who questions (written communication, 1980) such a statement of procedure in the strongest terms. Hey states that he has carried out the experiment and finds that ignition below approximately 900°C gives a residue of Co₃O₄ which corresponds to a total weight loss of 66.27%, or to a weight loss on ignition minus the Cl content of 36.5%. At approximately 900°C Co₃O₄ loses oxygen and is converted to CoO. Ignition above this temperature results in a total weight loss of 68.5%, which minus chlorine, yields a value of 38.7%. Neither result corresponds to the weight percentages of water (45.61 and 45.16). Hey notes that only if the ignition product is pure cobalt metal would their value for H₂O be correct, “and to get a residue of Co metal it would be necessary for the ignition to take place in a reducing atmosphere—H₂, or possibly CO—and then the metal would very likely be pyrophoric.” We therefore conclude that the analytical data for H₂O reported for “albrittonite” are invalid.

Hey has also pointed out (written communication) that there appears to be a problem with the optical data for “albrittonite” and he states that: “γ - β 0.026, β - α 0.025 is not consistent with 2V 53°” (the values given by Crook and Marcotty.)

In summary we note that the reported crystallographic data, geologic occurrence, and chemical analyses for “albrittonite” are questionable, and do not exist as presented. It is for these reasons that “albrittonite” has been discredited as a valid mineral species.

Nickelbischofite

Nickelbischofite was described by Crook and Jambor (1979). In the preceding section we raised serious questions regarding “albrittonite” data, and at the same time noted that many of those questions
were equally applicable to nickelbischofite. We shall therefore not repeat them here. However, we emphasize that those questions relate only to the occurrence of nickelbischofite at the Oxford serpentinite quarry, Llano County, Texas, and to data reported by Crook and Jambor (1979) from Texas material. Independently reported occurrences of nickelbischofite are those of Shima (1957), who detected a compound which may be NiCl₂·6H₂O in volcanic sulfate-rich sublimates, and Jambor (1975) who noted the mineral on drill core from the Du-mont ultramafic intrusion, Amos, Quebec. Jambor (1975) obtained an excellent X-ray powder pattern for the Amos mineral and noted at that time that it corresponded to NiCl₂·6H₂O. Subsequent confirmation of Ni and Cl in the Amos mineral, as reported in Crook and Jambor (1979), was obtained at the Geological Survey of Canada in 1977, and communications concerning the naming of the compound had been initiated by Jambor with the Chairman, Commission on New Minerals and Mineral Names, I.M.A., well prior to Crook's independent submission of data for NiCl₂·6H₂O. The status of nickelbischofite as a naturally occurring mineral therefore should be retained. Occurrence of a highly soluble mineral such as nickelbischofite is not surprising in drill cores or in volcanic sublimates, unlike the surface exposures at the Oxford quarry. Soluble minerals are less likely to be dissolved inside a solid rock sample. The rapid supersaturation attendant upon volcanic degassing allows transient formation of highly soluble and perhaps unstable minerals. We believe that only the occurrence in Llano County, Texas and data from such material are invalid. Physical properties and X-ray crystallography of nickelbischofite as given in Crook and Jambor (1979) conform with data for synthetic NiCl₂·6H₂O as given by Groth (1906), Kleinberg (1969), Swanson et al. (1974), and Winchell and Winchell (1964) except as follows (Jambor, written communication, 1980): morphological forms, stated as {100}, {100}, {011} should read {100}, {110}, {001}, {201}; natural occurrence of these crystals up to 15 mm long is improbable; pleochroism of α pale green, γ green should be α greenish yellow, γ green; absorption γ > α, not α > γ.

Rowlandite

We refer here to the data relating to rowlandite in a paper given by Crook et al. (1978). The principal conclusion of the paper is that rowlandite has the formula RE₃(SiO₄)₂(F,OH). We are concerned here only with the chemical data which was reported by Crook et al. (1978).

Two chemical analyses of rowlandite are given in Table 2 of Crook et al. (1978). These are similar and sum to 99.11 and 98.51 weight percent, respectively. We have normalized analysis 1 to 2.215 Si (as given in the "calculated formula") and find reasonable agreement for most elements with values in the "calculated formula" except for fluorine. Our value for molar fluorine is 1.85 times as large as that given, and shows the latter to be in serious error, based on the reported weight percent of fluorine. Whereas values for F, Cl, and OH are summed to 0.997 in the paper, they sum to 1.556 according to our calculations, thus suggesting serious error either in the analyses or in the formula obtained from it. If the formula is correct, on the other hand, the summation for the analysis (given the necessary change in fluorine and oxygen) would be unsatisfactory. We note that the same kinds of errors involving fluorine were made by Crook (1977c) for analyses of rare-earth fluorides in his Master's Thesis (Crook, 1977c). This implies a pattern of errors in which the formula content of fluorine is generally that expected for the mineral, yet the elemental weight percent from which the formula was supposedly derived is not in agreement with the formula given. This is the kind of error expected during hand calculation, for instance, of a weight percent analysis from a previously assumed formula, but not the kind of error obtained using the EMPADR VII program which produces a correct molar formula for F from the element weight percent as obtained from a microprobe analysis.

The electron microprobe standards for rare-earth elements Crook has used are subject to question. A statement is made (Crook et al., 1978) that: "Analyzed specimens of gadolinite and yttrialite (Rode Ranch pegmatite, Texas) and synthetic rare-earth salts were used as electron microprobe standards." No mention of the correspondence between specific elements and specific standards is made, as would be proper. Moreover, Crook (written communication, 1979) has indicated that gadolinite was used for the majority of the rare-earth elements. Because gadolinite is subject to metamictization it generally should be a poor probe standard. Indeed, a probe standard of the Rode Ranch gadolinite as prepared by Crook and donated to us by him was examined by us on the electron microprobe. It proved to be
extremely heterogeneous in the rare-earth elements and entirely unsuitable as a probe standard. Results obtained with it would therefore be unacceptable. Furthermore, Crook’s sources of the gadolinite and yttrialite standard analyses are not documented. Lastly, neither gadolinite nor yttrialite contain significant fluorine so we assumed that a rare-earth salt or fluorite was used as a fluorine standard. The nature and source of that standard should have been listed. In fact identical analyses of rowlandite appear in Crook’s thesis where he lists only fluorapatite and fluorite as fluorine standards.

Interferences of rare-earth element emission lines on the electron microprobe are generally serious. Only a brief comment about a general method of dealing with such interferences is given by Crook (1977c) and Crook et al. (1978), and no specific details are given despite their significance. Crook (1977c) notes (p. 32) that the Kα₁ line was used for the element yttrium at 15 kV operating voltage. Interferences notwithstanding, there is a serious problem in that Y Kα₁ radiation has an energy of 14.956 kV. Because a significant overvoltage is required to excite emission lines, Crook’s analytical values for Y are subject to question, especially in view of the fact that Y is a major element in his analyses of rowlandite (29.44 and 26.18% Y₂O₃). We have tuned Y Kα using synthetic Y₂O₃ on the electron microprobe at 0.015 μa and plotted counts vs. operating kilovoltage. It is clear that Y is not efficiently excited below 25 kV and that no counts above background levels are generated below 18 kV. No yttrium Kα X-rays can be excited at all below 17.037 kV (Liebhafsky et al., 1972), yet Crook has claimed (written communication, 1977, 1978) that he was able to generate 48,716 counts above background for yttrium Kα, on Y₂O₃ at 15 kV in an evaluation of interferences he provided for the authors. Crook’s (1977b, 1977c, 1978a, 1978b, 1979) and Crook et al.’s (1978) data for yttrium could not have been measured as claimed and it appears that they were derived without measurements ever actually having been made.

Yttrocrasite

We refer here to the paper on yttrocrasite by Crook (1977b). The analysis given in Crook’s Table 2 and implied to be of yttrocrasite from Clear Creek, is duplicated in Table 18 of Crook (1977c). There the analysis is reported to be of yttrocrasite from the type locality obtained from specimen NMNH #R5031, not from Clear Creek; a second analysis is ascribed to a specimen from Clear Creek. Furthermore, the analysis of yttrocrasite in Crook (1977c) said to be for material from Clear Creek is duplicated by Crook (1976). These assertions seem to be clear inconsistencies.

We have normalized the yttrocrasite analysis given in Crook’s (1977b) Table 1 to 1.838 Ti atoms, the number given as his normalized value. In our Table 2 we compare Crook’s values with those which we calculate from the analysis of his paper. Major differences are apparent, as are apparently random small differences.

Using the lattice parameters stated by Crook (1976) to have been obtained by least-squares refinement, we have calculated d-values (Table 3 of this paper). Our Table 3 also contains the d(Obs) and d(Calc) values given by Crook in his Table 1. There are discrepancies in values of d(Calc) due simply to an interchange in the lattice parameters b and c. Aside from the fact that this is an error which should be noted for the record, it is difficult to understand how Crook’s erroneous values could have occurred since the lattice parameters are based on a least squares refinement.
A more serious problem with the X-ray diffraction data concerns the magnitudes of the unit cell parameters \( a = 12.862, b = 4.810, c = 4.571 \text{Å} \). These were said by Crook (1977b) to have been obtained by least-squares refinement of single-crystal data, such data serving also to identify the space group as \( Pbcn \). All of Crook’s data (chemical, X-ray, etc.) imply that yttrocrasite is a member of the euxenite group. The lattice parameters of typical members of this group are more than ten percent greater than those given for yttrocrasite. Crook notes that “The shortened cell parameters in yttrocrasite must be due to considerable corrugation of the zig-zag chains of \( B \) (Ti) octahedra resulting in an overall contraction of the structure.” Given Crook’s lattice parameters and composition showing 24 \((\text{O,OH})\) per unit cell, each \((\text{O,OH})\) ion occupies an effective volume of 11.8Å³ (Cell volume/24). However, we calculate, assuming an anion radius of 1.3Å, that in an ideally closest-packed array of anions an anion effectively occupies 12.4Å³. This is 0.6 Å³ larger than the value for yttrocrasite, implying that the parameters for yttrocrasite are incorrect. In addition, as cations are substituted into a closest-packed array, expansion generally occurs. In cubic-closest-packed spinel (with sites occupied by Mg and Al) the effective volume per oxygen atom is 16.6Å³ for example. In euxenite it is approximately 17Å³. Lastly, distortions such as the “corrugations” hypothesized by Crook result in increases in effective anion volumes in ideal closest-packed arrays. From these and other points of view, it is clear that the unit cell parameters given by Crook for yttrocrasite lead to a cell volume which is incompatible with the euxenite-group structure and formula. As the other data given in the paper in question appear to show that yttrocrasite is a member of the euxenite group, some or all of the data must be invalid at best, and this must include the X-ray diffraction data. Because the X-ray results are based on least-squares refinement of single-crystal data, and because the parameters so-derived lead to satisfactory indexing of the separate powder diffraction data (with corrections as noted above), the data are contradictory and incompatible. We therefore question the validity of all of the X-ray diffraction data.

The chemical analysis of yttrocrasite given by Crook (1976, 1977b, 1977c) is subject to the same general problems (i.e., interferences, inappropriate standards, etc.) as have been discussed in the case of rowlandite. Again, Crook (1977c) claims analysis of yttrium using \( Y \) Ka at 15 kV which is impossible.

We summarize the data for yttrocrasite by noting that there are serious inconsistencies reported by Crook (1977b) on yttrocrasite, especially concerning X-ray diffraction and analytical data. This raises serious questions regarding the validity of all of the data in the paper in question. Whether or not yttrocrasite is a valid member of the euxenite group therefore still remains to be shown.

"Yttromicrolite"

We refer to the paper by Crook (1979) in which he purports to show that hjelmite should be redefined as "yttromicrolite." We obtained the cotype sample of "yttromicrolite" from the Smithsonian Institution. X-ray examination showed that it was largely amorphous with only two lines near to the principal lines of quartz presumably derived from a small transparent inclusion on one side of the sample. Electron microprobe analysis revealed it to be a mixture of different phases. At least 95 percent of the material contained only Ca and S as elements with \( Z \geq 10 \) and is presumably a calcium sulfate. Contained in it were five micron blebs of heterogeneous niobotantalates, with variable Ca, U, T, Th, and Fe. While difficult to analyze due to their heterogeneity, these appeared to have at least two compositions, one similar to niobian tantalite and one similar to a niobian microlite. The microlite contained variable amounts of uranium, yttrium, titanium, cerium, neodymium, samarium, and thorium but always had \( \text{Ca} \geq \text{Y}, \text{U} > \text{Th} \). Crook did not identify a calcium sulfate, nor did he describe heterogeneous niobotantalates. His method of analyzing yttrium in other papers on yttrian compounds suggests that the "yttromicrolite" analysis is also in
Fig. 1. Morphological development of cuprohydromagnesite. Forms are $b = \{010\}$, $c = \{001\}$, and $x = \{310\}$.

Fig. 2. Copy of Fig. 1 from Oswald and Crook (1979) (on the left) said to be “morphological development of cuprohydromagnesite,” together with a copy of a figure of a pinakiolite crystal (on the right) from Vol. II of The System of Mineralogy.

error. We conclude that the type “yttromicrolite” is an amorphous mixture of calcium sulfate, tanta-lite and heterogeneous microlite. It has been discredited by the I.M.A. Commission on New Minerals and Mineral Names (Kato, written communication).

“Cuprohydromagnesite” and “Cuproartinite”

We refer here to the paper by Oswald and Crook (1979). Figure 1 of the paper is reproduced as part of our Figure 2. It was labelled as “Morphological development of cuprohydromagnesite. Forms are $b = \{010\}$, $c = \{001\}$, and $x = \{310\}$.” We have also reproduced as part of our Figure 2 the crystal drawing of pinakiolite figured on page 325 of Vol. II of The System of Mineralogy (Palache et al., 1951). On page 324 the form indices are given as $b = \{010\}$ and $x = \{310\}$. The similarity in figures is even more remarkable given the significant difference in lattice parameters (and axial ratios) between those reported for “cuprohydromagnesite” and those of drawing of pinakiolite figured on page 325 of Vol. II as being orthorhombic with $a:b:c = 0.8339:1:0.5881$ (Palache et al., 1951) whereas cuprohydromagnesite is reported to be monoclinic with $a = 10.653$, $b = 9.141$, $c = 8.570\AA$ and $\beta = 115^\circ23'$ (Oswald and Crook, 1979) for which the corresponding axial ratio is $a:b:c = 1.165:1:0.938$. There is therefore no correspondence between axial ratios even though the crystal morphologies appear to be identical. This apparent copying of a figure of another mineral without reference, and apparent inaccurate description of so-called “cuprohydromagnesite” crystals in part led us to re-examine other data regarding “cuprohydromagnesite” and “cuproartinite.”

A type specimen with both minerals was deposited by Crook in the Smithsonian Institution (NMNH #114188). Mr. Pete J. Dunn of the Smithsonian Institution has informed us (personal communication) that in his study of this specimen he had been able to detect neither of the minerals in question. His study included the use of Gandolfi photographs. He reported that he did detect X-ray peaks characteristic of nakauriite. We have independently been unable to detect X-ray lines of either “cuprohydro-
values appear to be exceptionally accurate. Oswald tent,” respectively. The reported H2O and CO2 weight loss on ignition minus analyzed CO2 content tubes and acid decomposition” and “by total analyses. However CO2 and H2O were said to be performed thus necessitating electron microprobe analyses. Indeed, the statement is made of both minerals that in small grains and are thoroughly intergrown.

Oswald has confirmed (personal communication, 1980) to be identical to material originally supplied by him to Crook.

Both “cuproartinite” and “cuprohydromagnesite” are described by Oswald and Crook (1979) as occurring as small crystals, sprays, etc. intimately associated with hydromagnesite and artinite. Oswald has confirmed (personal communication, 1980) that these and other associated minerals occur in small grains and are thoroughly intergrown. Indeed, the statement is made of both minerals that the available quantities of both minerals were so small that standard chemical analyses could not be performed thus necessitating electron microprobe analyses. However CO2 and H2O were said to be “measured by gravimetric analysis using absorption tubes and acid decomposition” and “by total weight loss on ignition minus analyzed CO2 content,” respectively. The reported H2O and CO2 values appear to be exceptionally accurate. Oswald has also provided us with copies of Crook’s notes (personal communication, 1980) showing that Crook obtained four separate, complete analyses of cuprohydromagnesite and three of cuproartinite, all of apparently exceptional accuracy. We question that such small amounts of materials which are exceedingly difficult to separate could lead to such accurate analytical values. This is especially significant in that even Oswald’s carefully chosen “best” examples of these minerals turned out to be largely nakauriite, as we explain below. Such samples of nakauriite could not possibly lead to such apparently exceptionally accurate analyses of two other minerals. It is even reported that DTA, TGA, and DTG analyses were carried out and the results appear to be highly accurate (i.e., close to theoretical values). Given the statement that very small quantities of material are available and that it is subject to contamination, these statements of methods of analysis for H2O and CO2 must be invalid.

Data are presented in the paper in question which imply significant solid solution between Cu and Mg. The ion Cu2+ does not generally enter into solid solution with ions such as Mg2+. This is because of the very different coordination requirements of Cu2+, in part related to the well-known Jahn–Teller effect in Cu2+ compounds. It would, nevertheless, be reasonable to expect ordered substitution of Cu2+ for Mg. However the significant solid solution of Cu2+ for Mg as described in the paper in question is difficult to accept as valid.

In view of the above relations we carried out a detailed investigation of the materials and samples kindly provided by Oswald. These included vials of carefully selected material remaining from Oswald’s original qualitative research done in 1972 which had led to his conclusion that this specific material was probably a new mineral. (Indeed, our work shows that it was a new mineral at that time in that Oswald’s research preceded publication of a description of nakauriite). Our X-ray diffraction patterns of this material are in no way related to those of “cuprohydromagnesite” or “cuproartinite,” but they are very similar to those of nakauriite (Suzuki et al., 1976). We obtained Gandolfi patterns for two specimens of “cuprohydromagnesite” and two of “cuproartinite,” said by Oswald to be his best, carefully selected examples of each. All four patterns are nearly identical. They in no way resemble the published patterns (Oswald and Crook, 1979) for “cuprohydromagnesite” and “cuproartinite,” but they are very similar to the pat-
terns reported for nakaurite. Table 4 compares our powder data with that for "cuproartinite" and "cuprohydromagnesite." These data strongly indicated that "cuproartinite" and "cuprohydromagnesite" do not exist as valid species, and that specimens purported to be of them are all nakaurite.

The best of the specimens of so-called "cuprohydromagnesite" consisted largely of rather platy, blue material, thus matching the original description of "cuprohydromagnesite" and appearing to be fibrous. A very small, light-blue, transparent crystal of "nakaurite," both of which are described as being separated from this material. Precession and Weissenberg photographs show that it is monoclinic with lattice parameters $a = 9.62(1)$, $b = 6.231(6)$, $c = 7.857(8)$Å and $\beta = 111.82(7)^\circ$, these final lattice parameters having been obtained by least-squares refinement of powder diffraction data for nakaurite. The latter was obtained using a polycrystalline mount in a Gandolfi camera. The indexed powder pattern is given in Table 4. The indexing is satisfactory for this pattern which was obtained using the best "cuprohydromagnesite" specimen provided by Oswald. This shows definitively that "cuprohydromagnesite" specimens are in fact specimens of nakaurite. The single-crystal data that we obtained for the sample labelled "cuprohydromagnesite" are for nakaurite. Both the unit cell and powder data represent a significant improvement over those of Suzuki et al. (1976).

There are minor differences between powder patterns of different specimens. This is especially true for diffractometer patterns as obtained using samples spread on glass slides as opposed to Gandolfi powder patterns, but we assume that is largely due to preferred orientation effects. We have often observed a line having $d = 7.8$Å in diffractometer patterns, as also noted by Suzuki et al., as being characteristic of Type II nakaurite). Indeed, this line and other 001 reflections appear with much greater intensity in diffractometer patterns, where preferred orientation would naturally produce such a result.

The single-crystal data show that there is a pronounced substructure having $B = 6.23/2$Å. As reflections with $K = 2N + 1$ are exceptionally weak, and as the crystal was very small, we are unable to determine whether or not there is an extinction rule for 0k0 reflections. We were able to conclude that the lattice is primitive, however, and that there is no glide plane. In addition, the crystal was twinned by reflection on {001}.

In summary we conclude that the above data imply that "cuprohydromagnesite" and "cuproartinite" do not exist as they were described by Oswald and Crook (1978). There is no direct evidence for their occurrence, and all specimens of them that have been studied, including the type specimen, have been shown to be nakaurite. We therefore urged that they be discredited. It is especially significant that Oswald was also in agreement (personal communication, 1980) with this reconn-
mendment. He has informed us that in view of our extensive data he can only conclude that “cuproar-tinite” and “cuprohydromagnesite” are not valid species and that all specimens of these materials are apparently nakaurite or a structurally closely related phase. The I.M.A. Commission on New Minerals and Mineral Names has therefore discredited “cuprohydromagnesite” and “cuproar-tinite” as valid minerals (Kato, written communication, 1981).

Sources of published data
We are unable to determine the source of much of Crook’s data as it is presented in the papers discussed above. It is generally acknowledged that the author of a paper will indicate which laboratories and facilities served as the source of data in one of three ways. First, the author’s institutional address may generally be inferred to be the direct source, when no other reference is given. Second, it is common practice to include such references under “acknowledgments” where laboratory facilities other than the author’s own are utilized. Third, and less commonly, reference to specific laboratories, equipment, etc. may be found at appropriate places in the text, especially under sections with titles such as “Procedures.” These or other references to sources of data are generally not clearly defined in the papers in question.

For example, in the paper describing “cuprohydromagnesite” and “cuproar-tinite” we are unable to determine the sources of any of the data, including X-ray, electron microprobe, optical goniometer, DTA, TGA, etc. Although the address of the authors is given as Mobil Oil Corporation, we have determined that at least single-crystal X-ray and electron microprobe equipment was not available through that source. Oswald has informed us that he does not know the sources of these data (personal communication, 1980) and there are no acknowledgments. There are many similar examples in the papers which we have cited above which are much too numerous to list here, but this single example will suffice to illustrate our concern.

It is important that the nature of the laboratories, facilities, etc. used as a source of data be properly identified. Only in this way can the quality of the data be correctly evaluated. This is especially significant in the cases discussed in this paper, in view of the substantial questions about the validity of data whose sources are not documented. The lack of such identification of sources of data by Crook in his published papers is therefore a serious omission.

Recommendations to the I.M.A.
It is not unusual for a mineral to be discredited or to have the status of some species questioned. However, our recommendation to simultaneously discredit five individual, apparently well-defined minerals may be without precedent. This is in part because of the inherent difficulties in characterizing the minerals described herein. It is also because it is difficult to prove, beyond a shadow of a doubt, that a mineral does not exist as a valid species once the cloak of authenticity is granted to it by acceptance by the I.M.A. Commission on New Minerals and Mineral Names. The authors recommend that some revisions be made in current procedures used by the I.M.A. for accreditation of new minerals. We have two such recommendations, each of which could be implemented without causing considerable extra effort. They are:

1. A type sample of each new mineral should be required (rather than recommended) to be deposited in a suitable national collection, such as the Smithsonian Institution. This sample should be clearly in matrix in order that synthetic materials might be guarded against. It should be large enough to act as a source for duplicating the determination of characteristic properties. That is, there should be sufficient material to provide electron microprobe and X-ray diffraction data, at the very least. If so little of a proposed mineral exists that this cannot be done, then the mineral should receive species accreditation only under the most unusual circumstances.

2. The completed paper, preparatory for publication, should be submitted in its entirety to the I.M.A. Commission, not just an abstract. This should be studied by at least selected members of the commission while taking into consideration both the large amount of work expended by commission members and the problem of translating a full paper into many languages. This simultaneously solves many problems: the article once evaluated by the I.M.A. Commission might actually appear faster, eliminating the present, not uncommon cases where a mineral is accredited but is not described in press for several years. More importantly, the complete paper is crucial for a fully informed evaluation of the mineral species. An abstract alone may contain only a formula and only a very brief description of the methods of analysis or source of the laboratory. The X-ray data presented in an abstract may have insufficient detail to allow proper inferences to be made. A complete manuscript should
help prevent invalid materials being given species accreditation.

We believe that these two recommendations will considerably reduce the number of discreditations necessary in the future. They are simple and do not require extensive modifications in current procedures. No system will ever be absolutely perfect, but where procedures are easily implemented, and where they may have a significant, positive effect, their implementation should be seriously considered. We emphasize that these are only our own personal recommendations and that they have not been considered by the I.M.A. Commission on New Minerals and Mineral Names.

Note added in proof. A sample of nakauriite kindly provided by Dr. J. Suzuki was studied on the electron microprobe. It contains little or no S (as is true of specimens from Gabbs) and thus the formula of Suzuki et al. may be in error, in part.

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