Bergvesenet

Postboks 3021, N-7441 Trondheim

Rapportarkivet

Bergvesenet rapport nr	Intern Journal nr	Internt arkiv nr	Rapport lokalisering	Gradering
5702				

Kommer fra ..arkiv	Ekstern rapport nr	Oversendt fra	Fortrolig pga	Fortrolig fra dato:
AS Megon	Hazen research inc.	Elkem AS		

Tittel

Determination of Quantitative rare eart mineralogy of core no.2 from the Fen deposit

Forfatter	Dato	År	Bedrift (oppdragsgiver og/eller oppdragstaker)
Hazen research, inc.	12.5.	1971	AS Megon

Kommune	Fylke	Bergdistrikt	1: 50 000 kartblad	1: 250 000 kartblad
Nome	Telemark		17134	Skien

Fagområde	Dokument type	Forekomster (forekomst, gruvefelt, undersøkelsesfelt)
Geologi		Fensfeltet

Råstoffgruppe	Råstofftype
Malm/metall	RE

Sammendrag, innholdsfortegnelse eller innholdsbeskrivelse

I 1970 ble Hazen research engasjert av AS Megon for å foreta en mineralogisk studie av prover fra borhull DBH F-2 fra Fensfeltet. Den primære oppgaven var å bestemme kvantiteten av mineralogien hvor sjeldne jordartselementer inngår, med spesiell vekt på Yttrium og Epropium.

Det beskrives syreopplosning av provene og parallellt nedknusing av for å fastslå den beste metoden for å finne innholdet av yttrium og europium . Rontenanalyse av opploste prover og nedknuste prover viser at forholdet mellom La, Ce og Y ikke er konstant. Resultatet indikerer at Y kanskje ikke forekommer i fluorkarbonater som antatt. Mistanken bekreftes ved å undersoke polerslip som viser at Y forekommer sammen med Niob og ikkr i fluorkarbonatmineralene.

Det er videre utfort omfattende undersokelser med mikroskopering av tynnslip og ronkenanalyse i forskjellige fraksjoner og med forskjellige gangbergarter.

DETERMINATION OF QUANTITATIVE RARE EARTH MINERALOGY OF DRILL CORE NO. 2 FROM THE FEN DEPOSIT

FOR

A/S MEGON

METAL EXTRACTOR GROUP OF NORWAY

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DETERMINATION OF QUANTITATIVE RARE EARTH MINERALOGY OF DRILL CORE NO. 2 FROM THE FEN DEPOSIT

for

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INTRODUCTION AND SUMMARY

On December 4, 1970, Hazen Research, Inc. was engaged by A/S Megon to undertake a mineralogical study of a composite sample prepared from drill core DBH F-2 from the Fen deposit. The primary objective of this study was to determine the quantitative rare earth mineralogy with particular emphasis on the distribution of yttrium and europium. The method chosen for the achievement of this objective was selective leaching of the gangue minerals, at a coarse particle size, in order to obtain a quantitative recovery of the rare earth minerals so that the rare earth distribution and the natural particle size range could be determined. The feasibility of this approach was based on the assumption that yttrium and europium probably occurred associated with the fluo-carbonate minerals. That the yttrium might occur with synchisite was indicated by the similarity of the optical properties of hand picked synchisite crystals with yttrosynchisite. Because bastnäsite is rather insoluble in acids, it was further assumed that these fluocarbonate minerals would also be relatively insoluble in acids.

Before proceeding with selective leaching of the ore, it was necessary to establish the conditions under which dolomite, the major constituent of the composite, would dissolve completely with the least effect on the rare earth minerals. In a series of chemical dissolution tests using a wide variety of organic acids, diluted mineral acids, and complexing agents, it was found that under identical conditions, 5% formic acid will dissolve 100% dolomite and only 2-2.5% bastnäsite (Madagascar material). It was also determined that concentrated HF will dissolve only 2.8% bastnäsite in the same length of time that most silicates and iron oxides will dissolve.

Based on these promising results, percolation leaches were conducted on minus 6-mesh Fen ore. X-ray fluorescence analyses of the insoluble residue showed, however, that significant amounts of the rare earths had dissolved. Yttrium, particularly, showed extractions of over 50%.

Physical separations were made simultaneously with the acid leaches to ascertain the mode of occurrence of yttrium and europium and to provide an alternate means of determining the distribution and quantitative rare earth mineralogy should the acid leach approach be unsuccessful. X-ray fluorescence analyses of the various separation products revealed that the ratio between La, Ce, and Y is not constant. These results aroused the suspicion that yttrium might not occur associated with the fluocarbonates after all. The suspicion was strengthened by preliminary electron microprobe analyses of polished sections which showed yttrium occurring with niobium and, indeed, not in the fluocarbonate minerals. This, of course, changed the whole aspect of the investigation and forced the emphasis to be placed on the determination of the mode of occurrence of the yttrium since obviously it is necessary to know first in which mineral the yttrium occurs before the quantity of that mineral can be determined. Therefore, another series of mineral separations employing gravity, low and high intensity magnetic separation, and flotation, was conducted on a larger sample. These separations produced ≈50% concentrates of the fluocarbonate minerals and a fraction in which the yttrium was upgraded from 0.026% in the feed material to 0.19%. However, because the ore is very fine grained and the minerals finely interlocked, none of the mineral fractions were sufficiently pure to be suitable for quantitative determination of the rare earth mineralogy. Nevertheless, certain conclusions can be drawn from the work so far accomplished:

1. Yttrium has at least two modes of occurrence. The first is with niobium as the mineral kobeite $(Y,Fe,U)(Ti,Nb,Ta)_2(O,OH)_6$. The second is in xenotime $Y(PO_4)$. The occurrence of fergusonite $(YNbO_4)$ is

doubtful; the material described in Progress Report No. 2 is probably kobeite. Electron probe analyses indicate that kobeite is responsible for the majority of the yttrium in the ore. The identification of kobeite is based on individual X-ray diffraction patterns of two-minute handpicked single particles which were located by electron probe scanning techniques. These scans were made on polished sections of the -100 +400 mesh "yttrium concentrate" containing 0.19% Y.

The two particles which were located and extracted for the diffraction work contained about 10% Y and >10% Nb. Other elements occurring in minor to trace amounts were: Sm, Nd, Dy, Gd, Ti, and P. Europium was looked for but could not be detected.

The kobeite occurs as essentially isotropic (probably partially metamict) yellowish to brown particles. Generally it is finely admixed with gangue constituents. So far kobeite is known from very few localities and apparently is always associated with granitic rocks.

The presence of xenotime was also determined by X-ray diffraction of a single particle which analyzed about 25% Y by electron probe analysis.

2. By means of X-ray diffraction studies of pure-appearing, handpicked particles from the crude "fluocarbonate" concentrates, (-100 +400M), bastnäsite, parisite, monazite, and a synchisite-like mineral were identified. The X-ray pattern of the latter mineral resembles synchisite except that one major line is missing and another is very weak. Because of this discrepancy, no positive identification has been made. However, for lack of a more accurate term, this mineral will be called "synchisite" in this report.

In an attempt to separate the bulk fluocarbonate concentrate into its component minerals for determination of the rare earch distribution, crude "synchisite-parisite" and "bastnäsite-monazite" concentrates were produced by heavy liquid separation at sp. gr. 4.05. Because of the presence of much inseparable barite, the concentrates were separately dissolved (Na_2CO_3 fusion); the rare earths separated from the Ca and the bulk of the Ba by precipitation at pH 2 as oxalates; ignited to oxides; and analyzed by X-ray fluorescence. These analyses showed about 80% total rare earths but no europium in either concentrate. With the detection limit of the instrument at about 50 ppm, it must be concluded that the rare earth fluocarbonate minerals and the monazite have a europium content of less than 50 ppm. Furthermore, europium was never detected in any of the numerous rare earth scans performed on the products of the mineral separations. Thus, the occurrence of the europium is still a mystery.

3. By comparison of the Y/Nb ratios, it appears that a significant amount of the niobium is associated with yttrium in the mineral kobeite, however, since a direct correlation does not exist, other niobium minerals must be present. Some of the niobium may occur in what appears to be fine grained niobium-bearing rutile. Pyrochlore, which is reported to occur in the Fen deposit, although it has not been definitely identified in this study, may account for some of the niobium also.

4. From consideration of the overall study, but particularly from analyses of the mineral separation products and from microscopic studies, it has become clear that conventional ore dressing methods, including flotation, should give very poor recoveries and grades of the rare earth minerals. The main reason for this is the fine grained nature of the ore minerals which at a particle size suitable for flotation, are still badly locked. In addition, the main source of the yttrium is probably a very fine grained kobeite whose flotation properties are unknown.

The following comparison shows the poor liberation of rare earth minerals from some of the gangue minerals at different particle sizes:

Sample	Particle Size Range	Rare Earth Content La	Ce	Y
Dolomite-Float @ sp. gr. 3.17	- 35 +400 Mesh	0.34	0.67	0.020
Dolomite-Float @ sp. gr. 3.17	-100 +400 Mesh	0.17	0.77	0.015
Dolomite-Float @ sp. gr. 3.17	-400M +10 Microns	0.35	0.78	0.022
Celsian Feldspar Float @ sp. gr. 3.8	- 35 +400 Mesh	3.3	3.6	0.054
Celsian Feldspar Float @ sp. gr. 3.8	-400M +10 Microns	0.51	1.0	0.032
Magnetite Sink @ sp. gr. 3.17	- 35 +400 Mesh	2.2	2.2	0.025
Magnetite Sink @ sp. gr. 3.17	-400M +10 Microns	0.51	0.78	0.032

It was determined microscopically that the rare earth content of these gangue minerals is primarily due to locked R.E. minerals. Because of this locking problem, it would be very difficult to establish to what extent the rare earths might be in substitution for other elements in the gangue minerals.

In view of the overall findings, only a few extractive metallurgical processes present any possibility of successful recovery of the values. For example, it might be possible by means of selective flocculation to flocculate and reject dolomite at a very fine particle size; or through hydrometallurgy, the rare earths could perhaps be selectively dissolved by certain organic acids or complexing agents. Any such approach would require considerable basic research inasmuch as there is little known about such processes except in very specific applications.

MINERAL SEPARATIONS AND ANALYSES

In Progress Report No. 3, a flowsheet was presented showing the type of mineral separation carried out in order to produce sufficient amounts of fluocarbonate minerals for determination of the rare earth content and the rare earth distribution in these minerals. It was also necessary to produce an yttrium concentrate for further analysis. The yttrium and fluocarbonate concentrates were analyzed by X-ray fluorescence. The results are given in Tables 5 and 8 in the next section of this report. In addition to these analyses, the more important gangue mineral products were also analyzed by X-ray fluorescence in order to see the effect of decrease of particle size on liberation of rare earth minerals from gangue minerals.

As had been found in previous separations, yttrium was concentrated again to a maximum of about 0.2% in the -100 +400 mesh, sp. gr. >3.8, 0.45 amps magnetic fraction. Because of the fine grain size of the yttrium mineral, it was anticipated that the highest concentration of yttrium would occur in the -400 mesh (38 μ) +10 μ range. This, however, was not the case, probably because of the difficulty of making clean cut separations at such fine particle size even with increased liberation of discrete metals.

Only the yttrium distribution in the various mineral separation products has been calculated. These calculations show that nearly 60% of the yttrium still occurs in the dolomite float products, yet about 17% occurs in the minus 10-micron slimes. The balance of the yttrium is more or less evenly distributed throughout the other fractions. The details of these data are summarized in Table 1. The designations of the fractions listed under "Test Product" in the table correspond to designations used in the flowsheet in Progress Report No. 3. For convenience, the mineral fractions produced from each size range are grouped together. The complete X-ray fluorescence scans of the dolomite, celsian feldspar, magnetite, and barite products, as well as the -10μ slimes, are given in Tables 2-4. These are arranged in the sequence established in Table 1.

The tables show that every product is infested with rare earths. This poor distribution is due to the complex locking of almost all of the mineral constituents. This is particularly apparent in the celsian and magnetite products. The photomicrographs, Figures 1 through 5, illustrate this locking problem.

Table 1

Distribution of Yttrium in the Various Mineral Separation Products
(See flowsheet of Progress Report No. 3)

Test Product	Weight g	Weight %	Assay % y	Content g y	Distribution % y
-35 +400 Mesh Products					
Dolomite and Calcite *	625.4	48.85	0.015	0.0938	31.42
Float @ sp. gr. 3.17					
Celsian Feldspar	55.9	4.37	0.054	0.0302	10.12
Float @ sp. gr. 3.8					
Magnetite-sink	62.3	4.87	0.025	0.0156	5.23
@ sp. gr. 3.17					
-35 +400 Mesh Products					
of sink @ sp. gr. 3.8					
Mag @ 0.25A	1.2	0.09	0.066	0.0008	0.27
Mag @ 0.45A	4.3	0.34	0.21	0.0090	3.02
Mag @ 0.9A	0.9	0.07	0.11	0.0010	0.33
Float @ sp. gr. 4.05					
Mag @ 0.9A	0.9	0.07	0.13	0.0012	0.40
Sink @ sp. gr. 4.05					
Non-mag @ 0.9A	1.2	0.09	0.005	0.0001	0.03
-100 +400 Mesh Products					
of sink @ sp. gr. 3.8					
Mag @ 0.25A	5.2	0.41	0.055	0.0029	0.97
Mag @ 0.45A	0.9	0.07	0.12	0.0011	0.37
Float @ sp. gr. 4.05					
Mag @ 0.45A	0.6	0.05	0.19	0.0011	0.37
Sink @ sp. gr. 4.05					
Mag @ 0.9A	0.5	0.04	0.13	0.0007	0.23
Float @ sp. gr. 4.05					
Mag @ 0.9A	0.8	0.06	0.091	0.0073	2.44
Sink @ sp. gr. 4.05					
Non-mag @ 0.9A	2.9	0.23	0.007	0.0002	0.07
-400 +10μ Products					
Dolomite and calcite	356.6	27.86	0.022	0.0785	26.30
Float @ sp. gr. 3.17					
Celsian feldspar	2.9	0.23	0.032	0.0009	0.30
Float @ sp. gr. 3.8					
Magnetite-sink @	4.7	0.37	0.032	0.0015	0.50
sp. gr. 3.17					
Sink @ sp. gr. 3.8					
Mag @ 0.25A	3.0	0.23	0.035	0.0011	0.37
Mag @ 0.45A	0.7	0.05	0.11	0.0008	0.27
Mag @ 0.9A	0.4	0.03	0.12	0.0005	0.17
Non-mag @ 0.9A	2.5	0.19	0.018	0.0005	0.17
-10 μ Slimes	146.3	11.43	0.034	0.0497	16.65
Calculated Head	1280.1	100.0	0.0233	0.2985	100.0
Assayed Head			0.026	0.333	

*The +100 Mesh dolomite-calcite product was reground to -100 mesh in order to liberate more heavy minerals.

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Table 2

Semiquantitative X-ray Fluorescence Analyses of Gangue Minerals

Dolomite and Calcite Float 3.17-100 + 400 M		Celsian Feldspar Float 3.8-35 + 400 M		Magnetite Sink 3.17-35 + 400 M	
Cu	0.011	Cu	0.010	Cu	0.028
Au	0.009	Zn	0.050	Zn	0.045
Zn	0.026	Tl	0.060	Pb	0.024
Pb	0.006	Pb	0.010	Fe	18.0
Fe	4.6	Se	0.007	Ni	0.015
Ni	0.006	Fe	4.4	Ba	6.9
Ba	1.8	Ni	0.005	Sr	0.078
Sr	0.28	Ba	12.0	Ti	0.24
Ti	0.033	Sr	0.12	Zr	0.022
Th	0.045	Ti	0.48	Th	0.15
Nb	0.024	Th	0.20	Nb	0.11
Mn	1.1	Nb	0.13	Mn	0.36
La	0.17	Mn	0.46	La	2.2
Ce	0.77	La	3.3	Ce	2.2
Pr	0.072	Ce	3.6	Pr	0.22
Nd	0.15	Pr	0.22	Nd	0.63
Y	0.015	Nd	1.0	Sm	0.20
		Sm	0.20	Y	0.025
		Y	0.054		

Table 3

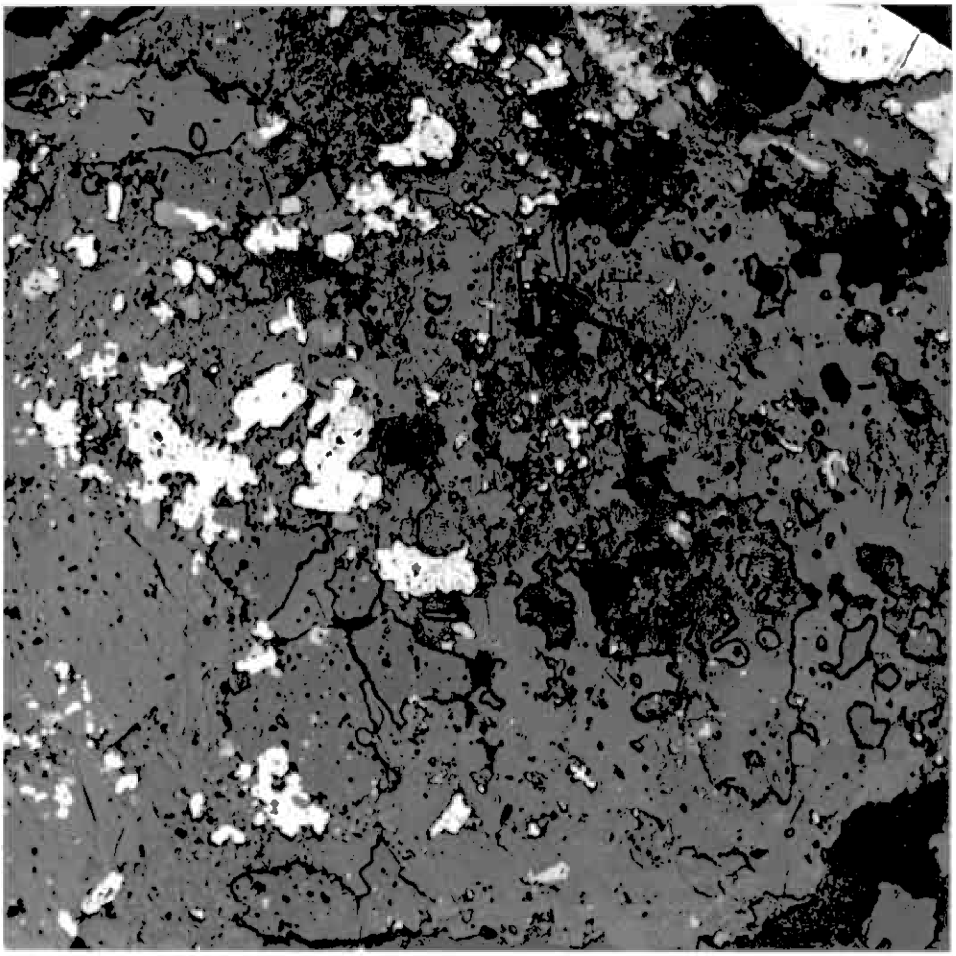
Semicuantitative X-ray Fluorescence Analyses of Gangue Minerals

Dolomite and Calcite Float 3.17 -400M +10 u		Celsian Feldspar Float 3.8 -400M +10 u		Magnetite Sink 3.17 -400M +10u	
Cu	0.007	Cu	0.019	Cu	0.015
Zn	0.029	Zn	0.082	Zn	0.050
Sn	0.004	Tl	0.017	Pb	0.048
Pb	0.019	Pb	0.033	Fe	35.0
Fe	4.5	Fe	5.2	Ni	0.013
Ni	0.006	Ni	0.011	Ba	4.8
Ba	2.8	Ba	3.2	Sr	0.043
Sr	0.23	Sr	0.25	Ti	0.36
Ti	0.18	Ti	0.20	Zr	0.032
Th	0.054	Zr	0.018	Th	0.20
Nb	0.036	Th	0.099	Nb	0.076
Mn	0.93	Nb	0.12	Mn	0.15
La	0.35	Mn	0.85	La	0.51
Ce	0.78	La	0.51	Ce	0.78
Pr	0.072	Ce	1.0	Nd	0.30
Nd	0.22	Nd	0.26	Y	0.032
Sm	0.086	Y	0.032		
Y	0.022				

Table 4

Semiquantitative X-ray Fluorescence Analyses of Gangue Minerals

Barite Sink 3.8 Non-magnetic at 0.9A -100 + 400 M		Slimes -10 Microns	
Cu	0.006	Cu	0.023
Zn	0.047	Zn	0.043
Fe	1.3	Sn	0.009
Co	0.007	Pb	0.043
Ni	0.005	Fe	4.8
Ba	37.0	Ni	0.006
Sr	0.24	Ba	2.8
Mn	0.018	Sr	0.22
Y	0.007	Ti	0.099
		Zr	0.018
		Th	0.13
		Nb	0.082
		U	0.012
		Mn	0.79
		La	0.86
		Ce	1.3
		Pr	0.072
		Nd	0.37
		Sm	0.098
		Y	0.034

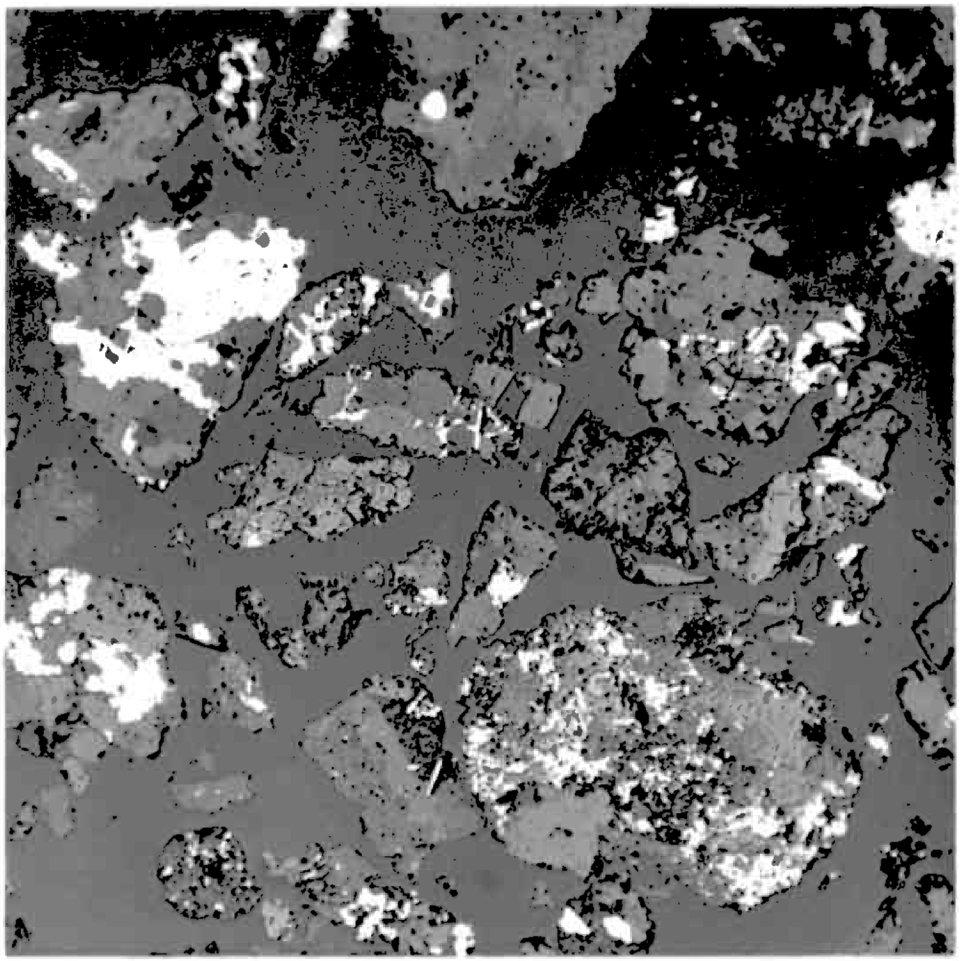


Head Sample

Polished section showing large grain consisting of an interlocked aggregate of carbonates and fluocarbonates (various shades of grey, general matrix), celsian feldspar (grey, but showing black relief), iron oxides and pyrite (white).

Plain incident light. Dry objective. 240X

Figure 1



Celsian Feldspar-Float at sp. gr. 3.8
-35 +100 Mesh

Polished section showing grains consisting of intimately admixed celsian feldspar, carbonates, fluocarbonates (all various shades of grey) and iron oxides (white).

Plain incident light. Dry objective. 85X

Figure 2

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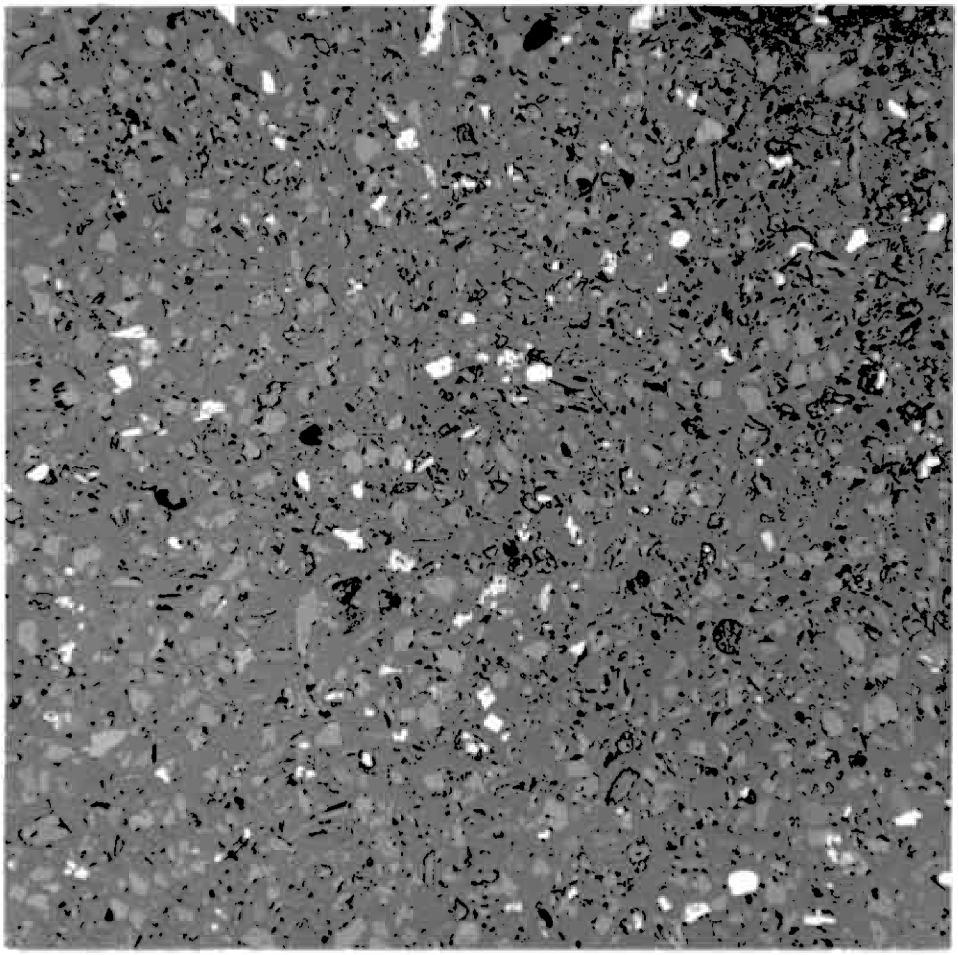
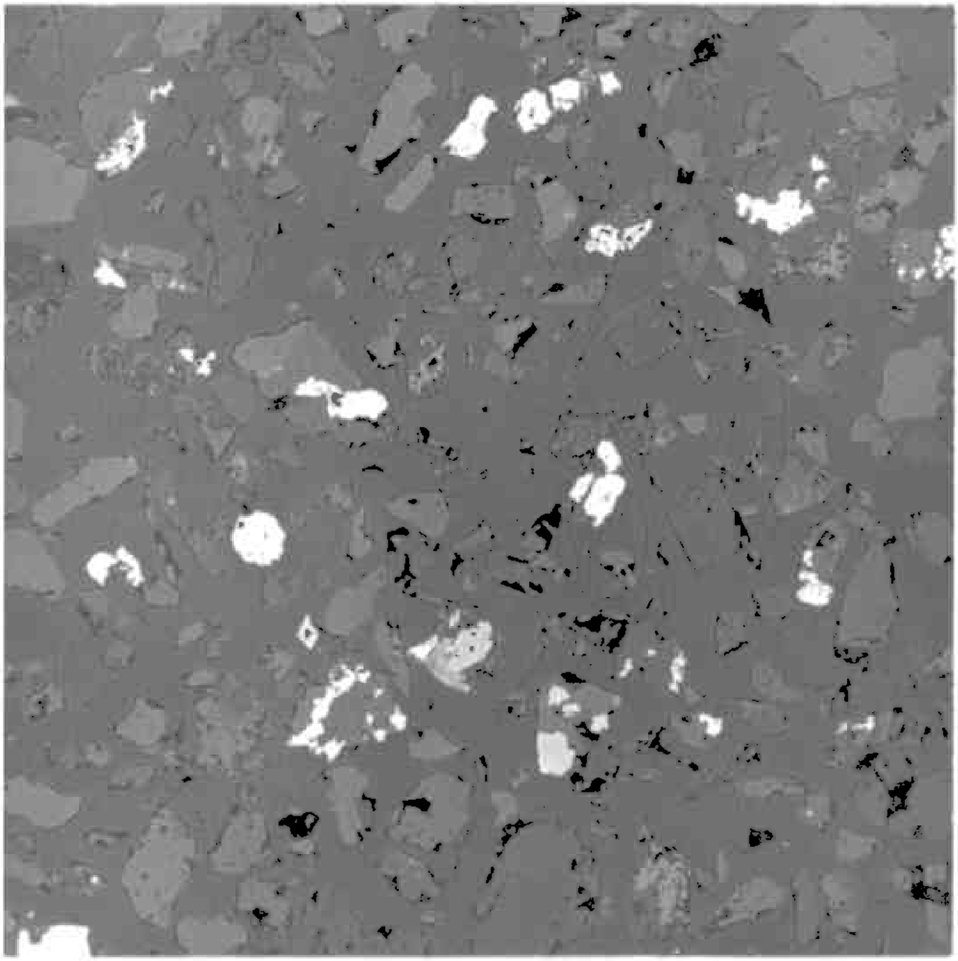


Celsian Feldspar-Float at sp. gr. 3.8
-400 Mesh +10 Microns

Grain mount still showing considerable locking even at this fine particle size. Celsian, carbonate, fluocarbonates and barite are various shades of grey. Iron oxides are white.

Plain incident light. Dry objective. 85X

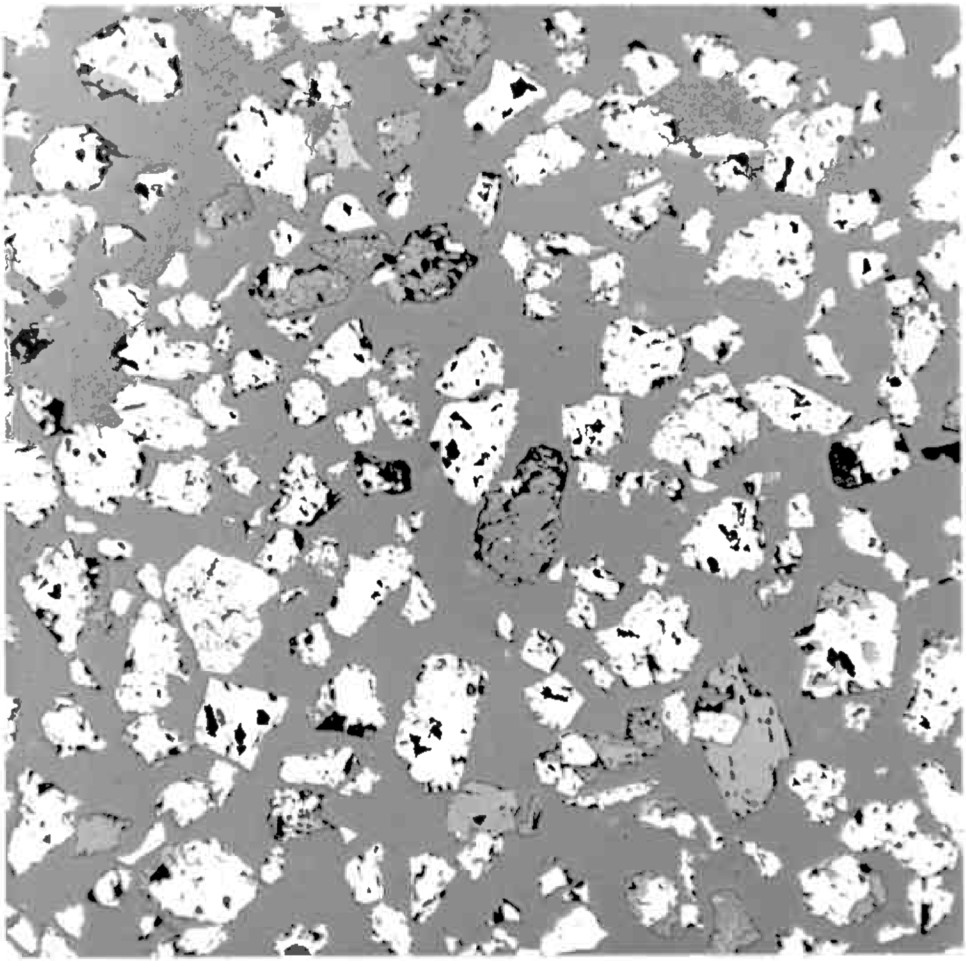
Figure 3



Same sample as given in Figure 3, except at higher magnification to show that most of the iron oxides (white) are interlocked with the celsian and other low reflecting minerals.

Plain incident light. Dry objective. 300X

Figure 4



Magnetite-Sink at sp. gr. 3.17
-100 +400 Mesh

Polished section of magnetite product showing mostly free magnetite (white) but also many intergrowths and inclusions in carbonates, fluocarbonates, etc. (medium grey). Inclusions of this type are responsible for the unsharp separations which have occurred in all the magnetic separations.

Plain incident light. Dry objective. 100X

Figure 5

MINERALOGICAL INVESTIGATION

The mineralogical examinations were confined to establishing the identity of the yttrium mineral and to further defining the nature of the fluocarbonate minerals, particularly with respect to their europium content. By means of electron microprobe analyses three particles rich in yttrium were found in polished sections prepared from the yttrium "concentrate" containing 0.19% Y (magnetic at 0.45A, sink at sp. gr. 4.05, -100 +400 M). For a complete X-ray fluorescence scan of this sample see Table 5. The first two particles contained about 10% Y and >10% Nb while the third particle contained about 25% Y (Nb was not measured). After these particles had been located and marked by the electron beam, they were carefully extracted and mounted on a glass fiber for X-ray diffraction (powder camera) identification. The patterns of the first two particles correspond to the pattern of kobeite (ASTM 11-0259)[1/]. One pattern is quite good and the other is poor. The third particle gave an excellent pattern with a virtually perfect match to xenotime (ASTM 11-254 from Shelby, North Carolina, and ASTM 9-377, synthetic). Approximately one-third of the lines in the sample pattern do not match xenotime but these are all weak to extremely weak in intensity and should represent impurities.

Figures 6, 7, and 8 are photomicrographs of the particles analyzed with the electron probe and by X-ray diffraction. Copies of the X-ray patterns obtained on kobeite particle No. 2 and on the xenotime particle are attached in Figure 9. A listing of the measured d-spacings of these patterns and the kobeite particle No. 1 pattern is given in Tables 6 and 7.

Since yttrium and europium were originally expected to be found in the fluocarbonates a "concentrate" of these minerals was made which

1/ Hutton, C.O., Kobeite from Paringa River, South Westland, New Zealand. Am. Min. 42, 342, 1957.
In this article the author also gives data on the original kobeite from Japan.

was reported as "synchisite" concentrate in Progress Report No. 2. A complete X-ray fluorescence analysis of this "concentrate" showed about 25% total rare earths but no europium. However, it was found that barite occurred as a major contaminant in the sample and could be a potential diluent to the europium should it be there. It was also found (and reported in Progress Report No. 3) that this "synchisite" concentrate was really a mixture of several fluocarbonates and monazite thus making the true source of the europium ever more nebulous.

Because of these findings it was necessary to produce a second and larger bulk fluocarbonate concentrate from which barite could be removed and the individual rare earth minerals separated. The next step was to separate the "synchisite" (sp. gr. 3.9) from the heavier bastnäsite, monazite, and barite by heavy liquid separation at a specific gravity of 4.05. However, microscopic examination of the float product still revealed significant amounts of barite. Removal of barite by chemical methods was then tested using small portions of these two heavy liquid products. Complexing of the barite with 10% EDTA + 2% NaOH as well as metathesis with Na_2CO_3 (100 g/l) were investigated in several tests. Both of these methods were successful in removing barite but microscopic examination showed that the rare earth minerals had been severely attacked; therefore these approaches were abandoned. Several fatty acid flotation tests also failed to remove the barite sufficiently. Since these methods were unsuccessful, the last resort was to dissolve the concentrates and coprecipitate the rare earths for analyses. As a precaution, the two concentrates were analyzed by X-ray fluorescence prior to dissolution. Also, from each concentrate,a portion of what appeared to be pure rare earth minerals was handpicked under the microscope for X-ray diffraction studies.

In general, the analytical procedure was as follows: the sample was decomposed by Na_2CO_3 fusion; the fusion melt was water leached and SO_4 and soluble P_2O_5 were determined on the filtrates. The carbonate residue was digested and fumed with $HClO_4$ and diluted to volume. After adjusting an aliquot to pH 2, the rare earths were precipitated as oxalates. The oxalates were ignited to oxides and then analyzed by X-ray fluorescence. Aliquots were also taken for determination of the balance of the P_2O_5 and for Ca.

The results of these analyses and the mineral composition calculated from them for the two crude concentrates are:

Float Product at sp. gr. 4.05

% RE_2O_3	30.7	% Fluocarbonates	62.4
% SO_4	11.2	% Barite	27.3
% P_2O_5	3.12	% Monazite	10.3
% CaO	6.85	(belongs to fluocarbonates)	

Sink Product at sp. gr. 4.05

% RE_2O_3	37.8	% Fluocarbonates	30.3
% SO_4	18.5	% Barite	44.9
% P_2O_5	7.45	% Monazite	24.8
% CaO	3.14	(belongs to fluocarbonates)	

The above calculations do not include other impurities such as iron oxides. The barite determinations are based on SO_4 assays rather than Ba assays because of celsian feldspar which is undoubtedly present in the float fraction. The fluocarbonate values were calculated by difference after the barite and monazite values were established. The RE_2O_3 cannot be used to calculate the fluocarbonate values because of the presence of monazite. The monazite contents were calculated from the P_2O_5 assays assuming a 30.27% P_2O_5 content in pure monazite.

The X-ray fluorescence scans on the crude float and sink concentrates are given in Table 8. The analysis of the rare earth oxide precipitate is given in Table 9. In Table 10 the calculated content and distribution of the rare earths in the R.E. minerals occurring in the float and sink products is given. The latter values are based on the analysis of the rare earth oxide precipitate after adjusting for the barite only, which was determined by chemical assay.

From Table 10, it can be seen that the rare earth minerals in the float product contain 0.26% Y and in the sink product 0.34% Y. This, however, does not mean that this amount of yttrium is necessarily chemically associated with the fluocarbonates and/or monazite. As was indicated in Progress Report No. 2, the yttrium mineral concentrates along with the fluocarbonate minerals to a certain extent. The tables also show that europium is absent from the two concentrates. With a detection limit of about 50 ppm, it is concluded that the europium content must therefore be less than 50 ppm.

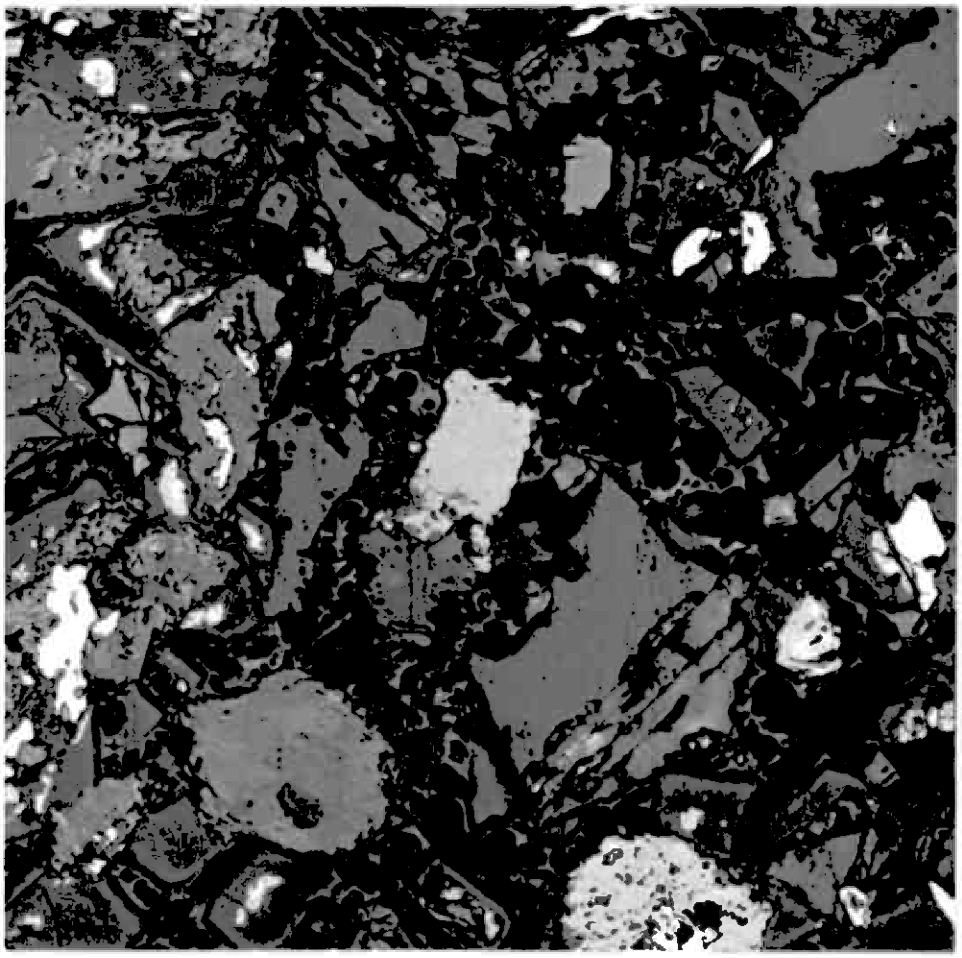
X-ray diffraction patterns were made of homogeneous appearing fluocarbonate crystals which were handpicked from the float and sink products before they were dissolved for assay. Using a special sample preparation technique, it was possible to obtain patterns with the diffractometer. These patterns showed that the float product consists primarily of "synchisite", parisite, barite, and minor amounts of bastnäsite and monazite, and the sink product of bastnäsite, monazite, barite, and minor amounts of synchisite and parisite. The fact that these crystals appeared homgeneous under the microscope and that minerals of high specific gravity such as bastnäsite are present in the float, while those of low specific gravity, such as "synchisite," are in the sink, indicates how intimately these minerals are intergrown with each other.

The "synchisite" pattern resembles the published synchisite pattern (ASTM 18-284, Predazzo, Italy) except for the absence of a 50 R.I. line and the faintness of one of the 100 R.I. lines. This makes the identification as synchisite uncertain. For comparison, a pattern of the "synchisite" crystals designated "No. 1 bastnåsite group minerals" (from A/S Megon)[1/], was also run. This pattern also corresponds with the published synchisite pattern except for the absence of the same 50 R.I. line and the low intensity of the 100 R.I. line.

Photomicrographs of the float and sink products are shown in Figures 10 and 11 to illustrate the type of material used for all of these determinations. The three described X-ray patterns will be found in a pocket in the back of this report.

During the examination of polished sections a tentative identification of a very fine grained rutile has been made. The greenish internal reflections of this rutile suggest that it might be Nb bearing. Figure 12 is a photomicrograph of this rutile.

1/ This is the sample received from Mr. O. Braaten during his visit to Hazen Research in October 1970.

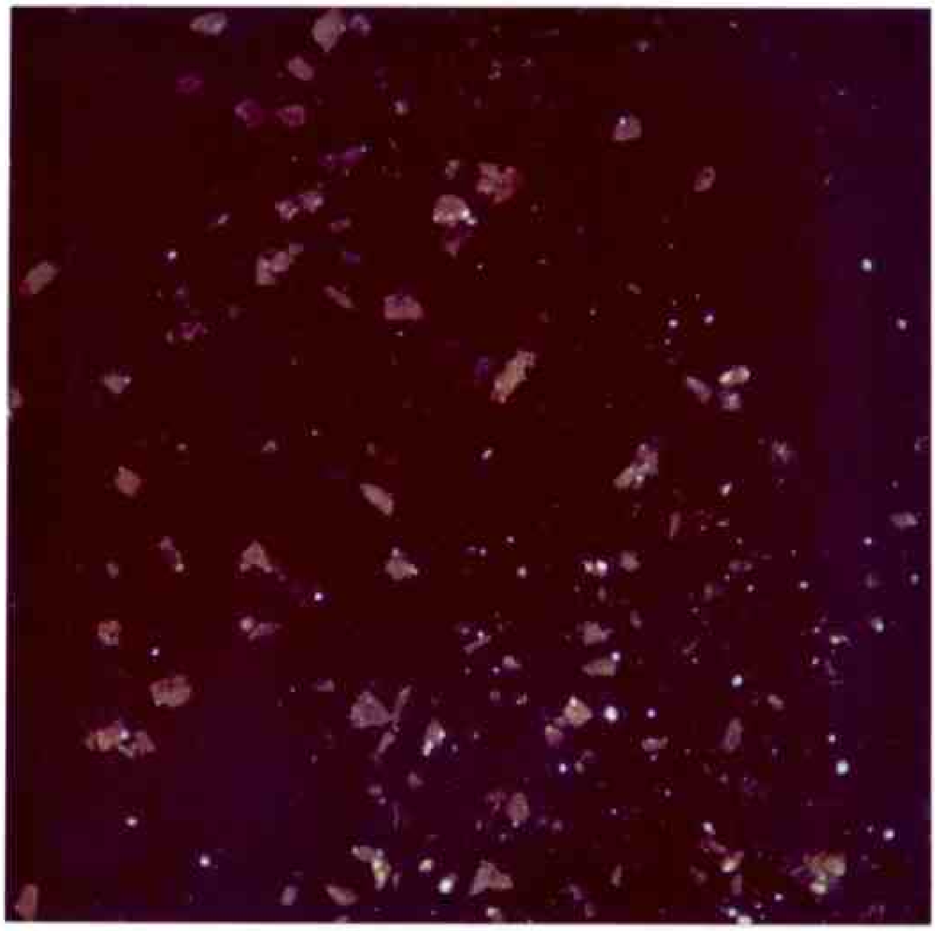


"Yttrium Concentrate"
Magnetic at 0.45 amps. Sink at 4.05 sp. gr.
-100 +400 Mesh

Polished section showing actual particle of kobeite (Particle No. 1 center, light grey) located by electron microprobe analysis. This particle which contained about 10% Y and >10% Nb, was removed from this section, mounted on a spindle and analyzed by x-ray diffraction. The resulting pattern, although of poor quality, corresponded reasonably well with the published pattern for kobeite. (ASTM 11-259) The dark lines and holes in the above photo are the tracks of the scanning electron beam.

Plain incident light. Dry objective. 290X

Figure 6



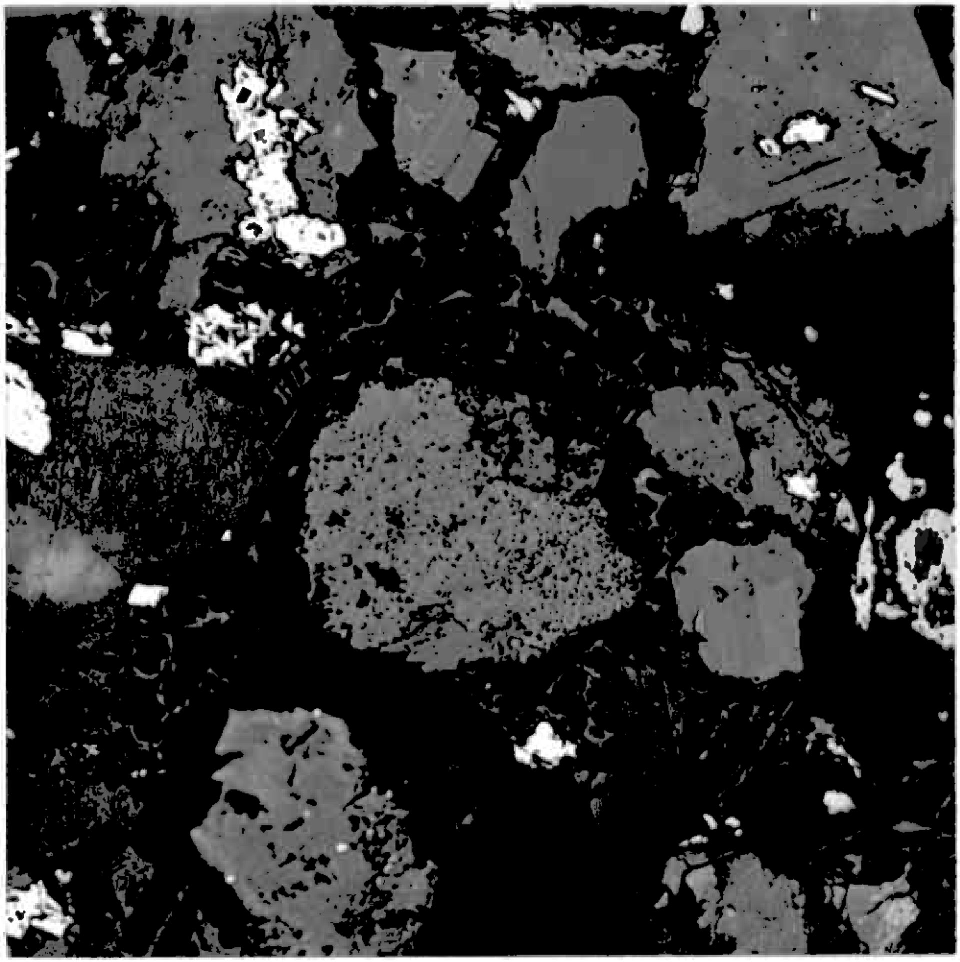
Yttrium "Concentrate"
Magnetic at 0.45 amps. Sink at sp. gr. 4.05
-100 +400 Mesh

Crushed fragments of kobeite particle No. 2 (yellowish-isotropic) with admixed carbonates (white). This particle was also located with the electron probe and then identified by x-ray diffraction. The pattern obtained corresponds well with the published pattern for kobeite. After completion of the x-ray diffraction pattern the single particle was crushed and examined by transmitted polarized light techniques, which showed that the mineral is essentially isotropic, probably because of metamictization.

Polarized, transmitted light. 145X

Figure 7

(hri)



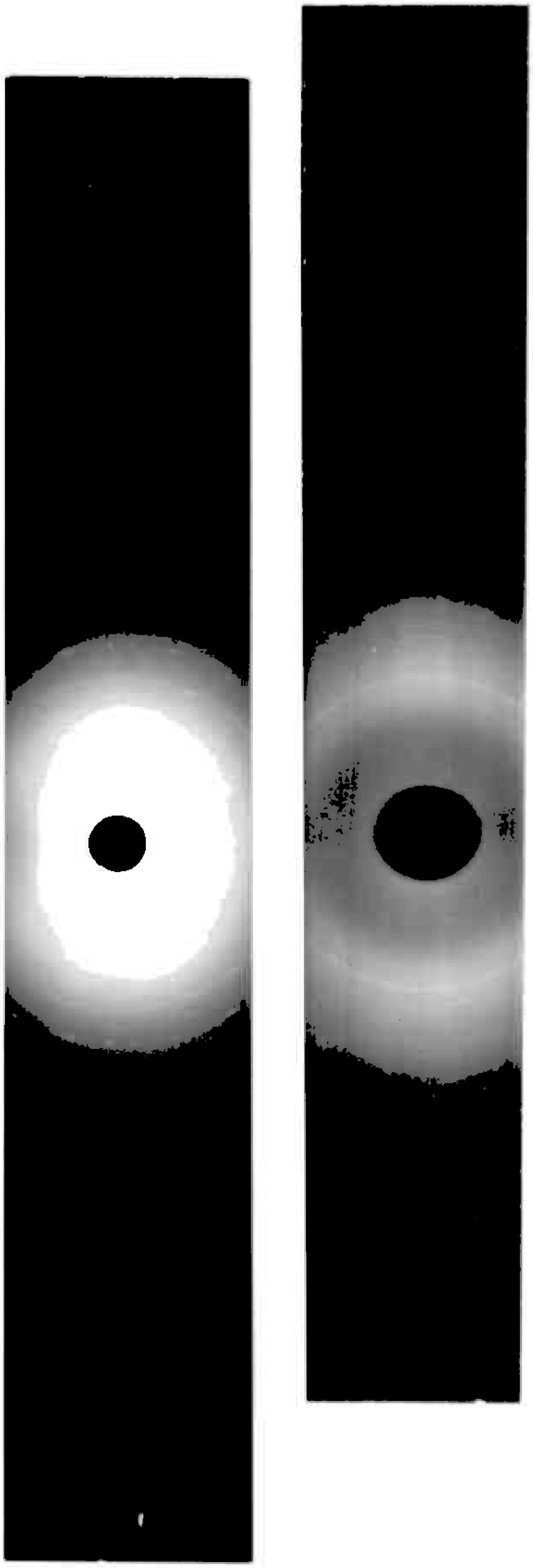
"Yttrium Concentrate"
Magnetic at 0.45 amps. Sink at 4.05 sp. gr.
-100 +400 Mesh

Xenotime particle (center) located by electron probe analysis. This particle which contained about 25% Y, was subsequently identified by x-ray diffraction. The pattern almost perfectly matches the published xenotime patterns (ASTM 11-254 and ASTM 9-377). The dark holes surrounding the xenotime were produced with the electron beam so the particle could be located under a low power microscope and removed for x-ray diffraction.

Plain incident light. Dry objective. 290X

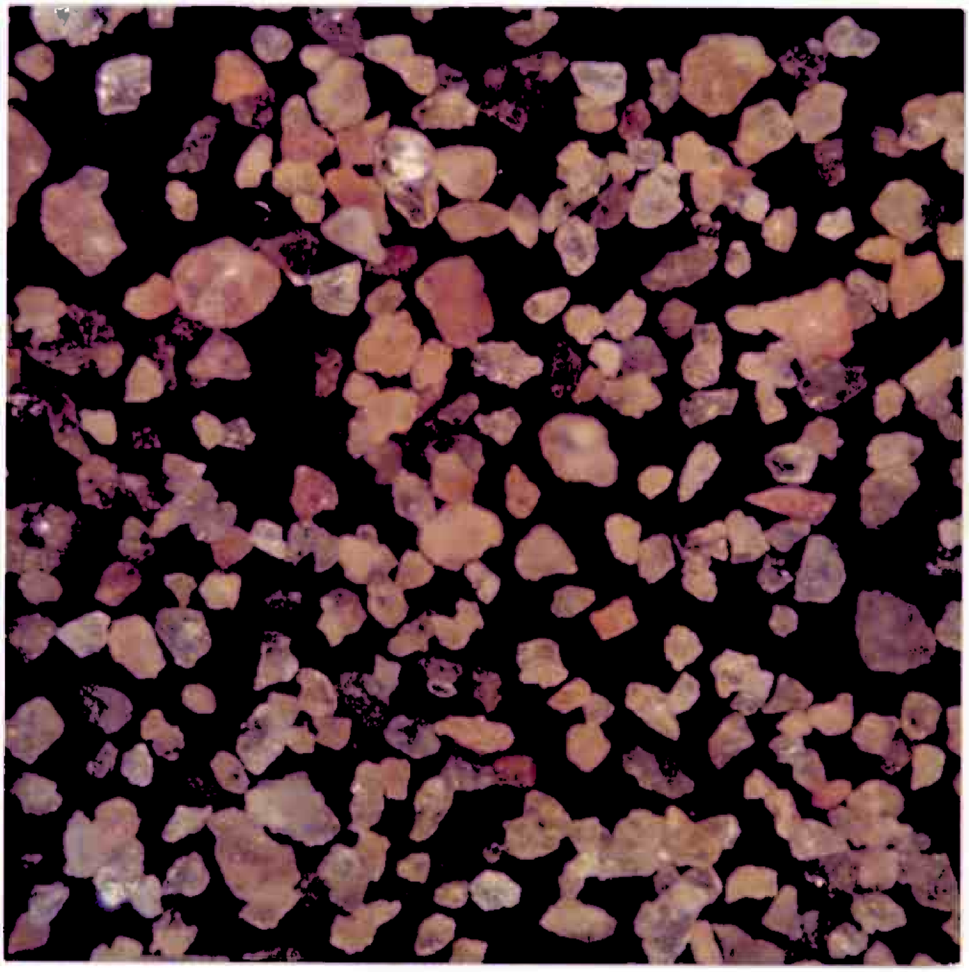
Figure 8

hri



X-ray diffraction patterns obtained on kobeite particle No. 2 (right photo) and xenotime (left photo). The kobeite was run with a regular 114.6 mm powder camera, using Ni filtered Cu Kα radiation, while the xenotime particle was run with a 100 mm camera. The mm line distances were multiplied by a factor of 1.146, converted to degrees 2θ and then to d-spacings. Many of the lines visible in the negative did not reproduce in the given copies. A listing of the measured d-spacings and relative intensities obtained from these films is given in Tables 6 and 7.

Figure 9

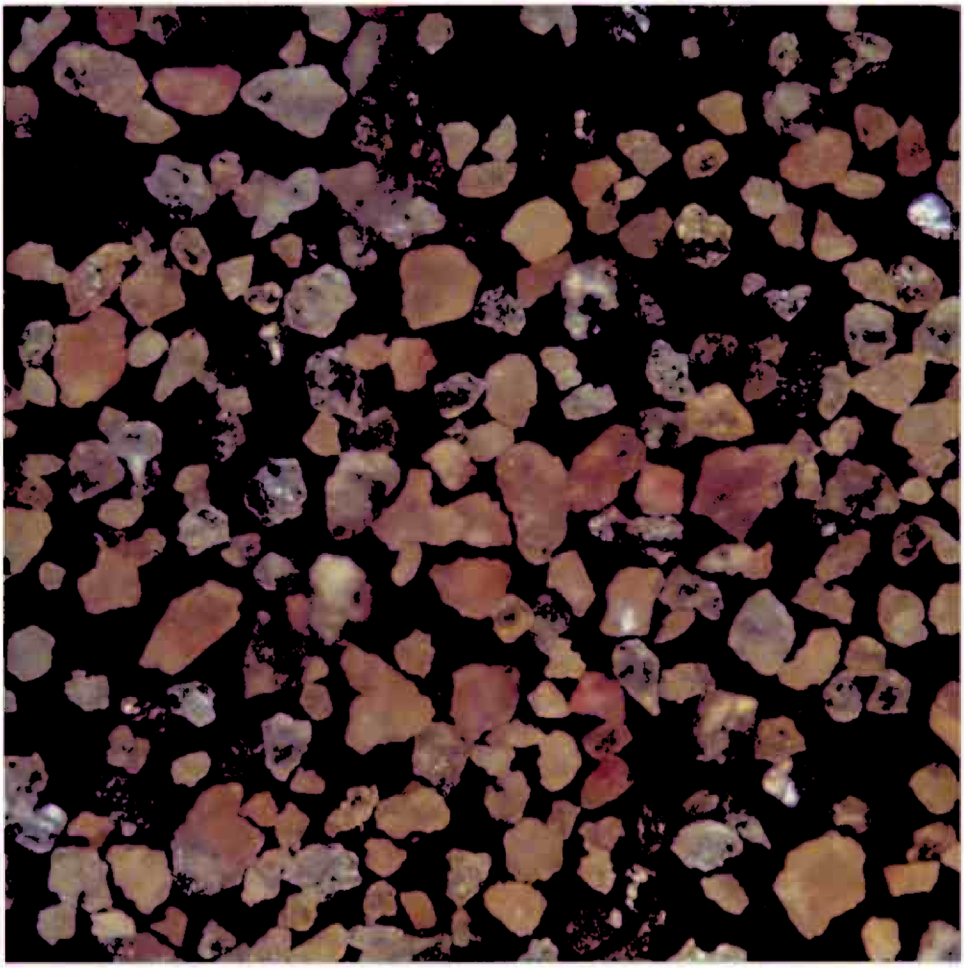


"Synchisite-Parisite Concentrate"
Magnetic at 0.9 amps. Float at sp. gr. 4.05
-100 +400 Mesh

The yellowish particles are primarily synchisite and parisite with minor bastnäsite and monazite. These minerals are finely intergrown with each other since all of them appeared in an x-ray pattern of a seemingly pure sample handpicked from the above product. The sample is also contaminated by considerable barite (white) iron oxides (black and brown) and other minerals.

Oblique reflected light. 50X

Figure 10

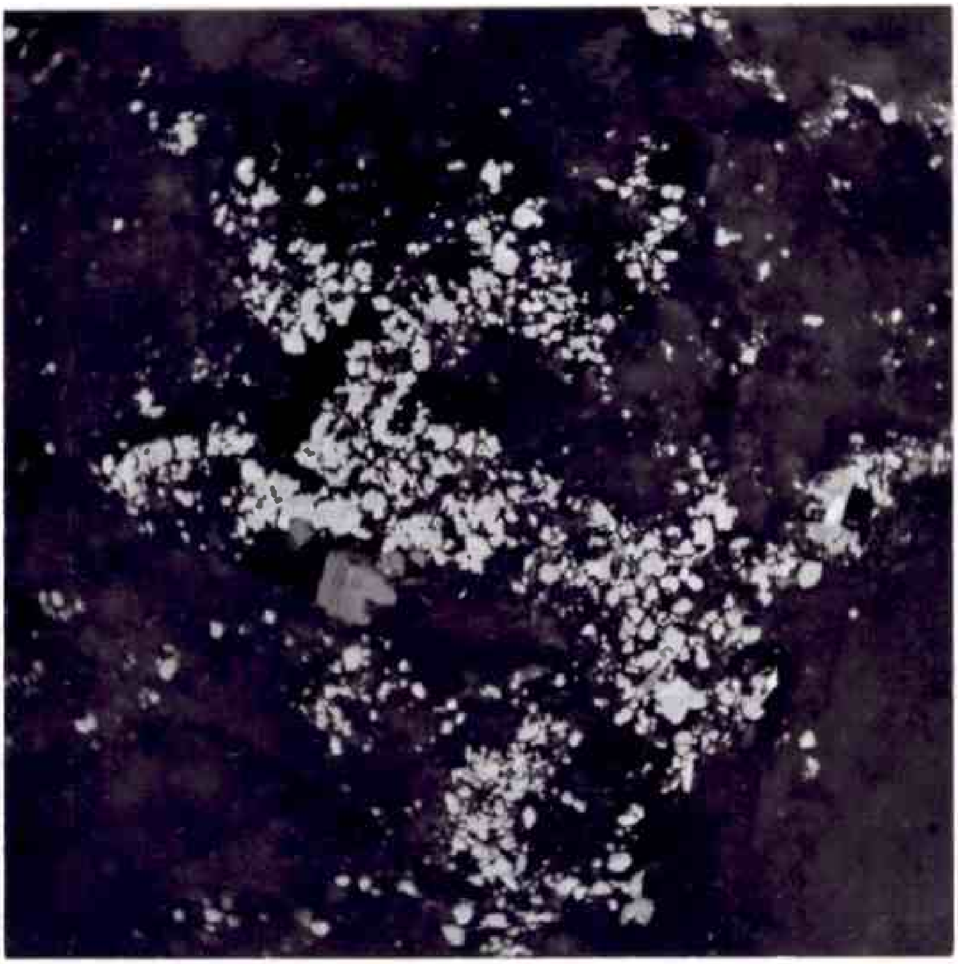


Bastnäsite-Monazite Concentrate
Magnetic at 0.9 amps. Sink at sp. gr. 4.05
-100 +400 Mesh

Bastnäsite, monazite with minor admixed synchisite and parisite (yellowish particles). Otherwise the same comments given in Figure 10 apply to this sample.

Oblique reflected light. 50X

Figure 11



Head Sample

Polished section showing Nb-rutile (white, tentatively identified) and kobeite (medium grey) finely admixed with carbonates (cloudy background). The possible occurrence of Nb-rutile is significant since it would contribute to the Nb values in the ore. The reason for the indistinct background is that with oil immersion the low reflecting minerals tend to disappear.

Plain incident light. Oil immersion. 360X

Figure 12

Table 5

Semiquantitative X-ray Fluorescence Scan of "Yttrium Concentrate" *

Zn	0.018
Tl	0.061
Sn	0.006
Pb	0.049
Fe	3.0
Ni	0.006
Ba	6.2
Sr	0.12
Ti	0.64
Zr	0.021
Th	0.27
Nb	1.3
Mn	0.30
La	6.9
Ce	10.0
Pr	1.2
Nd	1.9
Sm	0.20
Gd	0.13
Y	0.19

*Magnetic at 0.45 amps. Sink at sp. gr. 4.05. Sample used for identification of yttrium mineral.

Table 6

Listing of d-Spacings Obtained for Kobeite

Kobeite (Fen Deposit) Particle No. 1		Kobeite (Fen Deposit) Particle No. 2		Kobeite ASTM 11-259	
d (Å)	I/I_1	d (Å)	I/I_1	d (Å)	I/I_1
10.1	Spot?				
5.84	EW				
4.69	W			4.50	20
4.19	MW	4.15	100	4.16	100
3.77	EW	3.75	20	3.74	60
		3.55	5	3.50	20
3.34	EEW?	3.31	20	3.35	20
3.12	M-MW	3.10	15	3.13	60
3.01	MW				
3.84	EEW??	2.90	15		
2.48	EW??	2.60	5	2.55	20
				2.18	20
1.96	EW??			2.10	20
1.88	EW??				

M = Medium
W = Weak
EW = Extremely weak

Table 7

Listing of d-Spacings Obtained for Xenotime

Xenotime (Fen Deposit) Particle No. 3		Xenotime ASTM 11-0254 *		Xenotime ASTM 9-377**	
d (Å)	I/I_1	d (Å)	I/I_1	d (Å)	I/I_1
10.1	Spots				
4.64	EW ?	4.55	25	4.54	25
3.74	EW				
3.42	S	3.45	100	3.44	100
3.29	W				
3.11	MW				
2.92	W				
2.87	MS (spotty)	2.75	9	2.74	9
2.55	MS	2.56	50	2.56	60
2.43	W	2.44	13	2.43	20
2.19	EEW (spot)	2.27	6	2.27	9
2.14	W	2.15	25	2.14	25
2.03	EEW (spot)				
		1.92	9	1.93	13
1.81	EW	1.82	13	1.82	17
1.782	EEW (spot)				
1.764	M	1.77	50	1.76	45
		1.73	18	1.72	17
1.541	MW	1.54	9	1.54	11
1.469	EEW ? (spot)				
1.427	VW	1.43	9	1.428	3
1.390	EEW ? (spot)				
1.373	EW	1.38	7	1.379	11
1.334	EW	1.35	5	1.342	7
1.281	W	1.283	9	1.280	11
1.234	VW	1.235	9	1.232	11
1.096	EW				
1.075	EW				

S = Strong
M = Medium
W = Weak
VW = Very weak
EW = Extremely weak

* Xenotime Shelby, North Carolina
** Synthetic Xenotime

Table 8

Semiquantitative X-ray Fluorescence Analyses of Crude Rare Earth Mineral Concentrates

Float at Sp. Gr. 4.05		Sink at Sp. Gr. 4.05	
Zn	0.051	Zn	0.014
Tl	0.028	Pb	0.022
Sn	0.006	Fe	0.91
Fe	0.77	Ni	0.006
Ni	0.007	Ba	16.0
Ba	15.0	Sr	0.15
Sr	0.12	Ti	0.62
Ti	0.75	Th	0.32
Th	0.29	Nb	0.13
Nb	0.14	U	0.016
U	0.012	Mn	0.056
Mn	0.10	La	11.0
La	6.9	Ce	12.0
Ce	10.0	Pr	1.9
Pr	1.6	Nd	2.8
Nd	2.5	Sm	0.49
Sm	0.30	Gd	0.17
Gd	0.24	Y	0.11
Y	0.11		

Table 9

Semiquantitative X-ray Fluorescence Analyses of Rare Earth Oxalate-Oxide Precipitates

Obtained from dissolution of crude R.E. mineral concentrates.

Float at Sp. Gr. 4.05		Sink at Sp. Gr. 4.05	
La	25.0	La	25.0
Ce	41.0	Ce	39.0
Pr	3.2	Pr	3.9
Nd	12.0	Nd	10.0
Sm	0.89	Sm	0.68
Gd	0.44	Gd	0.44
Dy	0.11	Dy	0.11
Er	0.062	Er	0.094
Yb	0.079	Yb	0.053
Y	0.69	Y	0.50
Ba*	3.3	Ba*	4.8
Th	0.76	Sr	0.010
		Th	0.92

*Some Ba was coprecipitated as the oxalate. Because of the relatively small amount no attempt was made to remove the Ba entirely by reprecipitation.

Table 10

Rare Earth Content and Distribution in R.E. Minerals

Values calculated from analyses of rare earth oxalate-oxide precipitates less $BaSO_4$ basis.

Float at Sp. Gr. 4.05		Sink at Sp. Gr. 4.05	
%		%	
La	9.4	La	17.2
Ce	15.4	Ce	26.7
Pr	1.2	Pr	2.7
Nd	4.5	Nd	6.9
Sm	0.33	Sm	0.47
Gd	0.17	Gd	0.30
Dy	0.040	Dy	0.070
Er	0.023	Er	0.064
Yb	0.030	Yb	0.036
Y	0.26	Y	0.34

SYNCHISITE – PARISITE CONCENTRATE
FLOAT PRODUCT @ sp gr. 4.05


MONAZITE 25
BASTNÄSITE 40
MONAZITE 25
ALUMINUM (HOLDER) 47
BASTNÄSITE 40
BASTNÄSITE 40
MONAZITE 25
ALUMINUM (HOLDER) 100
MONAZITE 18
MONAZITE 18
BASTNÄSITE 3
BARITE 53
MONAZITE 70
BASTNÄSITE 100
MONAZITE 100
BARITE 97
MONAZITE 50
BARITE 100
MONAZITE 25
BASTNÄSITE 70
Cu Kα 1°/min 1"/min
T.C = 2 sec
F.S = 1 x 10² (X1)
1° - .006" - 1° Slit
50 Kv 40 Ma
BARITE 57
MONAZITE 25
MONAZITE 19
BASTNÄSITE 40
MONAZITE 13
BASTNÄSITE - MONAZITE CONCENTRATE
SINK PRODUCT @ sp. gr 4.05

No. 1 BASTNÄSITE GROUP MINERALS (FROM A/S MEGON)