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A NUMERICAL APPROACH TOWARD THE CLASSIFICATION OF COMPLEX, ORTHORHOMBIC, RARE-EARTH, AB_2O_6-TYPE Nb-Ta-Ti OXIDES

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ABSTRACT

Since their initial description in the early 1800's, orthorhombic, rare-earth, AB_2O_6-type Nb-Ta-Ti oxides have been a "mineralogical headache". Due to complex compositions, their metamict state and pervasive alteration, as well as errors of previous workers, the nomenclature of this mineral group is often inconsistent and ambiguous.

To test the consistency of the nomenclature of previous workers and to develop a consistent chemical nomenclature, five groups (euxenite, polycrase, priorite, blomstrandine and aeschynite) were subjected to stepwise discriminant analysis. The literature data set contained 91 cases. Each case consisted of a maximum of 58 chemical, physical, optical and structural variables. Output for the discriminant analysis was plotted in the plane of the two principal canonical variables using various combinations of seven chemical variables: TiO_2, Nb_2O_5, Ta_2O_5, ΣCe_2O_3, ΣY_2O_3, $\Sigma U_3O_8 + UO_2 + UO_3$, ThO_2. The results of the multivariant analysis and a program of classification improvement indicate at least a three-fold chemical classification — aeschynite, euxenite and polycrase.

SOMMAIRE

Depuis leur description originelle, au début des années 1800, les oxydes orthorhombiques de terres rares Nb-Ta-Ti du type AB_2O_6 ont causé bien des "maux de tête minéralogiques". A cause de leur composition complexe, leur état métamicte et leur profonde altération, à cause aussi des erreurs commises par les premiers chercheurs, les espèces de ce groupe minéral ont reçu une nomenclature ambigüe et contradictoire.

Dans le but d'établir une nomenclature chimique unifiée des oxydes orthorhombiques de terres rares Nb-Ta-Ti du type AB_2O_6, cinq groupes (euxénite, polycrase, priorite, blomstrandite et aeschynite) ont été soumis à une analyse discriminante (procédure pas-à-pas). L'ensemble des données de la litérature contenait 91 cas. Chaque cas était composé d'un maximum de 58 variables chimiques, physiques, optiques et structurales. Les résultats de l'analyse discriminante ont été reportés dans le plan des deux variables canoniques principales utilisant diverses combinaisons de sept variables chimiques: TiO_2, Nb_2O_5, Ta_2O_5, ΣCe_2O_3, ΣY_2O_3, $\Sigma U_3O_8 + UO_2 + UO_3$, ThO_2. Les résultats de l'analyse multivariante et d'un programme d'amélioration de la classification indiquent une classification chimique comportant au moins trois groupes: aeschynite, euxénite et polycrase.

(Traduit par le journal)

INTRODUCTION

The orthorhombic metamict Nb-Ta-Ti oxides of the type formula AB_2O_6 (A = REE, Fe^{+2}, Mn, Ca, Th, U, Pb; B = Nb, Ta, Ti, Fe^{+3}) are generally assumed to include the following minerals: euxenite, polycrase, priorite, blomstrandine and aeschynite (=eschynite) as well as the varieties eschwegite, lyndochite (= eschynite, Fleischer 1966), kobeite, tanteuxenite (=delorenzite, Butler & Embrey 1959), tant-polycrase, sinicite, fersmite, polymignyte and khlopinite (=samarskite, Kuz'menko *et al.* 1969). In many descriptions of their occurrences, these minerals are simply described qualitatively as "euxenite"

(Bjørlykke 1935) or "eschynite-priorite" (Čech *et al.* 1972). This is appropriate when there is insufficient data to distinguish one from the other, and the question still remains whether present evidence justifies the use of even four major mineral names.

These complex oxides have caused problems since their initial description in the early 1800's. Their complex and variable compositions combined with their often metamict state and pervasive alteration have resulted in a contradictory nomenclature and inconsistent values for many mineralogic parameters. The purpose of this study is two-fold: (1) to examine the internal consistency of past chemical systematics, and (2) to develop a consistent chemical nomenclature for the minerals of this group.

NOMENCLATURE

The inconsistencies in nomenclature are, in part, historical. The mineral initially described as euxenite by Scheerer (1840) from Jölster, Sönd Fiord of western Norway, is certainly not the same mineral as that to which the name is now applied; further, Scheerer's (1844) reasons for the introduction of polycrase (Hitterö, Norway) as a valid mineral species distinct from euxenite are certainly in error (Broegger 1906).

Additionally, there is confusion between the minerals aeschynite, blomstrandine and priorite. Aeschynite from Miask, Ilmen Mts. of the U.S.S.R. was originally described by Berzelius (1828). Broegger (1879) published the first crystallographic description of blomstrandine under the title "Über Aeschynit von Hitterö, nebst einigen Bermerkungen über die Krystallform des Polykras." In the text, Broegger clearly states that the mineral, which from his crystallographic examination he referred to as aeschynite, might possibly be another mineral and that only an exact chemical analysis would be definitive. Prior (1899), in a description of a previously unknown metamict niobium titanium oxide from Swaziland, re-examined the "aeschynite" from Hitterö because its crystal form corresponded precisely with that of the Swaziland material. Qualitative analysis of both mineral specimens showed them to be similar in composition and more nearly related to euxenite than aeschynite. A chemical analysis by Blomstrand confirmed Prior's findings. Broegger (1906) published Blomstrand's analysis and named the new mineral blomstrandine, suggesting that the most useful, although by no means proven, nomenclature involved dividing euxenite, polycrase, priorite and blomstrandine into two orthorhombic dimorphous series, the euxenite-polycrase series and the priorite-blomstrandine series. The basic criterion for separating the two series is the ratio of the crystallographic axes. Morphological data have shown that euxenite *(Pcan)* is orthorhombic, with axial ratios of $a:b:c$ = 0.3789:1:0.3527, whereas priorite *(Pbnm)*, also orthorhombic, has axial ratios of $a:b:c$ = 0.4746:1:0.6673. The relation of the axes of the priorite-blomstrandine series may, by an appropriate transformation, be brought very near that of the euxenite-polycrase series, with different forms developed in the two series (Adamson 1942; Komkov 1959). Thus it was not clear whether euxenite and priorite were polymorphous varieties of the same composition or whether the slight structural difference is due to a variable content of ismorphous cations (*e.g.*, uranium and thorium). By Broegger's (1906) definition each series is structurally distinct but chemically equivalent, and polycrase and blomstrandine represent the titanium-rich end members of the two series. Based on analyses compiled by Hintze (1938), the solid-solution series were defined by the following molecular ratios:

euxenite and priorite: $(Nb+Ta)_2O_5 : TiO_2 \geqq 1:4$

polycrase and blomstrandine:

$(Nb+Ta)_2O_5 : TiO_2 \leqq 1:4$

Aeschynite was considered to be a cerium-rich isomorph of the priorite-blomstrandine series, and fersmite, the calcium-rich isomorph of the euxenite-polycrase series.

Three difficulties have resulted from this initial confusion:

(1) Museum specimens of "aeschynite" collected from Hitterö between 1879 and 1906 are invariably blomstrandine and the occurrence of aeschynite at Hitterö remains unconfirmed (Adamson 1942).

(2) Blomstrandine and priorite are considered by some (Broegger 1906; Adamson 1942; Komkov 1959) to be end members of a solid-solution series whereas others consider them equivalent mineral names with priorite being the more common usage (Palache *et al.* 1944; George 1949; Lima-de-Faria 1964). More rarely priorite is considered a synonym for euxenite (Vlasov 1966).

(3) Two mineral species were named after G. W. Blomstrand (1862-1936), Professor of Chemistry at the University of Lund:

blomstrandine
blomstrandinite
blomstrandite

The first two mineral names are different spellings for the mineral first described from Hitterö (Broegger 1906). The third is a variety of betafite from Tongafeno, Madagascar (Palache *et al.* 1944; George 1949).

Work since the original descriptions has resulted in the proliferation of varietal names and contradictory mineralogic systematics. The IMA, to lessen confusion, has accepted Levinson's (1966) suggestion that the name priorite be discontinued in favor of the notation aeschynite-(Y); but Levinson's nomenclature has not been universally adopted (Bouska 1970). Levinson based this nomenclature on the assumption that aeschynite and priorite are the end members of a solid-solution series involving the rare earths. This was later confirmed by experimental synthesis (Komkov *et al.* 1962; Komkov & Belepol'skii 1966).

Compositionally, only two of the mineral species may be unambiguously defined: euxenite, $YNbTiO_6$, and aeschynite, $CeNbTiO_6$, with polycrase considered a titanium-rich variety of euxenite and all three phases subject to cationic substitution, particularly U, Th, Ca and the rare earths. The relation of euxenite to aeschynite is further complicated by a pseudomonotropic structural transition from the priorite-aeschynite structure to the euxenite structure for intermediate rare-earth compositions (Seifert & Beck 1965).

DISCRIMINANT ANALYSIS AND CLASSIFICATION IMPROVEMENT

As is apparent from the previous discussion or from comparison of standard references such as *Dana's System of Mineralogy* (Palache et *al.* 1944) or *Mineralogy of Rare Elements* (Vlasov 1966) the systematics of these Ti-Nb-Ta oxides is confusing and contradictory. Because these minerals are metamict, they can be considered pseudomorphs of a particular kind that can only be distinguished on the basis of chemical composition, but a consistent view of their compositional characteristics is lacking. Although X-ray and annealing studies (Komkov 1959, 1963; Lima-de-Faria 1964; Mitchell 1972; Ewing & Ehlmann 1975) are critical for the proper identification and classification of minerals in this group, it is advisable to examine the possibility and limitations of a chemical classification. Structural distinctions between species based on external morphologies cannot always be determined from what are commonly subhedral crystals, and X-ray data from annealing studies, which are not always consistent in procedure, will not distinguish between chemically similar species that have become completely metamict.

A data set of 129 cases, each consisting of 58 variables summarizing the chemical, structural and physical parameters of species and varieties that are considered orthorhombic Nb-Ta-Ti oxides of the type formula AB_2O_6 was compiled from the literature. Sources from which these analyses were taken are marked by an asterisk in the references. The data for most cases were partial and the chemical analyses were either incomplete or not accompanied by complementary determination of physical or structural parameters. Ninety-one cases (excluding varieties) contained values for the weight percentages of TiO_2 (X_1), Nb_2O_5 (X_2), Ta_2O_5 (X_3), ΣCe_2O_3 (X_4), ΣY_2O_3 (X_5), $\Sigma U_3O_8 + UO_2 + UO_3$ (X_6), and ThO_2 (X_7). These seven variables are of particular interest because:

(1) These variables have been important in previous systems of classification and their combined wt. % generally account for 80 to 90 wt. % of most analyses. When other elements are abundant, previous authors have preferred varietal names (*e.g.*, polymignyte, approximately 30% ZrO_2).

(2) The TiO_2, Nb_2O_5, and Ta_2O_5 content in minerals are the least susceptible to weathering or late-stage alteration (Van Wambeke 1970; Ewing 1975); therefore, the quality of the mineral-separation techniques used to separate altered from unaltered zones is not as important a consideration in using published chemical analyses. Those analyses for which the % Ta_2O_5 is greater than 25% when the wt. % of TiO_2, Nb_2O_5, and Ta_2O_5 are normalized to 100% have been excluded because they are more properly considered varieties (*e.g.* tantpolycrase or tanteuxenite).

(3) Soboleva & Pudovkina (1961) have considered the relative wt. % of uranium oxides and thorium oxide as important in distinguishing between euxenite and priorite. Although Komkov (1963) has indicated that this correlation was fortuitous, these variables have been included in this study. Since uranium is usually given in one of three possible oxidation states, these values were simply summed into a variable which will be referred to as ΣU (X_6).

To test the consistency of nomenclature by previous workers, all five groups — euxenite, polycrase, priorite, blomstrandine, aeschynite — were subjected simultaneously to a discriminant analysis on the basis of variables X_1 through X_7 (for a brief description of discriminant analysis see Davis 1973, pp. 442-456). The particular discriminant analysis program used was BMDO-7M (Sampson 1970). In this stepwise procedure each of the seven variables is brought into the analysis one at a time (beginning with the variable with the largest *F*-value), and at the end of each step the cases are reclassified on the basis of previously-used variables. The output is summarized in Figure 1 which shows each of 91 specimens plotted in the plane of the two principal canonical variables. Examination of Figure 1 and Table 1 suggests that the aeschynite samples, because of their high content of ΣCe_2O_3 (X_4), are readily discriminated from the other four groups, and in the first iteration of the program 19 of 22 cases of aeschynite were classified in a manner consistent with their use in the literature The other four groups — euxenite, polycrase, priorite, blomstrandine — are not clearly separated; the tabulation below summarizes the final classification at the end of seven iterations:

	NUMBER OF CASES CLASSIFIED AS					
	EUX.	POL.	PRI.	BLO.	AES.	TOTAL
EUX.	21	2	2	3	0	28
POL.	3	6	0	4	0	13
PRI.	0	1	18	1	0	20
BLO.	0	1	3	4	0	8
AES.	0	0	0	0	22	22
TOTAL						91

Of the five groups, euxenite, priorite and aeschynite were generally classified correctly in the literature on the basis of the seven variables examined, but as shown in Figure 1, there is still considerable overlap among euxenite, polycrase, priorite and blomstrandine groups. In Broegger's (1906) original distinction between these four species, euxenite and priorite were considered chemically similar to one another and distinct from polycrase and blomstrandine, their Ti-rich equivalents. The canonical projection from the multivariant discriminant analysis of these four groups using three variables — TiO_2, Nb_2O_5, and Ta_2O_5 — is shown in Figure 2. The overlap between groups is apparent, and priorite (P) is not equivalent to euxenite (E). Figure 2 is the most graphic representation of the inconsistent nomenclature for this mineral group. The tabulation below summarizes the final classification after three iterations:

	NUMBER OF CASES CLASSIFIED AS				
	EUX.	POL.	PRI.	BLO.	TOTAL
EUX.	23	1	2	2	28
POL.	4	3	1	6	14
PRI.	6	1	9	4	20
BLO.	0	1	2	4	7
TOTAL					69

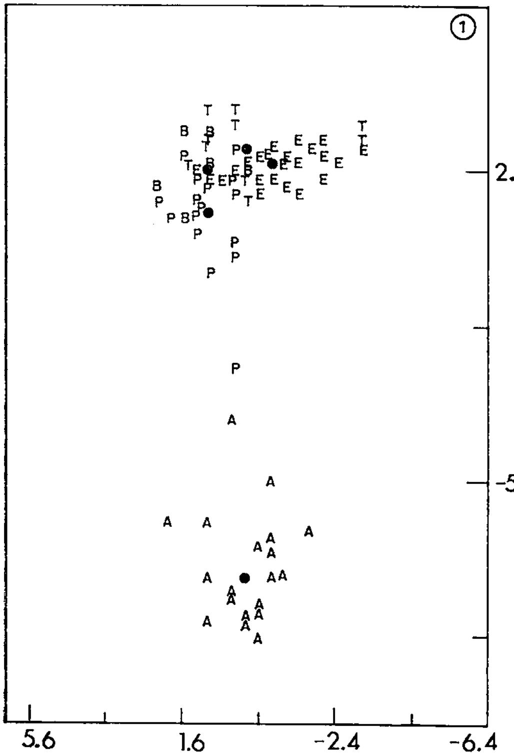
TABLE 1. MEANS AND STANDARD DEVIATIONS OF PRINCIPAL CHEMICAL COMPONENTS FOR RARE-EARTH AB_2O_6-TYPE Nb-Ta-Ti OXIDES FROM THE LITERATURE.

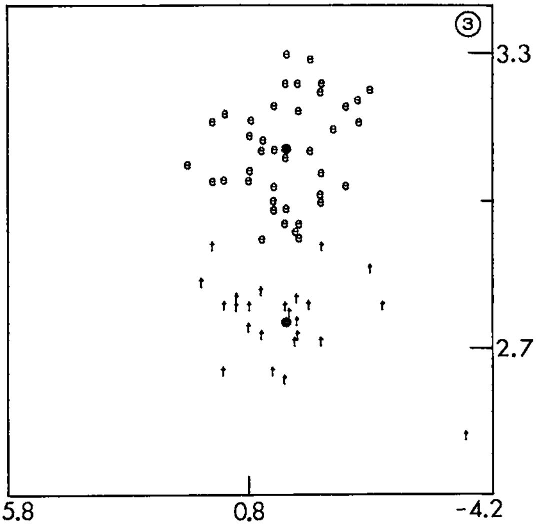
Variable	Eux.	Pol.	Pri.	Blom.	Aes.
X_1	22.47 ±4.28	27.63 ±5.91	27.26 ±4.21	32.56 ±6.84	21.05 ±5.09
X_2	26.52 ±4.53	21.92 ±3.52	21.43 ±9.29	20.66 ±3.94	30.57 ±9.25
X_3	2.58 ±2.06	2.23 ±2.27	2.88 ±3.11	2.16 ±1.35	3.73 ±6.95
X_4	1.81 ±0.87	1.50 ±1.08	3.14 ±1.32	2.89 ±0.88	20.95 ±6.63
X_5	24.50 ±3.08	24.65 ±6.08	25.91 ±5.30	25.34 ±3.74	3.09 ±2.77
X_6	9.31 ±3.58	9.39 ±6.51	4.05 ±2.48	5.25 ±1.82	1.21 ±0.99
X_7	3.08 ±1.52	3.47 ±2.14	5.93 ±3.93	3.75 ±2.75	10.73 ±8.14
No. of Cases	28	13	20	8	22

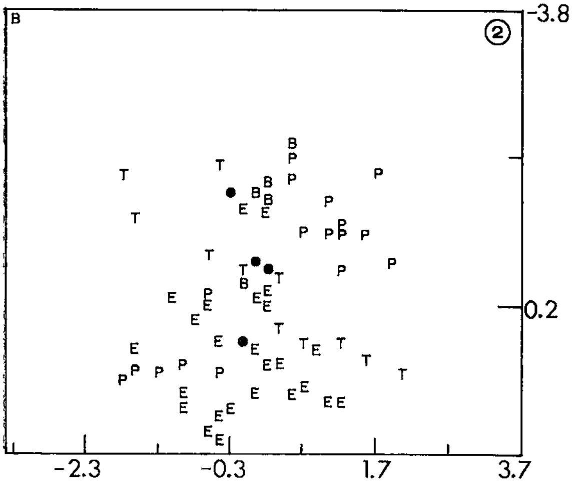
It is interesting to note that, with respect to these three variables, euxenite has most often been used in a consistent manner by previous authors; when the variables X_4, X_5, X_6 and X_7 are disregarded, far fewer cases of priorite are correctly classified although these four variables were not considered important in the original distinction between euxenite and priorite (Broegger 1906; Adamson 1942).

Since the multivariant analysis demonstrated inconsistencies in nomenclature, mineralogical labels were disregarded and a program of multivariant evaluation and improvement of classifications by Demirmen (1969) was used to reclassify the data on the basis of TiO_2, Nb_2O_5 and Ta_2O_5. The program first calculates group means in discriminant space. Each case is then reclassified according to its minimum distance from the group means and a new group mean is calculated. At the end of ten iterations only one case remained in an inappropriate group. Multivariant stepwise analysis of these four new groups showed that TiO_2 was the most important discriminating variable, with the four groups showing a sequential increase in TiO_2 content. These four groups were simplified into two larger groups — "euxenite" and "polycrase". The multivariant stepwise analysis (BMDO7M) was again repeated and is shown in Figure 3. The cases are now classified into two internally consistent groups — "euxenite" and "polycrase", and the break between the two groups is approximately at the point suggested by Hintze (1938). The means and standard deviations of principal components of the two groups are summarized below:

	"EUXENITE"	"POLYCRASE"
TiO_2	22.28 ± 3.06	32.47 ± 4.01
Nb_2O_5	26.91 ± 5.22	18.71 ± 5.04
Ta_2O_3	2.48 ± 2.54	1.68 ± 1.75
No. of cases	40	29







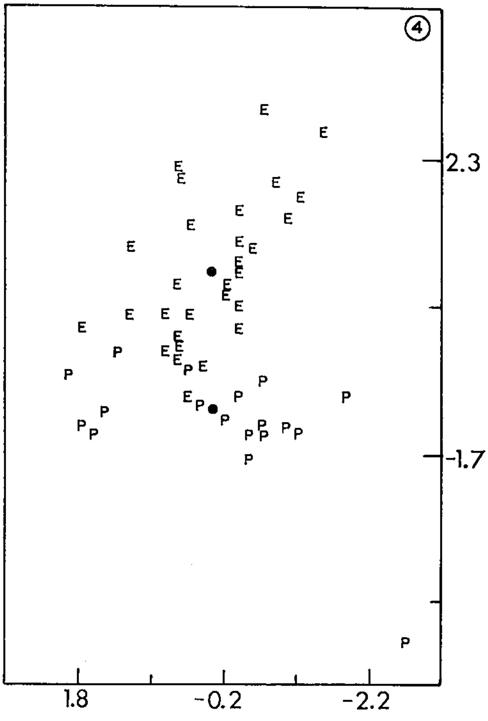


FIG. 1. Plot for the first (ordinate) and second (abscissa) canonical variables for the groups: aeschynite (A), euxenite (E), blomstrandine (B), priorite (P) and polycrase (T) using variables X_1 through X_7. Group means = ●

FIG. 2. Plot of the first (ordinate) and second (abscissa) canonical variables for the groups: euxenite (E), blomstrandine (B), priorite (P) and polycrase (T) using the variables X_1 (TiO_2), X_2 (Nb_2O_5) and X_3 (Ta_2O_5). Group means = ●

FIG. 3. Plot of the first (ordinate) and second (abscissa) canonical variables for the groups: "euxenite" (e) and "polycrase" (t) using the variable X_1 (TiO_2), X_2 (Nb_2O_5) and X_3 (Ta_2O_5). Group means = ●

FIG. 4. Plot of the first (ordinate) and second (abscissa) canonical variables for the groups: euxenite (E) and priorite (P) using variables X_6 (ΣU) and X_7 ,ThO_2). Group means = ●.

Although there is still overlap between the two groups on the basis of the Nb_2O_5 and Ta_2O_5 content, the two groups can be readily distinguished on the basis of the TiO_2 content.

In the original description Broegger (1906) maintained that priorite and euxenite were dimorphous chemical equivalents. Most authors (Adamson 1942; Mitchell 1972) have followed this convention, but Soboleva & Pudovkina (1957) suggested that euxenite contained more uranium than thorium, whereas priorite contained more thorium than uranium. Komkov (1963) showed that this criterion was not consistent and suggested that the correlation was fortuitous. In Figure 4, values for X_6 and X_7 of euxenite and priorite samples from the literature are plotted in the plane of the two canonical variables. Although there is a continuous gradation from one group to another and there is some overlapping of the groups, the two species can be fairly accurately classified on the basis of uranium and thorium content, and Figure 4 supports the distinction suggested by Soboleva & Pudovkina (1957). This would explain why more priorite cases were correctly classified using all seven variables (these include ΣU and ThO_2) than in the case where only three variables were used — TiO_2, Nb_2O_5, and Ta_2O_5.

CONCLUSIONS

Multivariant analysis and classification improvement using published chemical values for the variables X_1 through X_7 indicate at least a three-fold chemical classification — aeschynite, "euxenite" and "polycrase". The quotation marks are used to indicate that these are compositions defined by Demirmen's (1969) program of classificiation improvement. As shown in Figure 5, these three groups can be separated on the basis of their wt. % ΣCe_2O_3, TiO_2 and Nb_2O_5

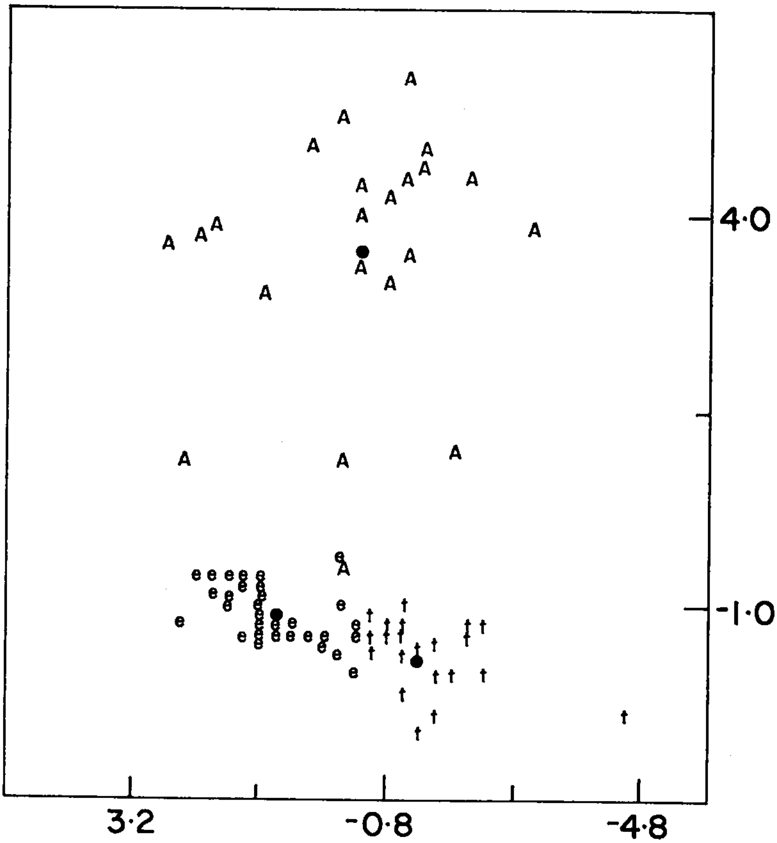


FIG. 5. Plot of the first (ordinate) and second (abscissa) canonical variables for the groups: "euxenite" (e), polycrase (t) and aeschynite (A) using variables X_1 (TiO_2), X_2 (Nb_2O_5) and X_4 (ΣCe_2O_3). Group means = ●

and the values and standard deviations of these critical variables are summarized below:

	"EUXENITE"	"POLYCRASE"	AESCHYNITE
TiO_2	22.28 ± 3.06	32.47 ± 4.01	21.05 ± 5.09
Nb_2O_5	26.91 ± 5.22	18.71 ± 5.04	30.57 ± 9.25
$\Sigma\ Ce_2O_3$	2.13 ± 1.28	2.47 ± 1.17	20.95 ± 6.63

Some authors (Lima-de-Faria 1964) have suggested a solid-solution series between aeschynite and euxenite. Fleischer (1966) has shown a regular and continuous variation between (La +Ce+Pr) and the ratio 100 Yt/(Yt+*RE*) for euxenite-priorite and aeschynite. Horne & Butler (1965) have pointed out that the composition of rare earths in euxenites and priorites are similar, but different from that of aeschynite. Komkov & Belopol'skii (1966) found continuous miscibility between synthetic priorite and aeschynite under hydrothermal conditions, but none between euxenite and aeschynite. The determination of extent of solid solution between euxenite and aeschynite will rest on structural determinations of synthetic analogues, but is certainly not proved on the basis of currently-available chemical data of natural minerals.

Present data indicate that ΣU and ThO_2 content may be critical to the chemical distinction between euxenite and priorite. If priorite and aeschynite are the end members of a solid-solution series, the thorium content of priorite may be particularly important. The ionic radius of thorium may allow for the easy substitution of cerium earths. Presently, *euxenite* should be used in preference to *priorite* in the absence of structural or morphological data or if ΣU is greater than ThO_2. In the event ΣU is less than ThO_2, the present nomenclature is ambiguous. At present, euxenite should be considered chemically equivalent to priorite (=aeschynite–(Y)); polycrase, chemically equivalent to blomstrandine. Further study will be required to establish the chemical relationship between euxenite and priorite.

As a final note, it should be emphasized that this study and its conclusions are derived from a statistical analysis of the *literature*, and as such, cannot be used to directly ascertain the chemical and structural relations of minerals in this group. Structural and chemical criteria must ultimately be the basis for the definition of mineral species, but in the case of metamict minerals of complex compositions (*e.g.*, the pyrochlores) a statistical approach can be both convenient and illuminating. Such an approach allows for ready access to the literature, and provides a quick means for testing ideas or spotting discrepancies among workers. In a more expanded form such an approach could include physical-optical properties and structural data.

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*References marked with asterisks are sources of the chemical analyses used in the present study.