Nonmetamict Nioboeschynite-(Ce) from Alaska

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Abstract

Crystals of essentially nonmetamict nioboeschynite-(Ce), a complex oxide with the general formula AB_2O_6, were identified in an alluvial concentrate from the Tofty tin belt in central Alaska. The mineral grains are mostly sand-size (0.2 to 1.5 mm), red-brown to dark brown, generally tabular, slender to stubby striated prisms. Crystal parameters of this rare orthorhombic mineral are: $a = 5.396$, $b = 11.085$, $c = 7.585$ Å, $V = 453.7$ Å³, space group = *Pbnm*, $Z = 4$; D = 5.04 g/cm³; RI: $\alpha = 2.27$; $\beta = 2.32$, $\gamma = 2.34$, (−) $2V$ (av.) = 84.6°; γ, α, and β are parallel to crystallographic a-, b- and c-directions, respectively; prominent d values (and relative intensities) = 2.98 **10**, 3.05 **7**, 3.13 **4**, 5.53 **2**, 3.44 **2**, 2.82 **2**, 2.70 **2**, 2.04 **2**, 1.72 **2**, and 1.61 **2**. Spectrochemical analysis shows (in percent): CaO 4.52, FeO 1.09, La₂O₃ 4.9, Ce₂O₃ 15.8, Pr₂O₃ 1.9, Nd₂O₃ 5.6, Y₂O₃ 0.18, ThO₂ 1.33, U₃O₈ 0.01, SiO₂ 3.51, Nb₂O₅ 47.0, TiO₂ 20.2, Σ = 106; minor elements (in parts per million) include Ba 2000, Be 10, Cr 300, Cu 30, Mg <10, Mn 40, Pb 100, Sb 50, and Ta <1000. The structural formula computed from the analysis is $(Ca_{0.27}Fe_{0.05}La_{0.10}Ce_{0.32}Pr_{0.04}Nd_{0.11}Y_{0.01}Th_{0.02})_{0.92}(Nb_{1.17}Ti_{0.83})_{2.00}O_{5.78}$.

Nonmetamict eschynite is rare; only five crystalline to partly metamict specimens were described in the literature. The physical, chemical, and X-ray data of Alaskan eschynite are compared with data for such eschynites from the Ural Mountains in the USSR, and northern China.

Introduction

Eschynite, a complex oxide composed of rare-earth elements, thorium, niobium, and titanium, is generally found in the metamict state (Pabst, 1952; Bouška, 1970; Mitchell, 1973). Eschynite is an uncommon mineral. Owing to its variable composition, present nomenclature makes use of suffixes such as −(Ce) and −(Y) to designate varieties of eschynite (Levinson, 1966; Sommerauer and Weber, 1972). The mineral has long been known in nepheline syenite and granite pegmatite at localities in Norway, the USSR, and Germany (Palache, Berman, and Frondel, 1944, p. 796), but eschynite was not recognized in Alaska until the early 1930's (Waters, 1934, p. 239–240). Other eschynite localities in Brazil, Canada, China, Kenya, Switzerland, and in Connecticut, Montana, and New Mexico in the U.S.A. have been discussed in the literature, but most of these have furnished metamict crystals. Only five crystalline to partly metamict nioboeschynites-(Ce) and one eschynite-(Y) have been described prior to the discovery of the crystalline character of this nioboeschynite-(Ce) from Alaska. Hereinafter, we will refer to this material as eschynite, except when specification is required.

Geologic Setting

Eschynite in Alaska was first reported by Waters (1934) in four placer deposits in the Tofty tin belt of the Hot Springs district in the southeast part of the Tanana quadrangle, central Alaska. Figure 1 shows the locations of the sites described by Waters where eschynite was first found, and the locality of the crystals under discussion in sample 2298 from the Alaskan concentrate file (collected by R. M. Moxham, 1954).

The geology of this area was described by Eakin (1913), Mertie (1934), and Waters (1934), and briefly summarized by Moxham (1954) and Wayland (1961). Recent mapping in the area by Robert M. Chapman (written communication, 1974) has been done to better define the major geologic units. The district is mainly underlain by weakly metamorphosed to unmetamorphosed conglomerates, sandstones, shales, and limestones of Cretaceous age that have been intruded by Cretaceous to Tertiary granitic and quartz monzonitic stocks to the north, northeast, and southeast of the district. Small bodies of gabbro and diabase flank a quartz monzonite stock just north of the area shown in Figure 1, and some small lenses of serpentine are conformable to the east-northeast regional strike. Much of the surface is covered by Quaternary gravel, silt, clay, and muck of several erosion-and-deposition cycles.

Eschynite was not mentioned by Waters as an accessory mineral in any of the rocks, and its origin is not known. If eschynite is locally derived from the granitic rocks or the contact zones, these sites are not
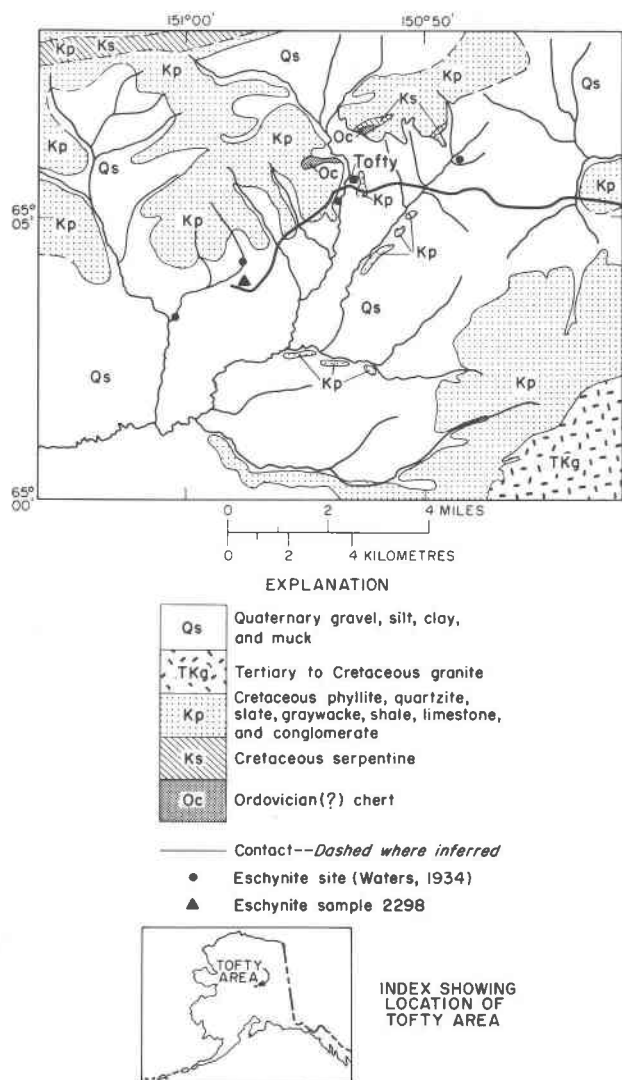
FIG. 1. Geologic sketch map showing locality of nioboeschynite-(Ce) in the Tofty area, Alaska. (Geology from Wayland, 1961, and R. M. Chapman, written communication, 1974.)

curves were made from spectrographically pure Nb_2O_5, CeO_2, TiO_2, and La_2O_3 to bracket the concentration ranges previously reported for Nb-rich eschynite.

A standard matrix was then prepared using the results obtained from the analyses of the above-noted oxides. A series of standards containing Fe_2O_3, ThO_2, SiO_2, CaO, Pr_2O_3, Nd_2O_3 and Y_2O_3 were prepared in this matrix to bracket suspected concentration levels in the mineral. The average of the five replicate analyses is shown in Table 1. We think that the total (106 percent) is a good value for the emission spectrographic procedure considering the small sample and the difficulties met in preparing matrices of sample and standards. Additional analyses from the literature are presented in Table 1 for comparison.

X-ray diffraction data were obtained from powdered eschynite on a collodion film in a Picker diffractometer. Table 2 lists unit-cell parameters determined from 23 reflections indexed and refined using a least-squares unit-cell refinement program (Evans, Appleman, and Handwerker, 1963); comparable physical, optical, and unit-cell data from the literature are also shown. Table 3 presents the X-ray powder diffraction data for Alaskan eschynite plus comparable data on samples from USSR and China.

Mineralogy

Eschynite in sample 2298 occurs as brittle, red-brown to dark brown, striated, slender prismatic, sand-size (0.2-1.5 mm) grains, mainly tabular but often stubby with a prominent cross fracture (Fig. 2). The mineral is generally translucent to opaque in the sand sizes, but thin fragments (0.01-0.1 mm) show red and red-brown colors when viewed against a glazed white-porcelain background. The streak is orange to light reddish brown; the hardness is estimated between 4 and 5 in the Mohs scale; the density (g/cm³) obtained on the Berman balance by averaging 7 determinations (range = 4.99 to 5.11) is 5.04; and the best extraction range in the Frantz magnetic separator (side tilt 15°, forward tilt 20°–23°) is 0.7 to 0.8 amp.

Under the microscope, in methylene iodide (n = 1.73), eschynite grains about 30 microns thick show an intense red-brown color and sparse turbidity due to opaque dust that is optically indeterminate. Pleochroism is weak to nil despite strong anisotropy; α is slightly darker than γ in a few grains. Refractive indices shown in Table 2 were estimated by comparison with selenium-sulfur mixtures (Merwin and Larsen, 1912) whose indices were determined by the

yet recognized or are now covered by Quaternary gravels and silts.

Analytical Procedures

The sample was analyzed by optical emission spectroscopy using a Jarrell-Ash 1.5 m Wadsworth-mounted spectrograph. A 2 mg sample was ground to a fine powder and mixed 1:10 with graphite containing 2 percent Ta_2O_5 as an internal standard. Five replicate samples were burned to completion; a 3-second preburn and a 105-second exposure were used, and the spectra were recorded on 35 mm Kodak Spectrum-Analysis No. 1 film. Analytical standard

TABLE 1. Analyses of Eschynite (in Percent)

Sample Reference	2298 This paper	1 Zhabin et al (1962)	2 Chang (1962)	3 Zhabin et al (1961)	4 Gorzhevskaya and Sidorenko (1964)	5 Borneman-Starynkevich (1941)
Location	Central Alaska	Urals, USSR	North China	Urals, USSR	China	Urals, USSR
CaO	4.52	2.60	3.54	4.82	5.34	2.65
FeO	1.09	N.R.*	6.12	N.R.	0.26	1.14
Fe_2O_3	N.A.*	1.43	N.R.	2.75	1.28	1.56
Al_2O_3	N.D.*	N.R.	0.15	0.35	3.80	N.R.
$[RE]_2O_3$	N.A.	31.93**	N.A.	28.17***	27.03†	N.A.
La_2O_3	4.9	**	19.61††	***	†	13.43†††
Ce_2O_3	15.8	**	11.56	***	†	10.84⁻
Pr_2O_3	1.9	**	††	***	†	†††
Nd_2O_3	5.6	**	††	***	†	†††⁼
Y_2O_3	0.18	**	0.70⁼	***	†	0.89⁻
U_3O_8	0.01	0.30	0.83	N.R.	0.08≡	N.R.
ThO_2	1.33	0.72	2.15	2.52	3.75	17.42
SiO_2	3.51	0.65	0.55	0.35	3.84	0.50
TiO_2	20.2	22.53	12.13	18.73	17.10	23.79
Nb_2O_5	47.0	33.70	41.13	41.41	35.90	25.35
Ta_2O_5	<0.1	N.R.	0.51	N.R.	None	0.94
H_2O+	N.D.	1.30	0.64	0.41	0.57	0.94
H_2O-	N.D.	0.36	0.10	0.04	0.40	0.14
Total	106‡	100.52	99.77‡‡††	99.55	100.99‡‡‡	99.96ˣ

* N.R. = not reported; N.A. = not applicable; N.D. = not determined.

** Rare-earth oxides = La_2O_3, 13; Ce_2O_3, 42; Pr_2O_3, 8; Nd_2O_3, 25; Sm_2O_3, 3.6; Eu_2O_3, 0.5; Gd_2O_3, 0.2; Tb_2O_3, 0.2; Dy_2O_3, 1.2; Ho_2O_3, 0.1; Er_2O_3, 0.5; Yb_2O_3, 0.1; Y_2O_3~4; Σ = 98.4.

*** Rare-earth elements = La, 14; Ce, 42; Pr, 9; Nd, 25; Sm, 3.1; Eu, 0.4; Gd, 1.3; Tb, 0.2; Dy, 1.2; Ho, 0.3; Er, 0.3; Yb, 0.2; Y~3; Σ = 100.00.

† Rare-earth oxides = La_2O_3, 21.2; Ce_2O_3, 48.7; Pr_2O_3, 4.1; Nd_2O_3, 19.4; Sm_2O_3, 1.7; Gd_2O_3, 3.6; $(Y, Dy, Ho)_2O_3$, 1.3; Σ = 100.00.

†† Reported as $[Ce]_2O_3$, cerium-group oxides.

††† Reported as $(La, Nd, Pr)_2O_3$.

⁻ Reported as CeO_2.

⁼ Reported as $[Y]_2O_3$, yttrium-group oxides.

≡ Reported as UO_3.

‡ Also, minor elements (in parts per million) are: Ba, 2000; Be, 10; Cr, 300; Cu, 30; Mg <10; Mn, 40; Pb, 100; and Sb, 50.

‡‡ Includes MgO, 0.05; MnO, trace.

‡‡‡ Includes MgO, 0.56; MnO, trace; Na_2O, 0.06; K_2O, 0.06; P_2O_5, 0.39; F, 0.15; and ignition loss, 0.42.

ˣ Includes MnO, 0.10; PbO, 0.12; SnO_2, 0.05.

minimum-deviation method (Ford, 1932, p. 242–243).

The range of values for $2V$ is for 10 measurements made on the universal stage either by direct readings from one optic axis to the other or by plotting one axis and the principal optic directions on a stereonet and computing the $2V$. The median value is (−) 85; the average value is (−) 84.6. The γ-direction is parallel to the striations, and these are in the crystallographic *a*-direction according to Palache *et al* (1944, p. 794). The tabular form is apparently in the (010) plane. The β-direction is in this plane and perpendicular to the striations; therefore, it is parallel to the crystallographic *c*-direction, and α must be

TABLE 2.   Physical, Optical, and Unit-Cell Data on Nioboeschynite-(Ce)

Sample Reference Location	2298 This paper Central Alaska	1* Zhabin et al (1962) Urals, USSR	2 Chang (1962) North China	3 Zhabin et al Urals, USSR	4 Gorzhevskaya and Sidorenko (1964) China
Density (g/cm^3)	5.04	4.97	5.056	5.132	4.55-4.82
Pleochroism, α β γ	reddish brown reddish brown reddish brown	yellowish brown brown darkish brown	yellowish red N.R.** dark reddish brown	red N.R. brown	golden brown N.R. reddish brown
R.I.(Na), α β γ n	2.27 2.32 2.36 N.A.**	2.28 N.R. 2.34 N.A.	N.R. N.R. N.R. >2.00	N.R. N.R. N.R. 2.26	N.R. 2.375 2.43-2.50 N.A.
2V α	75°-97°	N.A.	ca. 80°	N.R.	N.A.
2V γ	N.A.	75°	N.A.	N.R.	large
unit cell (Å), a b c	5.396 [1]*** 11.085 [2] 7.585 [3]	5.38 11.08 7.86	5.32 11.09 7.52	5.355 11.01 7.516	5.33 10.97 7.50
cell volume, Å^3	453.7 [1]	468.5	443.7	443.1	438.5

* Sample numbers same as in Tables 1 and 3.
** N.R. = not reported; N.A. = not applicable.
*** Standard errors in brackets refer to the last decimal place.

parallel to *b*. Sommerauer and Weber (1972, p. 80) indicated that α and γ are reversed in an eschynite-(Y) from Switzerland. This may be due to the dominant yttrium in that sample as compared to the dominant cerium in our Alaskan mineral. Also, Zhabin *et al* (1962, p. 86) indicated that γ lies along *c*, and α along *a* in a nonmetamict eschynite from the Urals. This would make the optic axial plane (010) instead of (001) as in the Alaskan and Swiss minerals.

Discussion

Nomenclature

Four main mineral species—euxenite, polycrase, priorite, and eschynite—have similar chemistry and morphology. Additional names, such as blomstrandine (≡ priorite) and lyndochite (≡ niobium-rich eschynite), were introduced respectively by Brögger (1906, p. 99) and Ellsworth (1927, p. 212). Adamson (1942, p. 108–110) and Fleischer (1966) showed that blomstrandine and lyndochite are varieties of what was lately called the eschynite-priorite series and was more recently designated the eschynite group (Levinson, 1966).

Brögger (1906, p. 92, 104) divided the minerals euxenite, polycrase, eschynite, and blomstrandine into two series, both orthorhombic: the euxenite-polycrase series and the eschynite-blomstrandine series. Each was said to form an isomorphic series, and the two series had different morphologies. The concept of two series proposed by Brögger from chemical considerations was questioned by Adamson

(1942, p. 109), Aleksandrov and Pyatenko (1959), and Lima de Faria (1964, p. 45), the latter two using X-ray studies of heated metamict minerals.

There has been little or no ambiguity on the identity of eschynite. Hintze (1938, p. 2) stated that it is essentially a niobate and titanate (thorate) of the cerium metals, with calcium and iron. Adamson (1942, p. 110) indicated that eschynite characteristically is enriched in cerium and the cerium-subgroup metals, and Komkov (1963) and Fleischer (1966, p. 804) adopted the concept of the eschynite-priorite series, in which the cerium-dominant mineral is eschynite and the yttrium-dominant mineral is priorite. This series name was apparently first employed by Palache *et al* (1944, p. 793–796).

The general formula of the eschynite-group minerals is AB_2O_6, with A = rare-earth elements, Th, Ca, Fe^{2+}, Mg, Mn, and other divalent ions; B = Nb and Ti; and O may include OH and F. The rare-earth elements have been divided into different Ce- and Y-groups by different investigators (Murata *et al,* 1957, p. 148; Semenov and Barinskii, 1958, p. 418), but for our purposes we use the division of the rare-earth elements by Rankama and Sahama (1950, p. 517) into the cerium group from lanthanum to europium (atomic nos. 57–63), and the yttrium group from gadolinium to lutetium (64-71) plus yttrium (39). As noted above, when cerium or cerium-group elements are dominant the mineral was called eschynite, but where yttrium or yttrium-group elements are domi-

nant the mineral was referred to as priorite. These are now called eschynite-(Ce) and eschynite-(Y), respectively, following Levinson's (1966) system of nomenclature. For the Alaskan material, the dominant Ce and La plus high Nb indicate the name should be nioboeschynite-(Ce) according to Levinson's system. Apparently the term nioboeschynite was first used by Zhabin, Mukhitdinov, and Kazakova (1960, p. 67).

Chemical Composition

This central Alaskan eschynite is similar in composition to the 5 nioboeschynites-(Ce) reported in the literature. These latter average about 3 percent less TiO_2 and 6 percent less Nb_2O_5. Only the nioboeschynite-(Ce) reported by Vlasov (1964, p. 490; 52.22 percent) and by Heinrich and Levinson (1961, p. 1436; 49.4 percent) have higher Nb_2O_5 contents. Ce and La oxides are not always re-

ported separately, and Ce-group metals (in X-ray fluorescence and electronmicroprobe analyses) are often lumped together; hence comparisons of these are not easily made. Total Ce-group oxides of Alaskan eschynite is 7 percent less than the average of the 5 nioboeschynites-(Ce). The Ce_2O_3 content of Alaskan eschynite is slightly less than the average for all eschynites; the Ce_2O_3 content of these eschynites ranges from 10.84 (Borneman-Starynkevich, 1941) to 25.4 percent (Heinrich and Levinson, 1961). Y_2O_3, ThO_2, U_3O_8, and Fe_2O_3 are considerably below average, but CaO is slightly above average, and SiO_2 (probably a contaminant) is considerably above average. Small amounts (generally less than 1 percent) of PbO, MnO, MgO, SnO_2, Al_2O_3, P_2O_5, F, and H_2O were noted in eschynite by previous authors (Hintze, 1938; Borneman-Starynkevich, 1941; Zhabin *et al*, 1961, 1962; Chang, 1962; Gorzhevskaya and Sidorenko, 1962).

The structural formula computed from the chem-

TABLE 3. X-Ray Powder Diffraction Data for Nioboeschynite-(Ce)

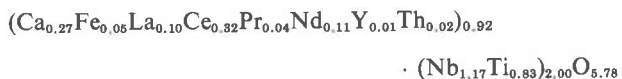| Sample Reference	2298* This paper			1 Zhabin et al (1962)		2 Chang (1962)	
h k l	d(calc)	d(obs)	I	d(obs)	I	d (obs)	I
020	5.543	5.539	2	--	--	5.36	2
021	4.475	4.479	1	--	--	4.37	3
111	4.087	4.082	1	--	--	4.04	2
002	3.793	3.789	1	--	--	--	--
120	--	--	--	--	--	3.78	2
121	3.445	3.445	2	--•	--	--	--
022	3.130	3.132	4	--	--	--	--
130	3.049	3.048	7	3.04	6	3.01	9
112	2.988	2.983	10	2.98	10	2.912	10
131	2.829	2.827	2	2.83	2	2.78	3
040	2.771	2.773	1	--	--	--	--
200	2.698	2.699	2	2.69	3	2.645	5
041	2.603	2.602	1	--	--	--	--
140	2.465	2.464	1	2.47	1	2.428	3
221	2.311	2.309	1	2.29	2	--	--
103	2.290	2.289	1	--	--	2.260	4
042	--	--	--	2.23	1	--	--
113	--	--	--	--	--	2.210	3
230	--	--	--	2.165	1	--	--
231	2.094	2.091	1	--	--	--	--
222	2.044	2.044	2	2.036	2	--	--
151	1.980	1.980	1	1.971	1	--	--
004	1.896	1.896	1	1.891	3	--	--
043	--	--	--	1.866	1	--	--
061	--	--	--	1.785	1	--	--
242	1.722	1.722	2	1.713	3	--	--
062	--	--	--	1.661	2	--	--
134	1.610	1.611	2	1.604	5	--	--
322	1.559	1.560	1	1.550	3	--	--
170	1.520	1.521	1	1.509	1	--	--

* Nickel-filtered CuKα radiation, $\lambda = 1.5418$ Å.
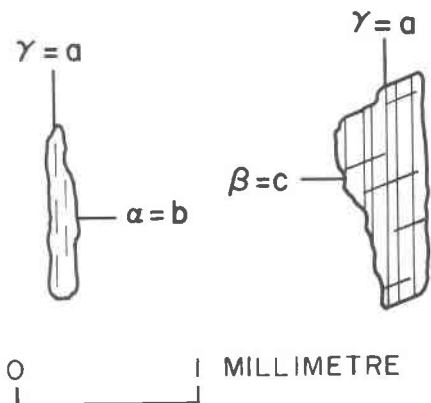
FIG. 2. Orientation diagram for nonmetamict nioboeschynite-(Ce) from Alaska.

ical analysis by subtracting SiO_2 and setting (Nb, Ti) to 2.00 is:

$$(Ca_{0.27}Fe_{0.05}La_{0.10}Ce_{0.32}Pr_{0.04}Nd_{0.11}Y_{0.01}Th_{0.02})_{0.92}$$
$$\cdot (Nb_{1.17}Ti_{0.83})_{2.00}O_{5.78}$$

Optical Properties

A brief comment is required on the range of the optic axial angle. This angle is sensitive to changes in the refractive indices relative to one another. At least 15 grains were crushed to tiny fragments to find good orientations for measurement of the angle. They are considered a heterogeneous mixture although they were picked from the same alluvial concentrate. The optic axial angle results indicate that more work is required on single crystals to determine the ranges in each analytical set.

X-ray Data

Lima de Faria (1964) discussed the effects of heating on the X-ray patterns of eschynites, especially metamict varieties. In our study of the effects of heat on Alaskan eschynite, a few grains were powdered, an X-ray pattern was obtained, the sample was carefully transferred to a crucible and heated at 800°C for one hour, and another X-ray pattern run. The pattern for heated eschynite showed a small shift (0.01–0.03 Å) to smaller d values for all peaks, relative to the peaks of a fluorite internal standard; three peaks increased and eleven decreased in relative height, but eight remained unchanged; and one small peak disappeared. The data compare very well with those of Komkov (1960, p. 585) for artificial eschynite, but are somewhat different from those of two eschynites presented by Lima de Faria (1964, Table 39). Cell parameters for heated Alaskan eschynite are $a = 5.342[2]$, $b = 11.007[3]$, $c = 7.508[2]$ Å, $V =$

441.4[2] Å^3 (standard errors in brackets refer to the last decimal place).

A laboratory approach to the unit-cell parameters of the several rare-earth element niobotitanates has been taken by at least two groups of investigators. The unit-cell parameters of the artificial La- to Lu-niobotitanates produced by Komkov *et al* (1964, p. 151) are almost identical to those plotted on a diagram by Seifert and Beck (1965, p. 16). On this diagram, Alaskan eschynite *b*- and *c*-dimensions are close to the flat slopes of the values for these parameters, but the *a*-dimension plots on the slope of the *a*-values for artificial eschynites between cerium and praseodymium values.

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