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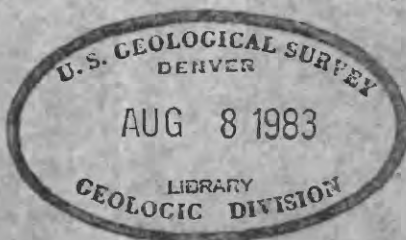
UNITED STATES DEPARTMENT OF THE INTERIOR  
GEOLOGICAL SURVEY

TRACE ELEMENTS INVESTIGATIONS  
THE OCCURRENCE OF COLUMBIUM AND TANTALUM

by

Michael Fleischer and James O. Harder

Trace Elements Investigations--Report No. 14



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TRACE ELEMENTS INVESTIGATIONS  
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ABSTRACT

Columbium and tantalum occur most abundantly in certain minerals which form late in the general process of magmatic differentiation, including the late hydrothermal stages. These minerals occur almost without exception in granitic and syenitic pegmatites and placers derived therefrom. Among the metallic elements, columbium and tantalum are most closely associated with titanium, zirconium, uranium, rare earths, and tin, as might be expected from their ionic radii. The  $Cb_2O_5$  +  $Ta_2O_5$  content of the minerals varies from a trace to 86 percent. The high value is for columbite-tantalite, the most common ore mineral of columbium and tantalum.

The domestic reserves of columbium-tantalum minerals are estimated to be 875,800 pounds of all minerals as of January 1, 1945. No grade figures are reported.

Products of tin smelters and plants which treat minerals chemically for elements such as titanium and tungsten should be investigated for columbium on the basis of association.

INTRODUCTION

This report on the occurrences of columbium and tantalum is the fourth in a series of similar reports to be prepared by the Geological Survey discussing possible sources of several rare or uncommon elements for which there are war uses. Other elements will be discussed in forthcoming reports.



Columbium<sup>1</sup> and tantalum are chemical elements with atomic numbers 41

<sup>2</sup>/ Niobium (Nb) is generally used in place of columbium (Cb) in the European literature.

and 73, and atomic weights 92.91 and 180.88, respectively. They belong to the fifth group of the periodic table and are quinquivalent in most of their compounds. The history of these elements, their properties and those of their compounds, are summarized by Mellor (<sup>19</sup>15). Their chemistry and metallurgy have been summarized recently (1, 20).

### GEOCHEMICAL CONSIDERATIONS

#### General statement

Columbium and tantalum are relatively rare elements, whose exact position in the order of abundance of the elements is uncertain. Analysis for these elements is unusually difficult, which may explain the paucity of data on their distribution and the widely varying estimates of their abundance. Thus Clarke and Washington in 1924 (5) gave 0.003 percent as the average columbium plus tantalum content of igneous rocks, whereas two years earlier they had given it as  $n \times 10^{-6}$  percent. More recently, von Hovey and coworkers (14, 15) gave  $3.2 \times 10^{-5}$  percent Cb and  $2.4 \times 10^{-5}$  percent Ta for the average contents of eruptive rocks, Goldschmidt (12) gave 0.002 percent Cb and 0.0015 percent Ta, and van Tongeren (24) estimated 0.001 percent Cb to be the average content of the rocks of the Dutch East Indies.

Columbium and tantalum are lithophilic elements that occur almost exclusively in igneous rocks. In contrast to dispersed elements such as gallium and indium, columbium and tantalum are so concentrated during the processes of magmatic differentiation that they are major constituents of many minerals. These minerals, almost without exception, occur in granitic and syenitic pegmatites.

The empirical ionic radius of columbium is given as 0.69A. and that of tantalum must be nearly the same. Columbium and tantalum are associated closely with titanium and zirconium, and to some extent with tin and tungsten. These associations would be anticipated from the ionic radii (titanium 0.64, tin 0.74, tungsten approximately 0.6), except for zirconium, whose ionic radius is given as 0.87A., but may well be smaller. Molybdenum minerals have apparently not been tested for the presence of columbium and tantalum, although the ionic radius of molybdenum (0.6 - 0.7) suggests a possible association especially in molybdenite from pegmatites. Some molybdenum ores tested spectrographically by Rabbitt (25) did not contain measurable amounts of columbium, but no molybdenum mineral was tested.

#### Occurrences in specific minerals and rocks

The following list includes all the minerals that are reported to contain columbium or tantalum which are recognized as valid species and some that are of dubious validity. The complexity of composition and the difficulty of analysis make it impossible to systematically classify all these minerals accurately. They have been divided into two groups: (1) Minerals in which columbium or tantalum is a major constituent, and (2) Minerals in which these elements are minor constituents. Within each group, the arrangement is roughly in order of possible importance as a source of columbium and tantalum. For the many rare and ill-defined species, a rough grouping of like minerals has been attempted. Unless otherwise indicated, the data given are from volume 1 of the Seventh Edition of Dana's System of Mineralogy (6) and from the mineralogical file of the Section of Chemistry and Physics, Geological Survey, Department of the Interior.

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(1) Minerals in which columbium and/or tantalum is a major constituent

Columbite-Tantalite Series  $(Fe, Mn) (Cb, Ta)_2O_6$  varying from columbite containing little tantalum to tantalite containing little columbium. The  $Cb_2O_5 + Ta_2O_5$  content is 75 to 86 percent. This is the principal ore of columbium and tantalum. Columbite-tantalite and tapiolite are the ores from which 95% or more of these metals are produced outside the U.S.S.R. (See loparite, below.) Columbite-tantalite occurs almost exclusively in granite pegmatites, especially in those rich in lithium minerals. Although of widespread occurrence, it is generally sparsely disseminated, and relatively few of the occurrences contain large amounts. It has also been found in some placer deposits, and occurs in the cryolite deposits at Ivigtut, Greenland. A summary account of columbite-tantalite deposits has been published by Mathieu (18), and an account of the Russian deposits by Gerasimovskiy (10).

Tapiolite-Mossite Series,  $(Fe, Mn) (Ta, Cb)_2O_6$ , varying from tapiolite with little columbium to mossite with  $Cb:Ta$  approximately 1:1. The  $Cb_2O_5 + Ta_2O_5$  content is 79 to 85 percent. This tetragonal dimorph of orthorhombic columbite-tantalite is generally considered to be a rare mineral, but is actually of common occurrence. Much of the so-called tantalite has been found by x-ray study to be tapiolite [Geological Survey Laboratory (25)]. It occurs in granitic pegmatites, generally associated with tantalite.

Loparite, essentially  $(Ce, Na, Ca)_2 (Ti, Cb)_2O_6$ , a member of the perovskite group. The  $Cb_2O_5 + Ta_2O_5$  content is 9 to 11 percent, with columbium predominating over tantalum. Loparite is a rare mineral, known only from nepheline syenite pegmatites of the Kola Peninsula, U.S.S.R. where, however, it occurs in large amounts and may be an important source of columbium. The deposits have been described (8,9,10).

Perovskite,  $\text{CaTiO}_3$ , with columbium and tantalum substituting for titanium, and rare earths and sodium for calcium (compare loparite). The columbian variety is called dysanelyte. The  $\text{Cb}_2\text{O}_5 + \text{Ta}_2\text{O}_5$  content ranges from traces to 26 percent and is particularly high in samples from contact zones of nephelinitic rocks. The columbian variety of perovskite is an uncommon mineral that generally occurs in small amounts.

Pyrochlore-Microlite Group (including the varieties columbomicrolite, kop-pite, hatchettolite, ellsworthite). Formula essentially  $(\text{Na,Ca})_2 (\text{Cb,Ta})_2\text{O}_6 (\text{O,OH,F})$ , but with variable amounts of uranium and rare earths replacing calcium and with various elements, especially titanium, replacing columbium and tantalum. The  $\text{Cb}_2\text{O}_5 + \text{Ta}_2\text{O}_5$  content is generally 56 to 78 percent, but may be as low as 40%, particularly in uranian varieties. Tantalum commonly predominates over columbium. These minerals are of uncommon occurrence and are present in small amounts in most occurrences. However, at a few localities, they occur in sufficient amounts to be mined as a source of tantalum. They occur in granitic pegmatites and in nepheline syenites and related rocks, also in placers derived from such rocks.

Scheteligite is a very rare complex mineral related to the Pyrochlore-Microlite Group. It contains 29 percent  $\text{Cb}_2\text{O}_5 + \text{Ta}_2\text{O}_5$ . It occurs in granitic pegmatite.

Hjelmsite is a very rare, chemically complex mineral similar to microlite in composition and containing 62 to 70 percent  $\text{Cb}_2\text{O}_5 + \text{Ta}_2\text{O}_5$ . It occurs in granitic pegmatites.

Rutile,  $\text{TiO}_2$  (columbian and tantalian varieties, also called ilmenorutile and strueverite.) These are solid solutions of  $\text{TiO}_2$  containing variable amounts of the tapiolite-mossite molecules,  $\text{Fe}(\text{Ta,Cb})_2\text{O}_6$ . The  $\text{Cb}_2\text{O}_5 + \text{Ta}_2\text{O}_5$  content ranges from traces to 47 percent. The varieties of rutile that contain appreciable percentages of columbium and tantalum are rare minerals, occurring in

granitic and syenitic pegmatites.

Tantalum, Ta. Tantalum metal, containing 1.5% Cb, has been reported to occur as a rarity in some placers, but the occurrences need verification.

Simpsonite,  $\text{Al}_5\text{Ta}_4\text{O}_{19}$ , containing up to 6%  $\text{Cb}_2\text{O}_5$ . The  $\text{Cb}_2\text{O}_5 + \text{Ta}_2\text{O}_5$  content is 70 to 75 percent. A rare mineral occurring in granitic pegmatites. It has been reported to occur in placers in sufficient amount to serve as a minor source of tantalum.

Stibiotantalite-Stibiocolumbite Series,  $\text{Sb}(\text{Cb}, \text{Ta})\text{O}_4$ , nearly the entire series being known. The  $\text{Cb}_2\text{O}_5 + \text{Ta}_2\text{O}_5$  content is 50 to 60 percent. It is a rare mineral, occurring in small amounts in lithium-rich granitic pegmatites and in some placers derived therefrom.

Bismutotantalite,  $\text{Bi}(\text{Ta}, \text{Cb})\text{O}_4$ , with tantalum predominating. The  $\text{Cb}_2\text{O}_5 + \text{Ta}_2\text{O}_5$  content is 45 to 47 percent. It is a very rare mineral that occurs in granitic pegmatite.

Thoreaulite,  $\text{SnTa}_2\text{O}_7$ , containing  $\text{Cb}_2\text{O}_5$  trace,  $\text{Ta}_2\text{O}_5$  73 percent. It is a very rare mineral, occurring in granitic pegmatite.

Following is a group of complex, poorly defined columbates and tantalates of the rare earths.

Euxenite-Polycrase Series, essentially  $\text{AB}_2\text{O}_6$ , with A = chiefly yttrium and calcium, also cerium, uranium, and thorium; B = chiefly columbium, tantalum, and titanium. The  $\text{Cb}_2\text{O}_5 + \text{Ta}_2\text{O}_5$  content ranges from 20 to 51 percent. It is an uncommon mineral occurring in granitic pegmatites, but only rarely in appreciable amounts. It has also been found in placers.

Khlopinite and Eschwegeite are very rare minerals similar to euxenite in composition and occurrence.

Eschynite-Priorite Series, including blomstrandine. These minerals are similar to the euxenite-polycrase series in composition, but differ in crystal



structure. The  $\text{Cb}_2\text{O}_5 + \text{Ta}_2\text{O}_5$  content ranges from 16 to 37 percent. Columbium generally predominates over tantalum. These are uncommon minerals that occur, generally in small amounts, in nepheline-syenite and related rocks and in granitic pegmatites.

Samaraskite is similar in composition to euxenite, but differs in crystal structure. The  $\text{Cb}_2\text{O}_5 + \text{Ta}_2\text{O}_5$  content ranges from 43 to 60 percent. Columbium generally predominates over tantalum. Samarskite is an uncommon mineral that occurs, generally in small amounts, in granitic pegmatites.

Fergusonite-Ferrosite Series, essentially  $\text{Y}(\text{Cb,Ta})\text{O}_4$ , with variable amounts of calcium, cerium, and uranium substituting for yttrium, and with titanium and other elements substituting for columbium and tantalum. Columbium generally predominates over tantalum. The  $\text{Cb}_2\text{O}_5 + \text{Ta}_2\text{O}_5$  content ranges from 42 to 58 percent. Fergusonite is an uncommon mineral that occurs, generally in small amounts, in granitic pegmatites, and in placers derived therefrom.

Yttrotantalite is similar to fergusonite in composition, but differs in crystal structure. The  $\text{Cb}_2\text{O}_5 + \text{Ta}_2\text{O}_5$  content is 55 to 60 percent. It is a very rare mineral that occurs in granitic pegmatites.

Ishikawaite is a very rare mineral similar to yttrotantalite in composition and occurrence. The  $\text{Cb}_2\text{O}_5 + \text{Ta}_2\text{O}_5$  content is 52 percent.

Polymignite and Loranskite are very rare, ill-defined minerals similar to yttrotantalite in composition, but containing much zirconium. The  $\text{Cb}_2\text{O}_5 + \text{Ta}_2\text{O}_5$  contents reported range from 13 to 48 percent. These are very rare minerals, occurring in small amounts in granitic pegmatites, nepheline-syenites, and augite-syenite pegmatites.

Betafite Group (including betafite, samiresite, dialumite and amangebeite). A group of complex, ill-defined minerals of doubtful formula, containing chiefly columbium, tantalum, titanium, calcium, and uranium. The  $\text{Cb}_2\text{O}_5 + \text{Ta}_2\text{O}_5$  content

ranges from 18 to 73 percent. These rare minerals all occur in granitic pegmatites.

Fersmanite,  $(\text{Ca}, \text{Na})_2 (\text{Ti}, \text{Cb}) \text{SiO}_5 \text{F}$ . The  $\text{Cb}_2\text{O}_5 + \text{Ta}_2\text{O}_5$  content is about 15 percent, nearly all of which is columbium. It is a rare mineral that occurs in nepheline syenite pegmatite.

Murmenite, approximately  $(\text{Na}, \text{Ca})_3 (\text{Ti}, \text{Cb})_5 \text{Si}_5\text{O}_{19} (\text{OH})_7 4\text{H}_2\text{O}$ . It contains about 8 percent  $\text{Cb}_2\text{O}_5$  and 1 percent  $\text{Ta}_2\text{O}_5$ . It is a rare mineral that occurs in nepheline syenite pegmatite with loparite and fersmanite and is therefore a minor source of columbium in the treatment of loparite.

Epistolite is a sodium columbium silicate of uncertain formula. The only analysis gave 34 percent  $\text{Cb}_2\text{O}_5$ . It is a very rare mineral that occurs in nepheline syenite pegmatite.

Wöhlerite, perhaps  $\text{NaCa}_2 (\text{Zr}, \text{Cb}) \text{Si}_2\text{O}_7 (\text{O}, \text{OH}, \text{F})_2$ . The  $\text{Cb}_2\text{O}_5 + \text{Ta}_2\text{O}_5$  content is 13 to 15 percent, with columbium predominating over tantalum. Wöhlerite is a very rare mineral that occurs in nepheline syenite pegmatites.

Lävenite and Hjortdahlite are sodium calcium zirconium silicates, like wöhlerite, but are reported to contain up to 5.2 percent and 2 percent, respectively, of  $\text{Cb}_2\text{O}_5 + \text{Ta}_2\text{O}_5$ . They are very rare minerals that occur in nepheline syenite pegmatites.

## (2) Minerals in which columbium or tantalum is a minor constituent

Wolframite,  $(\text{Fe}, \text{Mn})\text{WO}_4$ . Few analyses of wolframite report columbium or tantalum, but these may have been overlooked (16). The content of columbium and tantalum might be expected to be low, except for samples from pegmatites, but the data in the literature are too few to allow any estimate of average content. The maximum content recorded is 2.2 percent  $\text{Cb}_2\text{O}_5 + \text{Ta}_2\text{O}_5$ .

Scheelite,  $\text{CaWO}_4$ , has been reported to contain 0.10 percent  $\text{Cb}_2\text{O}_5 + \text{Ta}_2\text{O}_5$  (4), but the method of analysis used has been challenged (22). No other analysis of scheelite shows columbium or tantalum.

Ilmenite,  $\text{FeTiO}_3$ . Columbium and tantalum have been reported in only a few analyses of this very common mineral, but may not have been looked for. The maximum content of  $\text{Cb}_2\text{O}_5 + \text{Ta}_2\text{O}_5$  reported is 1.3 percent from syenitic pegmatite, which would be expected to be relatively high in columbium + tantalum content (14, 21, 26, 27). Two black sands from Norway and India contained 0.18 and 0.02 percent, respectively, of  $\text{Cb}_2\text{O}_5 + \text{Ta}_2\text{O}_5$  (14). These were presumably the "ilmenite" sands of commerce, which may be ilmenite, arizonite, or amorphous material. The only analysis of arizonite shows neither columbium nor tantalum, but the similar mineral,

Kalkowsk<sup>s</sup>ite contains 1.7 percent  $\text{Cb}_2\text{O}_5 + \text{Ta}_2\text{O}_5$ .

Cassiterite,  $\text{SnO}_2$ . This very common mineral has been reported to contain columbium and tantalum in amounts ranging from spectrographic traces to over 10 percent  $\text{Cb}_2\text{O}_5 + \text{Ta}_2\text{O}_5$ . Some of the samples analyzed probably were not homogeneous and contained intergrown columbite-tantalite or tapiolite, but there is evidence that cassiterite may contain columbium and tantalum in solid solution (7). Cassiterite from pegmatite veins contains more columbium than cassiterite from hydrothermal sulfide-bearing veins (3, 17).

Brookite,  $\text{TiO}_2$ , has been reported to contain spectrographic traces of columbium and tantalum. No quantitative data are available.

Zircon,  $\text{ZrSiO}_4$ . Most analyses of this very common mineral do not report columbium or tantalum, which may have been overlooked. However, zircons that contain appreciable amounts of rare earths and uranium also generally contain columbium and tantalum, the maximum content reported being 7.7 percent  $\text{Cb}_2\text{O}_5 + \text{Ta}_2\text{O}_5$ . This variety of zircon (to which several special names such as cyrtolite, naegite and hagatelite have been given) is of rare occurrence in granitic pegmatites.

Sphene,  $\text{CaTiSiO}_5$  commonly contains columbium and tantalum in amounts ranging from traces to about 2 percent  $\text{Cb}_2\text{O}_5 + \text{Ta}_2\text{O}_5$  (2, 14). The highest



percentages reported are for samples from nepheline syenite pegmatites. Columbium generally predominates over tantalum.

Astrophyllite,  $(K, Na) Fe_3TiSi_3O_{11}(OH)$ , has been reported to contain up to 0.8 percent  $Ta_2O_5$ . It is a very rare mineral that occurs in nepheline syenite pegmatites.

Rudialyte, approximately  $(Na, Ca)_5 (Zr, Fe)_2 Si_6O_{18}(OH, Cl)$ . It has been reported to contain up to 3.5 percent  $Cb_2O_5 + Ta_2O_5$ , with tantalum predominating over columbium. Rudialyte is a rare mineral that occurs in nepheline syenite pegmatites.

Lorenzenite (including the variety ramsayite),  $Na_2 (Ti, Zr) Si_2O_9$ , is reported to contain columbium, but no quantitative data have been published. It is a very rare mineral that occurs in granitic and nepheline syenite pegmatites.

Tritomite, a complex borosilicate of cerium, lanthanum, thorium, and calcium, is reported to contain up to 3.1 percent  $Ta_2O_5$ . It is a very rare mineral that occurs in nepheline syenite pegmatites.

Caryocerite-Melanocerite, a complex borosilicate of the rare earths and calcium, is reported to contain up to 3.5 percent  $Cb_2O_5 + Ta_2O_5$ . It is a very rare mineral that occurs in nepheline syenite pegmatites.

Steunstrupine, a complex phosphate-silicate of the rare earths, thorium, iron, and calcium, is reported to contain up to 4 percent  $Cb_2O_5 + Ta_2O_5$ . It is a very rare mineral that occurs in nepheline syenite pegmatites.

Polyolithionite,  $KLi_2AlSi_4O_{10}F_2$ , contains up to 1.5 percent  $Cb_2O_5$  (23). It is a very rare mica that occurs in granitic pegmatites.

## Rocks

### *Geochemical*

As stated above under General Considerations, p. 2, data on the columbium and tantalum content of rocks are not in good agreement. The recent published values (11,12,15,24) range from  $3.2 \times 10^{-5}$  percent Cb to 0.002 percent

Cb for the average content of igneous rocks. The highest values reported are 0.005 percent Cb for granite, 0.02 percent Cb for nepheline syenite (11). Less information is available on the tantalum content, which appears to be approximately two-thirds that of the columbium content.

#### Results of Mill Product Studies

The columbium content of more than 1000 samples from over 200 mines, mills, and geological occurrences, was determined spectrographically by Rabbitt (25) as part of the Geological Survey's Trace Elements investigations. Three of the samples each contained 0.2 percent  $\text{Cb}_2\text{O}_5$ . Two of these were from areas of alkali-syenite intrusives, some of which contain eudialyte. The third of these three samples is a placer concentrate from the Yentna district in Alaska. In addition to the three samples described above, 22 samples contained columbium in amounts ranging from 0.005 to 0.008 percent  $\text{Cb}_2\text{O}_5$ . All of these samples are listed in Table 1 on page 20 of this report.

It is noteworthy that excepting the placer sample the highest content of columbium was in the samples from alkali-syenite intrusive areas. This is in accord with previous experience which has shown columbium to be generally concentrated in the late differentiates of the magma. The only other important conclusion to be drawn from the mill product spectrographic study is a negative one. The remaining thousand odd samples which were examined and found to contain less than 0.005 percent  $\text{Cb}_2\text{O}_5$  included products from nearly every type of valuable mineral deposit and a variety of other geological occurrences. This evidence strengthens the relationship between columbium and late magmatic differentiates and makes hydrothermal deposits appear unattractive as possible columbium sources.

### Best Sources of Columbium and Tantalum

At present, columbium and tantalum are produced almost entirely from columbite-tantalite and tapiolite, with minor amounts from microlite and simpsonite, all from granitic pegmatites. The minerals, such as leoparite, associated with nepheline syenites appear to have been utilized only in the U.S.S.R., where the deposits are large. These minerals have low columbium + tantalum contents and are probably not attractive sources, even if titanium is also recovered.

Some production and reserve figures on the domestic columbium-tantalum supply were obtained by the Geological Survey. The figures were assembled from data supplied by various men. All of the figures are production or reserves for granitic pegmatites in the states indicated.

The production is given in pounds of mineral produced. Records are not available, and probably not existent, to show the proportion of columbium to tantalum in the various minerals listed and there are no figures to show the amount of pegmatite material from which the minerals were obtained. In nearly all cases the columbium-tantalum minerals are produced as by-products of the mining of a pegmatite body for other valuable minerals such as mica, feldspar, and spodumene. With one possible exception, it is doubtful if there is a single domestic mine which could operate for columbium-tantalum alone.

Reserve figures given in pounds of mineral are in part based on production records and estimates of volumes of pegmatite. Neither grade figures to show percentage of mineral in the pegmatite nor figures to show the ratio of columbium to tantalum have been included. As in past production, the reserves are chiefly dependent on the chance that they can be recovered as a by-product of pegmatite mining for other minerals.



Tabulation of domestic production and reserves of columbium-tantalum  
minerals:

Columbite--Production and reserves (U.S.)  
(Jan. 1, 1945)

State	Production		Reserves (lbs. columbite)		
	Period	Wt. lbs.	Measured	Indicated	Inferred.
Arizona					500
Colorado	1943	1,422		200	
New Mexico	1943	1,500			2,000
North Carolina	1943	4,219			6,000
South Dakota	1903-1908	66,000			120,000
Virginia	1934	50			
Totals		73,191		200	128,500
TOTAL				128,700	

Microlite--Production and reserves (U.S.)  
(Jan. 1, 1945)

State	Production		Reserves (lbs. columbite)		
	Period	Wt. lbs.	Measured	Indicated	Inferred.
Colorado				6,193	2,347
New Mexico			500	400,000	200,000
South Dakota					3,000
Totals			500	406,193	205,347
TOTAL				612,040	

Tantalite--Production and reserves (U.S.)  
(Jan. 1, 1945)

State	Production		Reserves (lbs. tantalite)		
	Period	Wt. lbs.	Measured	Indicated	Inferred
New Mexico	1942-1944	800	300	750	750
South Dakota	incomplete	1,381		30,000	60,750
Virginia	1929-1941	1,390		19,900	
Wyoming	1942	85		13,780	830
Totals		3,556	300	64,430	62,330
			TOTAL	127,060	

Mixed ores--Production and reserves (U.S.)  
(Jan. 1, 1945)  
(columbite, tantalite, microlite, hutchettolite, samarskite, etc.)

State	Production		Reserves (lbs. tantalite)		
	Period	Wt. lbs.	Measured	Indicated	Inferred
Idaho	?	500			
New England	1930-1942	<100			
New Mexico	?	700			8,000
North Carolina	1930-1942	4,000			6,000
South Dakota	1943	630			
Totals		5,930			8,000

Summary--Production and reserves (U.S.)  
of all columbium and tantalum minerals  
(Jan. 1, 1945)

Mineral	Production		Reserves (lbs. of ore mineral)		
	Period	Wt. lbs.	Measured	Indicated	Inferred
Columbite		73,191		200	128,500
Microlite		10,250	500	406,193	205,347
Tantalite		3,956	300	54,430	62,330
Mixed ore		5,930			8,000
				5823	
Totals		93,327	800	470,823	404,177
			TOTAL	875,800	



### Suggestions for Further Investigations

Further investigation should be directed to those minerals that contain columbium and tantalum, and that are treated chemically. Such minerals as rutile and zircon, that are marketed as such, are not possible sources.

Products of tin smelters should be investigated to learn the fate of the columbium and tantalum content of cassiterite during smelting, although the Bolivian ore used at the Longhorn Smelter is presumably low in columbium and tantalum.

The fate of the columbium and tantalum content of wolframite during the recovery of tungsten should be studied.

Products of plants producing titanium dioxide from "ilmenite" sands should be studied, though it seems likely that the columbium and tantalum would probably be concentrated in the final titanium dioxide product.

However, none of the foregoing possibilities appears now to offer a good chance for supplying important quantities of columbium. No better source is evident than the granitic pegmatites, such as now supply nearly all of the columbium. Furthermore the general distribution and mode of occurrence of these rocks in the United States appears to be quite well known and their content of columbium-bearing minerals is seldom more than one pound of mineral per ton of pegmatite material. In most cases it is much less. Therefore, the prospect for finding large quantities of columbium is not good. Probably an increase of price would stimulate production from known sources but, because most columbium is a by-product anyway the effect would undoubtedly be limited unless the price increase was very great.

Table I. Best columbium values resulting from mill products spectrography  
(Unpublished data of the Geological Survey)

Name of plant or occurrence	Location	Product	Columbium-bearing material	Percent Cb <sub>2</sub> O <sub>5</sub>
Sierra Diablo Intrusive	Van Horn, Texas	none	Gray porphyry	0.2
Wind Mtn. Intrusive	Corndas Peaks, N. M.	none	Altered limestone cut by dikes	0.2
Alaskan placer	Yentna dist., Alaska	Gold	Placer concentrates	0.2
Sierra Diablo Intrusive	Van Horn, Texas	none	Gray-green porphyry	0.08
"	"	none	Purplish-gray porphyry	0.06
"	"	"	Manganese-bearing vein	0.05
Vitro Mfg. Co.	Cannonsburg, Pa.	Radium	Radioactive sludge	0.02
Alaskan placer	Yentna dist., Alaska	Sand	Placer concentrates	0.01
Leyden Coal Mine	Golden, Colo.	Coal	Quartz-carnotite veins	0.01
Lincoln mine (mill)	Hiko, Nev.	Tungsten	Composite conc. (scheelite)	0.01
Reynolds Metals Co.	Listerhill, Ala.	Aluminum	Slimes from filter	0.01
Ris Mineral Co.	Vero Beach, Fla.	Titanium	Rutile concentrates	0.01
Titanium Alloy Co.	Molvena, Ark.	Titanium	Rutile concentrates	0.01
Leyden Coal Mine	Golden, Colo.	Coal	Siliceous coal	0.008
Ris Mineral Co.	Melbourne, Fla.	Titanium	Tails, magnetic separation	0.008
Vitro Mfg. Co.	Cannonsburg, Pa.	" -	PbS sludge (African ore)	0.008
Amer. Cyanamid Co.	Lakeland, Fla.	Phosphate	Pebble concentrates	0.007

Table I (continued)

Name of plant or occurrence	Location	Product	Columbium-bearing material	Percent Cb <sub>2</sub> O <sub>5</sub>
Jacksonville Beach	Jacksonville, Fla.	none	Entile, from black sand	0.007
Leyden Coal Mine	Golden, Colo.	Coal	Grab sample from dump	0.007
Amer. Cyanamid Co.	Swamp Foodle, Ark.	Aluminum	Iron tails	0.006
Reynolds Metals Co.	Listerhill, Ala.	"	Sand from thickener	0.006
Alaskan placer	Yentna dist., Alaska	Gold	Placer concentrates	0.005
Dulin Zeurite	Sweet Home, Ark.	Aluminum	Iron tails	0.005
Jacksonville Beach	Jacksonville, Fla.	none	Ilmenite	0.005
Lincoln mine (mill)	Hiko, Nev.	Tungsten	Sulfide flotation tails	0.005



Annotated Bibliography of Occurrences of Columbium and Tantalum

Descriptions of individual deposits are not included.

Note: The original papers were seen except those for which reference to an abstract journal is given.

1. Balke, C. W. Columbium and tantalum.  
Ind. Eng. Chem. 27, 1166-1169 (1935).

A brief review of methods of extraction from ores is included.

2. Bohmstedt, E. M. Sphene from the Khibine tundras.  
Trav. Lomonosov Inst. Acad. Sci. U.S.S.R. No. 7, 49-78  
(in English 77-78) (1936).

Eight analyses of sphene from nepheline syenites of the Kola Peninsula.

U.S.S.R., show  $Cb_2O_5$  contents up to 2 percent.

3. Borovick, S. A. and Cotman, J. D. Content of rare and other elements in cassiterites of different genesis from U.S.S.R. deposits according to spectrum analysis data.  
Compt. rend. acad. sci. U.R.S.S. 23, 351-354 (1939).

Qualitative spectrographic study of 27 cassiterites showed strong tests for columbium in pegmatite vein samples, moderate to none in samples from sulfide-cassiterite veins.

4. Carobbi, G. Ricerche analitiche sulla scheelite di Traversella.  
Gazz. chim. Ital. 54, 59-64 (1924); mineralog. Abstracts 3, 105 (1926).

Analysis of scheelite from Traversella, Piedmont, gave  $Cb_2O_5$  0.08,  $Ta_2O_5$  0.02%, but these results have been questioned, see (22).

5. Clarke, F. W. and Washington, H. S. The composition of the earth's crust.  
U. S. Geol. Survey, Prof. Paper 127, 117 pp. (1924).

The average content of igneous rocks is estimated, from scanty data, to be 0.003%  $Cb+Ta$ . Earlier, widely diverging, estimates are summarized.

6. Dana's System of Mineralogy. Seventh Edition, Volume I. John Wiley and Sons (1944).
7. Edwards, A. B. A note on some tantalum-columbium minerals from Western Australia.  
Proc. Australasian Inst. Mining Met. No. 120, 731-744 (1940).

This includes optical and chemical study that indicates some solid solution

of tantalum and columbium in cassiterite.

8. Eliseev, N. A. and Nefedov, N. K. The loparite deposits of Ilyavrita. *Proizvod. Sil'y Kol'skogo Polubostrova* 1940, No. 1, 77-118; Chem Abstracts 37, 1679 (1943).

A description of pneumatolytic deposits in the Kola Peninsula, U.S.S.R.

The deposits are stated to contain the largest reserves in the world in total columbium content.

9. Gerasimovsky, V. I. Pegmatites of the Lovozero alkaline massif. *Trudy Inst. Geokhim. Nauk., Akad. Nauk. S.S.S.R.* 18, Mineral.-Geokhim. Ser. No. 5, 1-44 (in English 44-45) (1939); Chem. Abstracts 34, 4362 (1940).

A description of the loparite deposits.

10. Gerasimovsky, V. I. Columbium and tantalum in the U.S.S.R. *Trudy Inst. Geol. Nauk., No. 39, Mineral.-Geokhim. Ser. No. 8, 49-57* (1940); Chem. Abstracts 37, 6602 (1943).

A summary of data on Russian deposits, including the loparite deposits, syenite pegmatites containing ilmenorutile and ilmenite, and columbite-tantalite deposits.

11. Goldschmidt, V. M. The principles of distribution of chemical elements in minerals and rocks. *J. Chem. Soc. (London)* 1937, 655-673.

A general account. The average content of columbium in the earth's crust is stated to be 0.0015%, from unpublished work. The following data on the  $Cb_2O_5$  content of rocks <sup>are</sup> also from unpublished work: gabbro 0.001%, diorite, 0.003%, granite 0.005%, and nepheline syenite 0.02%.

12. Goldschmidt, V. M. *Geochemische Verteilungsgesetze der Elemente. IX. Die Mengenverhältnisse der Elemente und der Atom-arten.* Skrift. Norsk. Videnskaps-akad. )slo, Mat.-Nat. Klasse 1937, No. 4, 148 pp.

The estimates by von Hevesy (15) of the columbium and tantalum contents of the igneous rocks are considered to be too low. Unpublished work gives approximately 0.002% columbium for eruptive rocks and for clay-sand sediments. No data were obtained for tantalum, but if von Hevesy's Cb: Ta ratio is accepted, the content of tantalum would be 0.0015%. In many rocks, the columbium content varies with the zirconium content.

13. Hevesy, G. von and Wurstlin, K. "Über das Häufigkeitsverhältnis Zirkonium/Hafnium und Niob/Tantal.  
Z. physikal. Chem. 139A, 605-614 (1928).

From a discussion of the data in the literature, it is concluded that the amounts of columbium and tantalum in the earth's crust are approximately equal, and are related most closely to the titanium content.

14. Hevesy, G. von, Alexander, E. and Wurstlin, K. "Über die Häufigkeitsverhältnisse Niob-Tantal in Titanmineralien.  
Z. anorg. allgem. Chem. 181, 95-100 (1929).

Analyses were made by chemical concentration followed by x-ray spectroscopic determination of 6 rutiles (0.22 to 3.1%  $\text{Cb}_2\text{O}_5 + \text{Ta}_2\text{O}_5$ ), 10 sphenes (1.37 to less than 0.006%  $\text{Cb}_2\text{O}_5 + \text{Ta}_2\text{O}_5$ ), 1 brookite (0.23%  $\text{Cb}_2\text{O}_5 + \text{Ta}_2\text{O}_5$ ), perovskite (0.095%  $\text{Cb}_2\text{O}_5 + \text{Ta}_2\text{O}_5$ ), 3 ilmenites (0.006 to 0.001%  $\text{Cb}_2\text{O}_5 + \text{Ta}_2\text{O}_5$ ) and two "black sands" (0.18 and 0.02%  $\text{Cb}_2\text{O}_5 + \text{Ta}_2\text{O}_5$ ).

15. Hevesy, G. von, Alexander, E. and Wurstlin, K. Die Häufigkeit der Elemente der Vanadiumgruppe in Eruptivgesteinen.  
Z. anorg. allgem. Chem. 194, 316-322 (1930).

A "synthetic" eruptive rock was made by mixing 133 granites, 9 quartz-diorites, 35 diorites, 82 gabbros and norities, and 23 ultrabasic rocks. Analysis by chemical concentration followed by x-ray spectroscopic determination gave  $3.2 \times 10^{-5}$  Cb,  $2.4 \times 10^{-5}$  Ta. Biotite from Hittero, Norway, contained  $6 \times 10^{-5}$  Cb+Ta; phlogopite from Bamle, Norway, contained  $3.2 \times 10^{-5}$  Cb+Ta.

16. Hodge-Smith, T. Mineralogical notes. No. 6.  
Proc. Australian Museum 21, 244-256 (1943).

Analyses of 9 wolframites from New South Wales showed that 6 contained  $\text{Cb}_2\text{O}_5 + \text{Ta}_2\text{O}_5$ , the contents being 0.26 to 1.95%. It is suggested that most earlier analysts missed the presence of columbium and tantalum.

17. Larionov, J. and Tolmacer, J. M. On the chemical composition of cassiterites.  
Compt. rend. acad. sci. U.R.S.S. 14, 303-306 (1937).

Qualitative spectrographic study showed columbium to be present in 5 pegmatitic cassiterites, present in traces in 2 hydrothermal cassiterites.



18. Mathieu, F. F. Les gisements des minerais de tantale et de niobium. Publ. assoc. ing. faculté polytech. Mons, No. 64, 47-139 (1938).  
A review of occurrences of columbium and tantalum minerals.
19. Mellor, J. W. A comprehensive treatise on inorganic and theoretical chemistry. Vol. IX.  
Pages 837 to 925 deal with columbium and tantalum.
20. Myers, R. H. A review of the literature on the chemistry and metallurgy of tantalum and columbium.  
Proc. Australasian Inst.. Mining and Metallurgy No. 129, 55-79 (1943).
21. Panteleev, P. G. Titanium, columbium and tantalum in the alkaline complex of the Ilmeny Mountains in the Urals.  
Bull. acad. sci. U.R.S.S., classe sci. math. nat., Ser. geol. 1938, 827-836 (in English 835-836).

The deposits are described. They contain rutile (ilmeno-rutile) with 6-17%  $\text{Cb}_2\text{O}_5$ , 1-3%  $\text{Ta}_2\text{O}_5$ , and ilmenite with 0.6-1.3%  $\text{Cb}_2\text{O}_5$  and 0.1%  $\text{Ta}_2\text{O}_5$ . The occurrences of columbium and tantalum are discussed, with the general conclusions that they are most closely related to titanium in occurrence, and that the greatest concentrations occur in pegmatite veins of syenitic and miaschitic magmas.

22. Schoeller, W. R. and John, C. Investigations into the analytical chemistry of tantalum, niobium and their mineral associates VIII. The separative of tungsten from tantalum and niobium.  
Analyst 52, 506-514 (1927).

Chiefly analytical chemistry. The determination of 0.1%  $\text{Cb}_2\text{O}_5 + \text{Ta}_2\text{O}_5$  in scheelite by Carobbi (4) is doubted, because he used an unreliable method of analysis.

23. Stevens, R. E. New analyses of lepidolite and their interpretation.  
Am. Mineral. 23, 607-628 (1938).  
Two analyses of polyolithionite showed 0.14 and 1.52%  $\text{Cb}_2\text{O}_5$ .
24. Tongeren, W. Van. Contributions to the knowledge of the chemical composition of the earth's crust in the East Indian Archipelago. II. On the occurrence of rarer elements in the Netherlands East Indies. pp. 122-161. Amsterdam, 1938.

Quantitative spectrographic study of many samples. No details are given, but it is stated that as far as evidence is available, the columbium content

<sup>p</sup>  
 Parallels the zirconium content. A rough approximation is  $Zr:Cb = 10:1$ , which gives 0.001%  $Cb_2O_5$  for the average content.

25. U. S. Geological Survey.  
 Unpublished X-ray data by W. E. Richmond and J. M. Axelrod; unpublished spectrographic data by E. J. Murata, J. C. Rabbitt, and George Steiger.
26. Yurk, Y. Y. and Bresser, V. M.. Geochemistry of rare elements in the granitic pegmatites of the western Azov seacoast region.  
 J. Geol., Acad. sci. Ukrain. S.S.R. 5, No. 4, 131-161 (in English 161-163) (1940); Chem. Abstracts 35, 416 (1941).

The ilmenite sands of the Azov seacoast contain 0.25%  $Cb_2O_5$  and traces of  $Ta_2O_5$ .

Recovery and utilization are discussed.

27. Yurk, Y. Y. and Tsarovsky, I. D. Black-ore minerals of the Mariupol alkaline massif.  
 J. Geol., Acad. sci. Ukrain. S.S.R. 7, Nos. 1-2, 151-160 (in English 161-162) (1940); Chem. Abstracts 35, 3198 (1941).

Ilmenite from syenite pegmatite contained up to 0.4%  $Cb_2O_5+Ta_2O_5$ .