WODGINITE—A NEW TIN-MANGANESE TANTALATE FROM WODGINA, AUSTRALIA AND BERNIC LAKE, MANITOBA*

E. H. NICKEL, J. F. ROWLAND AND R. C. McADAM
Mineral Sciences Division, Mines Branch, Department of Mines and Technical Surveys, Ottawa, Canada

ABSTRACT

The name wodginite is proposed for a mineral found at two widely separated localities —Wodgina, Australia and Bernic Lake, Manitoba. The mineral is monoclinic, space group $C2/c$ or Cc, with cell parameters of $a = 9.52$ Å, $b = 11.47$ Å, $c = 5.10$ Å and $\beta = 91°18'$ (Wodgina) and $a = 9.47$ Å, $b = 11.42$ Å, $c = 5.09$ Å and $\beta = 91°02'$ (Bernic Lake). Its composition is expressed by the formula $A_{16}O_{32}$, where A represents chiefly Ta, Sn and Mn, with lesser amounts of Nb, Fe and Ti. The relationship of wodginite to columbite-tantalite, ixiolite and olovotantalite is discussed.

INTRODUCTION

During the past few years the pegmatitic lithium-cesium deposit at Bernic Lake in southeastern Manitoba, generally known as the "Montgary pegmatite", has been studied by a number of investigators. Occurring in this deposit as an accessory mineral is a dark, relatively fine-grained mineral that early investigators identified as columbite (Hutchinson, 1959) or as columbite-tantalite (Wright, 1961). Initial studies by Nickel (1961*a*) revealed that the mineral is high, not only in tantalum, but also in tin and it was tentatively called "stanniferous tantalite". Later, however, it became evident that the mineral is crystallographically distinct from columbite-tantalite (Nickel, 1961*b*) and that a new species name should be considered for it.

In an effort to relate this mineral to known species, the literature was searched for reports of similar minerals and attempts were made to obtain specimens for examination. These attempts were partially successful. Of a number of foreign samples that were examined, one from Wodgina, Australia produced an *x*-ray powder pattern that was almost identical to that of the Bernic Lake mineral. The Wodgina mineral was described many years ago by Simpson (1909), who called it ixiolite (?) because of its apparent similarity to the original so-called ixiolite from Finland (Nordenskiöld, 1857). As will be shown later in this paper, Simpson's mineral is different from Nordenskiöld's ixiolite, the latter actually being

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a mixture of several phases. Consequently, Simpson's nomenclature should be revised.

Subsequent work by the authors showed that the Bernic Lake and Wodgina minerals represent the same species. Because the mineral was first discovered at Wodgina, Australia, it is proposed that the mineral be named wodginite in recognition of its original locality. It will be referred to by this name throughout the remainder of this paper.

OCCURRENCES

Wodgina, Australia

Simpson (1909, p. 314) described the Wodgina occurrence as follows: "It occurs in a very fresh state in a matrix of granular albite, the latter with a little quartz and muscovite forming about 72 per cent of the ore. The remaining 28 per cent consists of a tantalite with a somewhat resinous lustre, and varying from pale cinnamon brown to dark brown. It occurs in indistinct crystalline aggregates, the system of which has not been determined".

A sample of this mineral (No. 8492) supplied by J. H. Lord of the Geological Survey of Western Australia conforms to this description. It was noted, furthermore, that the wodginite grains are roughly tabular in shape, and that some of them form partially radiating groups.

Bernic Lake, Manitoba

At Bernic Lake, Manitoba, wodginite occurs within a complex zoned pegmatite that is bordered by Precambrian amphibolite. The geology of the area has been described by Davies (1955), and the geology of the pegmatite has been described in detail by Hutchinson (1959) and Wright (1961). Briefly stated, the pegmatite is a gently dipping tabular body that varies from about 60 to 270 feet in thickness, and consists of irregular zones, each of which has a distinctive mineral assemblage. The mineralogy (Nickel, 1961*a*) is typical of that of cesium-lithium deposits found in various parts of the world, the principal minerals being quartz, feldspar, lithium micas, spodumene, pollucite, and amblygonite. Accessory minerals include beryl, lithiophilite, apatite, tourmaline, rhodochrosite, tantalite, tapiolite, cassiterite, microlite, and wodginite.

The wodginite has been found in two different mineral assemblages—in a coarse, partially sericitized perthitic microcline, and in a relatively unaltered, fine-grained, bluish-white aplitic albite. The wodginite occurs as disseminated grains varying from less than one to about ten millimetres in diameter. Some of the grains are sphenoidal in shape (Fig. 1), whereas others are entirely anhedral (Fig. 2). All the grains, whether sphenoidal or irregular, are highly serrated and contain numerous inclusions of feldspar and/or mica. In some samples the wodginite is closely associated with tapiolite and microlite.

PHYSICAL AND OPTICAL PROPERTIES

The wodginite, from both Wodgina and Bernic Lake, varies in colour from reddish brown through dark brown to almost black. Small splinters are clear and translucent in transmitted light and range from light yellow to reddish brown. In polished section under the ore microscope the wodginite is very similar in appearance to normal columbite-tantalite. With an air objective it is greyish-white in colour and weakly anisotropic, but is not perceptibly pleochroic. With an oil immersion objective it shows weak pleochroism and stronger anisotropism, and exhibits a great many internal reflections.

The specific gravity of the Wodgina mineral is reported by Simpson (1909) to be 7.36. That of the wodginite from Bernic Lake is 7.19 (pycnometer determination on analyzed concentrate).

CHEMICAL ANALYSIS OF BERNIC LAKE WODGINITE

Particular care had to be exercised in concentrating the Bernic Lake wodginite for analysis because of the fine texture and large number of

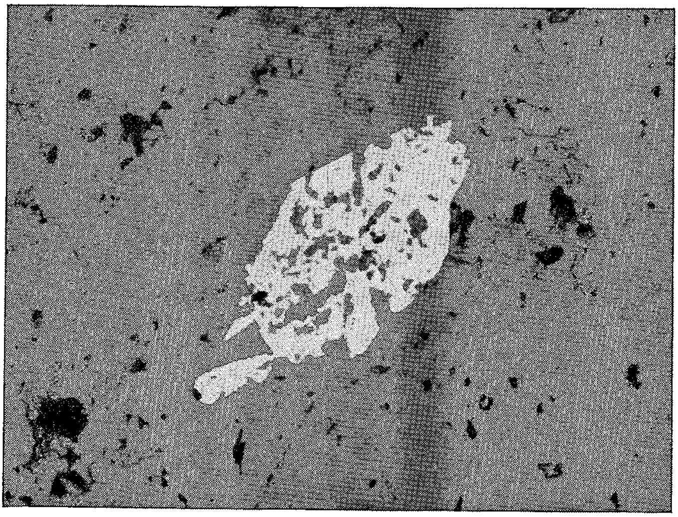

FIG. 1. Polished section of wodginite grain in partially sericitized perthite. The wodginite grain shows a tendency toward sphenoidal development. Vertical illumination, ×80.

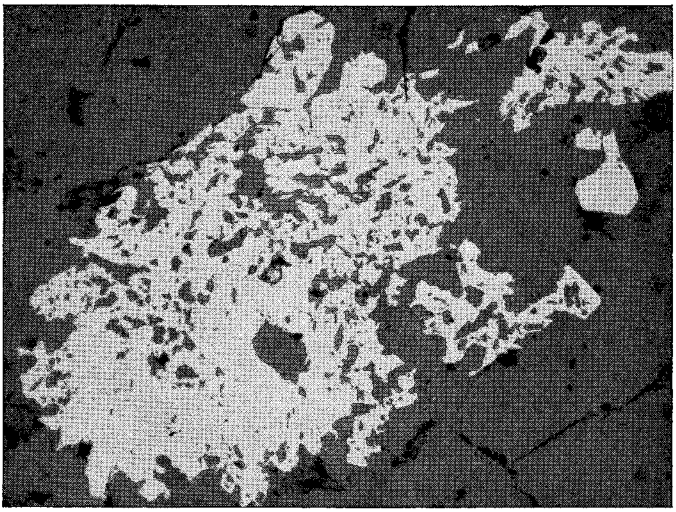

FIG. 2. Polished section of some highly irregular wodginite grains in partially sericitized perthite. Note the highly serrated edges and the large number of inclusions. Vertical illumination, ×80.

inclusions of matrix minerals (Figs. 1 and 2). The material was first crushed to minus 100-mesh. It was then separated into light and heavy fractions by means of Clerici solution with a specific gravity of 4.3, the wodginite going into the heavy fraction. Next, wodginite was purified by successive separations at high intensity on a Frantz isodynamic separator. Since microscopic examination revealed significant amounts of attached silicate minerals, the wodginite concentrate was then finely pulverized in a mechanical mortar and repeatedly centrifuged in Clerici solution. After material ceased to float off, the sample was assumed to be sufficiently pure for analysis, and an *x*-ray diffraction pattern exhibited no extraneous lines. However, the SiO_2 reported in the analysis shows that appreciable amounts of silicate impurities were present.

The chemical analyses of the Wodgina and Bernic Lake wodginites are shown in Table 1. It can be seen that their compositions are broadly similar, particularly with respect to the Ta_2O_5, MnO and FeO values. The Bernic Lake wodginite, however, is higher in SnO_2 and TiO_2, and lower in Nb_2O_5, than the Wodgina mineral.

The SiO_2 in the Bernic Lake analysis and the CaO and MgO in the Wodgina analysis can reasonably be attributed to impurities. The chief impurity in the Bernic Lake sample was found, by petrographic examination, to be muscovite and can therefore be assumed to be responsible for the majority of the SiO_2. Unfortunately the other muscovite consti-

TABLE 1. CHEMICAL ANALYSES OF WODGINITE

	Wodgina,* Australia	Bernic Lake,** Manitoba
Ta_2O_5	70.49%	70.05%
Nb_2O_5	7.63	1.35
SnO_2	8.92	13.20
MnO	10.87	9.04
FeO	1.34	1.87
TiO_2	—	2.39
CaO	0.42	—
MgO	0.37	—
SiO_2	—	0.60
Loss on ignition	0.18	—
	100.22	98.50

*From E. S. Simpson (1909).
**This paper; R. C. McAdam, analyst.

tuents (K_2O, Al_2O_3 and H_2O) could not be determined quantitatively because of the small amount of sample remaining after the other determinations were completed. However, aluminum was detected by emission spectrography and potash by flame photometry (potassium is not normally reported in spectrographic determinations). If the SiO_2 were due entirely to the presence of muscovite, then the remaining muscovite components would comprise an additional 0.73 per cent, bringing the total Bernic Lake analysis to 99.23 per cent, which would still be appreciably below 100 per cent. A semi-quantitative spectrographic analysis showed that, in addition to the constituents shown in Table 1, the following elements are present in excess of 0.01 per cent: Zr 0.2 per cent, Al 0.09 per cent, Ca 0.05 per cent and Mg 0.03 per cent.

The fact that the analysis totals less than 100 per cent could be explained if some of the constituents, possibly manganese or iron, were present in a more highly oxidized form than that shown in the analysis. Unfortunately, the oxidation states of these elements could not be determined analytically, although, as will be shown later, the view that higher states of oxidation exist is supported by the abnormally low specific gravities of both minerals.

X-RAY CRYSTALLOGRAPHY

Neither the Bernic Lake nor the Wodgina samples contained crystals directly suitable for single crystal *x*-ray diffraction analysis. However, after crushing the Wodgina mineral, some imperfect cleavage fragments were found to be suitable for this purpose. Single crystal films were obtained with unfiltered molybdenum radiation on a Buerger precession camera from a fragment that was predominantly a single crystal.

The precession films show a few extra diffractions which can be attributed to minor amounts of the mineral in other orientations. These extra diffractions could not be correlated with a twinning mechanism, although they are consistent with the existence of parallel crystallization. The imperfect cleavage faces do not appear to have a simple relationship to the orientation of the unit cell axes. The data obtained from the single crystal films are:

Crystal system: Monoclinic
Extinction rules: $(hkl)\ h + k = 2n$
$(h0l)\ l = 2n\ (h = 2n)$
$(0k0)\ (k = 2n)$
Space group: $C_{2h}^6 = C2/c$ or $C_s^4 = Cc$
Unit cell parameters:
$a = 9.52$ Å; $b = 11.46$ Å; $c = 5.11$ Å; $\beta = 91°15'$
Cell volume: 557.4 Å^3

The x-ray powder diffraction data for the two wodginites are shown in Table 2. The d values were calculated from measurements made on diffraction patterns obtained with 114.6 mm Debye-Scherrer powder cameras using iron-filtered cobalt radiation. The diffraction intensities of the Bernic Lake mineral were obtained by peak height measurements made on the diffractometer tracing shown in Fig. 3, recalculated to a scale of 100. The x-ray intensities of the Wodgina mineral were estimated by visual comparison of its powder pattern with that of the Bernic Lake mineral, since there was insufficient material for a diffractometer determination.

The powder patterns were indexed from the unit cell and space group data obtained from the single crystal determinations, and there was very good agreement between the calculated and measured d-values for both minerals. More accurate cell parameters were obtained by least-squares calculations using the indexed spacings of the powder patterns, and these parameters are shown at the top of Table 2.

On casual inspection, the wodginite powder pattern is quite similar to that of columbite-tantalite. The most obvious difference is the presence of the strong doublet at 2.95–3.00 Å (Table 2) that takes the place of the strong columbite-tantalite single peak in that region. This doublet can easily be overlooked on a small film, but on a large (114.6 mm.) film or on a diffractometer tracing, the difference between the patterns is unmistakable (see Fig. 3).

TABLE 2. WODGINITE X-RAY POWDER DIFFRACTION DATA

	Wodgina, Australia $a = 9.522$Å $b = 11.468$Å $c = 5.104$Å $\beta = 91°18'$			Bernic Lake, Manitoba $a = 9.475$Å $b = 11.423$Å $c = 5.095$Å $\beta = 91°02'$		
hkl	*I*(est).	*d*(meas.)	*d*(calc.)	*I*(meas.)	*d*(meas.)	*d*(calc.)
110	11	7.22Å	7.33Å	4	7.3Å	7.29Å
020	4	5.71	5.73	4	5.69	5.71
200	11	4.76	4.76	11	4.73	4.73
$\bar{1}11$	2	4.21	4.22	2	4.20	4.20
111	3	4.16	4.15	3	4.15	4.15
021	10	3.81	3.81	10	3.80	3.80
220	70	3.67	3.66	70	3.64	3.65
130	2	3.60	3.55	4	3.53	3.53
$\bar{2}21$	100	3.00	3.00	100	2.98	2.98
221	70	2.95	2.95	87	2.95	2.95
040	25	2.87	2.87	25	2.86	2.86
$\bar{3}11$	1	2.66	2.65			
311	1	2.60	2.60			
002	21	2.55	2.55	21	2.55	2.55
041	29	2.50	2.50	29	2.49	2.49
240	1	2.45	2.46	1	2.45	2.45
330			2.44			2.43
112	10	2.40	2.40			
400	10	2.38	2.38	11	2.37	2.37
$0\bar{2}2$	4	2.268	2.270	4	2.261	2.260
241	8	2.200	2.203	8	2.197	2.197
$\bar{2}22$	8	2.113	2.111	10	2.101	2.102
222	8	2.077	2.077	8	2.078	2.074
$\bar{4}21$	1	2.030	2.034	1	2.029	2.016
421	2	2.004	2.004	2	2.001	2.004
$\bar{3}12$	1	1.966	1.980			
312	1	1.933	1.939			
042	11	1.906	1.906	11	1.905	1.901
440	14	1.831	1.831	14	1.824	1.823
$\bar{2}42$	1	1.780	1.780			
$\bar{3}32$			1.779			
260	27	1.774	1.774	27	1.767	1.766
$\bar{4}02$	13	1.760	1.760	13	1.750	1.750
332	1	1.747	1.749			
$\bar{4}41$	Broad	1.733	1.733	26	1.723	1.724
402		to	1.721			1.719
441		1.715	1.714	24	1.711	1.709

NOTE: A weak line that could not be indexed occurs at $d = 6.05$Å in both powder patterns.

CRYSTAL CHEMISTRY

The general similarity between the powder patterns of wodginite and columbite-tantalite is due to their closely related structures. The unit cell of wodginite (monoclinic, with $a = 9.50$ Å, $b = 11.45$ Å, $c = 5.10$ Å, and $\beta = 91°10'$—average of Wodgina and Bernic Lake data) is related

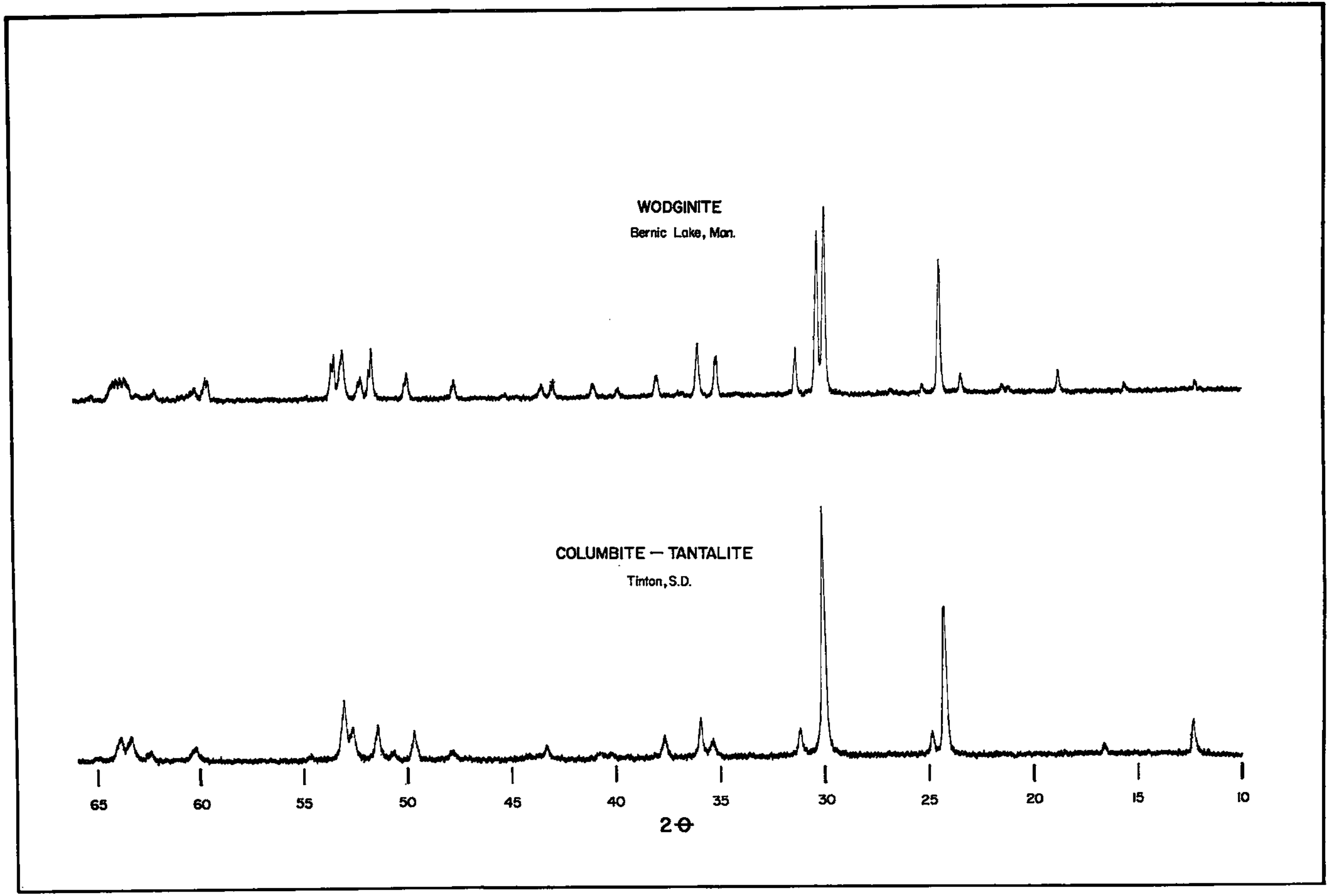

FIG. 3. Diffractometer tracings of wodginite and columbite-tantalite. Copper radiation, nickel filter; scanning speed 1/4 degree per minute.

to that of columbite-tantalite (orthorhombic, with $a = 5.73$ Å, $b = 14.24$ Å, and $c = 5.08$ Å—Tinton, South Dakota data) as follows: $a_W \equiv 2/3b_{CT}$, $b_W \equiv 2a_{CT}$, $c_W \equiv c_{CT}$, and $\beta_W \equiv \beta_{CT}$ (where the subscripts W and CT refer to wodginite and columbite-tantalite, respectively). The unit cell volume of wodginite is consequently 4/3 that of columbite-tantalite.

Since the unit cell of columbite-tantalite contains $(Fe, Mn)_4(Nb, Ta)_8O_{24}$ (Sturdivant, 1930), or a total of 12 cations and 24 anions, the wodginite unit cell should contain 4/3 as many, or 16 cations and 32 anions. On this assumption, the chemical analyses of the Wodgina and Bernic Lake wodginites have been recalculated to atomic proportions for a cell containing 32 oxygen ions. The results are shown in Table 3.

TABLE 3. ATOMIC PROPORTIONS IN WODGINITE

	Wodgina, Australia			Bernic Lake, Manitoba		
	Wt.%*	Atomic proportions**		Wt.%*	Atomic proportions**	
Ta	57.73	8.29		57.37	8.40	
Nb	5.33	1.49		0.94	0.27	
Sn	7.03	1.54	15.78	10.40	2.32	
Mn	8.42	3.98		7.00	3.38	15.85
Fe	1.04	0.48		1.45	0.69	
Ti	—	—		1.43	0.79	
O	19.70	32.00		19.31	32.00	
Total	99.25	47.78		97.90	47.85	

*Calculated from oxide percentages given in Table 1, with CaO, MgO, SiO_2 and loss on ignition excluded.

**Calculated to bring total oxygen atoms to 32.00.

The space group requirements for wodginite would group the 16 cations either in two sets of four equivalent positions and one set of eight equivalent positions (*C*2/*c*), or in four sets of four equivalent positions (*Cc*). These positions correspond approximately to those of the twelve cations in columbite-tantalite where they are grouped in one set of four equivalent positions (iron and manganese) and one set of eight equivalent positions (niobium and tantalum). It can be seen from Table 3, however, that the cations do not in general approximate whole numbers, and there does not appear to be any logical way of grouping the cations to conform to these requirements. The results of the analysis seem to suggest that the cations may be randomly distributed in the 16 available positions.

On the other hand, it might be speculated that if some of the cations exist in several degrees of oxidation and therefore have different ionic radii, there might then be an ordering on the basis of ionic size rather than purely on an elemental basis. Unfortunately, the oxidation states of the cations could not be determined analytically, so this must remain in the realm of speculation for the time being.

The atomic proportions shown in Table 3 give molecular weights of 2579 and 2594, respectively for the wodginites from Wodgina and Bernic Lake. Using these molecular weights and the cell parameters shown in Table 3, the calculated specific gravity of the wodginite from the type locality is 7.69, and that of the Bernic Lake wodginite is 7.81. These values are considerably greater than the measured values of 7.36 and 7.19. This discrepancy might be explained by postulating a higher state of oxidation for the cations than those indicated by the chemical analyses. Increasing the oxidation state, *i.e.* valency, of one or more of the components would result in fewer cations being required to electrostatically balance the 32 oxygen atoms in the unit cell. This would be reflected in lower gravities of the minerals. It would also explain the failure of the Bernic Lake mineral analysis to total 100 per cent.

Since the manner in which the cations are ordered can not at present be ascertained, the formula for wodginite will tentatively have to be expressed as $A_{16}O_{32}$, wherein A represents all the cations analyzed. If there is a cation deficiency, then the formula would be given more accurately by $A_{16-x}O_{32}$, with the value of x related directly to the average valency of the cations, and decreasing to zero as the average cation valency approaches four.

DISCUSSION

Since both samples of wodginite are distinguished chemically from normal members of the columbite-tantalite series by virtue of their high tin contents, it is tempting to conclude that tin is an essential constituent of wodginite. However, a tin-free phase giving the characteristic wodginite diffraction pattern has recently been synthesized by A. C. Turnock of the Mineral Sciences Division, Mines Branch (personal communication). The composition of his synthetic wodginite phase approximates $(Mn_{0.8}Fe_{0.2})Ta_2O_6$, which indicates that tin is not an essential component.

Mention should be made of several other related minerals that have recently come to light. These minerals, which will be referred to here as "polymorphs", can be distinguished by their x-ray powder diffraction patterns (Table 4).

The intensity data for ixiolite, columbite-tantalite and wodginite were measured from diffractometer tracings. The d-values were calculated from measurements made on 114.6 mm Debye-Scherrer films obtained with iron-filtered cobalt radiation. The patterns were indexed on the

TABLE 4. X-RAY POWDER DIFFRACTION PATTERNS OF COLUMBITE-TANTALITE "POLYMORPHS"

Ixiolite (R.O.M.) 6591 Skogböle, Finland			Columbite-Tantalite Tinton, S.D.			Olovotantalite U.S.S.R.			Wodginite Wodgina, Australia		
Int.	*d* (meas.)	*hkl*	Int.	*d* (meas.)	*hkl*	Int.	*d* (meas.)	*hkl*	Int.	*d* (meas.)	*hkl*
						40	11.1	010			
			12	7.13	020				11	7.22	110
									4	6.05	?
									4	5.71	020
			4	5.30	110						
						10	4.69	200	11	4.76	200
									2	4.21	$\bar{1}11$
									3	4.16	111
						20	3.79	021	10	3.81	021
32	3.65	011	48	3.66	130				70	3.67	220
			9	3.57	040						
									2	3.60	130
100	2.98	111	100	2.96	131	70	2.95	221	100	3.00	$\bar{2}21$
									70	2.95	221
5	2.87	020	10	2.86	200	20	2.88	040	25	2.87	040
						20	2.76	320			
									1	2.66	$\bar{3}11$
									1	2.60	311
13	2.57	200	6	2.53	002	40	2.56	002	21	2.55	002
20	2.51	120	12	2.49	201	70	2.51	041	29	2.50	041
									1	2.45	240, 330
									10	2.40	112
5	2.37	002	12	2.38	060	30	2.38	400	10	2.38	400
2	2.265	201	1	2.279	151	10	2.275	202	4	2.268	$\bar{2}02$
			3	2.236	032						
4	2.213	121	4	2.207	231	50	2.21	241	8	2.200	241
						30	2.12	411			
9	2.104	211	5	2.084	132	40	2.099	222	8	2.113	$\bar{2}22$
									8	2.077	222
						10	2.077	132			
			3	2.043	241						
									1	2.030	$\bar{4}21$
									2	2.004	421
						10	1.952	232	1	1.966	$\bar{3}12$
									1	1.933	312
4	1.915	220	5	1.898	202	60	1.917	042	11	1.906	042
6	1.826	022	9	1.831	260	50	1.835	449	14	1.831	440
			4	1.796	152, 171						
									1	1.780	$\bar{2}42, \bar{3}32$
13	1.772	031	14	1.772	330	80	1.780	260	27	1.774	260
									13	1.760	$\bar{4}02$
									1	1.747	332
17	1.746	202	12	1.735	062	70	1.743	402		1.733	$\bar{4}41$, 402
24	1.722	122	22	1.721	261	70	1.723	441	Broad	1.715	441

(See text for details of intensity measurements and indexing).

unit-cell parameters given in Table 5. The *d*-values and intensities of olovotantalite were taken from Matias (1961), with the intensities multiplied by a factor of ten to make them comparable to those of the other minerals. The indexing was done on the hypothetical unit cell shown in Table 5, since no single crystal data are available for olovotantalite.

TABLE 5. CELL PARAMETERS OF COLUMBITE-TANTALITE "POLYMORPHS"

	Ixiolite Skogböle, Finland	Columbite-Tantalite Tinton, S.D.	Olovotantalite* U.S.S.R.	Wodginite Australia
Unit-Cell Lengths	$a = 5.15$ $b = 5.73$ $c = 4.74$	$c = 5.08$ $a = 5.73$ $b = 14.24$ (3×4.75)	$c = 5.14$ $b = 11.59$ (2×5.75) $a = 9.59$ (2×4.75)	$c = 5.10$ $b = 11.47$ (2×5.73) $a = 9.52$ (2×4.76)
β	90°	90°	90°	91°18′
Cell Volume	140 Å^3	414 Å^3 (3×138)	561 Å^3 (4×140)	557 Å^3 (4×139)
Space Group	*Pnab*	*Pcan*	?	*C2/c* or *Cc*

*Deduced from *x*-ray powder diffraction data given by Matias (1961).

The close relationship between the unit-cell parameters of these "polymorphs" is shown in Table 5. The unit cells can all be expressed as multiples of the smallest unit cell—that of ixiolite.

Ixiolite, first described by Nordenskiöld (1857) and later discredited by Åmark (1941) and Quensel (1941) has recently been re-investigated. Nickel & Rowland (1962) found that ixiolite represents a substructure of tantalite, with the cations in disordered arrangement. It has an SnO_2 content of 12.27 per cent. The ixiolite powder pattern is distinguished from that of the other polymorphs by the absence of spacings at *d*-values greater than 3.7 Å.

Olovotantalite (literally "tin-tantalite") also has a high SnO_2 content (9.06 per cent). Although Fleischer (1962) suggests that this new name is unnecessary, the powder pattern is quite different from that of the other "polymorphs" and cannot be indexed on the columbite-tantalite unit cell. This suggests that olovotantalite is indeed a separate species. The powder data can be indexed on an orthorhombic wodginite cell, *i.e.*, a unit cell with the dimensions of wodginite but with $\beta = 90°$.

This proliferation of columbite-tantalite "polymorphs" warrants further study. Some of these varieties may be considerably more common that has been supposed, but may have been overlooked because of their similarity to columbite-tantalite. As an example, a sample of "manganotantalite" from the Benson Mine of Southern Rhodesia has been found to give the typical wodginite powder pattern*. Others will probably come to light in the future.

Phase equilibrium studies now being conducted by A. C. Turnock at the Mines Branch may be expected to provide information on the stability relations of wodginite and perhaps the other minerals, as well.

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*Information received from H. J. Martin of the Geological Survey of Southern Rhodesia after this paper went to press indicates that the Benson Mine wodginite contains only 0.30% SnO_2, thereby supporting the view that a high tin content is not essential to wodginite.

It is hoped that this work will assist in defining the formation conditions of these minerals.

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