Tetrawickmanite, Tetragonal $MnSn(OH)_6$ A New Mineral From North Carolina, And The Stottite Group

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In 1966 one of us (JSW) received a large specimen of pegmatite from the Foote Mineral Company's spodumene mine, Kings Mountain, North Carolina, sent by Mr. Ed Heard of Raleigh, North Carolina. It consisted of intergrown masses of curved, platy crystals of gray-colored albite. Throughout the mass were irregular vugs averaging about 1 cm in diameter wholly or partly filled with dense, white, chalky-looking bavenite. The vugs all appeared to be connected by thin fractures, usually lined with the same chalky bavenite. A large vug on one side of the specimen was partially filled with bavenite. Implanted upon the bavenite surface, and partlyembedded in it, were approximately ten rounded, rough-surfaced, globule-like masses of a translucent, honey-yellow mineral. They averaged about 1 mm in diameter. A few were selected for x-raying and, on the basis of the powder pattern, the mineral was identified as wickmanite, isometric $MnSn(OH)_6$, a mineral that had just been described[1] by Moore and Smith (1967) from Långban, Sweden. With thoughts of writing a short note about a *second* occurrence of wickmanite, we went on to examine some of the other minerals of the Foote mine.

Several months later, on another specimen, crystals of the same color were observed but these were better formed and clearly tetragonal. The basal pinacoid was obvious. An x-ray diffraction pattern of these also seemed a good match for wickmanite but close examination of back-reflection lines revealed splitting, characteristic of a tetragonal, pseudo-cubic phase. The mineral, therefore, was not wickmanite, but its tetragonal dimorph! To emphasize this dimorphic relationship the mineral has been named tetrawickmanite, and the name has been approved by the I. M. A. Commission on New Minerals and Mineral Names.

Tetrawickmanite is rare at the Foote mine and has been found on a very small number of specimens. In addition to the specimens mentioned above, it has been found attached to the side of a free-standing eakerite crystal and perched upon quartz and late albite druses in an open cavity in spodumene-albite pegmatite. These crystals are turbid, brownish-orange in color. It is also found in association with siderite-rhodochrosite on late albite druses nearly buried in very fine-grained, sugary quartz. The sharpest, most transparent, brightest yellow crystals are those of the latter association. All of this type were supplied by Mr. Jack Eaker of Kings Mountain.

CRYSTALLOGRAPHY

The habit is such that the crystals remind one of tiny wulfenite crystals (Figs. 1a, 1b). The only forms identified on them are the prominent pyramid {112}, the pinacoid {001}, and a very minor prism {100}. Under the microscope the faces appear sharp enough to produce good signals on the goniometer. However, no useful reflection could be obtained from the pyramid faces. The scanning electron microscope photograph at a magnification of 6500X (Fig. 1c) reveals why this is so. The pyramid faces are very irregular and consist of a stack of plate-like units reminiscent of a brick wall where the mortar has been squeezed out between the rows of bricks. The indices of the pyramid therefore were assigned on the basis of the approximate interfacial angle and the knowledge, from single crystal precession photographs, that the *a* axes emerge from corners rather than from the centers of edges, that is, the pyramid is of the first order. The cell parameters, determined from precession photographs and computer refined powder data, are $a = 7.7870$, $c = 7.797$ (both ± 0.001) Å.

Subtle differences in the powder patterns of tetrawickmanite and wickmanite, alluded to above, are discernible in the powder data table. These differences are of the type one would expect of a pseudo-cubic tetragonal dimorph and, together with the mineral's tetragonal morphology and its uniaxiality, force the conclusion that the mineral is tetragonal. At the same time all evidence indicates that wickmanite is unquestionably isometric.

The space group, by analogy with stottite (tetragonal $FeGe(OH)_6$), is probably C^4_{4h}-$P4_2/n$.

CHEMISTRY

The mineral was analysed by electron microprobe using cassiterite and pure tin as standards. The authors had in-

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[1] The authors were provided with a preprint in 1966.

tended to use *type* wickmanite as a standard but our analysis differed so from that of Moore and Smith (Table 2) that we felt we should not. This difference in analyses is perhaps due to the use of different wickmanite crystals, even though taken from the type specimen. We tried growing synthetic $MnSn(OH)_6$ crystals for use as standards but without success. The analysis of tetrawickmanite generates the formula $(Mn_{.94}Fe_{.045}Ca_{.01}Mg_{.005})(Sn_{.984}Si_{.011}Al_{.005})(OH)_6$, if it is assumed that the iron is ferrous. However, the iron may be ferric, substituting for tin. A corresponding amount of hydroxyl would have to be converted to water to maintain charge balance. As will be seen, the optics and density lend support to this interpretation.

OPTICAL PROPERTIES AND DENSITY

Tetrawickmanite is uniaxial, negative, with $nO = 1.724$, $nE = 1.720$. Wickmanite has an index of 1.705 ±0.003. One would not expect such a great difference in indices between the two minerals if the iron in tetrawickmanite were ferrous. Ferric iron, on the other hand, in the amount of 2.3% could very well produce an index escalation of this magnitude.

Much tetrawickmanite appeared turbid under the microscope but transparent crystals were found. No evidence of twinning or cleavage was seen.

Density was measured by Berman balance and by Clerici solution flotation using grains of minerals of known density for comparison. From calculations it is apparent that tetragonal $MnSn(OH)_6$ should have a slightly higher density than the isometric form, it has a smaller cell volume. Ferrous iron replacing manganese would increase the density even more, but ferric iron replacing tin would serve to lower the density. In Clerici solution tetrawickmanite floats slightly higher than wickmanite so once again the ferric iron for tin interpretation is supported.

An error in the calculated density reported by Moore and Smith (1966) for wickmanite was discovered during the course of determining that of tetrawickmanite. A new value is given in Table 3.

OCCURRENCE

The pegmatite in which the mineral was found is considered to be a part of a narrow tin-lithium belt which runs across the middle of North Carolina in a NNE direction. It is mined principally for spodumene. A number of minerals regarded by one of the authors (JSW) as late hydrothermal are enriched in tin and/or manganese. The tin silicates eakerite, unknown #7 (potassium-lithium-tin silicate), and stannian titanite (∽4-6% Sn) are found in open fissures and in vugs produced by dissolution of primary minerals, chiefly spodumene. Many secondary manganese phosphates have been found in addition to abundant bavenite, vivianite and siderite-rhodochrosite. Primary cassiterite and manganoan apatite are dispersed throughout the fresh pegmatite suggesting that these minerals provided tin, manganese and phosphorus to late solutions as they were dissolved out of what is now vuggy pegmatite. Beryl,

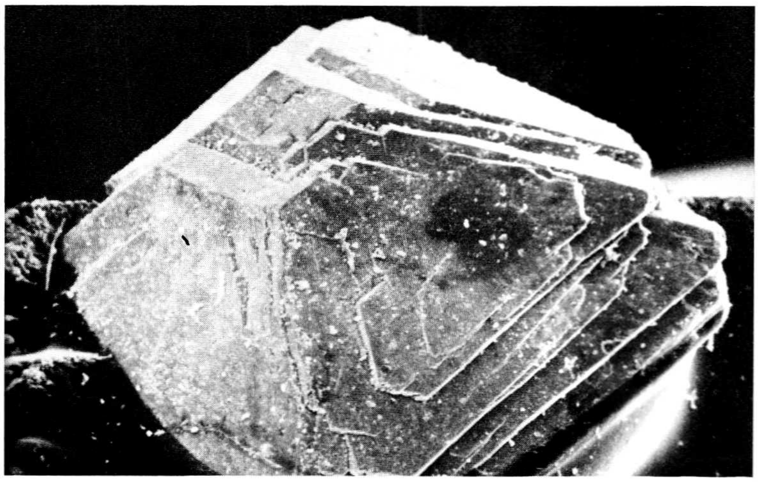

Fig. 1a.* Tetrawickmanite at 130X. *SEM photo.

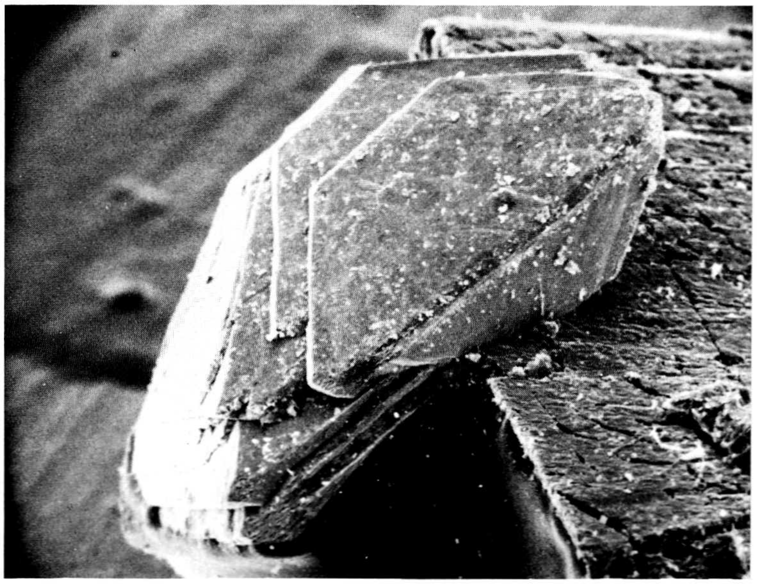

Fig. 1b.* Tetrawickmanite at 130X. *SEM photo.

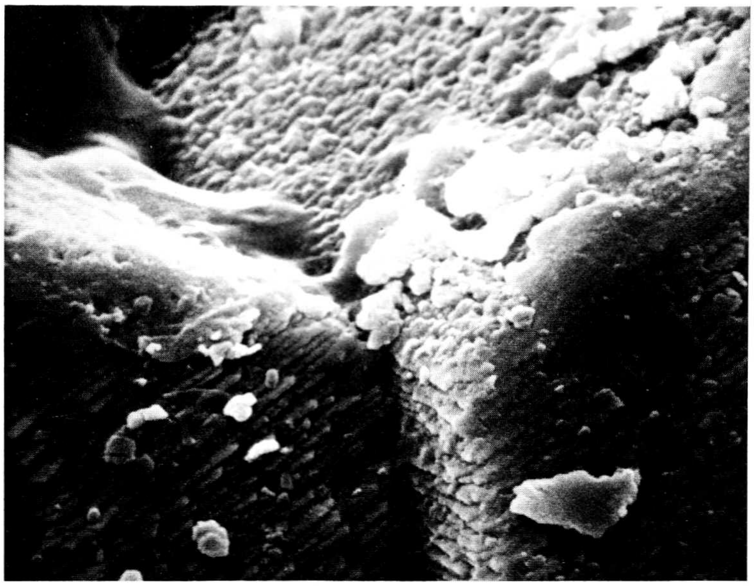

Fig. 1c.* Tetrawickmanite showing surface detail at 6500X. *SEM photo.

Table 1. Tetrawickmanite and wickmanite powder data.

TETRAWICKMANITE				WICKMANITE[1]	
I/Io	***dhklobs.***	***dhkl*calc.**	***hkl***	***dhkl*obs.**	***hkl***
30	4.518	4.529	111	4.552	111
100	3.939	3.935	200	3.931	200
30	3.880	3.898	002		
90	2.7698	2.7693	202	2.778	220
10	2.4820	2.4862	301	2.487	310
<5	2.4656	2.4678	103		
20	2.3697	2.3707	311	2.372	311
10	2.3535	2.3547	113		
25	2.2637	2.2647	222	2.273	222
5	2.1006	2.1018	321	2.104	321
20	1.9690	1.9674	400	1.9710	400
10	1.9493	1.9491	004		
				1.8060	331
50	1.7604	1.7600	420	1.7618	420
25	1.7458	1.7466	204		
40	1.6050	1.6039	422	1.6080	422
20	1.5970	1.5964	224		
				1.5138	511
10	1.3923	1.3912	440	1.4038	440
5	1.3850	1.3847	404		
5	1.3290	1.3270	531	1.3315	531
10	1.3222	1.3213	315		
				1.3130	600
5	1.3103	1.3102	442		
<5	1.3059	1.3061	424		
10	1.2434	1.2431	602	1.2452	620
5	1.2338	1.2339	206		
				1.2000	533
10	1.1851	1.1854	622	1.1882	622
5	1.1776	1.1774	226		
<5	1.1320	1.1323	444		
<5	1.0893	1.0882	604	1.0929	640
<5	1.0857	1.0843	406		
5	1.0660	1.0663	525		
<5	1.0453	1.0453	426	1.0536	642
5	.9522	.9522	644		
<5	.9504	.9496	446		
<5	.9443	.9453 .9411	724 427		
<5	.9252	.9274	660		
<5	.9226	.9231	606		
<5	.9180	.9188	616		
<5	.9005	,9023 .8987	662 626		
<5	.8970	.8964	832		
<5	.8560	,8560	119		
<5	,8505	,8502	706		
<5	.8352	.8357	646		
<5	.8334	.8334	843		
<5	.7998	.8019 .7982	844 448		
<5	.7978	.7980	904		
<5	.7961	.7963	656		
<5	.7852	.7849	339		
<5	.7829	.7828	942		
<5	.7804	.7804	816		

Cu/Ni radiation; 114.6 mm camera diameter; corrected for film shrinkage; visual intensity estimates.

[1] Moore and Smith (1966).

an uncommon mineral, certainly could have been the source of the beryllium that went into bavenite, bertrandite and milarite; and spodumene must have provided the lithium for bikitaite, eucryptite, lithiophilite and unk. #7. That tetrawickmanite is later even than siderite-rhodochrosite is demonstrated on one specimen where tetrawickmanite crystals have grown upon hemispheres of the carbonate. More than eighty species have been identified thus far at this exciting mineral locality.

The type specimens carry the catalog numbers 120239 and 121265 and are preserved in the collections of the National Museum of Natural History, Smithsonian Institution, Washington, D. C. There is so little tetrawickmanite on these samples that solicitations for specimens cannot be honored.

THE STOTTITE GROUP OF MINERALS

It may be an unprecedented phenomenon in mineralogy that an entire *group* of minerals made its appearance within the rather limited time span of about 15 years. Strunz *et al* (1958) published a description of stottite, iron germanium hydroxide ($FeGe(OH)_6$), found at Tsumeb, Southwest Africa. Although tetragonal, the mineral closely approximates cubic symmetry. Seemingly the discovery of this mineral helped to trigger a succession of new mineral descriptions. Strunz and Contag (1960) described a series of synthetic tin hydroxides (of iron, manganese, cobalt, magnesium and calcium) and it is perhaps true that this series was produced as a byproduct of an attempt to grow stottite, or its tin analog, in the laboratory. The synthetics, however, all turned out to be isometric.

Strunz then published (1965) a description of another Tsumeb mineral, sohngeite, given as $Ga(OH)_3$ and described as isotypic with dzhalindite but, when written as $GaGa(OH)_6$ and noting that it is cubic, the similarity to the cubic tin hydroxides of Strunz and Contag becomes apparent. The same may be said, of course, for dzhalindite (Genkin and Murav'eva, 1963), which may be written

Table 2. Electron microprobe analyses of tetrawickmanite and wickmanite.

	$MnSn(OH)_6$	Wickmanite (Moore & Smith)	Wickmanite (White & Nelen)	Tetrawickmanite (White & Nelen)
Mn	19.90	16.6	19.9	19.5
Fe	-	0.1	0.2	0.9
Ca	-	0.2	0.2	0.2
Mg	-	0.3	0.2	0.1
Sn	43.08	47.2	39.2	36.0
Si	-	-	0.05	0.4
Al	-	-	0.1	0.2

	$MnSn(OH)_6$	Wickmanite (M&S)	Wickmanite (W&N)	Tetrawickmanite (W&N)
(Mn,Fe,Ca,Mg)O	25.7	22.1	26.5	26.7
$(Sn,Si,Al)O_2$	54.7	59.9	50.0	46.4

$InIn(OH)_6$. Dzhalindite was found with cassiterite at the Dzhalindin deposit, Little Khingan Ridge, Far Eastern Siberia.

Little more than a year after Strunz's sohngeite came the description by Moore and Smith (1967) of wickmanite, cubic $MnSn(OH)_6$, from Långban, Sweden. Wickmanite is a mineral counterpart of one of the synthetic compounds of Strunz and Contag. Some five years later Faust and Schaller (1971) described schoenfliesite, $MgSn(OH)_6$, another of the cubic tin compounds made by Strunz and Contag. It occurs as a late-stage hydrothermal alteration product in a contact-metamorphic zone on Brooks Mountain, Seward Peninsula, Alaska.

The introduction here of tetrawickmanite, the tetragonal dimorph of wickmanite and the manganese-tin analog of stottite, further extends the list. Yet, the stottite group is even larger, but the existence of two more members is based in part upon indirect evidence so complete descriptions, and the naming of these two new minerals, will have to wait until new specimens are discovered. In his study of the minerals of the tin mines of Cerro de Llallagua, Bolivia, Gordon (1944) concluded with a note about an unknown that was in the form of an incomplete description:

> "*Dark brown twelvelings* (Text fig. 82). Tiny dark brown crystals of octahedral habit were seen on a single small fragment of a quartz veinlet. Perched upon druses of quartz crystals on a yellow felt-like aggregate of tourmaline needles, were isolated crystals of apatite, siderite, cubes of pyrite, and a few bladed crystals of franckeite.
>
> The unidentified crystals, rarely as much as 0.1 mm. in diameter, occurred upon the franckeite as a rule. They are slowly soluble in HCl, and consist of an isotropic core, slightly greenish-brown under the petrographic microscope, with an index of refraction of 1.745.
>
> The isotropic core has upon each of its twelve octahedral edges a prismatic crystal, four of them meeting on each cube face, and having exactly isometric angles. The prismatic crystals have about the same index of refraction as the isotropic core, and weak birefringence, with positive elongation. They are probably tetragonal, and uniaxial negative, with the crystal forms *a* (100), and *p* (111), and $c/a = 0.707$."

To the authors there seems little doubt that Gordon's material is $FeSn(OH)_6$ and belongs in the "stottite group." Wickmanite has an index of refraction of 1.705. One would expect the iron analog to have a higher index and the "isotropic core" and the "prismatic crystals" of Gordon have an index of 1.745.

The U. S. National Museum of Natural History inherited much of Gordon's Llallagua collection but the "dark brown twelvelings" could not be found. However, careful examination of other specimens of Gordon's revealed a few tiny brownish-orange crude spherules (up to 0.1 mm) with apatite, siderite, pyrite and franckeite. These produced a powder pattern very similar to that of wickmanite except that the lines are spread further apart reflecting the smaller cell of the iron analog. The strongest lines of the powder pattern are 3.83 (10)(200), 2.71 (9)(220), 1.710 (9)(420), 1.560 (8)(422), 4.40 (7)(111), 2.30 (5)(311), 2.21 (5)(222), and 1.910 (5)(400), intensities visually estimated and indices assigned by comparison with the data for synthetic $FeSn(OH)_6$ (Strunz and Contag, 1960). The pattern is clearly that of an isometric mineral, so the Llallagua mineral is an analog of wickmanite rather than tetrawickmanite. A microprobe analysis of one of these grains gave the following rough values:

	Ideal $FeSn(OH)_6$	Llallagua mineral	
Fe	20.2	19.0	31.4
Mn	-	1.4	
Ca	-	10.5	
Mg	-	0.5	
Sn	42.6	32.5	

There can be no doubt that the iron analog of wickmanite exists. Actually Gordon has shown that the iron analogs

Table 3. Tetrawickmanite and wickmanite densities.

	Moore & Smith (1967) Calc.	Moore & Smith (1967) Obs.	Present study Calc.	Present study Obs.
Wickmanite	3.75*	3.89 ±0.05	3.76	3.65
Tetrawickmanite	--	--	3.79	3.65(+)

*the calculated density of 3.82 reported in Moore & Smith (1967) was based on a faulty cell volume calculation.

of *both* wickmanite and tetrawickmanite exist, and on the same crystal! All evidence indicates that Gordon's crystals are composed of isometric $FeSn(OH)_6$ in the cores (simple octahedra) with oriented overgrowths along the edges of the tetragonal dimorph. He suggested that the overgrowths are tetragonal but he assigned the indices incorrectly. He should probably have realized that his 0.707 (a/c) equals sin 45°; a strong indication of misinterpretation. That the overgrowing phase is tetragonal is supported only by Gordon's statement that it shows weak birefringence and positive elongation and is probably uniaxial negative. Note that he reported the index of refraction to be the same as the core.

There are some problems in analysing the data from Gordon's material but they can be explained, even though the loss of the crystals he actually described precludes absolute proof. The chemical analysis, for example, shows that the sum of Fe, Mn, Ca, and Mg is far in excess of the ideal and the Sn content is much too low. Further, the refractive index is too high (1.745), for calculations using the Gladstone and Dale rule show that it should be about 1.723. Both the analysis and refractive index become reasonable, however, if we assume that some of the iron is trivalent and substituting for tin as described earlier in this paper in the case of tetrawickmanite. If we would calculate an analysis based upon one coupled Ca for Fe^{II} and one Fe^{III} for Sn substitution out of every four formula units, or:

$$Fe^{II}_2Ca_2Fe^{III}Sn_3(OH)_{23}H_2O,$$

the results are very reassuring.

	Hypothetical	Llallagua material
Fe^{II}	19%	17%
Ca	10.5	8
$Sn+Fe^{III}$	32.5	35

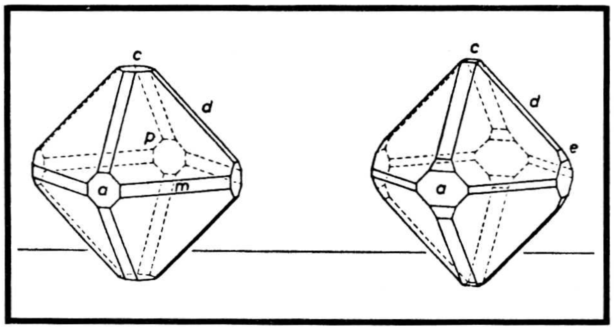

Fig. 2. **Drawing of stottite from Tsumeb, Strunz *et al* (1958).**

The presence of ferric iron will once again account for the higher than expected refractive index. All of this has only a minimal effect upon the size of the unit cell. The cell of the Llallagua mineral is 7.65Å (from powder data). Pure synthetic $FeSn(OH)_6$, as reported by Strunz and Contag (1960), is 7.79Å. Substitution of Ca^{II} for Fe^{II} (0.99Å vs. 0.76Å) is to some extent offset by the Fe^{III} for Sn^{IV} substitution (0.64Å vs. 0.71Å), so there is little overall cell size difference.

Therefore we may assume that the isometric and tetragonal iron hexahydroxostannates exist as minerals and that it will only be a matter of time before suitable crystals will be found to establish these new species. Any tin- and iron-rich locale would be a good place to look.

One such locality is Tsumeb. Geier and Ottemann (1970) provided brief descriptions of phases from Tsumeb which belong in the stottite group. They referred to these as *Minerals A, B, C,* and *D.*

Mineral A is a zincian stottite with the composition Fe 13.4, Zn 12.6, Co 0.9, Ni 0.2 and Ge 30.1. Cell parameters are *a* = 7.512 and *c* = 7.438Å.

Mineral B is the germanium analog of either wickmanite or tetrawickmanite, no powder or optical data provided. The analysis is Mn 23.2, Fe 1.2, Sn 2.7, and Ge 31.4.

Mineral C is isotropic so it is a germanian wickmanite with the composition Mn 21.5, Fe 3.5, Sn 39.6 and Ge 4.0.

Mineral D is described as a tin analog of stottite. It is essentially the same as either the isotropic or tetragonal mineral Gordon described from Llallagua. The analysis is Fe 14.2, Mn 3.5, Sn 43.4 and Ge 1.0. There is no powder or optical data provided.

There is still another locality reported for a mineral having the composition $FeSn(OH)_6$. Grubb and Hannaford (1966) identified extremely minute inclusions in Malayan

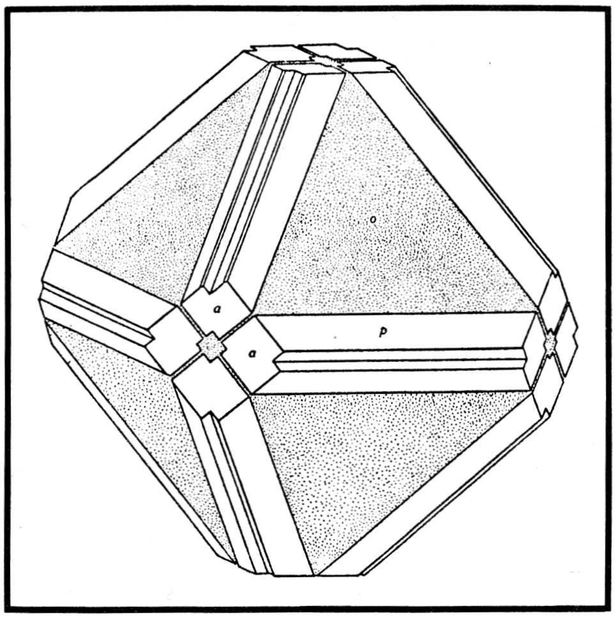

Fig. 3. **Drawing by Gordon (1944) of the "dark brown twelveling" from Llallagua.**

Table 4. Minerals of the stottite group and related synthetic compounds.

COMPOUND	SYNTHETIC		MINERAL			
	Cell Parameters	Reference	Mineral Name	Cell Parameters	Locality	Reference
ISOMETRIC						
$In(OH)_3$	7.958	Roy & Shafer*	dzhalindite	7.95	Far Eastern Siberia, USSR	Genkin & Murav'era
$Ga(OH)_3$			söhngeite	7.47	Tsumeb	Strunz
$FeSn(OH)_6$	7.79	Strunz & Contag	(unnamed)	7.65	Llallagua	Gordon White & Nelen
$MnSn(OH)_6$	7.88	Strunz & Contag	wickmanite mineral C	7.873	Långban	Moore & Smith Geier & Ottemann
$CoSn(OH)_6$	7.78	Strunz & Contag				
$MgSn(OH)_6$	7.77	Strunz & Contag	schoenfliesite	7.759	Alaska	Faust & Schaller
$CaSn(OH)_6$	8.13	Strunz & Contag				
$ZnSn(OH)_6$	7.80	Cohen-Addad**				
$NiSn(OH)_6$	7.711	Morganstern & Badnan***				
TETRAGONAL						
$FeGe(OH)_6$			stottite	a = 7.55 c = 7.47	Tsumeb	Strunz, Söhnge, & Geier
$(Fe,Zn)Ge(OH)_6$			zincian stottite (A)	a = 7.512 c = 7.438	Tsumeb	Geier & Ottemann
$MnSn(OH)_6$			tetrawickmanite	a = 7.870 c = 7.797	North Carolina	White & Nelen
$FeSn(OH)_6$			(unnamed)		Llallagua	Gordon
CRYSTALLOGRAPHY UNKNOWN						
$MnGe(OH)_6$			mineral B		Tsumeb	Geier & Ottemann
$(Fe,Mn)(Sn,Ge)(OH)_6$			mineral D		Tsumeb	Geier & Ottemann

* Roy & Shafer, *J. Phys. Chem.*, **58,** 372 (1954)

** Cohen-Addad, *Bull. Soc. franc. Mineral. Cristallogr.*, **90,** 32 (1967)

***Morganstern & Badnan, Lab. Chim. Mineral., Univ. of Paris, France (1968)

cassiterite as iron hexahydroxostannate but it is very unlikely that crystals of sufficient quality to properly characterize the species will ever be found in this material. The best prospects appear to be the Llallaguan and Tsumeb occurrences.

ACKNOWLEDGEMENTS

The authors gratefully acknowledge the important contribution of Walter R. Brown (Smithsonian Institution) in taking the SEM photographs of tetrawickmanite. The x-ray data refinement was done by Miss Judy Konnert, of the U. S. Geological Survey.

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Note: A second locality for wickmanite has been recently reported by R. Åmli and W. L. Griffin, "Three Minerals

New to Norway: Wickmannite, Leadhillite and Hydrocerussite", *Norsk Geologisk Tidsskrift*, **52**, 193-196, 1972. Wickmanite was found as a poorly developed 0.5 mm crystal which was deep golden yellow under the binocular microscope. Associated with it are analcime, fluorite and a chlorite. It was found in a syenitic pegmatite intruding larvikite at Tvedalen (Heia quarry) near Larvik, southern Norway.

It is unfortunate that the authors misspelled wickmanite in the title and throughout the article.

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