ART. XLVI.—*On Sipylite, a new Niobate, from Amherst County, Virginia; by J. W. MALLET.*

THE allanite found in Amherst County in this State, of which an analysis by Mr. J. A. Cabell, was published in the *Chemical News*, 1874, p. 141, occurs in large quantity, and furnishes an abundant source of supply of the cerium family of metals. In picking over a lot of three or four hundred pounds of it, I was struck with the appearance of a few fragments of an accompanying mineral, which on more careful examination turns out to be a new niobate.

The locality in question is on the northwest slope of Little Friar Mountain, about fifteen miles from the Virginia Midland Railway. The allanite is said to occur in a vein of more or less decomposed feldspar in a gneissoid rock, and is met with in large, but very imperfect crystals, and loose lumps of irregular shape, about four feet below the surface of the ground. Magnetite is found with it, the two minerals often forming parts of the same mass; and in going down the vein seems to become more compact, and tends to pass into solid magnetic iron ore. The vein is said to be about two feet wide, and runs about northeast and southwest, dipping at a large angle to the southeast.

Beside allanite, magnetite, and the new mineral now to be described, I have only noticed among the specimens which have reached me a few large crystals of hydrous zircon. One of these measured about $30 \times 18 \times 13$ mm., was doubly terminated, of sp. gr. = 4.217, and yielded on ignition 1.89 per cent of water.

The new mineral is decidedly rare; all the specimens I have collected were picked out from three lots of the allanite, two of them of several hundred pounds each, and would probably not weigh half a kilogram; the largest single piece weighs about forty grams; most of the fragments are much smaller. It is found imbedded in, or more commonly adherent to, the outside of the masses of allanite and magnetite, from which it is easily detached.

A few imperfect crystalline faces have been met with, but none of these brilliant, and only two dihedral angles that could be, even in a very rough way, measured with the application of a goniometer; each of these was about 125° , which is not far from $I \wedge I$ of the prism of yttrio-tantalite, samarskite, and euxenite. There were observed also a few very imperfect cleavage planes. For the most part the mineral appears in little, irregularly shaped masses, very brittle, and exhibiting small, but distinct, conchoidal, as well as uneven fracture.

The color of the mineral in mass is brownish black, in thin splinters a red brown, like that of dark pine-rosin; one or two small specimens display a gradual passage to a brownish orange, and even a yellow, but whether in these cases the chemical composition remains quite the same, there is not sufficient material to determine. The streak is light cinnamon-brown to pale gray. The luster resinous and pseudo-metallic. In general appearance to the eye the mineral is much like fergusonite from Greenland, euxenite from the neighborhood of Arendal, and samarskite from North Carolina, save that the last named is more distinctly pitchy black. Translucent in thin splinters. Hardness = nearly 6. Specific gravity may be considered = 4.89; one specimen gave 4.887 at 12°·5 C.; another 4.892 at 17°·5.

Heated alone in the ordinary blowpipe flame the mineral cracks, decrepitates, *glows brilliantly* (more brightly, I think, and at a lower temperature than any specimen of gadolinite I have ever seen), becomes pale greenish-yellow and opaque, like many specimens of good blast-furnace slag, and remains quite infusible. In the flame of one of Fletcher's hot-blast blowpipes, before which a stout blowpipe wire of platinum readily melts to a bead, thin splinters are fused merely on the edges. Heated in a closed glass tube, the same decrepitation, glowing and change of color are observed, and water is given off, which condensing on the surface of the tube is found to have an acid reaction, and slightly etches the glass. Fused with borax in the oxidizing flame, the mineral is dissolved, producing a yellow glass, which becomes pale on cooling, and assumes a greener tint in the reducing flame. With microcosmic salt, a yellowish green glass is obtained. Strong boiling hydrochloric acid attacks to some extent the mineral in fine powder, and the partial solution, if boiled with metallic tin and diluted with water, gives the fine sapphire-blue color due to niobium. This partial hydrochloric acid solution, if diluted, contains zirconium enough to brown turmeric paper to an extent quite sensible if a comparative experiment be made with similarly diluted hydrochloric acid alone. Boiling concentrated sulphuric acid decomposes the mineral completely, though somewhat slowly; and the diluted solution gives a blue color on addition of metallic zinc.

The chemical analysis was made, with much care and patience, under my direction by Mr. W. G. Brown, a student in this laboratory during the last winter. The details of the method used are given in a notice of his work in the *Chemical News*. Tantalum was found to be present, but in such small quantity, certainly less than one-twelfth of the niobium, that a satisfactory separation could not be obtained by Marignac's method. The sp. gr. of the mixed niobic and tantalic oxides

was 4·60. By determination of the yttrium and erbium first as oxides and then as sulphates it was found that the mixture contained almost exclusively the latter metal, of which the absorption spectrum is obtainable with great distinctness from the crude solution. Iron and uranium were proved to exist as ferrous and uranous compounds. The following results were obtained :

Nb ₂ O ₅ -----	} 48·66
Ta ₂ O ₅ * -----	
WO ₃ -----	
SnO ₂ -----	·16
ZrO ₂ -----	·08
Eb ₂ O ₃ -----	2·09
Y ₂ O ₃ † -----	} 27·94
Ce ₂ O ₃ ‡ -----	
La ₂ O ₃ § -----	
Di ₂ O ₃ -----	1·37
UO ₂ -----	3·92
MnO -----	4·06
FeO -----	3·47
BeO -----	trace
MgO -----	2·04
CaO -----	·62
Li ₂ O¶ -----	·05
Na ₂ O -----	2·61
K ₂ O -----	trace
F -----	·16
H ₂ O -----	·06
	3·19

 100·48

Throwing together, as Rammelsberg has done in his valuable paper** on the natural tantalates and niobates, the acid oxides of niobium, tantalum, tungsten, tin and zirconium, reducing all the basic oxides present to the equivalent amounts of dyad oxides, and leaving out the water, we have from the above figures the ratio,

$$R''O : M^v_2O_5 = 221 : 100$$

leading to the formula $R''_3M^v_2O_8 \cdot 4 R''_2M^v_2O_7$, or, applying the common phosphate nomenclature, a single atomic group of ortho-niobates with four of pyro-niobates; while samarskite, according to the calculation of Professor O. D. Allen†† from his analysis, contains one to one, or is represented by the formula

* Ta₂O₅ may be assumed = about 2 per cent.

† Y₂O₃ may be assumed = about 1 per cent.

‡ Cerous oxide, but with Cléve's formulæ and atomic weights for this and the corresponding oxides of lanthanum and didymium.

§ Containing a trace of Di₂O₃.

|| Containing a trace of Ce₂O₃.

¶ Spectroscopically detected.

** Jour. Chem. Soc., March, 1872, p. 189.

†† This Journal, August, 1877, p. 131.

$R''_3M_2^V O_8 \cdot R''_2M_2^V O_7$, and Rammelsberg makes pyrochlore from Fredriksvårn solely the pyro-niobate, $R''_2M_2^V O_7$, and fergusonite, tyrite, etc., solely the ortho-salt, $R''_3M_2^V O_8$.

If, however, the water be included in the calculation, and considered basic, placing it on an equivalent footing with the dyad oxides we have the ratio,

$$R''O : M_2^V O_5 = 311 : 100, \text{ or nearly } 3 : 1,$$

which gives the simple formula of an ortho-salt, $R''_3M_2^V O_8$. This I confess I am inclined to think more probable, and, if so, it may be allowable to suppose that the very remarkable glow exhibited by the mineral when heated is connected with the loss of basic water and the change from ortho- to pyro-niobate, as in the well known incandescence of ammonio-magnesian ortho-phosphate at the moment of change by heat to the pyrophosphate of the latter metal.*

Whichever formula be preferred, however, for the mineral now described, it differs essentially from that of any niobate hitherto on record, the one view making it the nearest approach to a simple pyro-niobate (since the Fredriksvårn pyrochlore contains largely of titanium) and the other making it an ortho-salt like fergusonite, etc., but one partially acid in character or containing basic hydrogen.

Not on chemical grounds alone, but in several respects as to physical properties, the mineral is new and distinct. Carrying out the fancy of Heinrich Rose, which led him to name niobium from the daughter of Tantalus, and remembering the number and complexity of the natural niobates which have been met with, I propose for this species the name *Sipylite*, from *Sipylus*, one of the numerous children of *Niobe*.

* It may be worth remarking that from the analysis of Professor Allen (loc. cit.) of Professor J. Lawrence Smith's new mineral, hatchettolite, which accompanies samarskite in North Carolina, water seems to be present in it in definite proportion; and, although Rammelsberg has considered the water found in his analyses of tantalates and niobates as non-essential, and the formula, $R''_3M_2^V O_8$, which he has assigned in common to fergusonite, ytthro-tantalite, tyrite and bragite, requires that water be excluded, if it be also taken into account his analyses of these minerals lead pretty closely to simple relations as to the extent of hydration (without considering the water basic), making

Fergusonite, from Greenland (with very little water)— $R''_3M_2^V O_8$,

or perhaps $2R''_3M_2^V O_8 \cdot H_2O$.

Brown ytthro-tantalite, from Ytterby	} $2 R''_3M_2^V O_8 \cdot 3H_2O$.
Yellow " " "	
Tyrite	
Bragite	
Gray ytthro-tantalite, from Gamle Kararfvet	$2R''_3M_2^V O_8 \cdot 5H_2O$.

University of Virginia, Sept. 3, 1877.