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MINERALOGICAL NOTES.¹

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Although the results of analysis of the herein-mentioned minerals, unsupported as they are, with one exception, by any crystallographic evidence, lead to complete identification of the species only in one case, they are deemed of sufficient importance to place on record, together with such information regarding their physical properties as could be gathered from the material at hand, which was in some cases rather scanty.

A few years ago in granitic débris on Devil's Head Mountain, Douglas County, Colo., in the Pike's Peak region, Mr. W. B. Smith found a number of minerals which appeared worthy of examination. Two of these, allanite and gadolinite, have already been analyzed by Mr. L. G. Eakins.² The following analyses, I-VI, represent the composition of the others, of which all but No. III were made in the laboratory of the U. S. Geological Survey, in Denver, over two years ago.

1. SAMARSKITE †.

The mineral first to be mentioned occurred in fragments of all sizes up to that of a chestnut, without any evidence of crystal form except on a small portion of one piece, where a structure resembling that shown in Fig. 9 was apparent. The color was pitch black, with brilliant vitreous luster, but pale brown in thin splinters; streak dirty brown. The pieces were very brittle, and when heated flew into fine, sharp fragments. Fracture subconchoidal. These characteristics agree very closely with those given by J. Lawrence Smith³ for the purest North Carolina samarskite, but the density is much greater, being 6.18 at 22° C. for I, as might be expected from the percentage of Ta_2O_5 , which is much higher than that as yet found in any recognized samarskite. Hardness, 5.5 to 6. Slight solubility in HCl was shown on prolonged boiling, and a somewhat greater in H_2SO_4 . Before the blow-pipe a splinter became dull, but did not fuse. Analysis I shows the composition.



FIG. 9.—Samarskite (†).

A single piece of but a few grammes weight, while in its general behavior and appearance resembling those just described, presented two points of difference. The streak was salmon colored, and the rather thin piece was bounded on two opposed sides by flat surfaces, which

¹ Read in part before the Colorado Scientific Society, March 5, 1888.

² Proc. Colorado Sci. Soc., vol. 2, p. 32.

³ Am. Jour. Sci., 3d series, vol. 13, 1877, p. 359; Ann. Chim., vol. 12, 1877, p. 253; Original Researches in Mineralogy and Chemistry, p. 197.

might possibly be the result of cleavage. These observed differences led to analysis II. The density at 25° C. was 6.12, and the hardness 5.5. As the analysis reveals a remarkable agreement in composition with the first-described specimens, there is no reason to suspect the presence of distinct species.

Analysis III gives the composition of the specimen shown in Fig. 9, which it was thought might be an altered state of the above-described mineral. It was evidently in an advanced stage of alteration, being very easily broken, and so much stained (brown) that scarcely any evidence of its original color and luster could be observed, though what little of these features remained indicated agreement with those above mentioned. As some water escaped at 100° C., and still more for each further slight increase in temperature, air-dried material was taken for the analysis. This shows in most respects so close an agreement with I and II, that the assumption of original identity with the brilliant black mineral does not seem far-fetched. The chief differences lie in the much lower percentage of Ta_2O_5 in III, and the presence of UO_3 instead of UO_2 . The density at 16° C. was 5.45.

Analyses of samarskite.

	I.	II.	III.
Ta_2O_5	27.03	28.11	19.34
Cb_2O_6	27.77	26.16	27.56
WO_3	2.25	2.08	5.51
SnO_2	0.95	1.09	0.82
ZrO_2	*2.29	*2.60	3.10 (with TiO_2)
UO_3	4.02	4.22	6.20 (UO_3)
ThO_2	3.64	3.60	3.19
Ce_2O_3	0.54	0.49	0.41
(Di, La) $_2O_3$..	1.80	2.12	1.44
Er_2O_3	†10.71	†10.70	†9.82
Y_2O_3	†6.41	†5.96	†5.64
Fe_2O_3	8.77	8.72	8.90
FeO	0.32	0.35	0.39 (or 0.74 UO_2)
MnO	0.78	0.75	} 0.77
ZnO	0.05	0.07	
PbO	0.72	0.80	1.07
CaO	0.27	0.33	1.61
MgO			0.11
K_2O	0.17	0.13	} 0.36
(Na, Li) $_2O$..	0.24	0.17	
H_2O	1.58	1.30	3.94
Fl	‡	‡	‡
	100.31	99.75	100.18

* Probably containing some TiO_2 .

† Calculated from the molecular weight of the mixed oxides of this group on the unsupported assumption that only Er_2O_3 and Y_2O_3 were present. The molecular weight in the case of I was 304.9; in that of II, 307.7.

The method of analysis followed was, with certain modifications, that devised by J. Lawrence Smith.¹ Two instead of five grammes of material were taken for greater convenience in manipulation.

A few remarks in regard to certain points in the course of analysis may prove of service to those having similar analyses to make. By the method of Smith the constituents of the mineral are divided at once into two portions, those soluble in HFl and those insoluble. The former portion includes Ta, Cb, Sn, W, U (if present as UO_2), Fe,² Ti, Zr in part, Zn, Mn, Mg, K, and Na, while the latter contains the earths of the cerium and yttrium groups, Th, U (if present as UO_2), part of the Zr when other earths are present, a small part of the Fe, and the Pb and Ca.

It was found, after converting the insoluble fluorides into sulphates and dissolving in water, that a white insoluble residue remained, which, according to Smith in the case of samarskite, should be Cb_2O_5 and Ta_2O_5 . Neither of these could be found in it, however, the whole being lead sulphate. After filtering, the remainder of the lead was thrown down by H_2S . The uranium then having been oxidized by nitric acid, the earths (except ZrO_2 and some CaO) were thrown down by oxalic acid (not ammonium oxalate) in excess. The use of oxalic acid was rendered necessary by the observed solubility of the earths of the yttrium group in ammonium oxalate. These when freed from bases of the cerium group were not precipitated by this reagent, or, more properly, the precipitate at first formed dissolved *completely* in excess of the precipitant on very gentle warming and was not again precipitated on cooling. This behavior indicates the presence of erbium in quantity, which may influence the yttrium present, for the latter by itself is stated to be only partially soluble in concentrated boiling ammonium oxalate and to be again thrown out on cooling. It should be remarked, however, that the solubility above mentioned was very much greater than when to a hot solution of erbium and yttrium sulphates,³ prepared according to the data furnished by the above analyses, ammonium oxalate was added in excess.

The filtrate from the insoluble fluorides was evaporated to dryness, dissolved in water and a few drops of HFl, and the tin was thrown down by H_2S , as done by Marignac.⁴ The filtrate was then evaporated with large excess of H_2SO_4 , heated till all HFl was certainly removed;

¹Am. Chem. Journ., vol. 5, pp. 44, 73; Chem. News, vol. 48, pp. 13, 29; Original Researches in Mineralogy and Chemistry, p. 350.

²Treatment with HFl at once reveals whether the iron is present as admixed hematite or limonite (as in analysis III, where it remained suspended in the liquid in an extremely fine state of division), or is chemically combined with the acids of the mineral. In the first case it resists the solvent action of HFl for some time after the tantalates and columbates are thoroughly decomposed.

³From T. Schuchhardt. Though marked *chem. pure*, it is not at all probable that they were so.

⁴Ann. Ch. Phys., 4th series, vol. 13, p. 5.

then, without driving off the excess of H_2SO_4 , diluted and boiled (stirring constantly to prevent bumping) in a very large platinum dish for ten or fifteen minutes. The precipitate collected on a filter was then dissolved in HFl , the filtrate evaporated with excess of H_2SO_4 and treated in every way as above described till after the third precipitation by boiling. The combined filtrates were then evaporated, most of the H_2SO_4 was driven off, the residue taken up with water and boiled for some time. The remainder of the metallic acids, except some TiO_2 , if present, is now entirely precipitated, but to free altogether from ZrO_2 , Fe_2O_3 , and from most of the TiO_2 the precipitate was again dissolved in HFl , the filtrate was evaporated with H_2SO_4 , the excess of the latter partly driven off, and the acids were again thrown down by boiling.

This process is somewhat tedious, but seemed to possess the advantage of entirely separating Ta, Cb, and W from Fe and Zr and in great measure from Ti, unless the latter is present in quantity. The point to be observed is that the first precipitations shall be made in strongly acid solutions, the latter in solutions less strong. The procedure of Smith—driving off nearly all H_2SO_4 at first and then boiling with HCl —is objectionable; the metallic acids become strongly caked during removal of the H_2SO_4 and retain much iron, which no amount of boiling with HCl can remove. The further treatment of the combined filtrates requires no special mention.

For the separation of WO_3 the ignited and weighed oxides were fused with Na_2CO_3 and S and separated as recommended by Rose. The method, however, leaves much to be desired, for a considerable quantity of the R_2O_3 acids goes into solution with the W. Cb_2O_5 , and Ta_2O_5 were separated by Marignac's method.

FeO was estimated by titration with $KMnO_4$ after solution of the very finely powdered mineral in H_2SO_4 by heating for two or three days in sealed tubes, a blank experiment being made with H_2SO_4 alone in another tube. The excess of $KMnO_4$ above that required for the conversion of all uranium (when assumed present as UO_2) to UO_3 was considered to be equivalent to the FeO . The accuracy of the determination is of course not affected by the fact that uranous salts reduce ferric salts in solution.

Fluorine could not be found quantitatively, but from the slightly acid reaction of the water evolved on heating the mineral, its presence is rendered probable.

From the two first analyses it is impossible to identify the mineral with any known species beyond doubt, although it appears to be more nearly allied to samarskite than to any other. The presence of the iron as Fe_2O_3 distinguishes it from most other tantalates and columbates containing much iron. That the greater part of the iron exists in the ferric condition is certain, for if the uranium existed as UO_3 instead of UO_2 , as assumed, the amount of $KMnO_4$ required to oxidize 4.02 per cent. of UO_2 (anal. J) would indicate but 2.13 per cent. of FeO .

2. AN ILL-DEFINED ZIRCONIUM MINERAL.

With the mineral just described occurred peculiar growths, from an inch to an inch and three-quarters in length, of a pale brown color, and having the forms shown in Figs. 10 and 11, of which analyses IV-VI represent the composition. Evidences of crystal faces were observed on the upper terminations of some specimens. It seemed not at all probable from its appearance that the substance was homogeneous, and a thin section showed plainly a mixture of two or more minerals. One of these is probably limonite, and the action of HFl supports this view; for, while the powder as a whole was instantly decomposed, brown oxide of iron remained for some time suspended in the liquid.



FIG. 10.—Cyr-
tolite (?).



FIG. 11.—Crytolite (?).

The specimen represented by analysis VI differed much in appearance in places from the others, fractured surfaces being coarsely granular, and showing clear reddish grains of some size which could easily be detached. The sp. grs. were 3.70 (IV), 3.60 (V), 3.64 (VI). The analyses give the composition of air-dried material, for it was found that at 100° C. and with each further increase in temperature water was lost. They are only of value as showing the composition of these characteristic forms, which are mixtures of perhaps cyrtolite, or some allied alteration product of zircon, with limonite and a phosphate.

Analyses.

	IV.	V.	VI.
Ta ₂ O ₅	} 47.99	0.71	} 51.00
SrO ₂		0.03	
ZrO ₂		47.81	
SiO ₂	20.06	20.64	19.21
ThO ₂	1.16	} 1.20	0.60
Ce ₂ O ₃	0.06		
(Di,La) ₂ O ₃	0.19		
Er ₂ O ₃	4.77	4.76	4.55
Y ₂ O ₃	2.27	2.48	3.13
Fe ₂ O ₃	5.53	5.97	4.86
MnO	0.47	0.57	0.33
CuO	1.99	1.93	2.15
MgO	0.13	0.11
K ₂ O	0.20	0.10	0.17
Na ₂ O	0.46	0.50	0.42
H ₂ O	12.87	12.00	12.97
P ₂ O ₅	1.64	1.75	0.93
Fl.	0.25	0.42	0.42
	100.04	100.98	100.74

3. WHITE BERYL.

The crystals analyzed were from a specimen in the National Museum found in a tin mine near Winslow, Me. The beryl is of interest on account of its color, which is white, with a faint greenish hue under certain conditions of light. Mr. J. S. Diller examined the crystals and reported as follows:

The white beryl which you gave me to examine is full of gas and liquid inclusions, most probably air and water, to which the light color of the beryl is due. Besides these it contains a considerable amount of fine ferritic dust, but on the whole is about as pure as minerals usually are. The beryl itself is fresh, and has suffered but little, if any, alteration.

The sp. gr. at 27°C. was 2.707.

VII.

SiO ₂	65.21	K ₂ O }	0.14
TiO ₂	trace	Cs ₂ O }	
Al ₂ O ₃	18.50	Na ₂ O	0.87
Fe ₂ O ₃	*0.33	Li ₂ O	0.16
BeO	†13.03	H ₂ O	†1.80
MgO	0.09	P ₂ O ₅	none
			100.13

* FeO, if present, not estimated.

† BeO and Al₂O₃ separated in the manner described by Penfield. (Am. Jour. Sci. [3], xxxii, 114).

; After drying at 110° C.

4. A SULPHIDE OF COPPER, SILVER, AND ZINC.

Mr. Richard Pearce gives an analysis¹ of a mineral from the Gagnon Mine, Butte, Mont., which he describes as massive, and in appearance much like bornite, with density 4.95 and hardness 3.5 to 4. A specimen received from Mr. Pearce, and analyzed at his request, had a density of 5.407 at 20° C. It was rather brittle, but flattened out in part under the pestle. From analysis VIII 0.12 per cent. of gangue has been deducted. Pearce's analysis is given under IX.

	VIII.	IX (Pearce.)
Cu	40.24	41.10
Ag	21.80	24.66
Pb	1.46
Zn	12.83	9.80
Fe	1.98	2.09
S	20.88	20.51
Insoluble	1.02
	99.19	99.18

¹ Proc. Colorado Sci. Soc., vol. 2, p. 70.

The general formula deduced from both analyses is RS , with the different constituents apparently in varying proportions. It is impossible from the specimens examined to decide whether the mineral is new or only a mixture.

5. AN ARGENTIFEROUS ARSENIDE OF NICKEL AND COBALT.

From Mr. W. George Waring, through Mr. Whitman Cross, a mineral from the Rose Mine, Grant County, N. Mex., was received, which has the composition given under X. It is said to occur also in the Black Hawk Mine, in the same county. It has a steel-gray color, and the particles abraded by a sharp blow of the hammer "take fire, and the pathway of each particle thus ignited as it falls to the ground is marked very distinctly by a trail of dense, white smoke," according to Waring. In a closed tube a heavy sublimate of arsenic is formed on very moderate heating. The gangue of the mineral is mainly carbonate of lime and iron with quartz. The carbonates may be extracted with HCl , when the arsenide is left entirely unaffected and showing evidences of crystalline structure ("pyritohedral," Waring). The sp. gr., after correcting for 4.80 per cent. of quartz, was 6.644 at $20^{\circ} C$.

	X.		X.
As	74.04	Ni }	*19.52
S	0.13	Co }	
Ag	4.78	CaO	0.09
Cu	0.04	MgO	0.05
Pb	0.03		99.12
Fe	0.44		

* The separation of Ni and Co miscarried. According to a blow-pipe analysis by Waring the proportion of Ni to Co is as 3 to 1.

The atomic ratio of Ni, Co, Ag, Fe to As is much above 1: 2.5, showing either that the substance is a mixture of two minerals with the general formulas RAs_2 and RAs_3 , or of arsenic with one of the formula RAs_2 .

6. A BASIC CUPRIC SULPHATE.

Two soft lumps of ore of a light green color (rubbing off on the fingers) from the Antler mine, Yucca Station, on the Atlantic and Pacific Railroad, in Mohave County, Ariz., gave the following composition, after deducting 8 and 6 per cent. respectively of gangue, which latter consisted mainly of quartz and a brownish foliaceous mineral. Sp. gr. at $16^{\circ} C$., 3.93, after correcting for gangue, the density of which was assumed to be 2.6.

	XI.	XII.
CuO.....	68.19	67.64
ZnO.....	0.29	0.04
CaO.....	0.05	0.04
SO ₃	20.46	21.49
H ₂ O.....	11.11	10.76
	100.10	99.97

The molecular ratios are:

	RO	SO ₃	H ₂ O
I.....	10.00	: 2.98	: 7.19
II.....	10.00	: 3.15	: 7.01

which lead to the formula $3\text{CuSO}_4 + 7\text{Cu}[\text{OH}]_2$, for which theory requires 68.45 CuO, 20.69 SO₃, 10.86 H₂O. If in time this should prove to be a well-defined species, which conclusion the absence of crystalline form and want of very close agreement in the analyses do not warrant at present, the name *antlerite*, from the mine in which it occurs, is suggested as appropriate.