

ART. XI.—On Danalite, a new Mineral Species from the Granite of Rockport, Mass.; by JOSIAH P. COOKE, Jr.

DISSEMINATED through the Rockport granite, which is quarried at the extremity of Cape Ann, Massachusetts, and much used for building in Boston and the vicinity, are occasional grains of a flesh-red mineral somewhat resembling Rhodonite.

The mineral has been at times found in masses of considerable size, and for a specimen of this sort I am indebted to the kindness of Mr. W. J. Knowlton, of the Lawrence Scientific School.

The characters of the mineral are as follows: Color, flesh-red to gray. Streak similar in color to the mineral but lighter. Lustre, vitreo-resinous. Translucent. Fracture subconchoidal uneven. Brittle. Hardness 5.5 to 6. Specific gravity—two determinations—8.427. The exterior portion of the mass showed no indication of crystalline form and there was no distinct cleavage; but on breaking it open a well developed octahedron of the regular system was found in the interior. The angle between the octahedral faces measured with an application goniometer $109^{\circ} 30'$. The edges of the octahedron were replaced by planes of a rhombic dodecahedron, strongly striated parallel to the longer diagonal of the face. The mineral, therefore, crystallizes in the *holohedral* forms of the monometric system.

Before the blowpipe the mineral readily fuses on the edges to a black enamel. Hence its fusibility is about 4 of von Kobell's scale. On charcoal with carbonate of soda it gives a slight coating of oxyd of zinc. In a closed tube it loses color, but gives off no water or any sublimate. It is perfectly decomposed after some time by hydrochloric acid, the silica partly gelatinizing. It is also decomposed by nitric acid; but then the silica separates as a powder. It is partially decomposed by dilute sulphuric acid, and even by acetic acid, sulphid of hydrogen gas being evolved.

In order to thoroughly decompose the mineral the material was finely pulverized and sealed up with some concentrated acid in a glass flask, which was then exposed for several hours to the heat of a water-bath. When hydrochloric acid was used a slightly greenish solution was obtained, frequently depositing crystals of protochlorid of iron on cooling, but showing no traces of sesquichlorid, and on opening the flask a strong odor of sulphid of hydrogen was observed. When nitric acid was used the flask became filled with nitrous vapors, and both the iron and the sulphur were completely oxydized. A qualitative analysis proved the mineral to be a compound of silica, glucina, protoxyd of iron, oxyd of manganese, and oxyd of zinc, mixed with the sulphids of the last three metals. The presence of

alumina could not with certainty be detected by any known tests. The precipitate of glucina perfectly redissolved in an excess of carbonate of ammonia, and no crystals of alum could be obtained from a solution of the sulphate when treated in the usual way with an excess of sulphate of potash, although they were sought for with a microscope.

As the sulphid of hydrogen which is evolved from the metallic sulphids, when the mineral is decomposed by hydrochloric acid in a closed flask, would necessarily reduce all the iron present to the condition of proto-chlorid, the following experiment was made to determine the original condition of the iron in the mineral. It is evident that any such reduction must be attended with the separation of free sulphur, and hence sulphur was sought for in the products remaining in the flask after the decomposition was finished. The sulphid of hydrogen and the greater part of the free hydrochloric acid having been first expelled, the residue was boiled with an excess of concentrated nitric acid, and as no trace of sulphuric acid was found it was concluded that the iron in the mineral, not united with sulphur, was all in the condition of protoxyd. The same experiment also proved that none of the varieties of iron pyrites could be present in the mineral in distinct grains, as was at first suspected; and this conclusion was confirmed by the fact that a powerful magnet failed to attract any portion of the mineral, even when reduced to the finest powder.

In the quantitative analysis no unusual methods were employed. The mineral was decomposed in a sealed flask as described above, sometimes by hydrochloric but usually by concentrated nitric acid. The silica was separated in the ordinary way and its purity tested by hydrofluoric acid, the small amount of residue, which was in some cases obtained, being carefully determined and estimated in calculating the final result. The iron was then precipitated as basic acetate, and this precipitate carried down with it a portion of the glucina. From the filtrate the manganese was separated by bromine, and the zinc was precipitated by sulphid of hydrogen. The filtrate from the sulphid of zinc was always found to contain the larger part of the glucina,¹ which was precipitated by ammonia. The glucina thus precipitated was weighed down by itself and the amount added to that subsequently separated from the iron. This last separation was effected by a simple modification of Deville's method which will be described at the end of this article. Lastly, the sulphur, which now remained in solution as sulphuric

¹ We have never succeeded in precipitating the whole of the glucina as basic acetate, although carefully attending to all the precautions which have been indicated by other analysts. But alumina is perfectly precipitated when the necessary precautions are observed.

acid, was determined as sulphate of baryta in the usual way. The results of my analyses were as follows:

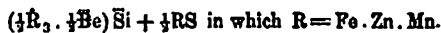
	1.	2.	3.	4.	Mean.
Silica,	31.74	31.54	31.96	31.69	31.73
Protoxyd of iron, -	25.71	29.09	27.40
Oxyd of zinc, -	17.90	16.90	19.11	16.14	17.51
Oxyd of manganese, -	5.83	6.64	6.17	6.47	6.28
Glucina,	13.86	13.79	13.83
Sulphur, -	5.93	5.02	5.48
			102.74	102.20	102.23
Oxygen equivalent to per cent of sulphur,			2.96	2.51	2.74
			99.78	99.69	99.49

For analysis 4 the material used was a portion of the crystal described above. When in mass it had a bright flesh-red color, and even in the powder the color was still quite decided. The material used in analysis 3 was taken from a wholly different portion of the specimen. The red tint was less marked and the color of the powder was decidedly gray. These two portions were selected as presenting the most decided contrasts of color observed. Analyses 1 and 2 were made before the composition of the mineral was correctly known, and the best method of analysis discovered. Hence, only a portion of the bases were accurately determined, and only those results are given which are known to be trustworthy. The material for all the analyses was selected with great care; but that used in 4 being a portion of a crystal from the center of a large mass was unquestionably the most pure.

The most natural theory of the constitution of the mineral, to which the above results and the crystalline form both point, is that the mineral is an isomorphous mixture of a monometric silicate with the simple sulphids of iron, zinc, and perhaps of manganese, all of which affect the same crystalline form. These different sulphids must be present in somewhat varying proportions; for while in 4 the sulphid of iron is evidently in excess, the sulphid of zinc equally predominates in 3, and such a difference is plainly indicated by the difference of color already mentioned. This view is also sustained by the action of different acids on the pulverized mineral. Dilute sulphuric acid attacks the powder even when cold, sulphid of hydrogen being evolved, while iron and zinc in large quantities, with some glucina, enter into solution. Even dilute acetic acid causes an evolution of sulphid of hydrogen dissolving an abundance of iron, but also some glucina. Since now sulphid of zinc is insoluble in acetic acid this last reaction points clearly to the presence of sulphid of iron in the mineral; but at the same time both experiments indicate that the metallic sulphids are so intimately associated with the silicate that the decomposition of the first involves to

a limited extent at least the breaking up of the last. This is what we should naturally expect in an isomorphous mixture, the sulphids not being present in separable grains; but diffused through the mineral in a state of imperfect chemical combination, and thus rendering even a firm silicate exceedingly susceptible of decomposition.

On examining the results of analysis given above it will further appear, in support of the same theory of the constitution of the mineral, that while the proportions of silica, glucina, and even of oxyd of manganese, are very constant, the proportions of the oxyds of zinc and iron vary very considerably; these metals, although in the analyses separated and determined as oxyds, being, in fact, combined to a greater or less extent with sulphur in the different portions of the mass examined. If now we calculate on this view the oxygen ratio of the silicate, deducting of course from the sum of the oxygen of the bases, an amount of oxygen equivalent in each case to the per cent of sulphur found, it appears, taking the mean result, that the sum of the oxygen of the bases is to the sum of the oxygen of the silica as 16.93 : 16.81, or very nearly as 1 : 1. Moreover, if we regard glucina as a sesquioxid base we shall have for the oxygen ratio between protoxyds, sesquioxys and silica, the proportion 8.22 : 8.71 : 16.81 or very nearly as 1 : 1 : 2. Again, the amount of sulphur in the metallic sulphids is very nearly one-sixth of the amount of oxygen in the silicate; so that for every twelve equivalents of oxygen in the silicate we have one equivalent of sulphur in the sulphids. Hence we deduce as the general formula of the mineral



The oxygen ratio of the new mineral is the type ratio of the garnet family, and to this family it undoubtedly belongs. Its mineralogical characters would place it intermediate between Willemite and Iron Garnet where by its composition it naturally falls. This relationship appears in the following table; but it is seen much more clearly on comparing the actual specimens :

	WILLEMITE.	DANALITE.	IRON GARNET.
Action of hydrochloric acid,	Gelatinizes easily.	Gelatinizes, but less easily and perfectly.	Decomposed, but does not gelatinize.
Before blowpipe,	Fuses on edges to white enamel.	Fuses more readily to black enamel.	Fuses easily to a bead.
Sp. gr.,	3.30 to 4.	3.427.	3.7 to 4.2.
Hardness,	5.5	5.5 to 6.	6.5 to 7.5.
Luster,	Vitreous-resinous, weak.	ditto, stronger.	ditto, brilliant.

It is true that Willemite and Garnet belong to different crystalline systems, but the ordinary form of Willemite really ap-

proaches very nearly a rhombic dodecahedron, the difference of angle only amounting to 5°, and there is no good reason for attaching a greater significance to this difference of angle between the corresponding forms of allied systems, than is attached to an equal difference between similar forms of the same system. In its peculiar constitution the new mineral is allied to Helvin, although the two species have no outward resemblance. The holohedral character of its crystallization, the very large amount of iron and zinc entering into its composition, its color, luster, hardness, and other physical as well as chemical properties, all distinguish it from Helvin and prove the mineral to be a new species. As such I take great pleasure in giving to it the name of *Danalite*, after Prof. James D. Dana, of New Haven, a name so honorably associated with American mineralogy.

Small fragments of Danalite are not unfrequently met with in the quarries at Rockport, and small grains of it, as I have already said, are quite generally disseminated through the granite ledges which form the extremity of Cape Ann. But large masses of the mineral have not been obtained for some time, the portion of the rock in which they were found having been long since quarried. The mineral was first supposed by the local collectors to be Rhodonite, and under this name good specimens of Danalite may probably be found in some of the collections of the country. Specimens of the mineral have also been found associated with green feldspar at the railroad cut near Gloucester, Mass. The mineral at this locality is more garnet-like in structure, and contains a considerable amount of alumina associated with the glucina. An analysis of a specimen from this locality gave the following results:

Silica, - - - - -	29.88
Protoxyd of iron, - - - - -	28.13
Oxyd of zinc, - - - - -	18.15
Oxyd of manganese, - - - - -	5.71
Glucina and alumina, - - - - -	14.72
Lime, - - - - -	0.88
Magnesia, - - - - -	tracea
Sulphur, - - - - -	4.32
	102.24
Oxygen equiv. to sulphur, - - - - -	2.41
	99.83

At Gloucester the Danalite is associated with fluor spar, which I have never recognized on the specimens from Rockport, although the granite, in which the mineral is imbedded, has at both localities a similar character. Danalite is also associated at both localities with two very remarkable varieties of lepidolite mica. These have also been analyzed and an account of the investigation will be given in a future paper.

Separation of Sesquioxyd of Iron from Alumina, Glucina, and most of the rare earths.—The method of Mr. H. Sainte-Claire Deville,¹ referred to in the above paper, is one of the most accurate processes of analytical chemistry, and would probably have been more generally used had it not been supposed that it required a furnace heat and involved the usual accompaniments of furnace work. The simple modification we have introduced is to substitute a tube of platinum for the tube of porcelain, and a Bunsen's blast lamp for the furnace. We use in the process the condenser of a small platinum still; but a smaller tube about 6" long and $\frac{1}{8}$ " in diameter would be better adapted to the purpose, and would serve many other useful ends in the laboratory. In addition to the tube, a small platinum nacelle would be required, as large as the tube will admit and about $1\frac{1}{2}$ " in length. With such an apparatus the method of conducting the process is as follows: The tube having been mounted horizontally on any convenient stand, one end of it, which is closed by a doubly pierced india rubber cork, is connected on one side with a small hydrogen generator and on the other with a small flask for generating hydrochloric acid gas. To the other end of the tube is fastened by an india rubber connector a small glass adapter, so curved that the end may dip under water. The mixed bases, whose total weight is known, having been placed in the nacelle in a finely pulverized condition, and the nacelle having been introduced into the tube, the heat of a single Bunsen burner is applied, while a gentle current of hydrogen is caused to flow through the apparatus. In the course of half an hour all the oxyd of iron is reduced to the metallic state. The current of hydrogen is then replaced by a much more rapid current of hydrochloric acid gas and the heat of a blast lamp applied. The reduced iron is now rapidly converted into chlorid, which, being volatilized by the heat and carried forward by the current, dissolves in the water. After a few minutes the action ceases, the heat is then withdrawn, and the current of hydrochloric acid gas being again replaced by a current of hydrogen, the apparatus is allowed to cool. The alumina, or whatever earth may be present, is left behind in the nacelle in a perfectly pure condition and can be at once weighed, while the weight of sesquioxyd of iron is known from the loss. If the product is not perfectly white the nacelle should be returned to the tube and the process repeated. The result can be controlled by also weighing the nacelle after the reduction of the iron, but it is not safe to estimate the amount of iron solely from the loss of weight at this time, since a very small error in this determination would cause an important error in the calculated amount

¹ Annales de Chimie et de Physique, Tome xxxviii.

of sesquioxyd. We give these details not as new, but because we feel assured that with the simple modification here described the process will be found far more expeditious, convenient and satisfactory, than any other process now in use. A small porcelain tube might be used instead of the tube platinum, but this cannot be recommended, as the porcelain is liable to break unless protected, and when properly protected sufficient heat can hardly be obtained without a furnace. The hydrogen gas is best obtained from a small automatic generator, and the hydrochloric acid gas may be generated in a small flask from coarse salt and sulphuric acid, which has been previously diluted with about one-third of its volume of water, and allowed to cool. This mixture when gently heated gives a constant flow of gas, which almost immediately stops when the lamp is withdrawn. Both gases should pass through a wash bottle containing strong sulphuric acid before entering the tube.