



XLVIII. Experiments on allanite, a new mineral from greenland

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P. S.—Some may wish to know, that
 [the *semitone* subminimis = $2t - T - S = 2.514561 \times \text{C}$
 = $25.165387 \times \Sigma$.
 C the *minor comma* = $2S - T = 10.0078624 \times \Sigma$
 = $.3976837 \times [$; and,
 Σ the *schisma* = $2T - t - 2S = .0999204 \times \text{C}$
 = $.03973712 \times [$.

The schisma-excessive minor *Fourth* $5 [+ 12 \text{C} + 10 \Sigma$, belongs to the scale of my Equal Temperament (mentioned in your 28th volume, p. 65; 35th volume, p. 452; and 36th volume, p. 48), and exceeds the true Isotonic Fourth or $\frac{1}{2}$ ths of the octave, by only about the $\frac{1}{168801}$ st part of a major comma: it is equal $3V + III - 44$ ths, and is tuned, or may be proved on this organ, by trying C, G D A' and *C upwards and thence *G', *D', *A' and *E' downwards, when C will be found to beat with this last note, 1.0836 times per second, if C of the tenor cliff line on the diapason stop vibrates 240 times per second, according to the true concert pitch.—J. F.

XLVIII. *Experiments on Allantite, a new Mineral from Greenland.* By THOMAS THOMSON, M.D.F.R.S. Fellow of the Imperial Chirurgo-Medical Academy of Petersburg*.

ABOUT three years ago, a Danish vessel † was brought into Leith as a prize. Among other articles, she contained a small collection of minerals, which were purchased by Thomas Allan, Esq. and Colonel Inrie. The country from which these minerals had been brought was not known for certain; but as the collection abounded in cryolite, it was conjectured, with very considerable probability, that they had been collected in Greenland.

Among the remarkable minerals in this collection, there was one, which, from its correspondence with gadolinite, as described in the different mineralogical works, particularly attracted the attention of Mr. Allan, who was confirmed in the idea of its being a variety of that mineral by the opinion of Count Bournon, added to some experiments made by Dr. Wollaston; and he was thus induced to give the description which has since been published in a preceding part of the present volume.

* From the Transactions of the Royal Society of Edinburgh.

† Der Fruhling, Captain Jacob Ketelson, captured on her passage from Iceland to Copenhagen.

About a year ago, Mr. Allan, who has greatly distinguished himself by his ardent zeal for the progress of mineralogy in all its branches, favoured me with some specimens of this curious mineral, and requested me to examine its composition,—a request which I agreed to with pleasure, because I expected to obtain from it a quantity of *yttria*, an earth which I had been long anxious to examine, but had not been able to procure a sufficient quantity of the Swedish *gadolinite* for my purpose. The object of this paper is to communicate the result of my experiments to the Royal Society,—experiments which cannot appear with such propriety any where as in their Transactions, as they already contain a paper by Mr. Allan on the mineral in question.

I. Description.

I am fortunately enabled to give a fuller and more accurate description of this mineral than that which formerly appeared, Mr. Allan having, since that time, discovered an additional quantity of it, among which he not only found fresher and better characterized fragments, but also some entire crystals. In its composition it approaches most nearly to *cerite*, but it differs from it so much in its external characters, that it must be considered as a distinct species. I have therefore taken the liberty to give it the name of *Allanite*, in honour of Mr. Allan, to whom we are in reality indebted for the discovery of its peculiar nature.

Allanite occurs massive and disseminated, in irregular masses, mixed with black mica and feldspar; also crystallized: the varieties observed are,

1. A four-sided oblique prism, measuring 117° and 63° .
2. A six-sided prism, acuminated with pyramids of four sides, set on the two adjoining opposite planes. These last are so minute as to be incapable of measurement. But, as nearly as the eye can determine, the form resembles Fig. 3, Plate VI; the prism of which has two right angles, and four measuring 135° .
3. A flat prism, with the acute angle of 63° replaced by one plane, and terminated by an acumination, having three principal facettes set on the larger lateral planes, with which the centre one measures 125° and 55° . Of this specimen, an engraving is given in the Plate, Fig. 4.

Specific gravity, according to my experiments, 3.533. The specimen appears to be nearly, though not absolutely, pure. This substance, however, is so very much mixed with mica, that no reliance can be placed on any of the trials which have been made. Count Bournon, surprised

at the low specific gravity noted by Mr. Allan, which was 3·480, broke down one of the specimens which had been sent him, in order to procure the substance in the purest state possible, and the result of four experiments was as follows: 4·001, 3·797, 3·654, 3·119.—In a subsequent experiment of Mr. Allan's, he found it 3·665. From these it appears that the substance is not in a pure state. Its colour is so entirely the same with the mica, with which it is accompanied, that it is only by mechanical attrition that they can be separated.

Colour, brownish-black.

External lustre, dull; internal, shining and resinous, slightly inclining to metallic.

Fracture, small conchoidal.

Fragments, indeterminate, sharp-edged.

Opake.

Semi-hard in a high degree. Does not scratch quartz nor feldspar, but scratches hornblende and crown-glass.

Brittle.

Easily frangible.

Powder, dark greenish-gray.

Before the blow-pipe it froths, and melts imperfectly into a brown scoria.

Gelatinizes in nitric acid. In a strong red heat it loses 3·98 per cent. of its weight.

II. *Experiments to ascertain its Composition.*

My first experiments were made, on the supposition that the mineral was a variety of gadolinite, and were pretty much in the style of those previously made on that substance by Ekeberg, Klaproth, and Vauquelin.

1. 100 grains of the mineral, previously reduced to a fine powder in an agate mortar, were digested repeatedly on a sand-bath in muriatic acid, till the liquid ceased to have any action on it. The undissolved residue was silica, mixed with some fragments of mica. When heated to redness, it weighed 33·4 grains.

2. The muriatic acid solution was evaporated almost to dryness, to get rid of the excess of acid, dissolved in a large quantity of water, mixed with a considerable excess of carbonate of ammonia, and boiled for a few minutes. By this treatment, the whole contents of the mineral were precipitated in the state of a yellowish powder, which was separated by the filter, and boiled, while still moist, in potash-ley. A small portion of it only was dissolved. The potash-ley was separated from the undissolved portion by the

the filter, and mixed with a solution of sal ammoniac, by means of which a white powder precipitated from it. This white matter being heated to redness, weighed 7.9 grains. It was digested in sulphuric acid, but 3.76 grains refused to dissolve. This portion possessed the properties of silica. The dissolved portion being mixed with a few drops of sulphate of potash, shot into crystals of alum. It was therefore alumina, and amounted to 4.14 grains.

3. The yellow matter which refused to dissolve in the potash-ley was mixed with nitric acid. An effervescence took place, but the liquid remained muddy, till it was exposed to heat, when a clear reddish-brown solution was effected. This solution was evaporated to dryness, and kept for a few minutes in the temperature of about 400°, to peroxidize the iron, and render it insoluble. A sufficient quantity of water was then poured on it, and digested on it for half-an-hour, on the sand-bath. The whole was then thrown upon a filter. The dark red matter which remained on the filter was drenched in oil, and heated to redness in a covered crucible. It was then black, and attracted by the magnet; but had not exactly the appearance of oxide of iron. It weighed 42.4 grains.

4. The liquid which passed through the filter had not the sweet taste which I expected, but a slightly bitter one, similar to a weak solution of nitrate of lime. Hence it was clear that no yttria was present, as there ought to have been, had the mineral contained that earth. This liquid being mixed with carbonate of ammonia, a white powder precipitated, which, after being dried in a red heat, weighed 17 grains. It dissolved in acids with effervescence: the solution was precipitated white by oxalate of ammonia, but not by pure ammonia. When dissolved in sulphuric acid, and evaporated to dryness, a light white matter remained, tasteless, and hardly soluble in water. These properties indicate carbonate of lime. Now, 17 grains of carbonate of lime are equivalent to about 9.23 grains of lime.

5. From the preceding analysis, supposing it accurate, it followed that the mineral was composed of

Silica	37.16
Lime	9.23
Alumina	4.14
Oxide of iron	42.40
Volatile matter	3.98
	<hr/>
	96.91
Loss	3.09
	<hr/>
	100.00

But

But the appearance of the supposed oxide of iron induced me to suspect that it did not consist wholly of that metal. I thought it even conceivable, that the yttria which the mineral contained might have been rendered insoluble by the application of too much heat, and might have been concealed by the iron with which it was mixed. A number of experiments, which it is needless to specify, soon convinced me that, besides iron, there was likewise another substance present, which possessed properties different from any that I had been in the habit of examining. It possessed one property at least in common with yttria; its solution in acids had a sweet taste; but few of its other properties had any resemblance to those which the chemists to whom we are indebted for our knowledge of yttria have particularized. But as I had never myself made any experiments on yttria, I was rather at a loss what conclusion to draw. From this uncertainty I was relieved by Mr. Allan, who had the goodness to give me a small fragment of gadolinite, which had been received directly from Mr. Ekeberg. From this I extracted about 10 grains of yttria; and upon comparing its properties with those of the substance in question, I found them quite different. Convinced by these experiments that the mineral contained no yttria, but that one of its constituents was a substance with which I was still unacquainted, I had recourse to the following mode of analysis, in order to obtain this substance in a pure state.

III. *Analysis of Allanite.*

1. 100 grains of the mineral, previously reduced to a fine powder, were digested in hot nitric acid till nothing more could be dissolved. The undissolved residue, which was silica, mixed with some scales of mica, weighed, after being heated to redness, 35.4 grains.

2. The nitric acid solution was transparent, and of a light-brown colour. When strongly concentrated by evaporation, to get rid of the excess of acid, and set aside in an open capsule, it concreted into a whitish solid matter, consisting chiefly of soft crystals, nearly colourless, having only a slight tinge of yellow. These crystals being left exposed to the air became gradually moist, but did not speedily deliquesce. The whole was therefore dissolved in water, and the excess of acid, which was still present, carefully neutralized with ammonia. By this treatment, the solution acquired a much deeper brown colour; but still continued transparent. Succinate of ammonia was then dropped

dropped in with caution. A copious reddish-brown precipitate fell, which being washed, dried, and heated to redness in a covered crucible, weighed 25.4 grains. It possessed all the characters of black oxide of iron. For it was attracted by the magnet, completely soluble in muriatic acid, and the solution was not precipitated by oxalate of ammonia.

3. The liquid being still of a brown colour, I conceived it not to be completely free from iron. On this account, an additional quantity of succinate of ammonia was added. A new precipitate fell; but instead of the dark reddish-brown colour which characterizes succinate of iron, it had a beautiful flesh-red colour, which it retained after being dried in the open air. When heated to redness in a covered crucible, it became black, and had some resemblance to gunpowder. It weighed 7.2 grains.

4. This substance attracted my peculiar attention, in consequence of its appearance. I found it to possess the following characters:

a. It was tasteless, and not in the least attracted by the magnet, except a few atoms, which were easily separated from the rest.

b. It was insoluble in water, and not sensibly acted on when boiled in sulphuric, nitric, muriatic, or nitro-muriatic acid.

c. Before the blow-pipe it melted with borax and microcosmic salt, and formed with both a colourless bead. With carbonate of soda it formed a dark-red opaque bead.

d. When heated to redness with potash, and digested in water, snuff-coloured flocks remained undissolved, which gradually subsided to the bottom. The liquid being separated, and examined, was found to contain nothing but potash. When muriatic acid was poured upon the snuff-coloured flocks, a slight effervescence took place, and when heat was applied, the whole dissolved. The solution was transparent, and of a yellow colour, with a slight tint of green. When evaporated to dryness, to get rid of the excess of acid, a beautiful yellow matter gradually separated. Water boiled upon this matter dissolved the whole. The taste of the solution was astringent, with a slight metallic flavour, by no means unpleasant, and no sweetness was perceptible.

e. A portion of the black powder being exposed to a red heat for an hour, in an open crucible, became reddish-brown, and lost somewhat of its weight. In this altered state, it was soluble by means of heat, though with difficulty,

culty, both in nitric and sulphuric acids. The solutions had a reddish-brown colour, a slight metallic astringent taste, but no sweetness.

f. The solution of this matter in nitric and muriatic acid, when examined by re-agents, exhibited the following phenomena :

- (1.) With prussiate of potash, it threw down a white precipitate in flocks. It soon subsided; readily dissolved in nitric acid; the solution was given.
- (2.) Prussiate of mercury. A light yellow precipitate, soluble in nitric acid.
- (3.) Infusion of nut galls. No change.
- (4.) Gallic acid. No change.
- (5.) Oxalate of ammonia. No change.
- (6.) Tartrate of potash. No change.
- (7.) Phosphate of soda. No change.
- (8.) Hydro-sulphuret of ammonia. Copious black flocks, Liquor remains transparent.
- (9.) Arseniate of potash. A white precipitate.
- (10.) Potash..... } Copious yellow-co-
- (11.) Carbonate of soda. } loured flocks; readily
- (12.) Carbonate of ammonia. } dissolved in nitric acid,
- (13.) Succinate of ammonia. A white precipitate.
- (14.) Benzoate of potash. A white precipitate.
- (15.) A plate of zinc being put into the solution in muriatic acid, became black, and threw down a black powder, which was insoluble in sulphuric, nitric, muriatic, nitro-muriatic, acetic, and phosphoric acids, in every temperature, whether these acids were concentrated or diluted.
- (16.) A plate of tin put into the nitric solution, occasioned no change.
- (17.) A portion being inclosed in a charcoal crucible, and exposed for an hour to the heat of a forge, was not reduced to a metallic button, nor could any trace of it be detected when the crucible was examined.

These properties were all that the small quantity of the matter in my possession enabled me to ascertain. They unequivocally point out a metallic oxide. Upon comparing them with the properties of all the metallic oxides known, none will be found with which this matter exactly agrees. Cerium is the metal the oxides of which approach the nearest. The colour is nearly the same, and both are precipitated white by prussiate of potash, succinate of ammonia, and benzoate of potash. But, in other respects, the two substances differ entirely. Oxide of cerium is precipitated

ecipitated white by oxalate of ammonia and tartrate of potash; our oxide is not precipitated at all: oxide of cerium is precipitated white by hydro-sulphuret of ammonia; while our oxide is precipitated black: oxide of cerium is not precipitated by zinc, while our oxide is thrown down black. There are other differences between the two, but those which I have just mentioned are the most striking.

These properties induced me to consider the substance which I had obtained from the Greenland mineral as the oxide of a metal hitherto unknown; and I proposed to distinguish it by the name of *junonium*.

In the experiments above detailed, I had expended almost all the oxide of *junonium* which I had in my possession, taking it for granted that I could easily procure more of it from the Greenland mineral. But, soon after, I was informed by Dr. Wollaston, to whom I had sent a specimen of the mineral, that he had not been able to obtain any of my supposed *junonium* in his trials. This induced me to repeat the analysis no less than three times, and in neither case was I able to procure any more of the substance which I have described above. Thus it has been out of my power to verify the preceding details, and to put the existence of a new metal in the mineral beyond doubt. At the same time, I may be allowed to say, that the above experiments were made with every possible attention on my part, and most of them were repeated at least a dozen times. I have no doubt myself of their accuracy; but think that the existence of a new metal can hardly be admitted, without stronger proofs than the solitary analysis which I have performed.

5. The liquid, thus freed from iron and *junonium*, was super-saturated with pure ammonia. A grayish-white gelatinous matter precipitated. It was separated by the filter, and became gradually darker-coloured when drying. This matter, after being exposed to a red heat, weighed about 38 grains. When boiled in potash-ley, 4.1 grains were dissolved, of a substance which, separated in the usual way, exhibited the properties of alumina.

6. The remaining 33.9 grains were again dissolved in muriatic acid, and precipitated by pure ammonia. The precipitate was separated by the filter, and allowed to dry spontaneously in the open air. It assumed an appearance very much resembling gum-arabic, being semi-transparent, and of a brown colour. When dried upon the sand-bath, it became very dark-brown, broke with a vitreous fracture, and still retained a small degree of transparency. It was tasteless,
felt

felt gritty between the teeth, and was easily reduced to powder. It effervesced in sulphuric, nitric, muriatic, and acetic acids, and a solution of it was effected in each by means of heat, though not without considerable difficulty. The solutions had an austere and slightly sweetish taste. When examined by re-agents, they exhibited the following properties:

- (1.) Prussiate of potash. A white precipitate.
- (2.) Oxalate of ammonia. A white precipitate.
- (3.) Tartrate of potash. A white precipitate.
- (4.) Hydro-sulphuret of potash. A white precipitate.
- (5.) Phosphate of soda. A white precipitate.
- (6.) Arseniate of potash. A white precipitate.
- (7.) Potash and its carbonate. A white precipitate.
- (8.) Carbonate of ammonia. A white precipitate.
- (9.) Ammonia. A white gelatinous precipitate.
- (10.) A plate of zinc. No change.

These properties indicated oxide of cerium. I was therefore disposed to consider the substance which I had obtained as oxide of cerium. But on perusing the accounts of that substance, given by the celebrated chemists to whose labours we are indebted for our knowledge of it, there were several circumstances of ambiguity which occurred. My powder was dissolved in acids with much greater difficulty than appeared to be the case with oxide of cerium. The colour of my oxide, when obtained from oxalate by exposing it to a red heat, was much lighter, and more inclined to yellow, than the oxide of cerium.

In this uncertainty, Dr. Wollaston, to whom I communicated my difficulties, offered to send me down a specimen of the mineral called *cerite*, that I might extract from it real oxide of cerium, and compare my oxide with it. This offer I thankfully accepted*; and upon comparing the properties of my oxide with those of oxide of cerium extracted from *cerite*, I was fully satisfied that they were identical. The more difficult solubility of mine was owing to the method I had employed to procure it, and to

* The specimen of *cerite* which I analysed, was so much mixed with actinolite, that the statement of the results which I obtained cannot be of much importance. The specific gravity of the specimen was 4.149. I found it composed as follows:

A white powder, left by muriatic acid, and presumed to be silica	47.3
Red oxide of cerium	44.
Iron	4.
Volatile matter	5.
Loss	1.7

100.0
the

the strong heat to which I had subjected it: whereas the oxide of cerium from cerite had been examined in the state of carbonate.

7. In the many experiments made upon this powder, and upon oxide of cerium from cerite, I repeated every thing that had been established by Berzelius and Hisinger, Klaproth and Vauquelin, and had an opportunity of observing many particulars which they have not noticed. It may be worth while, therefore, without repeating the details of these chemists, to mention a few circumstances, which will be found useful in examining this hitherto scarce oxide.

a. The precipitate occasioned by oxalate of ammonia is at first in white flocks, not unlike that of muriate of silver, but it soon assumes a pulverulent form. It dissolves readily in nitric acid, without the assistance of heat. The same remark applies to the precipitate thrown down by tartrate of potash. But tartrate of cerium is much more soluble in acids than the oxalate.

b. The solution of cerium in acetic acid is precipitated gray by infusion of nut-galls. Cerium is precipitated likewise by the same re-agent from other acids, provided the solution contain no excess of acid. This fact was first observed by Dr. Wollaston, who communicated it to me last summer. I immediately repeated his experiments with success.

c. Cerium is not precipitated from its solution in acids by a plate of zinc. In some cases, indeed, I have obtained a yellowish-red powder, which was thrown down very slowly. But it proved, on examination, to consist almost entirely of red oxide of iron, and of course only appeared when the solution of cerium was contaminated with iron.

d. The solutions of cerium in acids have an astringent taste, with a perceptible sweetness, which however is different from the sweetness which some of the solutions of iron in acids possess.

e. The muriate and sulphate of cerium readily crystallize; but I could not succeed in obtaining crystals of nitrate of cerium.

f. The best way of obtaining pure oxide of cerium is to precipitate the solution by oxalate of ammonia, wash the precipitate well, and expose it to a red heat. The powder obtained by this process is always red; but it varies very much in its shade, and its beauty, according to circumstances. This powder always contains carbonic acid.

g. I consider the following as the essential characters of cerium,

cerium,

cerium. The solution has a sweet astringent taste: it is precipitated white by prussiate of potash, oxalate of ammonia, tartrate of potash, carbonate of potash, carbonate of ammonia, succinate of ammonia, benzoate of potash, and hydro-sulphuret of ammonia: the precipitates are redissolved by nitric or muriatic acids: ammonia throws it down in gelatinous flocks: zinc does not precipitate it at all.

h. The white oxide of cerium, mentioned by Hisinger and Berzelius, and described by Vauquelin, did not present itself to me in any of my experiments; unless the white flocks precipitated by ammonia from the original solution be considered as white oxide. They became brown on drying, and when heated to redness were certainly converted into red oxide.

As cerium as well as iron is precipitated by succinate of ammonia, the preceding method of separating the two from each other was not unexceptionable. Accordingly, in some subsequent analyses, I separated the cerium by means of oxalate of ammonia, before I precipitated the iron. I found that the proportions obtained by the analysis above described, were so near accuracy that no material alteration is necessary.

8. The liquid, thus freed from iron, alumina, and cerium, was mixed with carbonate of soda. It precipitated a quantity of carbonate of lime, which amounted, as before, to about 17 grains, indicating 9.2 grains of lime.

From the preceding analysis, which was repeated no less than three times, a different method being employed in each, the constituents of allanite are as follows:

Silica	35.4
Lime	9.2
Alumina	4.1
Oxide of iron	25.4
Oxide of cerium	33.9
Volatile matter	4.

112.0

I omit the 7 grains of junonium, because I only detected it in one specimen of allanite. The excess of weight in the preceding numbers is to be ascribed chiefly to the carbonic acid combined with the oxide of cerium, from which it was not completely freed by a red heat. I have reason to believe, too, that the proportion of iron is not quite so much as 25.5 grains. For, in another analysis, I obtained only 18 grains, and in a third 20 grains. Some of the cerium was perhaps precipitated along with it in the preceding analysis, and thus its weight was apparently increased.

XLIX. *On*