

*On the identity of Guarinite and Hiortdahlite.*

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*With a note on the chemical composition of Guarinite.*

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*Literature.*

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**A**LTHOUGH more than fifty years have passed since Guiscardi published the first description of guarinite, the true nature of this mineral has never yet been ascertained. As regards the crystalline form, Guiscardi's first idea was that the crystals of guarinite are orthorhombic (he had himself reckoned the ratio  $a : b = 0.9896 : 1$ ; see literature, nos. 3 and 5); but, having observed crystals with a tetragonal habit, he finally decided to describe the mineral as tetragonal-hemihedral. V. v. Lang by his optical investigations appeared to place the orthorhombic symmetry of guarinite beyond doubt, and Des Cloizeaux later published a note on a combination observed by him.

Some years ago I published the results of some crystallographic and optical investigations on guarinite and also came to the conclusion that the mineral was orthorhombic, although I had observed irregularities in the angles on some crystals.

As regards the chemical composition of the mineral, Guiscardi's analysis (see under I) made it practically identical with sphene; Mauro, however, many years later, discovered that the mineral contains no titanium, and Rebuffat soon afterwards made a new analysis the result of which is given under II:—

	I.	II.	III.
	Guarinite (Guiscardi).	Guarinite (Rebuffat).	Hjortdahlite (Cleve).
SiO <sub>2</sub> ... ..	33.64 ...	34.84 ...	31.60
TiO <sub>2</sub> ... ..	33.92 ...	— ...	1.50
ZrO <sub>2</sub> ... ..	— ...	— ...	21.48
Al <sub>2</sub> O <sub>3</sub> ... ..	— ...	25.37 ...	—
Fe <sub>2</sub> O <sub>3</sub> ... ..	trace ...	1.69 ...	0.34
FeO ... ..	— ...	— ...	0.94
Y <sub>2</sub> O <sub>3</sub> (?) ... ..	— ...	1.23 ...	—
Ce <sub>2</sub> O <sub>3</sub> ... ..	— ...	3.45 ...	—
MnO ... ..	trace ...	— ...	0.96
CaO ... ..	28.01 ...	25.20 ...	32.53
MgO ... ..	— ...	— ...	0.10
Na <sub>2</sub> O ... ..	— ...	6.57 ...	6.53
K <sub>2</sub> O ... ..	— ...	1.56 ...	—
H <sub>2</sub> O ... ..	— ...	— ...	0.58
F ... ..	— ...	— ...	5.83
	95.57	99.91	102.39
		O for F <sub>2</sub>	2.43
			99.96

The view of Guiscardi that guarinite is an isomeric modification of the compound—CaTiSiO<sub>5</sub>—already known as titanite, had therefore to be given up. I had at my disposal (in 1902) only a very small amount of material and could establish only the absence of titanium and the presence of sodium. Krenner had remarked on the similarity of the crystalline form of guarinite and pseudobrookite; I, however, pointed out a closer resemblance between the mineral and danburite and was of the opinion that it should be placed in the danburite group. This view was adopted by Brögger.

My doubts as to the true nature of guarinite still remained, and I often sought on the ground of the above data to refer it to a known silicate, but without success. It was only at the end of 1906 that I discovered the clue to the problem. In the course of an investigation on the sanidinite of Monte Somma I found some small crystals which, by their crystallographic characters, could only be referred to hiortdahlite; and there was no difficulty in placing beyond doubt the identity of these small crystals with actual guarinite crystals in the mineralogical collection of the University of Naples. A comparison of the angles of guarinite and hiortdahlite then showed that these minerals can be regarded as crystallographically identical or very similar. If we take the forms  $\{0kl\}$ <sup>1</sup> observed by Guiscardi and me as  $\{h0l\}$ , we obtain the following table of comparison of angles for forms which have been observed on both minerals:—

Guarinite. <sup>2</sup>		Hiortdahlite. <sup>3</sup>
(100):(110) = 44° 47½'	...	{(100):(110) = 44° 54' {(100):(110) = 44 59½
(100):(210) = 26 23¾	...	{(100):(210) = 26 30 {(100):(210) = 26 32½
(100):(120) = 63 16	...	{(100):(120) = 63 18½ {(100):(120) = 63 25
(100):(101) = 69 41½	...	(100):(101) = 70 4

As seen in the table, the correspondence in angles is very satisfactory. There is also a similar correspondence in the colour, hardness, and specific gravity of the two minerals. In the chemical characters also guarinite and hiortdahlite are very similar: both fuse fairly easily before the blowpipe and are decomposed by acids. Finally, by comparing the analyses of guarinite with that of hiortdahlite which we owe to Cleve (see under III above) we remark at once, especially in the case of the analysis of Rebuffat, a very striking correspondence, if we consider that

<sup>1</sup> That this is so will be proved later. Guiscardi took the terminal form observed by him as  $\{0kl\}$  without optical examination; and the crystal on which I measured  $\{011\}$  was not transparent, and was therefore unsuitable for optical determination. In the course of the new investigation of guarinite, I had sections cut from the crystals which still remained in my possession, and so was able to convince myself easily that the terminal form is to be regarded as  $\{101\}$ . In fig. 3 of my paper of 1902 the axes  $a$  and  $b$  should be interchanged.

<sup>2</sup> According to my measurements.

<sup>3</sup> According to Brögger, Zeits. Kryst. Min., 1890, vol. xvi, p. 369.

the precipitate produced by ammonia should be referred, not really to titanium or aluminium as Guiscardi and Rebuffat respectively believed, but to zirconium. This idea is supported by the fact that Guiscardi did not test for zirconium at all and Rebuffat only did so on a small, separate part of the precipitate obtained by ammonia, and only by means of the turmeric-paper reaction. This view of the composition I was soon able to confirm, as I was fortunate enough to obtain proof by means of Ruer's microchemical test that zirconium is an essential constituent of guarinite. I then undertook a new and complete crystallographic and optical investigation of guarinite in order to determine whether the similarities between guarinite and hiortdahlite are sufficiently close for their specific identity.

In the present paper I content myself with giving only such data as will suffice in order to place the identity of the two minerals beyond doubt, leaving for a later publication a more detailed monograph on the properties, occurrence, &c., of the Vesuvian mineral.

The new investigation was rendered difficult by the fact that guarinite crystals are not only rare but almost always, unsuitable for exact measurements. Crystals provided with terminal faces which allow of a fairly exact determination are a rarity; the majority of the guarinite crystals consist of tufts with very complicated structure and imperfect parallel intergrowth or sheaf-like grouping. The faces of the zone  $\{001\}$ , which is the one most perfectly developed, are very often striated or notched or so small that they give broad or nearly unrecognizable images. It must also be mentioned that the guarinite crystals are generally cracked or bent and re-cemented again, in the same way as, according to Brögger, are crystals of hiortdahlite. In spite of all these difficulties, some crystals were found which were suitable for exact investigation.

As regards the crystallographic orientation, I have chosen as  $\{100\}$  that one of the two pinacoids approximately at right angles in the principal zone of guarinite which shows practically no pleochroism under the microscope. We see then that the guarinite crystals are very generally tabular to  $\{100\}$ ; only rarely does  $\{010\}$  predominate over  $\{100\}$ . Moreover, the crystals with pseudo-tetragonal habit, on which  $\{100\}$  and  $\{010\}$  are approximately of equal size, are very rare and consist mostly of the union of numerous crystals tabular to  $\{100\}$ . In the zone  $[001]$  I have observed, besides the two pinacoids  $a \{100\}$  and  $b \{010\}$ , also the forms  $m \{110\}$ ,  $M \{1\bar{1}0\}$ ,  $l \{210\}$ ,  $h \{2\bar{1}0\}$ ,  $g \{120\}$  and  $k \{1\bar{2}0\}$ . On many crystals each pinacoid  $\{hk0\}$  is associated with the corresponding  $\{h\bar{k}0\}$ , and then the zone  $[001]$  is pseudo-ortho-

rhombic; but generally this is not the case, and the zone [001] has clearly a triclinic development. When exact measurements are possible, differences are always observed in the angles which should be equal if guarinite were orthorhombic; in my work of 1902 I mentioned a crystal on which I measured

$$(010):(110) = 45^\circ 2'; (010):(\bar{1}\bar{1}0) = 45^\circ 32'.$$

At that time I explained this difference in angles as due to the geometrical variations so common in the minerals (sanidine, idocrase, &c.) of the volcanic bombs; but the new measurements, which have shown the constancy of the phenomenon, as well as the optical data, no longer allow such an explanation to hold good; they show, in fact, that guarinite is not to be regarded as orthorhombic with angular variations, but as really triclinic. Thus, on four crystals, measurement of the angle (100):(0 $\bar{1}$ 0) gave me  $90^\circ 7'$ ,  $90^\circ 2'$ ,  $90^\circ 7'$ ,  $90^\circ 6'$ ,  $90^\circ 5'$ : for hiortdahlite Brögger found  $90^\circ 5\frac{1}{2}'$ . The other angles of the zone [001] admitted of somewhat less exact measurement, still they agree quite well with the values which Brögger has calculated for hiortdahlite.

Guarinite (measured).	Hiortdahlite (Brögger).
(100):(110) = $44^\circ 52'$	... $44^\circ 54'$
(100):(2 $\bar{1}$ 0) = 26 36	... 26 $32\frac{1}{2}$
(010):(120) = 26 28	... 26 36

But more important than the angles of the zone [001] are those of the terminal forms, which gave me the clue to the identity of the minerals. Guarinite crystals with well-developed end-faces are extremely rare: the crystals are usually terminated quite irregularly or with a dull, rough, completely unmeasurable basal plane. The first crystal on which I was able to make approximate but satisfactory measurements of the terminal forms is the crystal represented in fig. 1. It is very small, and is tabular parallel to {100}: in the zone [001] were, besides  $a$  {100}, also the forms  $l$  {210} very small,  $m$  {110} somewhat larger,  $M$  { $\bar{1}\bar{1}$ 0},  $h$  {2 $\bar{1}$ 0}, and  $b$  {010}: a terminal face was also present. The crystal had a decidedly triclinic aspect, and was very similar to fig. 1, plate XVII of Brögger's paper, so that I supposed that the crystal must be hiortdahlite. In fact the measurements showed that the observed terminal face of the guarinite crystal exactly corresponds to the form  $e$  { $\bar{1}\bar{1}$ 1} of hiortdahlite.<sup>1</sup>

<sup>1</sup> Throughout this paper I refer always to the first mode of orientation of hiortdahlite adopted by Brögger.

Guarinite (measured).	Hiortdahlite (Brögger).
$(100):(1\bar{1}1) = 71^\circ 48' \dots$	$71^\circ 12'$
$(0\bar{1}0):(1\bar{1}1) = 70^\circ \text{ about}$	$71 \ 13$

The crystal represented in fig. 2 gave better measurements, the forms corresponding exactly to those of  $p$   $\{111\}$ ,  $e$   $\{1\bar{1}1\}$ ,  $r$   $\{\bar{1}11\}$ ,  $q$   $\{\bar{1}\bar{1}1\}$  of hiortdahlite, as the following measurements show.

Guarinite (measured).	Hiortdahlite (Brögger).
$(100):(11\bar{1}) = 72^\circ 27' \dots$	$72^\circ 12'$
$(100):(1\bar{1}1) = 72^\circ \text{ about}$	$71 \ 12$
$(0\bar{1}0):(11\bar{1}) = 71 \ 39 \dots$	$71 \ 10\frac{1}{2}$
$(010):(111) = 72 \ 23 \dots$	$72 \ 13\frac{1}{2}$

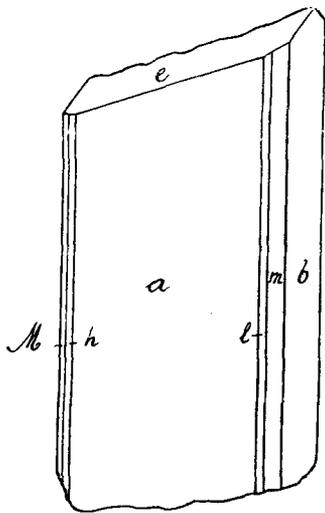


Fig. 1.

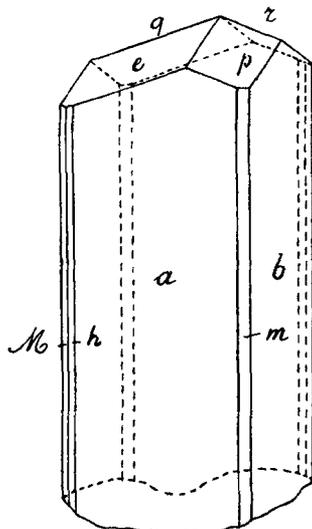


Fig. 2.

## Crystals of Guarinite.

The crystal shown in fig. 3 is very interesting: in the zone  $[001]$  it has a very pronounced triclinic habit on account of the complete failure of  $\{2\bar{1}0\}$  and  $\{110\}$ . The terminal forms are  $t$   $\{201\}$ , which is the largest face,  $v$   $\{101\}$ , and  $d$   $\{102\}$ : only  $\{201\}$  allowed of good measurement. I found:

Guarinite (measured).	Hiortdahlite (Brögger).
$(100):(201) = 53^\circ 40'$	$54^\circ 28'$
$(100):(101) = 70^\circ \text{ about}$	$70 \ 4$
$(100):(102) = 80 \ ,,$	$79 \ 26$
$(010):(201) = 90 \ 54$	$90 \ 27$

This crystal is like the figures of Guiscardi, which represent tabular guarinite crystals; the tabular face, however, is certainly  $\{100\}$ , as I have shown optically, so that the terminal forms are  $\{h0l\}$  and not  $\{0kl\}$ . It must be mentioned also that I have observed on some crystals  $\{0kl\}$  or forms vicinal to these, though always so imperfect as to allow only of rough measurements. It must not be forgotten also that the angles  $(010):(011) = 71^\circ 13'$  and  $(0\bar{1}0):(0\bar{1}1) = 70^\circ 5\frac{1}{2}'$  are not very different from the angles  $(010):(111) = 72^\circ 13\frac{1}{2}'$  and  $(0\bar{1}0):(\bar{1}\bar{1}1) = 71^\circ 13\frac{1}{2}'$ , so that if the faces of  $\{100\}$  are not measurable, it is not possible to distinguish with certainty the forms  $\{011\}$  and  $\{0\bar{1}1\}$  from  $\{111\}$  and  $\{\bar{1}\bar{1}1\}$ . On the crystal which is represented in fig. 4 of my work of 1902, according to the new measurements I have made, there appear to be present also the forms  $\{011\}$  and  $\{0\bar{1}1\}$ ; these were, however, scarcely measurable.

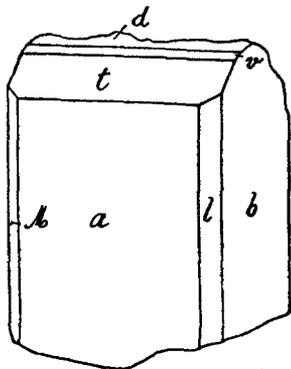


Fig. 3.

Crystals of Guarinite.

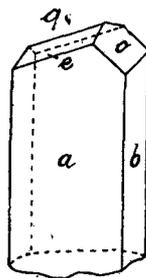


Fig. 4.

Lastly, the crystal represented in fig. 4 presents some remarkable features. Besides the forms represented, there are also present traces of pinacoids  $\{hk0\}$  and  $\{h\bar{k}0\}$ . The terminal forms are  $e\{1\bar{1}1\}$  and  $q\{\bar{1}\bar{1}1\}$ , both small, and one good face with the symbol  $o\{273\}$ , which gave good measurements, not only  $\{273\}$  but also  $\{100\}$  and  $\{010\}$  giving good reflections.

Guarinite (measured).				Hiortdahlite (Brögger).
$(100):(273) = 79^\circ 10'$	...	...	...	$79^\circ 9'$
$(010):(273) = 51 41$	...	...	...	$51 50\frac{1}{2}$
$(0\bar{1}0):(\bar{1}\bar{1}1) = 71 30$	about	...	...	$71 13$
$(100):(\bar{1}\bar{1}1) = 71 30$	„	...	...	$71 12$
$(\bar{1}00):(\bar{1}\bar{1}1) = 72$	„	...	...	$72 12$

Of the terminal forms mentioned up to the present  $p$  {111},  $e$  {111},  $r$  {111},  $q$  {111}, are new for guarinite; they are, however, characteristic faces on hiortdahlite<sup>1</sup>;  $t$  {201} is new for hiortdahlite, and  $d$  {102} and  $o$  {273} have not hitherto been observed either on hiortdahlite or on guarinite. Without giving further data from my measurements of other guarinite crystals, it appears to me that the above are quite sufficient in order to prove the crystallographic identity of hiortdahlite and guarinite. The small differences which were observed in some angles between the measured and calculated values are of little importance and for the most part are to be ascribed to the uncertainty of the measurements: the more exact measurements are in close agreement with the calculated. It is quite possible also that guarinite possesses geometric constants which vary a little from those of hiortdahlite; for it has been established that the crystals of a mineral which occur in volcanic bombs generally give axial ratios varying somewhat from those of crystals of the same mineral coming from older eruptive rocks.

We now come to the optical characters of guarinite<sup>2</sup>. I had already stated in 1902 that guarinite crystals do not generally extinguish uniformly between crossed nicols. At that time I sought to explain the phenomenon by the complicated structure of {100} and {010}, which are often divided into numerous facets. My new observations have, however, proved this view to be quite untenable, because the crystals with quite smooth {100} and {010} show exactly the same behaviour. Thin slices cut from good crystals parallel to {100} and {010} have, on the other hand, shown clearly that the failure to give a definite and regular extinction depends on the fine polysynthetic twinning of the guarinite crystals. Thin slices in fact afford clear proof, not only of the triclinic symmetry of guarinite, but also of the identity of guarinite and hiortdahlite.

<sup>1</sup> {111} is not given by Brögger in the list of forms, but occurs in the table of angles.

<sup>2</sup> These optical characters of the guarinite crystals, which I have repeatedly observed, and of the correctness of which there is no doubt, vary from those given in my work of 1902. My determinations were made at that time on a very beautiful crystal which came from the greyish-violet trachyte with melanite, and unfortunately was afterwards lost. That crystal was, however, not guarinite, but rather danburite, or a mineral near to danburite; this would explain the wonderful similarity which was found between the supposed guarinite and danburite. That the crystal which was examined was not guarinite is evident from the fact that it showed uniform extinction, and had the plane of the optic axes exactly parallel to {001}, with exactly perpendicular emergence of the mean lines on the forms regarded as {100} and {010}. These characters do not at all agree with those of true guarinite.

In sufficiently thin slices parallel to  $\{100\}$  one remarks between crossed nicols an apparently nearly homogeneous portion and another which consists of more or less broad lamellae in twin position to each other. The apparently almost homogeneous portions show, if a gypsum-plate or a stronger magnification be used, a very fine and complicated polysynthetic structure. The bounding lines of the polysynthetic lamellae are straight and parallel to the vertical axis. The individual lamellae extinguish right and left under an angle of  $22^{\circ}$ – $24^{\circ}$  with the vertical axis: in hiortdahlite the angle is about  $25^{\circ}$ . The polysynthetic structure of hiortdahlite crystals is often so fine that the extinction in thin sections is only imperfect and the single lamellae are not plainly recognizable: with stronger magnification and the use of the gypsum-plate, the polysynthetic structure becomes evident. Guarinite according to this behaviour must be regarded as a polysymmetric substance (in Groth's sense): according to the fineness of the polysynthetic lamellae there is a greater or less approximation to the optical behaviour of an orthorhombic crystal.

Thin slices parallel to  $\{010\}$  also showed a very pronounced polysynthetic structure. The twin-lamellae are much smaller than those which are observed in the slices parallel to  $\{100\}$ ; they also are parallel to the vertical axis. On the most distinct lamellae the extinction angle with the vertical axis amounts to  $14^{\circ}$  ( $15\frac{1}{2}^{\circ}$  on hiortdahlite according to Brögger).

All these observations agree perfectly with those which Brögger has made on hiortdahlite, and we can therefore conclude that the guarinite crystals, like the polysynthetically composed crystals of hiortdahlite, represent twins belonging to the triclinic system, according to the twin-law: twin-axis, the vertical axis; plane of composition,  $\{100\}$ . It must be remarked, however, that guarinite crystals generally show other lamellae, which are otherwise orientated than those mentioned above: they obey therefore other twin-laws: unfortunately no certain conclusions on this point could be drawn from the material at my disposal.

The acute bisectrix emerges through  $\{100\}$ , and the obtuse bisectrix through  $\{010\}$ . The acute bisectrix is the axis of least elasticity, so that guarinite is optically positive: the direction of vibration which makes the smallest angle with the axis  $c$  on the two pinacoids  $\{100\}$  and  $\{010\}$  approximates to the optic normal. The axial angle is large, the dispersion considerable, and probably  $\rho < v$ .

On  $\{100\}$  the pleochroism is scarcely observable; on  $\{010\}$ , on the other hand, it is distinct. We have  $c > b > a$ .  $c$  is orange-yellow,

wine-yellow, or very bright yellow, according to the intensity of colour of the crystal examined, and of course of the thickness of the plate. **b** is a very pale yellow, nearly colourless; **a** almost perfectly colourless, being almost equal to **b**. The correspondence with hiortdahlite is in this respect perfect.

Guarinite shows no good cleavage; thin slices parallel to {010} show numerous cracks, corresponding to the cleavage along {100} already observed in 1902. In thin slices parallel to {100} one sees occasionally straight cleavage-cracks which make with the axis *c* an angle of about 44° and correspond therefore to pinacoids {031} and {0 $\bar{3}$ 1}; generally cleavage-cracks are observed only parallel to one of these pinacoids, while according to Brögger in hiortdahlite they are parallel to both. Other rare and more obscure cleavage-cracks are fairly symmetrical to the vertical axis, with which they make an angle of about 37°; cleavage-cracks parallel to {010} are almost imperceptible.

Perfectly pure guarinite crystals, according to my determinations made by the suspension method, have a specific gravity = 3.196<sup>1</sup>: for hiortdahlite Brögger and Cleve found 3.267 and 3.235 respectively: the correspondence with guarinite is satisfactory. The value found by Guiscardi 3.487 is decidedly wrong, as was already shown in 1902.

From the preceding we can therefore conclude that guarinite and hiortdahlite possess the same crystallographic and physical characters: and since both are essentially silico-zirconates (having regard to my micro-chemical test on guarinite) of calcium and sodium, it is practically certain that the two minerals are identical. It appeared to me desirable, however, by a new and exact analysis of guarinite to establish the true quantitative composition of this mineral, more especially as we have only one analysis of hiortdahlite. To prepare sufficient material for the analysis was extremely difficult owing to the rarity of the mineral. After long and tedious picking out of crystals from numerous pieces of sanidinite, I was successful in collecting about 0.4 gram of almost perfectly pure material. The analysis was undertaken by my friend Dr. G. T. Prior, to whom it is my pleasant duty to offer my most hearty thanks.

Dr. Prior's investigations have completely established my view as to the identity of guarinite and hiortdahlite. Some of the numbers obtained by him on guarinite, especially as regards the fluorine, do not agree very well with those given by Cleve for hiortdahlite; but if we

<sup>1</sup> Prior's determination with the pycnometer gave 3.25 (see p. 257).

consider guarinite and hiortdahlite to be complicated solid solutions, we must conclude that the smaller content of fluorine and the presence of some niobic instead of titanitic acid do not suffice to justify the separation of guarinite as a variety of hiortdahlite.

*The chemical composition of Guarinite.<sup>1</sup>*

(By G. T. PRIOR.)

The material placed at my disposal for analysis consisted of small fragments of crystals. These were crushed to a coarse powder from which a little felspar was removed by suspension in methylene iodide diluted with benzene. The specific gravity of the material thus purified was 3.25, as determined by means of a 3 cc. pycnometer on 0.3601 gram.

Since previous analyses of guarinite have afforded such varying results, it seems advisable to give a somewhat detailed description of the methods adopted in the present analysis.

The finely powdered mineral was decomposed by concentrated sulphuric acid. In the course of this operation the attempt was made to collect any fluoride of silicon which might be given off, since the mineral presumably contained a large percentage of fluorine. For this purpose the material was placed in a platinum retort provided with inlet and outlet tubes of platinum, through which a current of dry carbon dioxide was passed during the decomposition. The narrow outlet tube was connected with a wider tube of platinum which dipped into a strong solution of potassium fluoride contained in a long narrow platinum crucible. Only about a milligram of potassium silico-fluoride, insoluble in alcohol, was, however, obtained in this way, a result which gave the first indication that the mineral did not contain such a high percentage of fluorine as hiortdahlite.

After the decomposition of the mineral, the solution was diluted, warmed, and filtered from the silica, which was weighed and afterwards tested for purity in the usual way by means of hydrofluoric acid. The filtrate from the silica was largely diluted and heated for many hours with renewal of water. A slight precipitate which separated was collected, and after testing for silica by hydrofluoric acid, was fused with acid sulphate of potassium. On treating with water, the whole remained as an insoluble residue, for in the filtrate no coloration was given with

<sup>1</sup> Communicated by permission of the Trustees of the British Museum.

hydrogen peroxide (absence of titanio acid) and no precipitate with ammonia. The insoluble residue consisted mainly of niobio acid, only a few needles of the tantalum double fluoride being obtained on treating the hydrofluoric acid solution with hydrogen-potassium fluoride.

After the separation of the precipitate of niobio acid from the original solution, the filtrate was evaporated to dryness and the excess of sulphuric acid was expelled. The residue dissolved to a clear solution in cold water, but on boiling a slight precipitate coloured red with iron separated. In the filtrate from this precipitate a double precipitation with ammonia was made. The precipitate obtained on boiling and the ammonia precipitate were dealt with separately but in the same way, and both were found to consist only of zirconia and oxide of iron. The method of treatment of the two precipitates was as follows. They were brought into solution in sulphuric acid, and the zirconia was separated from the iron by precipitation with sodium thiosulphate. The precipitate was converted into chloride, in the nearly neutral solution of which oxalic acid produced a bulky precipitate completely soluble in excess of the reagent ( $ZrO_2$ ). The filtrate from the thiosulphate precipitate after freeing from sulphur, oxidizing, &c., gave no precipitate with oxalic acid (absence of cerium and yttrium earths), but only contained a little iron.

The zirconia from the oxalic acid solution was tested in the following way. Converted into sulphate, it was precipitated by excess of potassium sulphate. The solution in hydrochloric acid gave on evaporation the characteristic needles of zirconium oxychloride. No alumina was obtained in the filtrate after precipitating with excess of potash. The potash precipitate was completely soluble in dilute hydrofluoric acid (absence of cerium, yttrium, &c.). The solution of the chloride, strongly acidified with sulphuric acid, gave with sodium phosphate a bulky gelatinous precipitate of zirconium phosphate.

In the filtrate from the ammonia precipitate of zirconia and oxide of iron, the lime, manganese, and alkalis were determined by the usual methods. A blank experiment was made for alkalis, using approximately the same amount of water and reagents (sulphuric acid, ammonia, &c.) as in the analysis. About a milligram of alkalis was obtained and deducted from the weight of alkalis obtained in the analysis. This main analysis was made on 0.3341 gram.

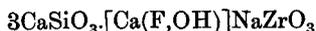
For the determination of water and fluorine only 0.0612 gram of material was available, and the numbers obtained are given for what they are worth. For the determination of water, the mineral was heated with oxide of lead. The loss of weight (corresponding with 0.81%  $H_2O$ )

only amounted to about half a milligram, and the result is uncertain, for no trace of water could be detected on heating a few grains of the pure mineral in a closed tube. For the determination of fluorine, the material mixed with oxide of lead which had served for the water determination was used. It was fused with sodium carbonate and treated by the Berzelius method as modified by Hillebrand. The slight precipitate of calcium fluoride obtained gave a distinct etching reaction on treatment with sulphuric acid. A blank experiment with approximately the same amount of reagents (oxide of lead, sodium carbonate, oxide of zinc, &c.) gave no trace of fluorine.

The result of the analysis is as follows:—

	Molecular ratios.
SiO <sub>2</sub> ...    ...    30.53    ...    ...	0.519
ZrO <sub>2</sub> ...    ...    19.70    ...    ...	0.161
Nb <sub>2</sub> O <sub>5</sub> ...    ...    1.68 <sup>1</sup> } 1.89    ...	0.007
Ta <sub>2</sub> O <sub>5</sub> ...    ...    0.21 <sup>1</sup> }	
FeO    ...    ...    1.91    ...    ...	0.026
MnO    ...    ...    1.56    ...    ...	0.022
CaO    ...    ...    35.80    ...    ...	0.639
MgO    ...    ...    0.57    ...    ...	0.014
K <sub>2</sub> O    ...    ...    0.43    ...    ...	0.005
Na <sub>2</sub> O    ...    ...    6.13    ...    ...	0.099
F    ...    ...    1.28    ...    ...	0.068
	99.80
less O for F <sub>2</sub> ...    0.54	99.26

The result agrees more closely with a formula



than with the formula  $4\text{Ca}(\text{Si}, \text{Zr})\text{O}_3 \cdot \text{Na}_2\text{ZrO}_2\text{F}_2$  suggested by Brögger for hiortdahlite. It would be rash, however, to press the claims of the above formula very strongly considering the small amount of material used in the analysis on which it is based. The weight of material used in Cleve's analysis of hiortdahlite is not stated, but was probably much greater.

<sup>1</sup> The individual numbers for Nb<sub>2</sub>O<sub>5</sub> and Ta<sub>2</sub>O<sub>5</sub> are the result of the Marignac separation, and can have little significance considering the small amount of material dealt with.