Gravimetric Microanalysis of Beryllium Silicate Rocks.*

The Determination and Separation of P_2O_5, Al, Fe, Mg, Be.

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The practical importance of the ability to carry out determinations of small amounts of silicates was recognized by F. EMICH in the initial stages of quantitative micro-analysis. In this connection his co-worker J. DONAU[1]) carried out investigations of the micro-determination of SiO_2, Fe_2O_3, Al_2O_3, CaO and MgO, using the micro balance of W. NERNST. He obtained satisfactory results but the work was discontinued.

In 1922 the work was again resumed[2]) this time using EMICH's beaker method and the micro balance of W. H. F. KUHLMANN. The simple technique of this method led to the belief that more complicated separations could be carried out. However, the work was again delayed due to difficulties encountered in the determination of aluminium.

The presentation of the problem of analysis of a beryllium phosphate silicate mineral by EMICH furnished the incentive for the present investigation. The authors believe that by employing the beaker method and by adapting the recently published macro methods for the determination[3]) and separation of aluminium

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[1]) J. DONAU, Monatsh. f. Chem., **32**, 1115 (1911).

[2]) A. BENEDETTI-PICHLER, Ztschr. f. analyt. Chem., **64**, 420 (1924).

[3]) R. BERG, Ztschr. f. analyt. Chem., **71**, 369 (1927); F. L. HAHN and K. VIEWEG, ibid. p. 122.

* Übersetzung bei den Separatabdrücken.

and beryllium, they have been able to make considerable progress toward solving the very difficult problem of analyzing silicate rocks by micro methods.

The methods for the determination of the individual elements or ions were first investigated to determine their accuracy and adaptibility to a system of separation. A tentative scheme of separation was set up as a guide. When a determination or separation proved to be impractical, it was discarded and another substituted and the scheme rearranged to permit the use of the substitute method.

The reagents used were of the purest quality obtainable and were tested qualitatively for the presence of the other ions before being used in the separation. A macro control analysis by approved methods was made of all compounds used as standards. A glass filter stick with an asbestos filtering mat was used in all filtering operations except where another type is specifically mentioned.

Determination of Phosphate.

The method used in the determination of phosphate was essentially that described by Dr. Hans Lieb in F. Pregl's book, "Die Quantitative Organische Mikroanalyse".

Certain changes were made which simplified the procedure and shortened the time of analysis. A micro beaker and filterstick were used in place of the Pregl filtering tube. The amounts of the reagents were reduced as was the volume of the solution. The most important change was in the method of drying the precipitate. Instead of using a vacuum dessicator as described by Lieb, the authors dried the precipitate in the drying tube devised by one of the authors[4]) after washing with alcohol and ether. The micro beaker and filter stick were wiped with wet flannel and then with chamois before placing in the drying tube, so that after being dried, they could be removed and weighed immediately according to Lieb.

The factor obtained by this method is slightly higher than that obtained by Lieb but is no less constant. All of the reagents

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[4]) A. Benedetti-Pichler, Mikrochemie, Pregl-Festschrift, 6 (1929).

used were prepared according to directions given by **Lieb** and v. **Lorenz**[5]). The complete procedure as well as the factors obtained by this method follow.

Procedure.

Two to five milligrams of the sample are dissolved in 1,5 cc of water in a beaker which has been previously weighed with its filter stick. 0,2 cc of the nitric acid containing sulphuric acid are added. The solution is heated to 70 degrees by placing on a water bath in which the water is just simmering. A thermometer must be placed beside the beaker so as to control the temperature accurately. It is very important that this temperature is not exceeded for a higher temperature will permit evaporation to take place and a precipitate of insoluble molybdenum oxide is formed which will spoil the determination. When the solution has reached 70 degrees it is removed, shaken and 1,5 cc of the molybdate reagent added. The latter is added drop by drop from a pipette which is held vertically over the beaker so as to have the reagent drop directly into the solution and not touch the sides of the beaker. This is to prevent the evaporation mentioned above. After standing on the water bath for 3 minutes it is again shaken and then allowed to stand for half an hour at 70 degrees. The solution is then filtered by means of the filter stick, washed twice with 2 cc of a 2% solution of ammonium nitrate, then once with 1 cc of absolute alcohol, and finally twice with 1 cc of ether. The outside of the micro beaker and the end of the filter stick are wiped with wet flannel and chamois and placed in the drying tube. The inlet[6]) of the tube is connected with a gas-washing bottle containing concentrated sulphuric acid. After 30 minutes of drying at room temperature the beaker is removed from the tube by pushing it out with the connecting tube into a clean crucible held at the end of the drying tube. The filter stick is then disconnected from the connecting tube and the crucible carried to the balance. The beaker and filter stick are placed on the pan by means of a forceps and weighed immediately.

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[5]) F. **Pregl**, Die quant. organ. Mikroanalyse (Berlin), 167 (1930).

[6]) „L“ in Fig. on p. 8, Mikrochemie, **Pregl-Festschrift** (1929).

Results

KH_2PO_4 taken	Ammonium Phospho-molybdate obtained		Per Cent P_2O_5 in precipitate
4,895 mg	76,13 mg		3,352%
4,928 mg	76,62 mg		3,355%
5,109 mg	78,81 mg		3,382%
3,124 mg	49,43 mg		3,379%
		Average	3,367%

Determination of Aluminium.

The determination of aluminium was carried out by the method described by one of the authors[7]), using 8-oxy-quinoline as the precipitating agent. Excellent results were obtained.

Results

$AlK(SO_4)_2 . 12H_2O$ taken	Oxy-quinoline compound obtained		% Al_2O_3 in alum
3,623 mg	3,498 mg		10,72%
3,720 mg	3,608 mg		10,77%
4,168 mg	4,055 mg		10,79%
		Theory	10,77%

Determination of Iron.

Iron gives a black amorphous precipitate with the 8-oxy-quinoline reagent under the same conditions as the aluminium. As the precipitate is very fine, it is very difficult to prevent some from passing through the filter. NIESSNER[8]) claims to have obtained a more coarsely crystalline precipitate by precipitating in the presence of tartaric acid, but it was found that the presence of tartaric acid caused an incomplete precipitation of the aluminium compound. For this reason NIESSNER's method was not used. It was also found that when aluminium and iron are present together in a solution, both are precipitated giving a coarser precipitate than the iron alone. The more aluminium present, the more coarsely crystalline the precipitate becomes. However, it is only when

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[7]) A. BENEDETTI-PICHLER, PREGL-FESTSCHRIFT, 9 (1929).

[8]) M. NIESSNER, Ztschr. f. analyt. Chem., **76**, 135 (1929).

the proportion of aluminium to iron is ten to one that a satisfactory precipitate is obtained which can be filtered and washed without loss of material.

If the iron is within the prescribed limits, the sum of the aluminium and iron oxyquinoline compounds is obtained. The iron can be determined volumetrically on a separate sample.

The method used for the determination of the aluminium and iron together as 8-oxy-quinoline compounds is the same as that used for the determination of aluminium alone. Some of the results obtained in mixtures follow.

Results

$AlK(SO_4)_2 . 12H_2O$ taken	$Fe(NH_4)_2(SO_4)_2$ taken	Oxy-quinoline compound obtained	Oxy-quinoline compound calculated
5,014 mg	0,256 mg	5,186 mg	5,178 mg
4,965 mg	0,154 mg	5,035 mg	5,002 mg
5,270 mg	0,247 mg	5,413 mg	5,413 mg

Determination of Beryllium.

Beryllium has always been a difficult metal to determine. The macro methods leave much to be desired and up to the present no gravimetric micro method had been developed. FISCHER[9]) has described a colorimetric-titrimetric method using chinalizarin but does not obtain very good results. When the beryllium is determined alone in a solution the average absolute error is about 2%. When determined in the presence of aluminium, the error is almost 4%.

Beryllium is precipitated macro-chemically as the hydroxide or ammonium phosphate or determined as anhydrous sulphate or volumetrically. Of these the precipitation as the hydroxide is the one most often used. The hydroxide is obtained by precipitation with ammonium hydroxide, potassium iodide and potassium iodate, methyl or ethyl amine, or ammonium nitrite and methyl alcohol. The hydroxide is then ignited to the oxide and weighed. All of these methods leave something to be desired besides having the usual disadvantages accompanying any determination which

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[9]) H. FISCHER, Ztschr. f. analyt. Chem., **73**, 54 (1928).

requires ignition, e. g. the change in weight of the vessels used and, especially in the case of BeO, the hygroscopic character of the precipitate.

Cupr[10]) describes a method for the precipitation of beryllium as ammonium beryllium phosphate and claims to have obtained a crystalline precipitate by a rather elaborate procedure. He ignites this crystalline precipitate to the pyrophosphate. L. Moser and J. Singer[11]) also describe a method for the precipitation of a crystalline precipitate of ammonium beryllium phosphate but owing to experimental difficulties it is impossible to adapt this method to micro analysis. One of the authors attempted to overcome these difficulties by precipitating in a sealed tube the ammonium beryllium phosphate, but, while a coarsely crystalline precipitate is obtained, the glass is attacked and an error is introduced.

Cupr also describes in the same paper a method for the determination of beryllium as the anhydrous beryllium sulphate and obtains good results.

After a careful study of these macro methods, it was decided that the one which presented the greatest opportunity for adaptation to micro methods was the last, i. e. the determination of the beryllium in the form of its anhydrous sulphate.

The $BeSO_4$ could be obtained in one of two ways. The beryllium solution could be purified by a careful separation and removal of all other ions present and evaporating the solution in a weighed beaker. The other way would be to precipitate the beryllium as hydroxide, filter, wash, and redissolve the precipitate in concentrated sulphuric acid and evaporate as before.

The method used in obtaining the anhydrous sulphate from the beryllium solution was similar to the one described by Cupr. The beryllium salt was dissolved in water and 0,5 cc aliquot portions were placed in a micro beaker. This beaker fits snugly into the metal container of the heating block designed by one of the authors[12]). (Fig. 1.) A gentle current of air was passed over the

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[10]) Cupr, Ztschr. f. analyt. Chem., **76**, 188 (1929).

[11]) L. Moser and J. Singer, Monatschr. f. Chem., **48**, 673 (1927).

[12]) This block can be procured from K. Schmitt, Mechaniker, Lessingstraße 25, Graz, Austria.

solution by means of a capillary tube (Fig. 2). The air serves two purposes; it aids the evaporation and keeps out the dust. A water pump must be used to supply the air. The authors had considerable trouble with a pump which ran in oil. The air supplied by this pump contained a slight amount of oil vapor and when this vapor came in contact with the hot beaker it charred and left a black residue which spoiled the analysis. Filters of cotton wool, concentrated sulphuric acid, etc. did not eliminate the oil entirely. The stream of air should not be strong enough to cause the solution to spatter. The heating block was kept at a temperature of 120 degrees until the solution was completely evaporated. Then the temperature was raised to 400 degrees where it was kept for fifteen minutes.

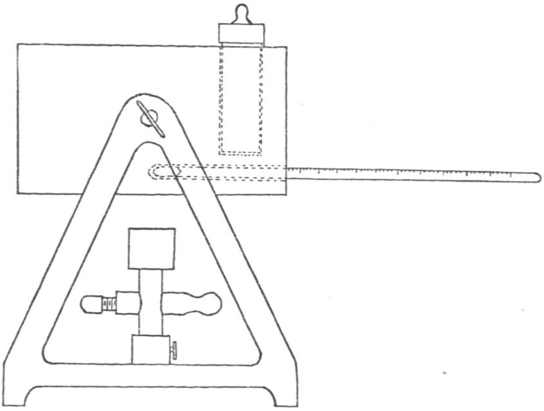

Fig. 1.

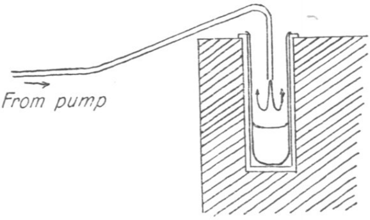

Fig. 2.

The results obtained with this method were too low. In seeking the reason for these low results, recourse was had to the literature, especially to the work of C. L. PARSONS[13]). This investigator states that $BeSO_4 . 4H_2O$ gradually loses water from 320 degrees up but from 350 degrees on loses SO_3 at the same time that it loses water. CUPR disputes this statement but the authors found that the residue from the evaporation was not completely soluble in water. This seemed to indicate that some decomposition of the anhydride had taken place. In order to prevent this decomposition, the solution was first evaporated to dryness which takes about half an hour. The beaker was then allowed to cool and then sufficient concentrated sulphuric acid was added to moisten the entire residue. The solution was then again evaporated at about 250 degrees to dryness and then heated at 320 degrees for fifteen minutes. The continual stream of air passing over the solution keeps the partial pressure of SO_3 low and so permits the evaporation of the acid below its boiling point. Some of the results obtained with this method follow.

Sample	Anhydrous Beryllium Sulphate obtained	BeO/cc
0,5 cc	1,500 mg	0,716 mg
0,5 cc	1,495 mg	0,715 mg
0,5 cc	1,493 mg	0,715 mg

These results checked with a macro analysis carried out according to L. MOSER and J. SINGER[14]) using ammonium nitrite and methyl alcohol as precipitants and weighing as BeO. Three consecutive determinations gave the following results: 0,714 mg/cc, 0,720 mg/cc, 0,716 mg/cc.

Determination of Magnesium.

Magnesium is usually determined by precipitation of the ammonium magnesium phosphate and ignition to the pyrophosphate. It is preferable, however, to use a method which does not

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[13]) C. L. PARSONS, Journ. Amer. Chem. Soc., **26**, 1433 (1904).

[14]) L. MOSER and J. SINGER, Monatsh. f. Chem., **48**, 673 (1927).

involve ignition. STREBINGER and REIF[15]) have described a micro method using 8-oxy quinoline as the precipitant. The authors of the present paper have tried this method and find that if the conditions are carefully fulfilled, very good results can be obtained. This method was not used, however, for reasons given later.

In seeking a method which could be used in the scheme of separation, the authors decided to try to adapt L. W. WINKLER's method of precipitation[16]) to micro determinations.

WINKLER weighs magnesium in the form of $NH_4MgPO_4 . 6H_2O$. This compound has the advantage over the pyrophosphate in having a larger molecular weight, the proportion of magnesium to the compound being about one to ten. WINKLER's method of precipitation gives a very coarsely crystalline precipitate of the correct composition.

The determination was carried out by the authors as follows.

A sample containing about 0,5 mg of magnesium was dissolved in 0,8 cc of water in a beaker which had been previously weighed with its filter stick. 0,2 cc of 15% ammonium chloride solution was then added. The solution was heated on a boiling water bath. It was then removed and 0,1 cc of 10% ammonia solution and 0,2 cc of 10% secondary sodium phosphate solution added. A cloudy precipitate forms almost immediately and on standing for fifteen minutes or half an hour this precipitate becomes coarsely crystalline. It is filtered, washed twice with 2 cc of 10% ammonia solution and twice with 1 cc of methyl alcohol. The alcohol is allowed to pass through the filter slowly so as to wash the precipitate thoroughly. The beaker and filter stick are then dried in the drying tube at room temperature. The inlet of the tube is connected to a tower containing crystalline calcium chloride (Fig. 3). After fifteen minutes the beaker and filterstick are removed, wiped with moist flannel and chamois and allowed to stand for about ten minutes before weighing. Some of the results obtained with this method follow.

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[15]) R. STREBINGER and W. REIF, Mikrochemie, PREGL-FESTSCHRIFT, 319 (1929).

[16]) L. W. WINKLER, Ztschr. f. angew. Chem., **31**, 211 (1918).

Results

$MgSO_4 . 7H_2O$ taken	$NH_4MgPO_4 . 6H_2O$ obtained		% Mg
3,852 mg	3,852 mg		9,90%
4,447 mg	4,449 mg		9,91%
3,296 mg	3,269 mg		9,82%
		Theory	9,86%

Separation of Beryllium and Aluminium.

One of the authors[17]) suggested that the 8-oxy-quinoline method for the micro determination of aluminium could be used for the separation of beryllium and aluminium since no insoluble compound of beryllium is formed under the conditions of the aluminium determination[18]). This idea was investigated using amounts of beryllium ranging from one to twentyfive times as much beryllium as aluminium. The results obtained were excellent and proved that this separation is satisfactory. The method of precipitation is the same as that used for the determination of aluminium alone. The results of the analyses follow.

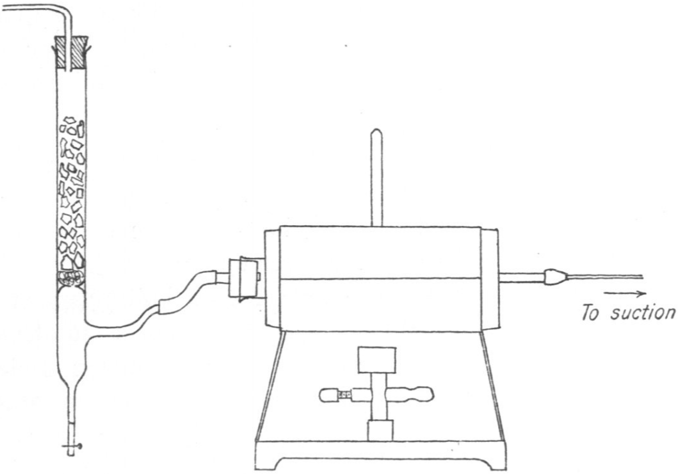

Fig. 3.

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[17]) A. **Benedetti-Pichler**, Mikrochemie, **Pregl-Festschrift**, 12 (1929).

[18]) I. M. **Kolthoff** and E. B. **Sandell**, J. A. C. S., **50**, 1900 (1928); M. **Niessner**, Ztschr. f. analyt. Chem., **76**, 135 (1929).

$AlK(SO_4)_2 . 12H_2O$ taken	$BeSO_4 . 4H_2O$ taken	Oxy-quinoline compound obtained	% Al_2O_3
4,168 mg	7,5 mg	4,055 mg	10,79%
2,439 mg	7,0 mg	2,367 mg	10,77%
3,603 mg	7,5 mg	3,317 mg	10,83%
2,143 mg	7,5 mg	2,068 mg	10,72%
1,927 mg	10,0 mg	1,878 mg	10,81%
3,189 mg	15,0 mg	3,103 mg	10,80%
1,622 mg	17,5 mg	1,572 mg	10,76%
2,412 mg	25,0 mg	2,345 mg	10,79%
2,690 mg	27,0 mg	2,602 mg	10,74%
2,022 mg	30,0 mg	1,965 mg	10,78%
1,869 mg	27,0 mg	1,823 mg	10,82%
2,402 mg	50,0 mg	2,335 mg	10,78%
1,933 mg	50,0 mg	1,879 mg	10,78%
1,962 mg	50,0 mg	1,900 mg	10,74%

Separation of Beryllium and Magnesium.

The authors first tried to apply the method of STREBINGER and REIF to the separation of magnesium and beryllium. It was thought that the addition of citric or tartaric acid would prevent the precipitation of beryllium on addition of the 8-oxy-quinoline and ammonium hydroxide. It was found that the beryllium did not precipitate in a solution which contained twice its equivalent weight of tartaric acid.

The precipitation was carried out exactly as described by STREBINGER and REIF only adding sufficient tartaric acid to prevent the precipitation of the beryllium.

Some of the results obtained by this method follow.

$MgSO_4 . 7H_2O$ taken	$BeSO_4 . 4H_2O$ taken	Oxy-quinoline compound obtained	% Mg
3,506 mg	0,35 mg	5,603 mg	11,15%
3,700 mg	0,35 mg	5,383 mg	10,16%
3,611 mg	0,35 mg	5,586 mg	10,79%
3,819 mg	0,35 mg	5,508 mg	10,06%
4,320 mg	0,35 mg	6,689 mg	10,78%

The high results obtained by this method are due to the fact that the precipitate of the 8-oxy-quinoline compound of magnesium was very fine when precipitated in this way and was very difficult to filter. The precipitate clogged the filter and prevented thorough washing. Drying the precipitate before washing or washing with ammonium nitrate solution did not improve the filtration.

The authors therefore turned to the separation of beryllium and magnesium by other means.

GLASSMAN[19]) describes a method for the precipitation of beryllium hydroxide with potassium iodide and potassium iodate which he claims will be unaffected by the presence of calcium or magnesium.

BLEYER and BOSHART[20]) precipitate beryllium with ammonium hydroxide in the presence of ammonium chloride and obtain good results. RUDISULE[21]) states that beryllium hydroxide is precipitated best by exactly neutralizing the solution with ammonia; any excess would introduce an error. A. KLING and A. LASSIEUR[22]) have found that it is possible to precipitate aluminium hydroxide completely without also precipitating magnesium even if no ammonia salts are present if the pH of the solution is exactly 7. It was thought that the beryllium would react in an analogous manner. It was found that the condition of exact neutrality could be most closely approached by blowing a stream of air containing ammonia over a hot solution of beryllium and magnesium[23]). The beryllium is precipitated quantitatively without precipitating any of the magnesium. The air contained only a small amount of ammonia and only a small part of this could dissolve in the hot solution, thus preventing an excess.

The complete procedure for the separation is as follows.

The beryllium and magnesium salts were dissolved in 1,5 cc of water in a micro beaker. Three drops of concentrated hydrochloric acid and a drop of methyl red solution added. The beaker was

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[19]) GLASSMAN, Ber. Deutsch. Chem. Ges., **39**, 3368 (1906).

[20]) BLEYER and BOSHART, Ztschr. f. analyt. Chem., **51**, 748 (1912).

[21]) RUDISULE, Nachweis, Bestimmung und Trennung der chemischen Elemente.

[22]) A. KLING and A. LASSIEUR, Ztschr. f. analyt. Chem., **71**, 135 (1927).

[23]) A. BENEDETTI-PICHLER, Ztschr. f. analyt. Chem., **64**, 425 (1924).

then placed on the water bath and heated. A stream of ammonia-carrying air was then passed over the solution by means of a capillary tube (Fig. 4) the end of which was about 1,5 cm above the surface of the solution. A 10% ammonia solution is used as the source of ammonia. The stream of air was just strong enough to cause a depression in the surface of the liquid and was directed to one side of the beaker. This caused a stirring of the solution and aided in the rapid precipitation of the beryllium hydroxide. In a few minutes the beryllium was completely precipitated and the solution became yellow. The air stream was then removed and the solution heated for a few minutes longer. It was then removed from the water bath and filtered by means of a porcelain filter stick. The filtrate was caught in a previously weighed beaker. The precipitate was washed twice with hot water containing ammonia[24]). The filtrate contains only the magnesium which can be determined as ammonium magnesium phosphate as described above.

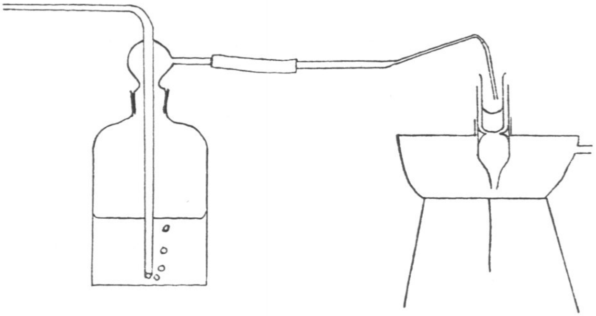

Fig. 4.

It is important to have the concentration of the magnesium not less than 0,5 mg per cc in the solution in which it is to be determined. If the solution is too dilute it can be quickly concentrated by heating on a water bath and blowing a gentle stream of air over the surface of the liquid.

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[24]) C. L. PARSONS and BARNES, Journ. Amer. Chem. Soc., **28**, 1589 (1906), recommend washing with water containing an electrolyte as they claim the beryllium hydroxide is otherwise converted to a colloidal form which would pass through the filter.

Some of the results obtained in mixtures of magnesium and beryllium follow.

$MgSO_4 \cdot 7H_2O$ taken	$BeSO_4 \cdot 4H_2O$ taken	$NH_4MgPO_4 \cdot 6H_2O$ obtained	% Mg
3,630 mg	7,0 mg	3,608 mg	9,85%
4,123 mg	7,0 mg	4,069 mg	9,78%
4,072 mg	7,0 mg	4,065 mg	9,88%
3,924 mg	70,0 mg	3,882 mg	9,80%
2,615 mg	55,0 mg	2,588 mg	9,81%
		Theory . .	9,86%

Elimination of Molybdenum.

The molybdenum remaining after the phosphate determination in the form of the excess ammonium molybdate reagent must be removed in order to prevent it from interfering with the subsequent determinations. It can be removed most easily by precipitating with hydrogen sulphide. The latter can easily be removed after the precipitation by boiling the solution. The strong nitric acid added in the phosphate determination must be removed prior to the precipitation with hydrogen sulphide as it will otherwise oxidize the latter and the coagulated mass of free sulphur may prevent proper washing of the precipitate.

The filtrate from the phosphate determination is evaporated on a water bath to a syrupy consistency in a quartz evaporating dish. One or two cc. of concentrated sulphuric acid are then added and the residue dissolved by heating. When the solution becomes clear and all of the residue has been dissolved, an equal volume of water is carefully added and the solution transferred to a large testtube (Fig. 5). The testtube is fitted with a stopper with a short glass tube which in turn is closed with a piece of rubber tubing and a pinchcock. The hydrogen sulphide is led in, first with a loosened stopper, then with a tightly closed stopper. In this way the air is displaced from the testtube and the hydrogen sulphide is admitted under pressure. After remaining attached to the source of gas for five minutes the pinchcock is closed and the tube heated in a water bath. It is then filtered and the filtrate again treated with hydrogen sulphide. This procedure is repeated until no further precipitate is obtained on adding hydrogen sulphide. The precipitate is then washed twice with hot water and the filtrate and washings evaporated to two cc.

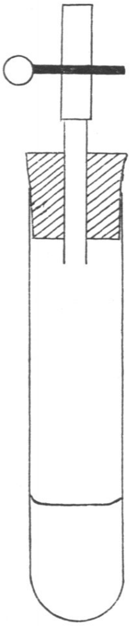

Fig. 5.

In solutions strongly acid with sulphuric acid, molybdenum will give a blue color to the solution and this may aid in showing when the precipitation is complete.

Complete Separation.

The various individual separations discussed above have been combined into a general scheme of separation. As mentioned in the introduction a tentative plan had been drawn up and the individual separations and determinations arranged as far as possible to fit into this general scheme. As yet the authors have not carried out a complete separation but are working on the problem. The following plan is suggested as the basis for such a complete separation. It is based on the individual separations which have been experimentally proven.

Mineral $+ H_2F_2 + H_2SO_4 \rightarrow SiF_4$ (driven off as gas)

$\downarrow$

$(NH_4)_2MoO_4 +$ residue $\rightarrow P_2O_5$ (precipitated)

$\downarrow$

$H_2SO_4 + H_2S +$ filtrate $\rightarrow MoS_2$ (precipitated)

$\downarrow$

8-oxy-quinoline + filtrate → Al + Fe (precipitated)

$\downarrow$

$H_2SO_4 +$ filtrate → oxy-quinoline (destroyed)

$\downarrow$

$H_2SO_4 +$ residue → Be + Mg (residue)

$\downarrow$

$NH_3 +$ residue → Be (precipitated)

$\downarrow$

$Na_2HPO_4 + NH_4OH +$ filtrate → Mg (precipitated)

The silicates are first decomposed and the silicon removed by treatment of the mineral with hydrofluoric and sulphuric acids in a platinum crucible which leaves a sulphuric acid solution of the other constituents. The phosphate determination is next carried out as described above. The molybdenum in the excess reagent is removed with hydrogen sulphide as it would interfere with the subsequent determinations. After removal of the molybdenum, the aluminium and iron can be determined with 8-oxy-quinoline. The excess of the latter reagent and the ammonium acetate is removed by treatment with concentrated sulphuric acid with the possible addition of a few drops of concentrated nitric acid. The beryllium and magnesium are now determined together as the anhydrous sulphates. On dissolving this residue the magnesium can be determined after precipitating the beryllium as the hydroxide by the method given above.

Summary.

The micro determination of P_2O_5, Al, Fe, Be, and Mg have been investigated to determine their applicability to a system of separation. In the case of magnesium and beryllium new methods of determination were worked out. A plan of separation of these ions has been suggested. The conclusions set forth in this paper are based on experimental data secured through numerous determinations. These determinations involved about one hundred macro and over three hundred micro weighings. The authors are at present continuing the work of carrying out a complete separation of all ions listed above.