

SILICATE ANALYSIS

by

Hugh Bogle

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Part I. Introduction

The composition of the fundamental ingredients of the earth's crust; that is, the different mineral species which are there found and of which its rocks are made up; was the favorite theme of the great workers in chemistry of the earlier half of the nineteenth century. Among the more important of these investigators were Berzelius and Wohler. Considering the limited facilities at their disposal in the way of laboratory equipment and reagents their work was exceptionally good.

Minerals were the first substances to receive attention, but as an outgrowth of and closely associated with the analysis of minerals came that of the more or less complex mixtures of them, the rocks. To aid the study of rocks by petrographers and geologists a host of chemists have for many decades turned out hundreds of analyses of all grades of quality and thoroughness. With the growth and extraordinary development of organic chemistry inorganic chemistry gradually fell into the background and relatively little work was done on it. In many European laboratories the course in mineral analysis, while maintained as part of the course of study, became but a preliminary to the ever-expanding study of the carbon compounds. These, multiplying rapidly and offering an easy and convenient field for original research and possible profit, proved more tempting to young chemists than the often worked over and apparently exhausted field of inorganic chemistry. For one student engaged in research on inorganic lines fifty perhaps were engaged in organic work. The instruction afforded the student in mineral analysis was confined to the ordinary separations of the commoner ingredients occurring in appreciable quantities with little regard to the supposed traces and still less attempt to find out if the tabulated list comprised all that the mineral or rock contained.

With the introduction of improved methods of examination by the

petrographer, especially as applied to thin rock sections and the use of heavy solutions, whereby, on the one hand, the qualitative mineral composition of the rock could be preliminarily ascertained with considerable certainty, and on the other, chemical examination of the more or less perfectly separated ingredients was rendered possible. Thus a great help and incentive was afforded those few chemists engaged in rock analysis. The microscope often obviated in part the necessity for tedious and time wasting qualitative analysis, and the heavy solutions, by permitting the concentration and separation of certain components, facilitated the detection of elements whose existence had long been overlooked.

Meanwhile in the general progress of chemistry improved equipment and reagents for qualitative detection and quantitative separation and estimation were gradually being devised and discovered. Many of the well established methods were tested and found to be inaccurate and they were either modified or discarded. With these improved facilities more interest was taken in analytical research and much important work was done on silicate analysis. The more important among those engaged in this work were Dr. H. S. Washington, Dr. M. Dittrich, Dr. Jannasch, and W. F. Hillebrand and his co-workers in the United States Geological Survey. Up to the beginning of the twentieth century the literature relating to silicate analysis was extensive but scattered and in no single paper up to this time was there to be found in the light of modern methods a satisfactory exposition of the procedures followed or the precautions observed, especially in the analysis of some of the rarer constituents. However in 1907 W. F. Hillebrand with the aid of his colleagues in the United States Geological Survey collected all the standard methods for the complete analysis of silicate and carbonate rocks in a pamphlet together with a thorough discussion of the principles involved and the precautions necessary to get good results.

It is to the decomposition of the original igneous rocks or their magmas that nearly all ore bodies owe their origin by one or another process of concentration. Hence it is the natural and inevitable conclusion that sufficiently careful examination of the rocks would show them to contain all or nearly all known elements, not necessarily in a given rock, but in one or another. Analytical difficulties have thus far stood in the way of experimental proof that this is so, owing to the great scarcity of many of the elements. However the following ingredients all may be found in weighable or easily discoverable quantities: Si O_2 , Ti O_2 , Zr O_2 , Al O_3 , Fe O_3 , Cr O_3 , V O_3 , Fe O , Mn O , Ni O , Co O , Mg O , Ca O , Sr O , Ba O , Zn O , Cu O , K O , Na O , Li O , H O , P O_5 , S , S O_3 , C , C O_2 , Fl , Cl , and N . To this list might be added certain others as the group of rare earths, besides Sn , Pt , Tl , Cb , B , Gl , and He . Some of these may occur at times in determinable amounts. Some other of the rarer elements may have been overlooked due to the absence of distinctive tests for their identification.

A complete and accurate analysis is of great importance to the petrographer or geologist for if many of the minor constituents are overlooked considerable error may be the result in the analysis. Then too its incompleteness may conceal points fruitful of suggestion, but, what is of greater importance they may be actually misleading.

Part II. Object of this work and its relation to previous work

The method of determining aluminum, iron, and ect. after first decomposing the silicate by hydrofluoric and sulfuric acids, silica being determined in a separate sample, is very attractive in principle and was one of the first used¹. It was abandoned however due to the presence of the constituents as sulfates rather than as chlorides as they were in other methods. For the sulfates are less soluble as is shown below³:

	Sulfate		Chloride	
	Solubility in 100 parts of cold water	Solubility in 100 parts of hot water	Solubility in 100 parts of cold water	Solubility in 100 parts of hot water
Al	36.1	89.1	40	very soluble
Fe	decomposes	slightly sol.	246	infinitely sol.
Ca	178	179	165.7	7141
Mg	26.9	73.8	167	367

This objection may be overcome by substituting perchloric acid for sulfuric since this leaves the constituents in a readily soluble form after the Si F and H F are driven off. After this the constituents may be separated and estimated by any of the standard methods. It is to determine the accuracy and practicability of this method that this work is devoted.

Part III. Methods used

The method used to decompose the silicate was as follows: If the sample is not ground finely, grind it to an impalpable powder in an agate mortar. Place the sample in a large platinum crucible or evaporating dish. The evaporating dish is preferable as the action with hydrofluoric acid is rather violent and some of the sample is apt to bubble out unless a large dish is used. Moisten the sample with water then add about ten c.c.'s of 1:1 perchloric acid and ten to fifteen c.c.'s of hydrofluoric acid, a few drops at first and after the first violent bubbling add the main portion of the hydrofluoric acid. Place on a hot plate and evaporate slowly to fumes of perchloric acid. If any gritty matter remains more hydrofluoric acid must be added and evaporated again. If none remains, continue the evaporation almost to dryness to insure driving off all the hydrofluoric acid. Add ten c.c.'s of hydrochloric acid and digest till solution is complete. Dilute with water and wash into a beaker. The solution is now complete and the various constituents may be separated and determined by any of the standard methods. Two were tried, the ammonia precipitation method

and the basic acetate precipitation method . The ammonia precipitation method is as follows: Dilute the solution after digesting with hydrochloric acid to 200 c.c.'s, heat to boiling, and add ammonium hydroxide till the solution is just alkaline to litmus paper. Boil for one minute, allow the precipitate to settle, and filter. Wash the precipitate four times with a dilute ammonium nitrate solution. Transfer the precipitate back to the beaker in which the first precipitation was made, add ten c.c.'s of hydrochloric acid, and heat till it dissolves. Dilute to 100 c.c.'s and add ammonium hydroxide till just alkaline, boil for two minutes and allow to settle. Filter and wash free from chlorides with the dilute ammonium nitrate solution. Place the paper and precipitate in a platinum crucible and burn off the carbon slowly then ignite over a Fisher burner for forty five minutes. Cool in a dessicator and weigh quickly as the aluminum oxide is highly hygroscopic. Calculate the combined percentage of aluminum and iron oxides. Place the oxides in a beaker, add ten c.c.'s of 1:1 hydrochloric acid, heat for several minutes then twenty c.c.'s of 1:1 sulfuric acid. Evaporate till only about five c.c.'s are left, cool, add thirty five c.c.'s of water and ten c.c.'s of hydrochloric acid, and heat till everything is dissolved. Add stannous chloride solution till the iron color disappears, then one drop more, dilute to 100 c.c.'s, cool, add ten c.c.'s of mercuric chloride solution, stir for two minutes then titrate with standard potassium permanganate solution. After calculating the percent ferric oxide, subtract this from the total percent of the oxides and this leaves the percent of aluminum oxide.

The filtrates from the ammonia precipitations contain the magnesium and calcium. The calcium is determined as follows :
4
Combine the filtrates from the two ammonia precipitations, heat to boiling, add ten c.c.'s of a saturated solution of ammonium oxalate slowly and with stirring. Digest on a hot plate for an hour, allow to settle, filter and wash with a one per-

cent solution of ammonium oxalate until free from chlorides. Place the paper and precipitate in a crucible, burn off the paper slowly, then ignite intensely for fifteen minutes. Cool, weigh as calcium oxide and calculate the percent. The filtrate from this determination contains the magnesium² which is determined by the following method: Add to the filtrate one fifth its volume of ammonium hydroxide and then add slowly and with constant stirring ten c.c.'s of a ten percent solution of sodium acid phosphate. Continue the stirring for several minutes to insure complete precipitation, then let stand for several hours, preferably overnight, filter, wash with a dilute solution of ammonium nitrate. Place paper and precipitate in a crucible, burn off the paper at a low temperature then ignite intensely for twenty minutes, cool and weigh as magnesium pyro phosphate, calculate as magnesium oxide.

The basic acetate method is as follows: The solution is carefully neutralized by adding a freshly prepared two molar solution of sodium carbonate. The end point can be recognized as when the precipitate thrown down by the final drop will not redissolve after stirring. Two or three grams of sodium acetate dissolved in a small amount of water are now added and then boiling water till the volume is about 400 c.c.'s. It is boiled for two minutes, allowed to settle, filtered, washed with a dilute solution of sodium acetate. The precipitate is dissolved in hydrochloric acid and reprecipitated ammonium hydroxide method described above. Any aluminum or iron that has passed through during the filtration is recovered by the following method: The filtrate from the acetate precipitation is evaporated to dryness and just enough hot water added to dissolve the residue. This is filtered and washed first with the filtrate from the ammonium precipitation which has been evaporated to a small volume then with hot water. Any precipitate recovered is burned with the other precipitate of the iron and a-

luminum.

Apparatus used: Platinum evaporating dishes were used to decompose the silicates in. Platinum crucibles were used to ignite the combined oxides of iron and aluminum and the calcium oxide in. Porcelain crucibles were used to ignite the magnesium pyro phosphate in. The beakers used were Pyrex.

Part IV. Results:

Table number 1

Sample number	1	2	3	4	5	6	7	8	Correct Analysis
% Al O + Fe O 2 3 2 3	21.02	20.51	19.21	19.46	19.10	20.32	18.97	19.23	19.14
% Fe O 2 3	1.04	.95	1.13	1.34	.91	1.21	.93	1.13	1.00
% Al O 2 3	19.98	19.56	18.08	18.12	18.19	19.11	18.04	18.10	18.14
% Ca O	.71	.65	.55	.59	.52	.60	.52	.62	.51
% Mg O	.75	.69	.61	.63	.79	.85	.88	.83	.76

Table number 2

Sample number	1	2	3	4	5	6	7	8	Correct Analysis
% Al O + Fe O 2 3 2 3	39.16	39.25	39.05	38.70	38.85	40.00	38.79	39.22	38.98
% Fe O 2 3	1.71	1.52	1.45	1.50	1.56	1.40	1.58	1.70	1.61
% Al O 2 3	37.45	37.73	37.60	37.20	37.29	38.60	37.21	37.52	37.37
% Ca O	.69	.81	.62	.87	.66	.70	.76	.58	.65
% Mg O	.53	.83	.55	.53	.62	.64	.70	.69	.51

Part V. Conclusions:

The principle of decomposing silicates by hydrofluoric acid and perchloric acid is practical. Its advantages are, that the constituents are present as perchlorates which are readily soluble in hydrochloric acid, its simplicity, and its quickness. For some purposes the percent silica is not desired so it is a waste of time to determine this and even if it is, it is

easier to determine it on a separate sample. The results obtained using this method of decomposition and the basic acetate method of separation are shown in tables one and two above. They are within the limits of allowable error thus showing that the method is accurate.

Part VI. Summary of Results

Table number 3

Weight of Sample	Weight of Al_2O_3	Weight of Fe_2O_3	% Al_2O_3 % Fe_2O_3	C.C. of K Mn O ₄ for Fe_2O_3	% Fe_2O_3	% Al_2O_3	Weight Ca O	% Ca O	Weight of $\text{Mg}_2\text{P}_2\text{O}_7$	% of Mg O
.5622	.1182	.1182	21.02	.95	1.04	19.98	.0040	.71	.0118	.75
.5208	.1066	.1066	20.51	.80	.95	19.56	.0034	.65	.0100	.69
.5758	.1108	.1108	19.21	1.1	1.13	18.08	.0032	.55	.0098	.61
.5738	.1116	.1116	19.46	1.3	1.34	18.12	.0034	.59	.0102	.63
.5300	.1012	.1012	19.10	.8	.91	18.19	.0028	.52	.0116	.79
.5254	.1068	.1068	20.32	1.05	1.21	19.11	.0032	.60	.0124	.85
.5236	.0996	.0996	18.97	.8	.93	18.04	.0029	.52	.0128	.88
.5286	.1018	.1018	19.23	1.00	1.13	18.10	.0033	.62	.0122	.83
Mean %			19.72		1.08	18.64		.59		.75
Correct %			19.14		1.00	18.14		.51		.76

Table number 4

Weight of Sample	Weight of Al_2O_3 Fe_2O_3	% Al_2O_3 Fe_2O_3	C.C. of K Mn O_4 for Fe_2O_3	% Fe_2O_3	% Al_2O_3	Weight of Ca O	% Ca O	Weight of $\text{Mg}_2\text{P}_2\text{O}_7$	% of Mg O
.5240	.2052	39.16	1.5	1.71	37.49	.0036	.69	.0080	.53
.5660	.2121	39.25	1.4	1.52	37.73	.0046	.81	.0130	.83
.5086	.1986	39.05	1.2	1.45	37.60	.0032	.62	.0078	.55
.5052	.1954	38.70	1.2	1.50	37.20	.0044	.87	.0074	.53
.5072	.1970	38.85	1.3	1.56	37.29	.0034	.66	.0088	.62
.5800	.2320	40.00	1.3	1.40	38.60	.0043	.70	.0104	.64
.6032	.2340	38.79	1.6	1.58	37.21	.0046	.76	.0118	.70
.5206	.2042	39.22	1.4	1.70	37.52	.0028	.53	.0100	.69
Mean %	39.12			1.55	37.57		.705		.63
Correct %	38.18			1.61	37.37		.65		.51

Bibliography

1. Hillebrand, W. F., The Analysis of Silicate and Carbonate Rocks,
United States Geological Survey Bulletin number 305.
2. Lord and Demorest, Metallurgical Analysis, McGraw Hill Book Company.
3. Hodgman and Lange, Handbook of Chemistry and Physics.
4. Treadwell and Hall, Analytical Chemistry, vol. II.

Part VII. Appendix

Sample 1

Weight tube + sample	14.1358 gm.
Weight tube	<u>13.5736 gm.</u>
Weight sample	.5622 gm.
Weight cru. + ash + Al_2O_3 + Fe_2O_3	7.6144
" " "	<u>7.4962</u>
Weight Al_2O_3 + Fe_2O_3	.1182 gm.
% Al_2O_3 + Fe_2O_3	21.02
Titre for Fe_2O_3 95 c.c. of .074 N. K Mn O_4	
% Fe_2O_3 1.04	
% Al_2O_3 19.78	
Weight cru. + ash + Ca O	7.4794
" " "	<u>7.4754</u>
Weight Ca O	.0040 gm.
% Ca O .7190	
Weight cru. + ash + $\text{Mg}_2\text{P}_2\text{O}_7$	7.4886
" " "	<u>7.4768</u>
Weight $\text{Mg}_2\text{P}_2\text{O}_7$	.0118 gm.
% Mg O .75	

Sample 2

Weight tube + sample	9.9528
" " "	<u>9.4320</u>
Weight sample	.5208 gm.
Weight cru. + ash + Fe_2O_3 + Al_2O_3	7.7218
" " "	<u>7.6142</u>
Weight Al_2O_3 + Fe_2O_3	.1066

% $\text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$	20.51
Titre for Fe_2O_3 .8 c.c. of .074 N. K Mn O_4	
% Fe_2O_3	.95
% Al_2O_3	19.56
Weight cru. + ash + Ca O	7.5252
" " "	<u>7.5218</u>
Weight Ca O	.0034 gm.
% Ca O	.65
Weight cru. + ash + $\text{Mg}_2\text{P}_2\text{O}_7$	7.3158
" " "	<u>7.3058</u>
Weight $\text{Mg}_2\text{P}_2\text{O}_7$	.0100
% Mg O	.69

Sample 3

Weight tube + sample	13.5736
" "	<u>12.9978</u>
Weight sample	.5758 gm.
Weight cru. + ash + $\text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$	7.6686
" " "	<u>7.5578</u>
Weight $\text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$	.1108 gm.
% $\text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$	19.21
Titre for Fe_2O_3 1.1 c.c. of .074 N. K Mn O_4	
% Fe_2O_3	1.13
% Al_2O_3	18.08
Weight cru. + ash + Ca O	7.1260
" " "	<u>7.1228</u>
Weight Ca O	.0032 gm.
% Ca O	.55

Weight	cru. + ash + $\text{Mg}_2\text{P}_2\text{O}_7$	7.4096
"	" "	<u>7.3998</u>

Weight	$\text{Mg}_2\text{P}_2\text{O}_7$	.0098
%	Mg O	.61

Sample 4

Weight	tube + sample	12.9978
"	" "	<u>12.4240</u>

Weight	sample	.5738
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Weight	cru. + ash + Al_2O_3 + Fe_2O_3	7.4486
"	" "	<u>7.3370</u>

Weight	Al_2O_3 + Fe_2O_3	.1116
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%	Al_2O_3 + Fe_2O_3	19.46
---	---	-------

Titre for Fe_2O_3	1.3 c.c. of .074 N. K Mn O_4
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%	Fe_2O_3	1.34
---	-------------------------	------

%	Al_2O_3	18.12
---	-------------------------	-------

Weight	cru. + ash + Ca O	7.6074
"	" "	<u>7.6040</u>

Weight	Ca O	.0034
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%	Ca O	.59
---	------	-----

Weight	cru. + ash + $\text{Mg}_2\text{P}_2\text{O}_7$	7.3992
"	" "	<u>7.3890</u>

Weight	$\text{Mg}_2\text{P}_2\text{O}_7$	.0102
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%	Mg O	.63
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Sample 5

Weight tube + sample	12.3852
" "	<u>11.8552</u>
Weight sample	.5300 gm.
Weight cru. + ash + $\text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$	7.5978
" " "	7.4966
Weight $\text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$	.1012 gm.
% $\text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$	19.10
Titre for Fe_2O_3 .8 c.c. of .074 N. K Mn O_4	
% Fe_2O_3	.91
% Al_2O_3	18.19
Weight cru. + ash + Ca O	7.4090
" " "	<u>7.4062</u>
Weight Ca O	.0028 gm.
% Ca O	.52
Weight cru. + ash + $\text{Mg}_2\text{P}_2\text{O}_7$	7.3180
" " "	<u>7.3064</u>
Weight $\text{Mg}_2\text{P}_2\text{O}_7$	.0116
% Mg O	.79

Sample 6

Weight tube + sample	11.8552
" "	<u>11.3298</u>
Weight sample	.5254
Weight cru. + ash + $\text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$	7.7214
" " "	<u>7.6146</u>
Weight $\text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$	.1068 gm.
% $\text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$	20.32

Titre for Fe_2O_3 1.05 c.c. of .074 N. K Mn O_4

% Fe_2O_3 1.21

% Al_2O_3 19.11

Weight cru. + ash + Ca O

7.4798

" " "

7.4766

Weight Ca O

.0036

% Ca O .60

Weight cru. + ash + $\text{Mg}_2\text{P}_2\text{O}_7$

7.4900

" " "

7.4776

Weight $\text{Mg}_2\text{P}_2\text{O}_7$

.0128

% Mg O .85

Sample 7

Weight tube + sample

11.3298

" "

10.8062

Weight sample

.5236

Weight cru. + ash + Al_2O_3 + Fe_2O_3

7.6578

" " "

7.5582

Weight Al_2O_3 + Fe_2O_3

.0996

% Al_2O_3 + Fe_2O_3 18.97

Titre for Fe_2O_3 .8 c.c. of .074 N. K Mn O_4

% Fe_2O_3 .93

% Al_2O_3 18.04

Weight cru. + ash + Ca O

7.3083

" " "

7.3054

Weight Ca O

.0029

% Ca O .52

Weight cru. + ash + $\text{Mg}_2\text{P}_2\text{O}_7$

7.4142

" " "

7.4014

Weight $\text{Mg}_2\text{P}_2\text{O}_7$

.128

% Mg O .88

Sample 8

Weight tube + sample	10.8026
" "	<u>10.2740</u>
Weight sample	.5286
Weight cru. + ash + Al_2O_3 + Fe_2O_3	7.4388
" " "	<u>7.3370</u>
Weight Al_2O_3 + Fe_2O_3	.1018
% Al_2O_3 + Fe_2O_3	19.23
Titre for Fe_2O_3 1.0 c.c. of .074 N. K Mn O_4	
% Fe_2O_3	1.13
% Al_2O_3	18.10
Weight cru. + ash + Ca O	7.5259
" " "	<u>7.5226</u>
Weight Ca O	.0033
% Ca O	.62
Weight cru. + ash + $\text{Mg}_2\text{P}_2\text{O}_7$	7.4116
" " "	<u>7.3994</u>
Weight $\text{Mg}_2\text{P}_2\text{O}_7$	.0122
% Mg O	.83

Sample 1

Weight tube + sample	11.8374
" "	<u>11.3134</u>
Weight sample	.5240
Weight cru. + ash + Al_2O_3 + Fe_2O_3	7.7118
" " "	<u>7.5066</u>
Weight Al_2O_3 + Fe_2O_3	.2052
% Al_2O_3 + Fe_2O_3	39.16

Titre for Fe_2O_3 1.5 c.c. of .074 N. K Mn O_4

% Fe_2O_3 1.71

% Al_2O_3 37.45

Weight cru. + ash + Ca O 7.5266

" " " 7.5230

Weight Ca O .0036

% Ca O .69

Weight cru. + ash + $\text{Mg}_2\text{P}_2\text{O}_7$ 7.6008

" " " 7.5928

Weight $\text{Mg}_2\text{P}_2\text{O}_7$.0080

% Mg O .53

Sample 2

Weight tube + sample 11.3134

" " 10.7474

Weight sample .5660 gm.

Weight cru. + ash + Al_2O_3 + Fe_2O_3 7.8585

" " " 7.6464

Weight Al_2O_3 + Fe_2O_3 .2121

% Al_2O_3 + Fe_2O_3 39.25

Titre for Fe_2O_3 1.4 c.c. of .074 N. K Mn O_9

% Fe_2O_3 1.52

% Al_2O_3 37.73

Weight cru. ash Ca O 7.4818

" " " 7.4772

Weight Ca O .0046

% Ca O .81

Weight cru. + ash + $\text{Mg}_2\text{P}_2\text{O}_7$ 7.5376

" " " 7.5246

Weight $\text{Mg}_2\text{P}_2\text{O}_7$.0130

% Mg O .83

Sample 3

Weight tube + sample	10.7474
" "	<u>10.2388</u>
Weight sample	.5086
Weight cru. + ash + Al_2O_3 + Fe_2O_3	7.7566
" " "	<u>7.5580</u>
Weight Al_2O_3 + Fe_2O_3	.1986
% Al_2O_3 + Fe_2O_3	39.05
Titre for Fe_2O_3	1.2 c.c. of .074 N. K Mn O_4
% Fe_2O_3	1.45
% Al_2O_3	37.60
Weight cru. + ash + Ca O	7.4798
" " "	<u>7.4766</u>
Weight Ca O	.0032
% Ca O	.62
Weight cru. + ash + $\text{Mg}_2\text{P}_2\text{O}_7$	7.4882
" " "	<u>7.4804</u>
Weight $\text{Mg}_2\text{P}_2\text{O}_7$	.0078
% Mg O	.55

Sample 4

Weight tube + sample	15.1132
" "	<u>14.6080</u>
Weight sample	.5052
Weight cru. + ash + Al_2O_3 + Fe_2O_3	7.5328
" " "	<u>7.3374</u>
Weight Al_2O_3 + Fe_2O_3	.1954
% Al_2O_3 + Fe_2O_3	38.70

Titre for Fe_2O_3 1.2 c.c. of .074 N. K Mn O_4

% Fe_2O_3 1.50

% Al_2O_3 37.20

Weight cru. + ash + Ca O 7.6108

" " " 7.6064

Weight Ca O .0044

% Ca O .87

Weight cru. + ash + $\text{Mg}_2\text{P}_2\text{O}_7$ 7.4846

" " " 7.4772

Weight $\text{Mg}_2\text{P}_2\text{O}_7$.0074

% Mg O .53

Sample 5

Weight tube + sample 14.6080

" " 14.1008

Weight sample .5072

Weight cru. + ash + Al_2O_3 + Fe_2O_3 7.6948

" " " 7.4978

Weight Al_2O_3 + Fe_2O_3 .1970

% Al_2O_3 + Fe_2O_3 38.85

Titre for Fe_2O_3 1.3 c.c. of .074 N. K Mn O_4

% Fe_2O_3 1.56

% Al_2O_3 37.29

Weight cru. ash Ca O 7.3804

" " " 7.3770

Weight Ca O .0034

% Ca O .66

Weight cru. + ash + $\text{Mg}_2\text{P}_2\text{O}_7$	7.0864
" " "	<u>7.0776</u>
Weight $\text{Mg}_2\text{P}_2\text{O}_7$	.0088
% Mg O .62	

Sample 6

Weight tube + sample	14.1008
" "	<u>13.5208</u>
Weight sample	.5800 gm.
Weight cru. + ash + Al_2O_3 + Fe_2O_3	7.8492
" " "	<u>7.6162</u>
Weight Al_2O_3 + Fe_2O_3	.2320 gm.
% Al_2O_3 + Fe_2O_3 40.00	
Titre for Fe_2O_3 1.3 c.c. of .074 N. K Mn O_4	
% Fe_2O_3 1.40	
% Al_2O_3 38.60	
Weight cru. + ash + Ca O	7.4061
" " "	<u>7.3998</u>
Weight Ca O	.0043 gm.
% Ca O .70	
Weight cru. + ash + $\text{Mg}_2\text{P}_2\text{O}_7$	7.3176
" " "	<u>7.3072</u>
Weight $\text{Mg}_2\text{P}_2\text{O}_7$	.0104
% Mg O .64	

Sample 7

Weight tube + sample	13.5078
" "	<u>12.9046</u>
Weight sample	.6032
Weight cru. + ash + Al_2O_3 + Fe_2O_3	7.7934
" " "	<u>7.5594</u>
Weight Al_2O_3 + Fe_2O_3	.2340
% Al_2O_3 + Fe_2O_3	38.79
Titre for Fe_2O_3 1.6 c.c. of .074 N. K Mn O_4	
% Fe_2O_3	1.58
% Al_2O_3	37.21
Weight cru. + ash + Ca O	7.3100
" " "	<u>7.3054</u>
Weight Ca O	.0046
% Ca O	.76
Weight cru. + ash + $\text{Mg}_2\text{P}_2\text{O}_7$	7.3870
" " "	<u>7.3752</u>
Weight $\text{Mg}_2\text{P}_2\text{O}_7$	.0118
% Mg O	.70

Sample 8

Weight tube + sample	12.9046
" "	<u>12.3840</u>
Weight sample	.5206
Weight cru. + ash + Al_2O_3 + Fe_2O_3	7.5428
" " "	<u>7.3386</u>
Weight Al_2O_3 + Fe_2O_3	.2042
% Al_2O_3 + Fe_2O_3	39.22

Titre for Fe_2O_3 1.4 c.c. of .074 N. KMnO_4

% Fe_2O_3 1.70

% Al_2O_3 37.52

Weight cru. + ash + Ca O

7.1198

" " "

7.1170

Weight Ca O

.0028

% Ca O .58

Weight cru. + ash + $\text{Mg}_2\text{P}_2\text{O}_7$

7.4110

" " "

7.4010

Weight $\text{Mg}_2\text{P}_2\text{O}_7$

.0100

% Mg O .69

APPROVAL

The foregoing thesis is hereby approved as a creditable study of an engineering subject, carried out and presented in a manner sufficiently satisfactory to warrant its acceptance as a prerequisite to the degree for which it has been submitted. It is to be understood that by this approval the undersigned does not necessarily endorse or approve any statement made, opinions expressed, or conclusions drawn therein, but approves the thesis only for the purpose for which it is submitted.

Name

Judson H. Robertson

Title

Professor of Chemistry