WHOLE ROCK ANALYSIS USING THE ELECTRON MICROPROBE

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ABSTRACT

Rock powders ranging in composition from a peridotite to a granite were fused on molybdenum and tantalum strips in a vacuum and a nitrogen atmosphere. A low voltage high amperage source was used to melt the rock powders. Samples weighing 200 mg and fused for 15 to 20 seconds produced glass beads suitable for analysis in the electron microprobe.

Good results were achieved on rocks with composition ranging from basalts to andesites. Relative errors were better than 5% for elements with concentrations greater than 1%. Elements with concentrations greater than 10% generally yielded relative errors better than 3%.

Rocks of a granitic or mafic composition produced good results when a flux was added. The relative error was generally better than 3% for elements with a concentration greater than 1%. The results for potassium in flux melts was poor. However, this should improve with the choice of suitable rock standards. Best results were achieved on flux melts when it was assumed that the difference between the oxide totals of the elements analysed and 100% had the composition of the flux.

CHAPTER 1

Introduction

Numerous authors have described the use of the electron microprobe for whole rock analyses. Techniques used to prepare samples have varied from pressed pellets to fusion of rock powders with and without a flux. Methods of melting rock powders have involved the use of quench furnaces, various metal strip heaters and an optical furnace, and melting of the rock powders has been carried out in vacuum as well as various inert gases.

The purpose of this work is to investigate the method of fusion of rock powders with and without a flux and to determine the precision and accuracy of the method over a range of various silicate rock compositions. Fusion was carried out on molybdenum and tantalum strip heaters under vacuum and in a nitrogen atmosphere. The analytical results obtained compared favorably with results obtained from XRF analyses and with results reported by other authors using similar methods. Finally the method is described in a step by step procedure in order that rapid and accurate whole rock analyses can be achieved with the electron microprobe.

History of whole rock analyses using the Electron Microprobe

Whole rock analyses using the electron microprobe have been reported by numerous authors. Gulson and Lovering (1968) described a method in which glass disks were prepared by a borate bead method which was identical to that used by Norrish and Chappell (1967) for XRF analyses. In this method lithium tetraborate, lanthanum oxide and ammonium nitrate were mixed with rock powders and fused in a gold-lined platinum crucible at 1000°C for several minutes. They reported that there was good correlation of the analytical results between microprobe, chemical and X-ray spectrographic determination on samples ranging in composition from basic gabbro through quartz mica diorite to granite. They experienced some problems, however, with Al_2O_3, FeO and Na_2O.

Rucklidge _et al._ (1970) prepared glasses by direct fusion of rock powders. The rock powders were fused in graphite crucibles in a platium wound quench furnace for 10 to 15 minutes at temperatures approximately 100°C above the liquidus temperature. Fusion difficulties were experienced with granites and peridotites. The advantage of direct rock powder fusion is that high count rates and peak to background ratios are obtained and therefore the method is more sensitive to minor elements. They reported good comparison of analytical results with wet chemical analyses although results for Al and Fe were considered less acceptable. EMPADR, a computer program was used to avoid the use of a wide range of rock standards.

Nicholls (1974) fused rock powders on an iridium strip heater. Samples were fused in air for 20 to 30 seconds and stirred with a Pt rod in order to make the melt homogeneous. The glasses were analyzed in the electron microprobe using an Ortec energy dispersive system. Individual analyses were corrected to 100 weight percent free of volatiles and unanalysed minor elements. Nicholls reported good agreement of analytical results obtained with international geochemical reference standards although the use of simple standards produced poor results for SiO_2 and Al_2O_3. Accuracy and precision for sodium and iron were also reported to be erratic. The use of standard rock glasses improved results. A beam diameter of 50 to 100 μm was used during analyses to smooth out local inhomogeneities.

Brown (1977) also used a strip heater but direct fusion was done in a container pressurized with argon. Various metal strips were investigated with Mo, Ir and Re selected for use but Mo was considered the first choice. Interaction of strip and melt at the contact were reported and Brown recommended that only regions of glass not in contact with the strip be analyzed. Sample size investigations suggested that the relationship between sample size, homogeneity and analytical error was complex. He reported analytical results compared favorably with those achieved by X-ray fluorescence and neutron activation for major elements in a wide range of silicate rocks. Analytical errors for major element concentrations were reported to be generally less than 5%.

Jezek _et_ _al._ (1979) directly fused rock powders on a molybdenum strip in a nitrogen atmosphere. Analytical results, when compared to analyses obtained by classical chemical methods, had relative accuracies better than 5% except for TiO_2. They reported that attempts to fuse granitic samples and MgO-rich low-silica samples to homogeneous glasses were unsuccessful and that MgO=rich low silica samples could not be prevented from growing skeletal olivine crystals during quenching. They also reported that prolonged fusion of samples resulted in Na_2O loss. They concluded that the required preparation of very fine powders and the increased possibility of Na_2O loss on fusion made routine fusion of high-silica samples impractical.

Schimann and Smith (1980) departed from the strip heater method and used an optical furnace to melt rock powders directly. Glasses produced by this method produced analyses which compared satisfactorily with quantitative analyses obtained by wet chemical analyses and X-ray fluorescence methods. Fusion is carried out in a partial vacuum and takes between 5 and 10 minutes. The authors reported that glasses cannot be prepared with this method from rocks with ultramafic composition or where the SiO_2 + Al_2O_3 content is greater than 85 to 90%. Also silicates with Fe_2O_3 greater than 20 to 25% commonly showed immiscibility phenomena. They advised that most of these problems could be avoided by using a $Li_2B_4O_7$ flux and stated that satisfactory accuracy could be achieved by assuming that the difference between oxide totals of the elements analysed and 100% has the flux composition.

Advantage of whole rock analyses using the Electron Microprobe

A number of advantages of fusing rock powders and analysing the glasses in the electron microprobe have been reported by several authors

1) Very small amounts of sample are required.
2) The method is fairly rapid.
3) The method is particularly attractive if the electron microprobe is the principal analytical instrument.
4) Accuracy for major elements (greater than 1%) is good when compared with wet chemical, X-ray fluorescence and neutron activation method.
5) Sample preparation is fairly simple and rapid.

Disadvantages

1) Volatiles are lost on fusion,therefore rocks with high contents of volatiles such as carbonates cannot be analyzed.
2) The results are generally poorer for elements with less than 1% concentration when compared with other methods and the electron microprobe limit of detection is quite high due to background noise therefore it cannot be used for trace elements.

Outline of this Work

The work consists of the following experiments:

1. Correlation of the temperature of tantalum and molybdenum strips against a variac.
2. Evaluation of tantalum and molybdenum strips as boats for melting rock powders in a vacuum and in a nitrogen atmosphere.
3. Investigation of melt temperature and variac setting for a number of rock types.
4. Investigation of the precision of the whole rock analysis procedure with the electron microprobe.
5. Test of accuracy of the method used to analyze rock powders.
6. To determine the effects on sodium and potassium loss as melt time of rock powder increases.
7. Investigation of the effects of adding a flux to rock powders prior to melting.
8. To determine accuracy and precision of the method of whole rock

analysis with the electron microprobe when a flux is added.

9. Investigation of the effect of structural H_2O on the scaling correction using a flux.

CHAPTER 2

Experiment 1.

Purpose

To correlate the temperature of tantalum and molybdenum strips against a variac.

Apparatus

A Minivac vacuum system with a coater module made by Vacuum Industries Limited was used in the experiment. A Leeds and Northrup Optical pyrometer was used to determine temperatures and an Edward penning gauge, model 8 was used for pressure measurements. Material included tantalum and molybdenum strips 0.01 cm in thickness and 5 cm by 1.5 cm in size.

Procedure

A tantalum or a molybdenum strip was clamped between the two electrodes in the bell jar of the Minivac coating unit. The two electrodes were connected to a power supply in the Minivac by a power transformer. Power to the transformer can be controlled with a variac; therefore electric current through the metal strip can be regulated.

Air was then evacuated from the bell jar until a vacuum of 10^{-3} torr was reached. The temperature of the metal strip was increased by raising the variac setting. At pre-determined variac settings the temperature of the strip was determined with an optical pyrometer and the results recorded. This procedure was repeated twice for each strip type.

In order to determine the precision and accuracy of the optical pyrometer, pure silicon and iron were in turn melted on a tantalum strip.

The procedure followed was to place the iron or silicon chip on a tantalum strip, evacuate air from the bell jar until a vacuum of 10^{-3} torr was reached and then to increase the variac setting slowly until the chip began to melt. Again melt temperature was determined with an optical pyrometer. This procedure was repeated a number of times for both silicon and iron.

Observations and Discussion

Observations are reported in Table 1-1 which shows the observed temperature of the metal strips as measured by the optical pyrometer for a number of variac settings. Four runs are shown, two runs each on a molybdenum and a tantalum strip. As seen on the table the observed temperature increases as the variac setting is increased. Also shown on Table 1-1 is the temperature of the strip at the melt temperature of pure silicon and pure iron. Since the melting temperature for pure silicon and pure iron are known these data were used as calibrations for the optical pyrometer.

Figure 1-1 is a plot of the data from Table 1-1 and shows a non-linear increase in strip temperature as the variac is increased in steps. The actual melt temperature for pure silicon and pure iron are also plotted on this diagram as well as the observed melt temperature for these two elements.

Conclusion

It is difficult to obtain accurate readings using the optical pyrometer as shown by the spread in readings when pure iron and silicon are melted. The optical pyrometer reads on the average 45°C low for the silicon melt and 85°C low for the iron melt. The wide spread of temperatures for both silicon and iron indicates poor precision. A plot of the data shows the relationship between strip temperature and variac settings to be non-linear. Also the observed temperature between two runs with the same metal strip and at the same Variac setting in some cases are over 100°C apart. This poor precision is probably due to instrument error and inconsistencies in operating the pyrometer. The average temperature for any given variac setting is generally higher using the molybdenum strip. This is to be expected since the electrical conductivity of molybdenum is higher than the electrical conductivity of tantalum.

TABLE 1-1: Variac settings vs Mo and Ta strip temperatures.

RUN #	VARIAC SETTING	OBSERVED TEMPERATURE °C	STRIP TYPE
1.	14	835	Mo
	16	985	
	18	1105	
	20	1160	
	24	1340	
	30	1450	
	35	1560	
2.	14	960	
	16	1065	
	18	1175	
	20	1280	
	24	1380	
	28	1505	
	32	1590	
	36	1655	
3.	14	860	Ta
	16	920	
	18	1025	
	20	1125	
	24	1270	
	28	1350	
	32	1450	
	34	1505	
	36	1565	
	40	1635	
4.	14	940	
	18	1145	
	20	1225	
	24	1385	
	28	1465	
	32	1550	
	34	1605	

CALIBRATION - STRIP TEMPERATURE VS. VARIAC SETTING FOR CALIBRATION OF OPTICAL PYROMETER

SAMPLE	VARIAC	AMPS	TEMP °C (OF STRIP)	STRIP TYPE
Pure Si	35	105	1425	Ta
	32	100	1360	
	33	95	1375	
	33	103	1390	
	32	95	1325	
Pure Fe	38	115	1475	
	35	105	1425	
	38	115	1450	

Melting Point for Si = 1420°C
Melting Point for Fe = 1530°C

Fig. 1-1. Plot of molybdenum and tantalum strip temperatures vs variac settings showing a non-linear increase in temperature as the variac is increased in steps. For comparison the actual melt temperatures of pure silicon and pure iron are shown (dashed lines) along with the observed temperatures.

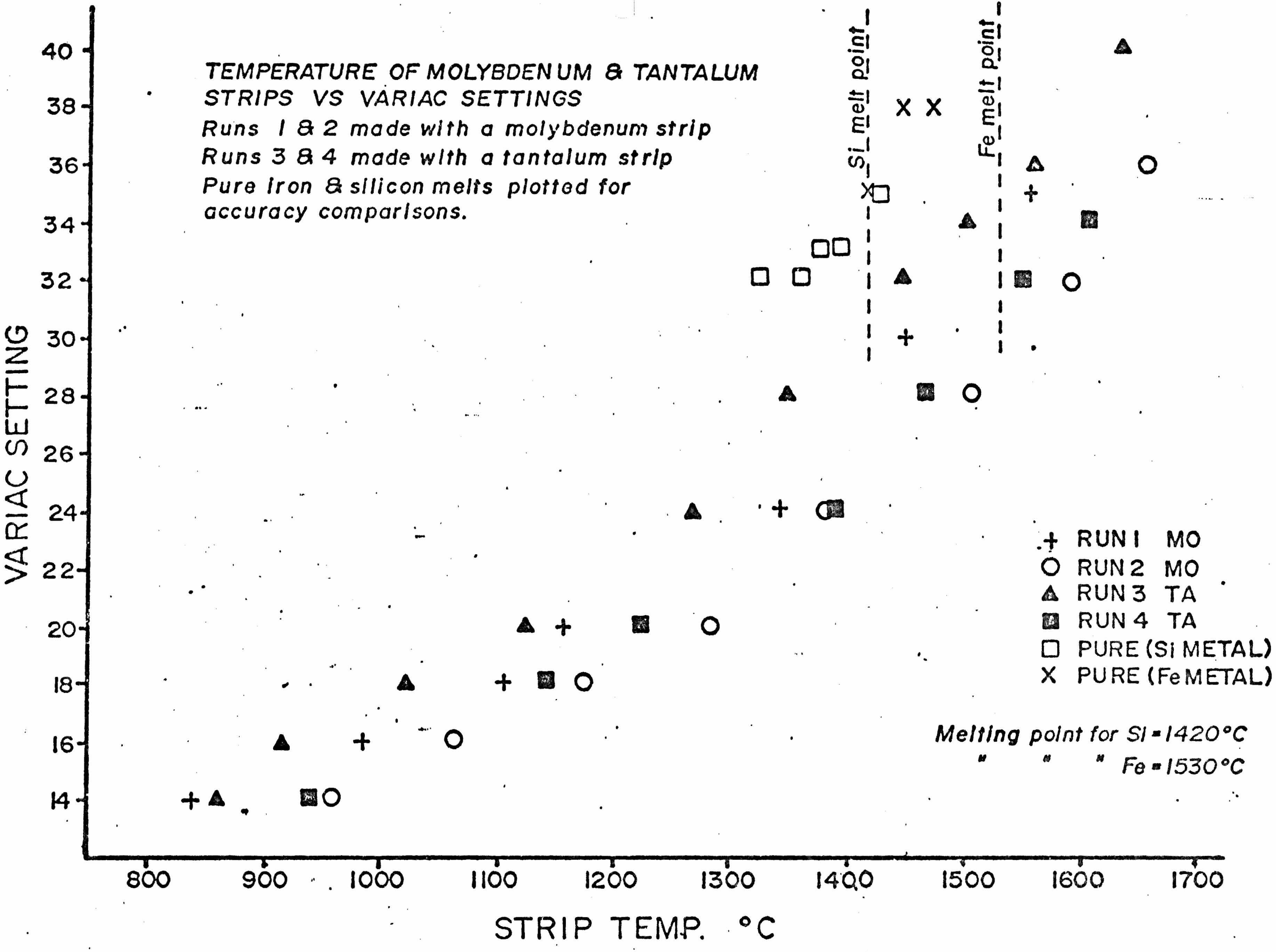

TEMPERATURE OF MOLYBDENUM & TANTALUM STRIPS VS VARIAC SETTINGS
Runs 1 & 2 made with a molybdenum strip
Runs 3 & 4 made with a tantalum strip
Pure iron & silicon melts plotted for accuracy comparisons.
Si melt point
Fe melt point
VARIAC SETTING
40
38
36
34
32
30
28
26
24
22
20
18
16
14
800
900
1000
1100
1200
1300
1400
1500
1600
1700
STRIP TEMP. °C
+ RUN 1 MO
○ RUN 2 MO
▲ RUN 3 TA
▨ RUN 4 TA
☐ PURE (Si METAL)
X PURE (Fe METAL)
Melting point for Si = 1420°C
" " " Fe = 1530°C

FIG. 1-1

Experiment 2

Purpose

To evaluate tantalum and molybdenum strips as boats for melting rock powders in a vacuum and in a nitrogen atmosphere.

Apparatus

The same equipment as used in Experiment 1 was also used in this experiment. The microprobe analyses were carried out on a Cambridge Mark 5 Electron Microprobe with an Ortec Energy Dispersive System. The resolution of the detector is rated at 149 ev on the Mn $K\alpha$ line. The microprobe was operated at 15 Kv acceleration voltage with a probe current of 5 nanoamperes. Data was corrected using the software program EDATA 2 (Smith and Gold, 1980). Rock powders used in the experiment included Z673, AZ70, E2 and DPL (Appendix 1).

Procedure

A metal strip was clamped between the two electrodes in the bell jar. The rock powder was placed on the strip and air was then evacuated from the bell jar until a vacuum of 10^{-3} torr was reached. At this point the rock powder was either melted in the vacuum, or nitrogen gas was first released into the bell jar and then the rock powder melted. Melting was accomplished by slowly increasing the variac setting until the rock powder began to melt, at which point the variac setting was increased slightly and held there until melting was completed. Melt times typically range from 25 to 35 seconds. Quenching of the melt to a glass was accomplished by quickly shutting off the power to the metal strip. This procedure was repeated for several rock types on tantalum and molybdenum strips.

The resulting glass beads were crushed and examined to determine their degree of optical homogeneity. Clear-looking pieces, free of inclusions, were mounted in an epoxy mould. This mould was then ground, polished and carbon coated following standard procedures. Finally the samples were probed to determine their chemical composition.

Observations and Discussion

Shown in Table 2-1 are microprobe analyses of sample Z673 and AZ70 along with absolute errors. Table 2-2 is derived from Table 2-1 and shows analyses from the glasses melted under one condition divided by analyses melted under another condition on an element basis in order to show any differences. Table 2-3 is additional information.

Figures 2-1, 2-2 and 2-3 are plots of oxide weight percent of sample Z673 from Table 2-1 for various combinations of strip types and atmospheres.

Optically most glasses produced from the melt looked similar. Sample Z673 however looked frothy and was difficult to melt because of its high SiO_2 + Al_2O_3 content. Sample E2 was also difficult to melt and one E2 sample did not appear to be homogeneous.

The rock powders melted in a vacuum tended to fly off the strip if the electric current through the strip was increased too quickly.

This effect was probably caused by water being liberated from the sample. The problem can be overcome by pre-heating the sample or by raising the strip temperature slowly.

Quenched glasses tended to stick to the tantalum strips more than to the molybdenum strips. This problem was more apparent with samples requiring a high melt temperature.

Contamination from the metal strips, if present, can usually be seen through a reflecting light microscope. Both tantalum and molybdenum have a high reflectivity compared to the surrounding silicate glass. Contamination by the metal strip is more pronounced in samples requiring a high melt temperature. Contamination, however, cannot always be detected optically. It can be kept to a minimum by analyzing areas of the sample away from the strip and by examining each X-ray spectrum for Mo and Ta peaks on the multichannel analyzer prior to processing the data. Tantalum produces a $L\alpha_1$ X-ray line at 8.15 Kev and molybdenum an $L\alpha_1$ X-ray line at 2.29 Kev. Both these lines can be readily identified from the X-ray spectrum. The Ta $M\alpha_1$ line falls close to the Si $K\alpha_1$ line and the Mo $L\alpha_1$ is close to the S $K\alpha_1$ line.

Samples AZ70 and Z673 were melted under four conditions. Each sample was melted on a tantalum strip and then on a molybdenum strip with two melts for each strip type, one in a nitrogen atmosphere and one under a vacuum. From the chemical analyses produced on the electron microprobe absolute errors were calculated in order that differences in the same sample could easily be seen (see Table 2-1).

If the melt condition for each sample is the same, then the microprobe analyses for all four samples should be identical. This was checked by dividing sample analysis by sample analysis on an element basis (see Table 2-2). For sample AZ70 most major elements deviated by no more than 3%.

The values obtained for sample Z673 were not good with 67% of the major elements within 5%. This sample,however, has a high SiO_2 + Al_2O_3 content and was difficult to melt. Also,the SiO_2 + Al_2O_3 content represents 85% of the sample,therefore most of the comparisons are between elements with low concentration in which minor fluctuations appear higher when shown as a relative percent.

Figures 2-1, 2-2 and 2-3 are plots of the oxide weight percent of sample Z673 for various combinations of strip type and atmosphere. From the diagrams it can be seen that the variation between strip types and 'atmosphere', are random.

Conclusions

From the data shown it appears little difference exists between melts on tantalum or molybdenum strips and melts in a nitrogen atmosphere or under a vacuum.

TABLE 2-1: Analyses of samples Z673 and AZ70 with absolute errors.

OBSERVATIONS

	1		2		3		4		5
SiO_2	65.53	-3.05	66.70	-1.89	65.76	-2.83	66.64	-1.95	68.59
TiO_2	.34	- .05	.28	- .11	.18	- .21	.32	- .07	.39
Al_2O_3	18.24	+1.34	17.24	+ .34	17.36	+ .46	17.57	+ .67	16.90
FeO	1.95	+ .17	1.92	- .20	1.93	- .19	2.11	- .01	2.12
MgO	.64	+ .19	.75	- .08	.76	- .07	.75	- .08	.83
CaO	3.70	+ .09	3.72	+ .11	3.84	+ .23	3.54	- .07	3.61
Na_2O	4.88	+ .40	4.77	- .01	4.58	+ .10	4.80	+ .32	4.48
K_2O	2.98	+ .30	2.82	+ .14	2.99	+ .30	2.95	+ .27	2.68
Other									.20

	6		7		8		9		10
SiO_2	46.88	- .70	46.95	- .63	46.82	- .76	46.27	-1.31	47.58
TiO_2	2.35	+ .16	2.29	+ .10	2.25	+ .06	2.31	+ .21	2.19
Al_2O_3	14.36	- .44	14.55	- .25	14.51	- .29	14.78	- .02	14.80
FeO	13.98	+ .62	13.37	+ .01	13.96	+ .60	13.57	+ .21	13.36
MgO	6.55	- .14	6.45	- .24	6.40	= .29	6.20	- .49	6.69
CaO	12.01	+ .02	12.04	+ .05	11.82	- .17	11.69	- .30	11.99
Na_2O	2.64	+ .09	2.63	+ .08	2.77	+ .22	2.73	+ .18	2.55
K_2O	.24	- .03	.25	- .02	.24	- .03	.28	+ .01	.27
Other									.57

1. Sample Z673(5) - Ta strip in nitrogen gas
2. Sample Z673(5) - Ta strip in a vacuum
3. Sample Z673(6) - Mo strip in nitrogen gas
4. Sample Z673(5) - Mo strip in a vacuum
5. Sample Z673 - accepted value
6. Sample AZ70(2) - Ta strip in nitrogen gas
7. Sample AZ70(2) - Ta strip in a vacuum
8. Sample AZ70(2) - Mo strip in nitrogen gas
9. Sample AZ70(2) - Mo strip in a vacuum
10. Sample AZ70 - accepted value

Microprobe analysis -

Each analyses includes absolute errors. Number of analyses for each sample in brackets. Accepted sample values calcuated to 100% volatile free.

TABLE 2-2: Analyses from Table 2-1, analysis from one melt divided by analysis from another melt.

	1	2	3	4	5
SiO_2		1.0118	1.0012	1.0131	47.58
TiO_2	1.0262	.9740	1.0044	1.0173	2.19
Al_2O_3	.9869	.9817	.9897	.9716	14.80
FeO	1.0456	1.0287	1.0014	1.0302	13.36
MgO	1.0155	1.0322	1.0234	1.0387	6.69
CaO	.9975	1.0111	1.0161	1.0273	11.99
Na_2O	1.0038	1.0146	.9531	.9670	2.55
K_2O	-	-	-	-	-

	6	7	8	9	10
SiO_2	.9825	.9847	.9965	.9826	68.59
TiO_2					.39
Al_2O_3	1.058	.9881	1.050	1.0381	16.90
FeO	1.015	.9147	1.010	.9242	2.12
MgO	.8533	1.013	.8421	.8533	.83
CaO	.9946	1.084	.9635	1.045	3.61
Na_2O	1.0901	.9542	1.0655	1.011	4.48
K_2O	1.056	1.013	.9967	1.010	2.68

1. Tantalum (N_2) ÷ Tantalum (vacuum)
2. Molybdenum (N_2) ÷ Molybdenum (vacuum)
3. Tantalum (N_2) ÷ Molybdenum (N_2) AZ 70
4. Tantalum (N_2) ÷ Molybdenum (vacuum)
5. AZ70 Book value
6. Tantalum (N_2) ÷ Tantalum (vacuum)
7. Tantalum (N_2) ÷ Molybdenum (vacuum) Z 673
8. Tantalum (N_2) ÷ Molybdenum (N_2)
9. Tantalum (N_2) ÷ Molybdenum (vacuum)
10. Sample Z673 - book value

TABLE 2-3: Analyses of samples E2 and DPL.

OBSERVATIONS

Additional Data

	1		2		3		4
SiO_2	56.54	-1.9	58.73	+ .29	57.19	-1.25	58.44
TiO_2	.39	- .02	.24	- .17	.39	- .02	.41
Al_2O_3	18.36	- .67	18.90	- .13	18.76	- .27	19.03
FeO	5.85	+ .20	4.88	+ .23	5.80	+ .15	5.65
MgO	.21	+ .11	.25	+ .15	.28	+ .18	.10
CaO	2.10	+ .14	1.83	+ .13	2.12	+ .16	1.96
Na_2O	6.48	- .21	6.02	- .67	6.24	- .45	6.69
K_2O	5.70	- .99	6.14	- .55	6.10	- .59	6.69

	5		6		7		8
SiO_2	50.70	- .06	50.53	- .23	51.04	- .72	50.76
TiO_2	1.09	- .03	1.01	- .11	1.01	- .11	1.12
Al_2O_3	14.68	- .17	14.81	- .04	14.86	+ .01	14.85
FeO	9.50	- .04	9.40	- .14	9.25	- .29	9.54
MgO	8.88	- .51	8.94	- .45	9.08	- .31	9.39
CaO	11.24	- .13	11.24	- .13	11.38	+ .01	11.37
Na_2O	2.21	- .11	2.16	- .15	2.13	- .19	2.35
K_2O	.21	- .14	.22	- .13	.23	- .12	.35

1. Sample E2 (2) - Ta strip in nitrogen gas
2. Sample E2 (3) - Ta strip in a vacuum
3. Sample E2 (2) - Mo strip in a vacuum
4. Sample E2 - given value
5. Sample DPL - Mo strip in nitrogen gas
6. Sample DPL - Mo strip in a vacuum
7. Sample DPL - Ta strip in nitrogen gas
8. Sample DPL - given value

Additional data - Microprobe analysis. Each analysis includes absolute errors. Number of analyses for each sample in bracket. Given sample values calculated to 100% volatile free.

Fig. 2-1. Plot of oxide weight percent of sample Z673 melted on a tantalum strip in nitrogen gas vs a melt on a molybdenum strip in a vacuum. Since all the points lie on a near straight line with a slope of unity, little difference exists between the two melting methods.

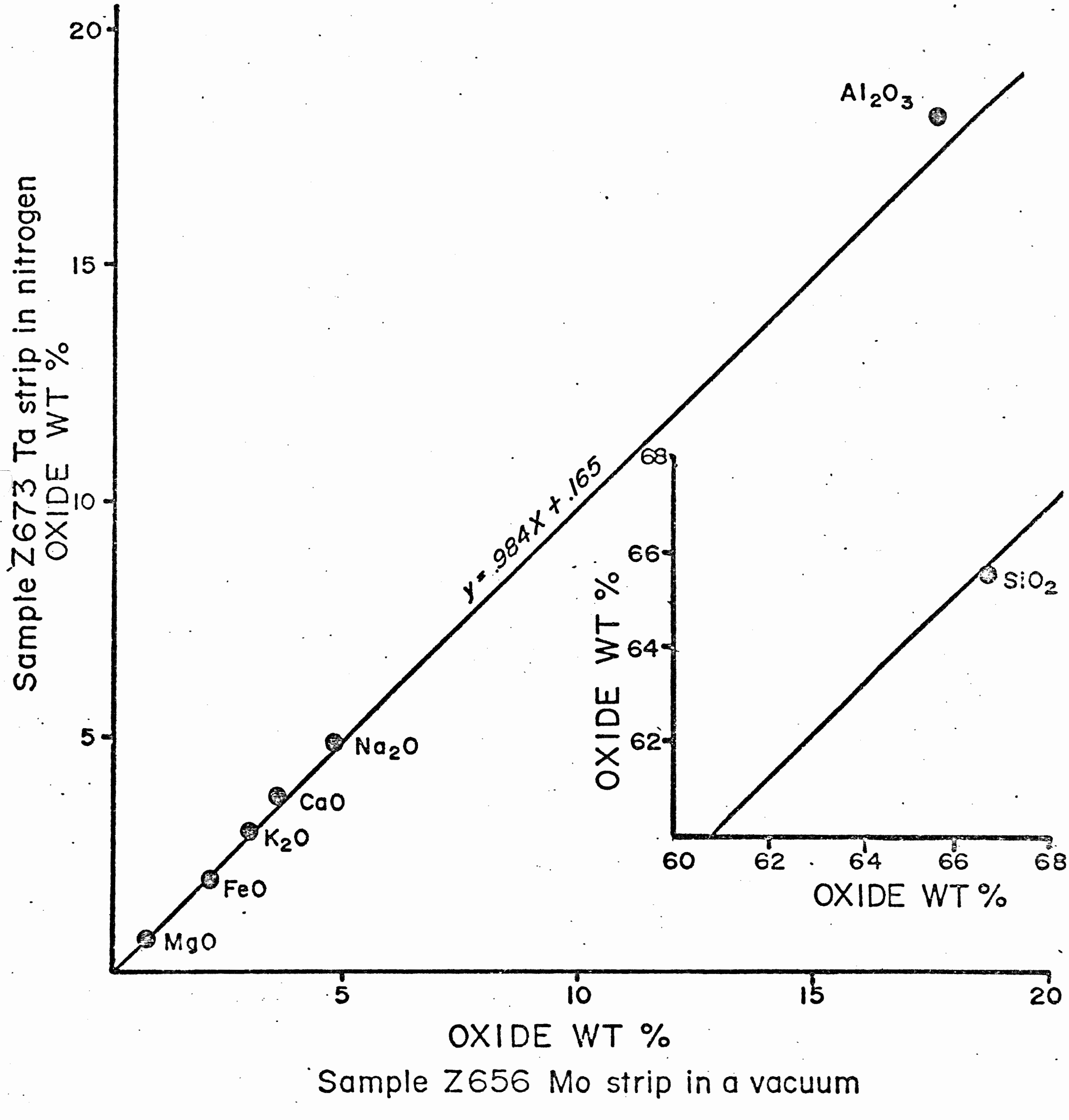

20
15
10
5
Sample Z673 Ta strip in nitrogen
OXIDE WT %
y = .984X + .165
Al_2O_3
Na_2O
CaO
K_2O
FeO
MgO
5
10
15
20
OXIDE WT %
Sample Z656 Mo strip in a vacuum
68
66
64
62
OXIDE WT %
SiO_2
60
62
64
66
68
OXIDE WT %

FIG. 2-1

Fig. 2-2. Plot of oxide weight percent of sample Z673 melted on a molybdenum strip in nitrogen gas vs a melt on a molybdenum strip in a vacuum. Since all the points lie on a near straight line with a slope of unity, little difference exists between the two melting methods.

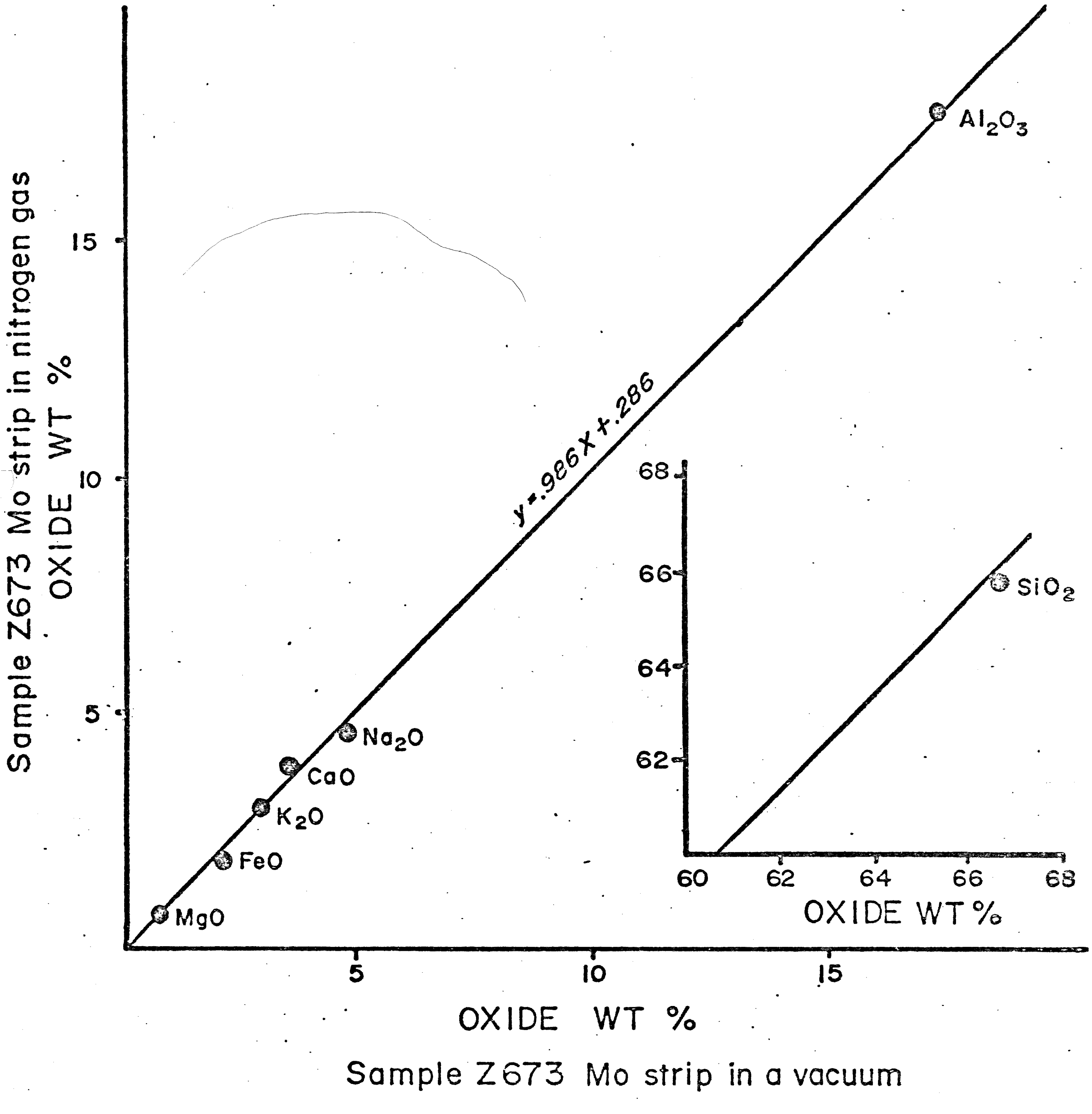

Sample Z673 Mo strip in nitrogen gas
OXIDE WT %
15
10
5
y=.986X+.286
Al$_2$O$_3$
Na$_2$O
CaO
K$_2$O
FeO
MgO
5
10
15
OXIDE WT %
Sample Z673 Mo strip in a vacuum
68
66
64
62
SiO$_2$
60
62
64
66
68
OXIDE WT %

FIG. 2-2

Fig. 2-3. Plot of oxide weight percent of sample Z673 melted on a tantalum strip in nitrogen gas vs a melt on a tantalum strip in a vacuum. Since all the points lie on a near straight line with a slope of unity, little difference exists between the two melting methods.

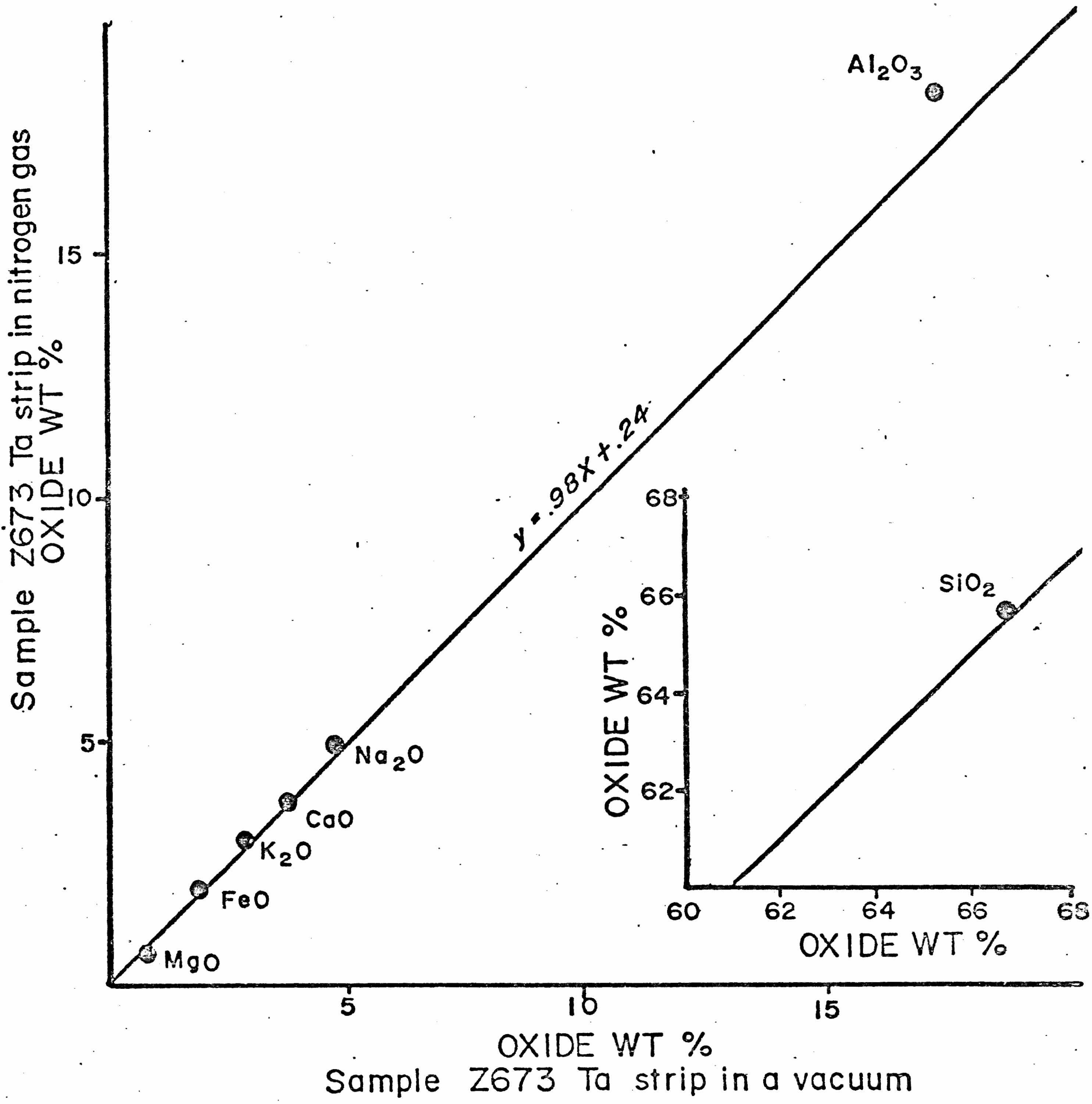

Sample Z673 Ta strip in nitrogen gas
OXIDE WT %
15
10
5
5
10
15
OXIDE WT %
Sample Z673 Ta strip in a vacuum
y = .98X + .24
Al_2O_3
Na_2O
CaO
K_2O
FeO
MgO
68
66
64
62
60
62
64
66
68
OXIDE WT %
OXIDE WT %
SiO_2

FIG. 2-3

Experiment 3

Purpose

To determine the melt temperature and the variac setting for a number of rock types.

Apparatus

Same equipment used as per Experiment 1.

Procedure

The rock powder was placed on a tantalum strip which was mounted between the two electrodes in the bell jar. Air was evacuated from the jar until a vacuum of 10^{-3} torr was reached. The temperature of the strip was increased by slowly increasing the variac settings until the rock powder began to melt. The melt temperature was then measured with the optical pyrometer. The procedure was repeated several times for each rock type and for a variety of rocks.

Observations and Discussion

Table 3-1 shows the melt temperature of the various rock samples as measured by the optical pyrometer. The variac setting at the point the rock powders began to melt is also recorded. The observed melt temperatures for pure silicon and pure iron are also shown for comparison and calibration purposes.

Figure 3-1 is a plot of the data from Table 3-1. It shows that a general linear relationship exists between the variac setting and the melt temperature of the various rock samples. Figure 3-2 is a plot of the $SiO_2 + Al_2O_3$ content of the melted rock samples against their melt temperatures and shows that as the $SiO_2 + Al_2O_3$ content of the rocks increases so also does the apparent melting temperature.

Conclusions

For calibration purposes data of pure silicon and iron melts from Experiment 1 are included. Figure 3-1 is a plot of rock melt temperature vs variac settings. Spread of the data points for each rock type is an indicator of the poor precision of measurement (see Experiment 2). Figure 3-2 is a plot of temperature vs $SiO_2 + Al_2O_3$ content in weight percent. From this diagram it can be seen that as the $SiO_2 + Al_2O_3$ content of the rock increases so also does the melt temperature increase.

Melt times are relatively fast with a typical melt time taking between 15 and 20 seconds. Possibly under these conditions each mineral phase melts out of equilibrium with other phases which could result in the most refractory mineral forming "pockets" of inhomogeneous melt. This effect would be most pronounced in rocks which contain a large percentage of a refractory minerals, such as quartz. Granitic rocks then, would not only require a high melting temperature, but would tend to form inhomogeneous melts.

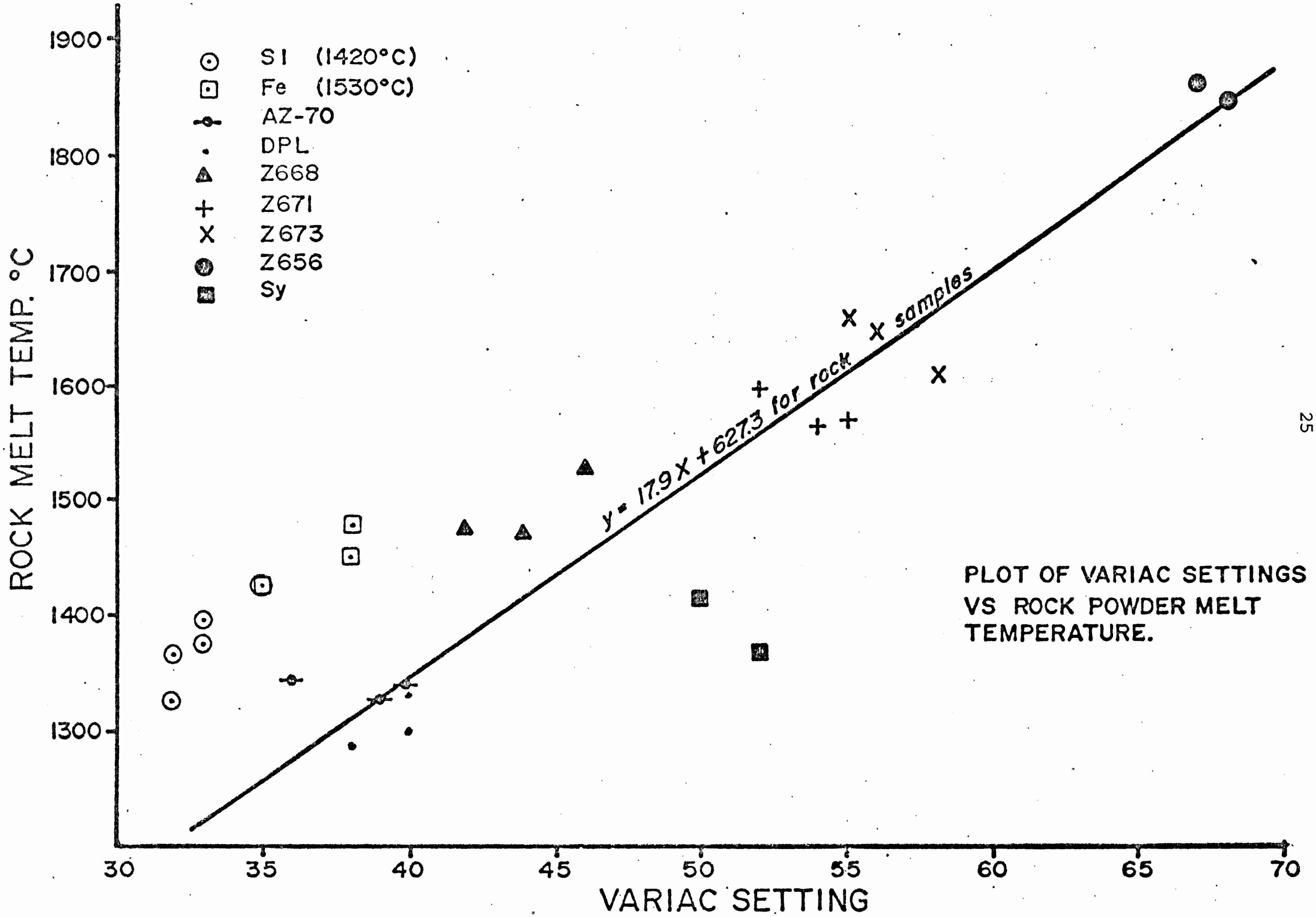
TABLE 3-1: Variac settings vs melt temperature for rock samples.

OBSERVATIONS

ROCK MELT TEMPERATURE

SAMPLE	VARIAC	AMPS	TEMP °C	COMMENT
Pure Si	35	105	1425	For calibration
	32	100	1360	
	33	95	1375	
	33	103	1390	
	32	95	1325	
Pure Fe	38	115	1475	
	35	105	1425	
	38	115	1450	
AZ-70	40	125	1335	Rock Powders
	36	115	1345	
	39	115	1325	
DPL	40	115	1300	
	40	120	1330	
	38	115	1285	
Z-668	44	130	1470	
	46	130	1525	
	42	145	1475	
Z-671	52	140	1590	
	55	145	1570	
	54	140	1565	
Z-673	56	150	1645	
	55	150	1655	
	58	150	1610	
Z-656	68	175	-	Strip broke
	68	175	1840	
	67	170	1855	
SY-3	52	140	1365	
	50	140	1420	

Fig. 3-1. Plot of rock melt temperature vs variac settings which (except for SyD shows a near linear relationship between the two. For comparison the observed melt temperatures for pure silicon and pure iron are also shown.



PLOT OF VARIAC SETTINGS VS ROCK POWDER MELT TEMPERATURE.

FIG. 3-1

Fig. 3-2. Plot of $SiO_2 + Al_2O_3$ content of the rock samples vs rock sample melting temperatures. The diagram shows that as the $SiO_2 + Al_2O_3$ content of the rocks increases so also does the apparent melting temperature.

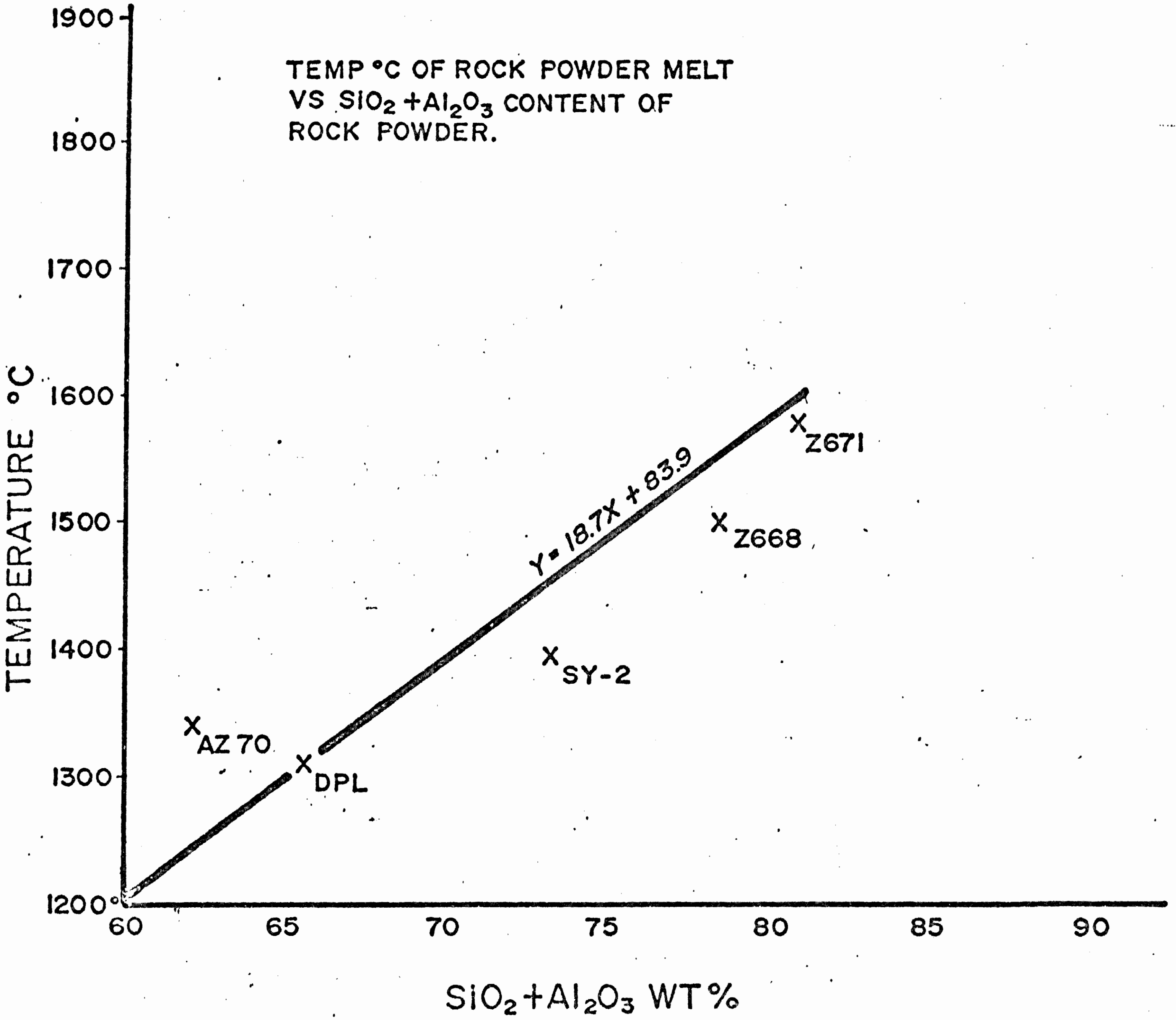

TEMP °C OF ROCK POWDER MELT
VS SiO2 +Al2O3 CONTENT OF
ROCK POWDER.
TEMPERATURE °C
1900
1800
1700
1600
1500
1400
1300
1200°
60
65
70
75
80
85
90
SiO2+Al2O3 WT%
Y= 18.7X + 83.9
Z671
Z668
SY-2
AZ 70
DPL

FIG. 3-2

Experiment 4

Purpose

To determine the precision of the whole rock analysis procedure with the electron microprobe.

Apparatus

The same equipment and material is used in this experiment as was used in Experiment 1 and Experiment 2. The rock powders melted included samples AZ70, AZ77, DPL, Z668, Z671, Z673 and Z656.

Procedure

The procedure used in this experiment was the same as in Experiment 2 except that only tantalum was used as a holder and all melts were carried out in a vacuum. Approximately 200 mg of rock powder were used for each melt and each rock powder was melted several times.

Each melt was then analysed in the electron microprobe at five different spots. Areas in the center of the sample and free from cracks and inclusions were chosen for analysis. In order to reduce the effect of local inhomogeneities the electron microprobe was placed in the raster mode and areas approximately 50 x 50 microns were analyzed. All analyses were referenced to a KK standard which was also rastered at the beginning of the run (see Appendix 2 for data on standards).

In order that the results could be compared with the internal precision of the electron microprobe, sideromelane, a standard basaltic glass, was rastered on the same spot 10 times.

Observations and Discussion

From the analyses, the mean ($\bar{x}$), standard deviation (s) and the coefficient of variation (CV) for each element was determined (Table 4-1). The coefficient of variation is the standard deviation times 100 divided by the mean and expresses the homogeneity of the glass for each element as well as any minor instrument drift.

Table 4-2 shows the mean, standard deviation and coefficient of variation for each melt of the DPL as well as for the sum of all the melts. From these data it can be seen how the error of precision is distributed among the five fusions. The standard deviation for the five melts is also expressed as a percent of the amount of the element present. From this it can be seen the precision of the melts fall within ±1.1% for all elements except for sodium, potassium and minor elements.

Figure 4-1 shows coefficient of variation for $SiO_2 + Al_2O_3$ vs wt % $SiO_2 + Al_2O_3$. From this figure it can be seen that the higher the concentration of an oxide except $SiO_2 + Al_2O_3$, the more homogeneous the melt. The exception is AZ77 which may not solidify as a glass but rather crystallizes even with rapid quenching, possibly because of its high FeO + MgO content. The higher the $SiO_2 + Al_2O_3$ content, the more inhomogeneous the melt, which affects the precision of these two elements plus all other elements.

Figure 4-2, 4-3 and 4-4 are plots of standard deviation vs oxide wt % for the samples plus sideromelane for comparison. In general as the $SiO_2 + Al_2O_3$ content of the rocks increase so also does the standard deviation increase.

Conclusion

For samples with low $SiO_2 + Al_2O_3$ (less than 70%) content the precision is good. As $SiO_2 + Al_2O_3$ content increases the melts become more inhomogeneous. This in turn results in a higher standard deviation and inferior precision. This inferior precision, however, is a reflection of the difficulty of melting rocks with high $SiO_2 + Al_2O_3$ content.

TABLE 4-1: Analyses of samples AZ77, AZ70, DPL, Z668, Z673 and Z656 - PRECISION TEST.
Sideromelane included for comparison.

		Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	TiO_2	MnO	FeO
Sideromelane - (rastered on same spot 10 times)											
	$\bar{x}$	2.80	6.93	13.82	49.94	.24	.19	10.95	1.75	.20	11.91
	s	.108	.092	.097	.144	.073	.031	.064	.080	.07	.145
	CV%	3.8	1.3	.70	.29	30.3	16.5	.59	4.6	35.1	1.2
Table values		2.62	6.71	14.06	50.81	.20	.19	11.12	1.85	.22	11.83
AZ-77 (average of 10)											
	$\bar{x}$	.41	23.43	8.27	45.21	.17	.04	8.52	.85	.17	11.77
	s	.128	1.72	.378	.497	.043	.04	.527	.085	.074	.338
	CV%	31.3	7.32	4.58	1.09	25.0	100.0	6.18	10.0	43.7	2.88
Table values		.47	24.61	7.67	45.76	.08	.07	8.18	1.25	.19	11.83
AZ-70 (average of 25)											
	$\bar{x}$	2.67	6.57	14.74	47.02	.26	.24	12.09	2.15	.23	13.56
	s	.128	.178	.196	.446	.075	.036	.171	.144	.073	.462
	CV%	4.8	2.7	1.33	.95	29.1	13.0	1.41	6.72	31.5	3.41
Table values		2.55	6.69	14.80	47.58	.22	.27	11.99	2.19	.24	13.36
DPL (average of 25)											
	$\bar{x}$	2.24	9.09	15.13	51.73	.13	.20	11.46	1.03	.17	9.17
	s	.138	.307	.224	.322	.09	.041	.122	.115	.052	.443
	CV%	6.15	3.38	1.48	.62	69.6	20.3	1.06	11.19	30.9	4.83
Table values		2.32	9.39	14.85	50.76	.12	.35	11.37	1.12	.18	9.54
Z - 668 (average of 24)											
	$\bar{x}$	3.94	2.59	18.03	60.10	.36	2.25	6.20	.70	.065	4.54
	s	.242	.479	1.04	1.93	.111	.206	.683	.243	.049	.737
	CV%	6.14	18.5	5.77	3.21	30.8	9.18	11.0	34.7	74.8	16.2
Table values		4.06	2.45	18.52	60.29	.33	2.13	6.42	.79	.07	4.46
Z-671 (average of 25)											
	$\bar{x}$	3.48	1.51	18.28	63.08	.25	2.28	5.97	.64	.07	3.51
	s	.502	.204	1.01	2.43	.09	.188	.488	.159	.064	.082
	CV%	14.45	13.49	5.50	3.86	36.3	8.25	8.18	24.9	92.1	22.86
Table values		4.07	1.45	17.55	63.50	.32	2.44	5.73	.64	.07	3.84
Z-673 (average of 25)											
	$\bar{x}$	3.75	.87	17.95	67.71	.16	2.88	3.89	.38	.07	1.77
	s	.729	.21	.849	2.19	.085	.279	.320	.199	.119	.306
	CV%	19.43	24.1	4.73	3.23	53.3	9.70	8.25	52.4	170.	17.27
Table values		4.48	.83	16.90	68.59	.16	2.68	3.61	.39	.04	2.12
Z-656 (average of 25)											
	$\bar{x}$	1.93	.21	13.74	77.44	.05	5.65	.79	.007		.44
	s	.728	.063	1.56	3.07	3.07	.647	.256	.0075		.164
	CV%	37.7	29.9	11.36	3.97	140.	11.45	32.4			37.3
Table Values		3.48	.41	13.80	75.57	.06	5.11	.77	.11	.02	.63

TABLE 4-2: Analyses of DPL for five separate melts for error of precision comparison.

		Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	TiO_2	MnO	FeO
Melt #1	$\bar{x}$	2.27	9.09	14.97	51.48		.18	11.14	1.02		9.29
	S	.155	.437	.233	.331		.034	.095	.094		.207
	CV%	6.8	4.8	1.6	.64		18.6	.86	9.2		2.2
Melt #2	$\bar{x}$	2.39	8.99	15.33	52.09		.21	11.48	1.02		9.30
	S	.075	.276	.131	.291		.040	.103	.138		.062
	CV%	3.1	3.1	.85	.56		19.0	.90	13.6		.67
Melt #3	$\bar{x}$	2.18	9.25	15.08	51.64		.20	11.41	1.07		9.16
	S	.056	.376	.119	.223		.046	.189	.094		.291
	CV%	2.6	4.1	.79	.43		23.0	1.7	8.8		3.2
Melt #4	$\bar{x}$	2.21	9.20	15.13	51.84		.23	11.49	1.02		8.81
	S	.160	.194	.339	.251		.045	.124	.196		.878
	CV%	7.2	2.1	2.2	.48		19.5	1.1	19.1		9.9
Melt #5	$\bar{x}$	2.16	8.92	15.14	51.60		.19	11.51	1.03		9.08
	S	.099	.152	.131	.188		.040	.093	.060		.179
	CV%	4.6	1.7	.87	.36		21.2	.81	5.8		2.0
For the 5 Melts	$\bar{x}$	2.24	9.09	15.13	51.73		.20	11.46	1.03		9.13
	S	.093	.138	.131	.239		.019	.153	.022		.200
	CV%	4.1	1.5	.86	.46		9.6	1.3	2.1		2.1
Standard Deviation as a percent of the amount of element present		4.1	1.5	.87	.46		9.5	1.3	2.1		2.2

Fig. 4-1. Plot of the coefficient of variation (cv), for SiO_2 + Al_2O_3 vs the weight percent of SiO_2 + Al_2O_3 in the rock samples (cv = standard deviation x 100/mean). The coefficient of variation reflects the homogeneity of the melted rock samples. As the SiO_2 + Al_2O_3 content of the rocks increases the melted samples become more inhomogeneous as shown by an increase in the cv values in the diagram.

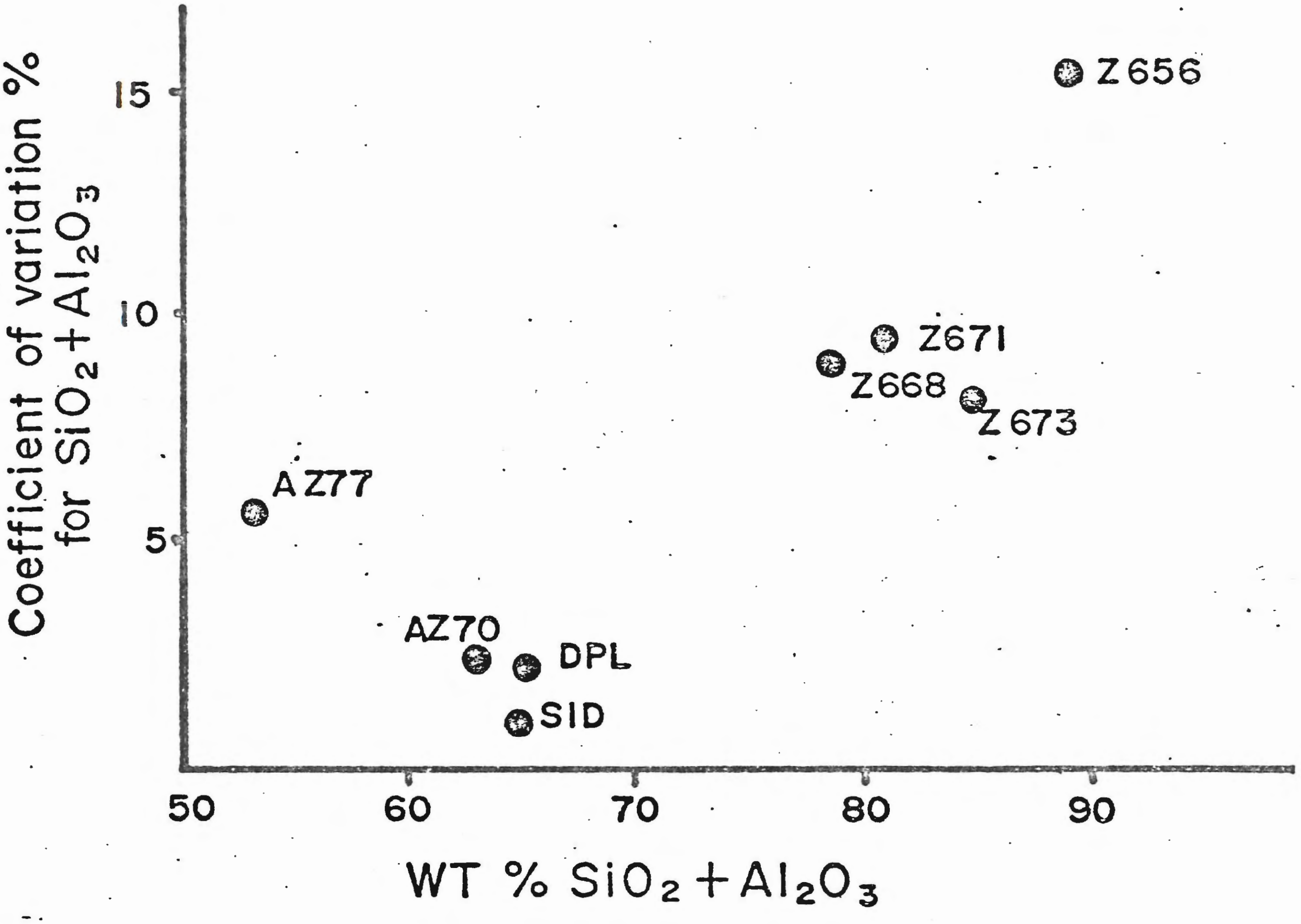

Coefficient of variation % for $SiO_2+Al_2O_3$
15
10
5
50
60
70
80
90
WT % $SiO_2+Al_2O_3$
Z656
Z671
Z668
Z673
AZ77
AZ70
DPL
SID

FIG. 4-1

Fig. 4-2. Plot of the standard deviation vs the oxide weight percent of samples DPL and Z668. For comparison SiD, a basaltic glass which was not melted is also shown. As the $SiO_2 + Al_2O_3$ content of the rocks increases, standard deviation also increases reflecting poorer precision.

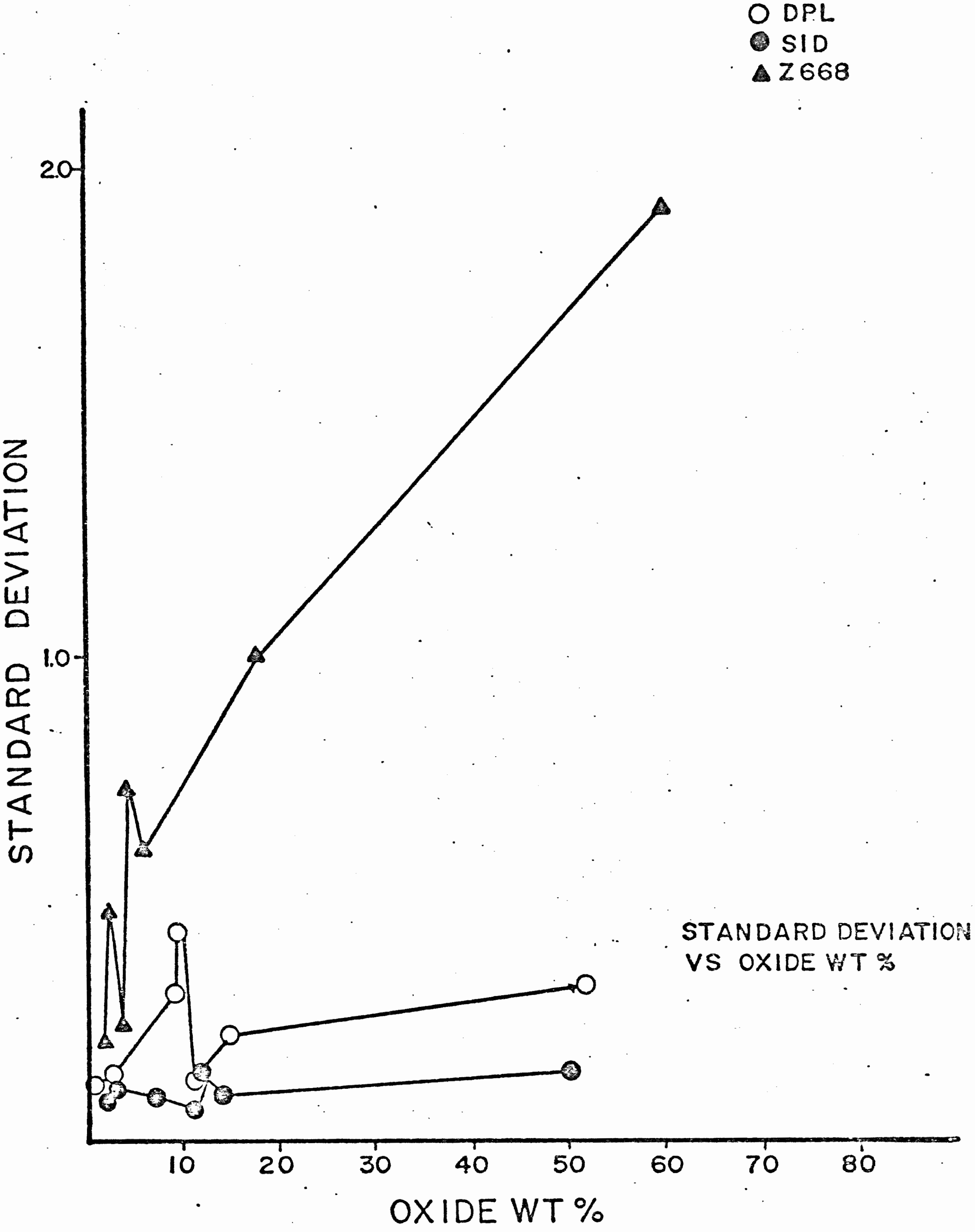

○ DPL
● SID
▲ Z668
STANDARD DEVIATION
2.0
1.0
STANDARD DEVIATION
VS OXIDE WT %
10
20
30
40
50
60
70
80
OXIDE WT %

FIG. 4-2

Fig. 4-3. Plot of the standard deviation vs the oxide weight percent of samples AZ77, AZ70 and Z671. For comparison SiD, a basaltic glass which was not melted is also shown. As the $SiO_2 + Al_2O_3$ content of the rocks increases, standard deviation also increases reflecting poorer precision.

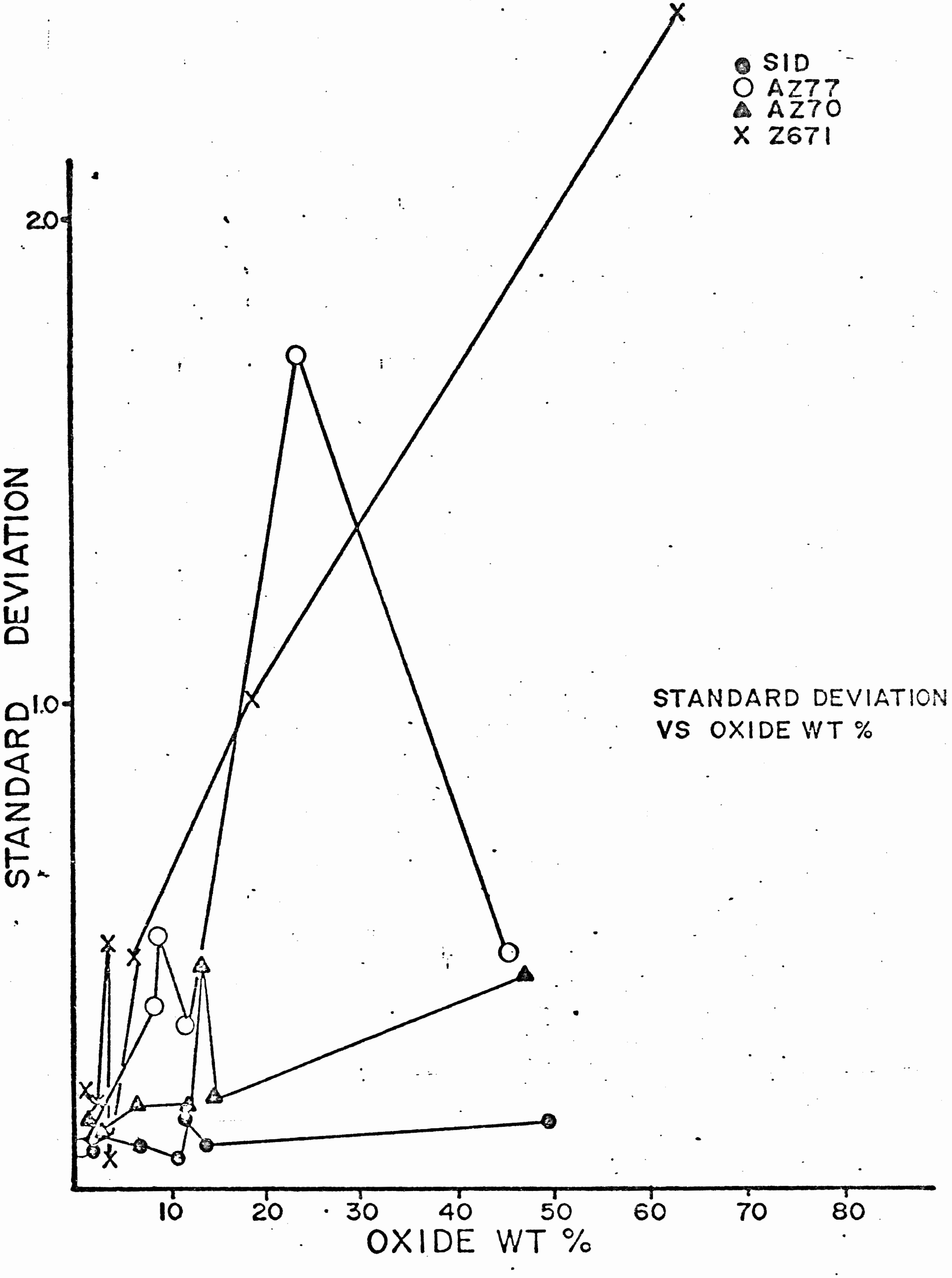
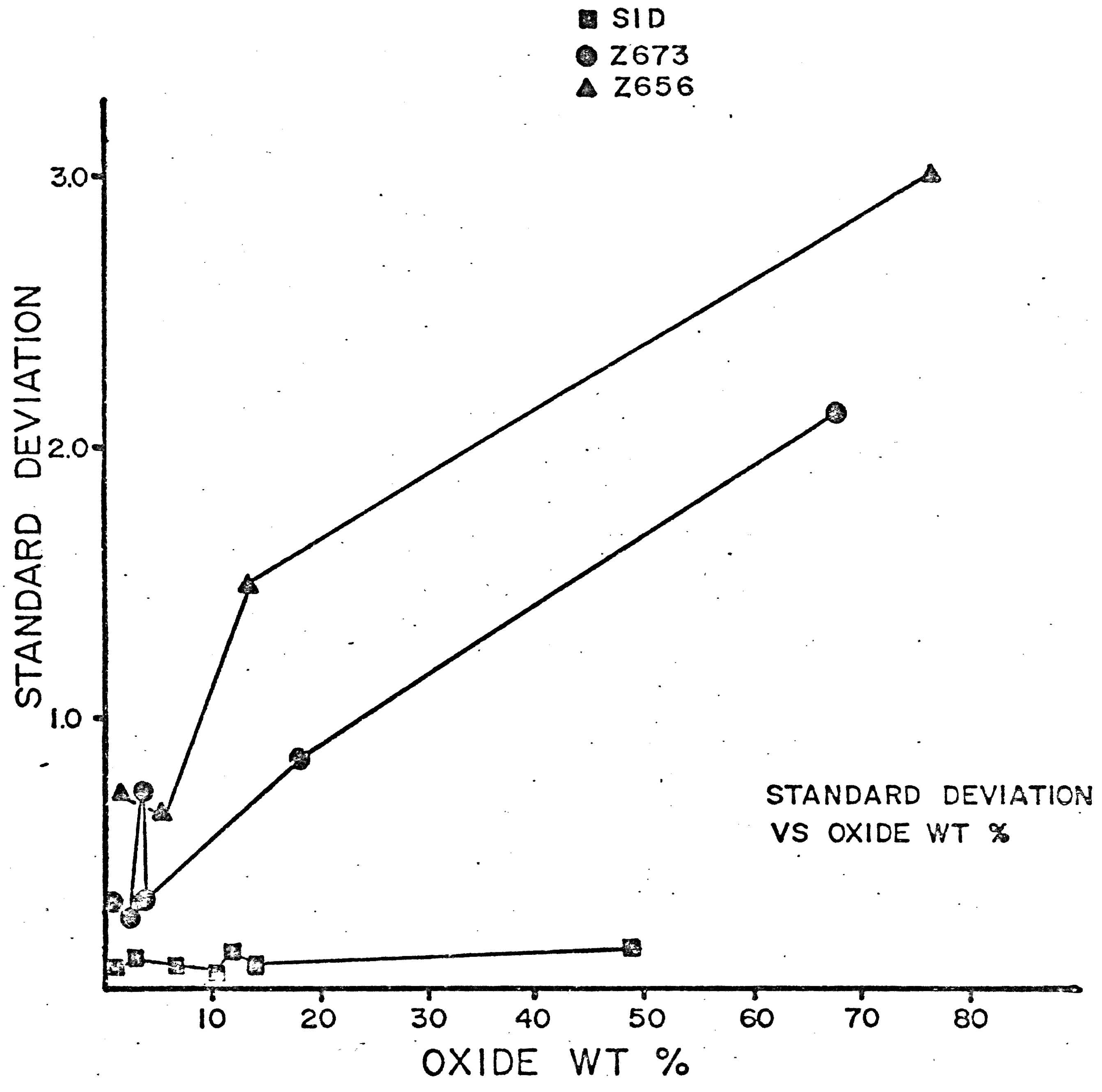

● SID
○ AZ77
▲ AZ70
X Z671
STANDARD DEVIATION
2.0
1.0
10
20
30
40
50
60
70
80
OXIDE WT %
STANDARD DEVIATION
VS OXIDE WT %

FIG. 4-3

Fig. 4-4. Plot of the standard deviation vs the oxide weight percent of samples Z673 and Z656. For comparison SiD, a basaltic glass which was not melted is also shown. As the $SiO_2 + Al_2O_3$ content of the rocks increases, the standard deviation also increases, reflecting poorer precision.


■ SID
● Z673
▲ Z656
3.0
2.0
1.0
STANDARD DEVIATION
10
20
30
40
50
60
70
80
OXIDE WT %
STANDARD DEVIATION
VS OXIDE WT %

Experiment 5

Purpose

To determine the accuracy of the method used to analyze rock powders.

Apparatus

The same equipment was used as in Experiment 2. The standard rock powders used in this experiment were BCR-1, AGV-1, SY-2, JG-1 and TB.

Procedure

The procedure used in this experiment was the same as in Experiments 2 and 4. In this experiment rock standards were melted, analyzed in the electron microprobe and the results compared to sideromelane, a basaltic glass.

Observations and Discussion

Table 5-1 shows the analyses of the five melted rock samples. For each sample the mean standard deviation and the coefficient of variation is shown as well as the accepted standard value for the sample. Relative and absolute errors are also reported for each sample.

Figure 5-1 through to Figure 5-8 shows the relative error against oxide weight percent for the rock samples. Figure 5-9 is a plot of relative error vs the log of oxide weight percent for samples BCR-1 and AGV-1. It shows that greater relative errors can be expected for minor elements than for major elements.

For comparison sideromelane was analyzed 10 times on the same spot. Sideromelane is a homogeneous glass and its chemistry is well known. The data on sideromelane therefore should represent the best results that can be achieved with the electron microprobe using the energy dispersive system.

In order that all analyses could be compared with each other and to assure uniformity, all samples were analyzed using KK as the standard. For samples BCR-1, AGV-1 and Sy-2, values for major elements compare favorably with listed book values. For samples JG-1 and TB, results compare poorly with listed values. Both samples, however, have a high $SiO_2 + Al_2O_3$ content and were difficult to melt,which resulted in an inhomogeneous glass. This is reflected in the high coefficients of variations for these two samples.

Conclusion

Rocks with a $SiO_2 + Al_2O_3$ content greater than 80% are difficult to melt and produce glasses that are inhomogeneous to various degrees. Good accuracy, however, can be obtained with this method from rocks with a $SiO_2 + Al_2O_3$ content which ranges between 60% to 80%. In these rocks the homogeneity of the melt is improved by the presence of CaO and increased amounts of MgO and FeO, which behave as natural fluxes.

For samples BCR-1 and AGV-1 the relative accuracy for most of the major elements (greater than 10 wt. %) is better than 3%. For minor elements (between 1% and 10%) the relative error is better than 5% for most of the elements analysed.

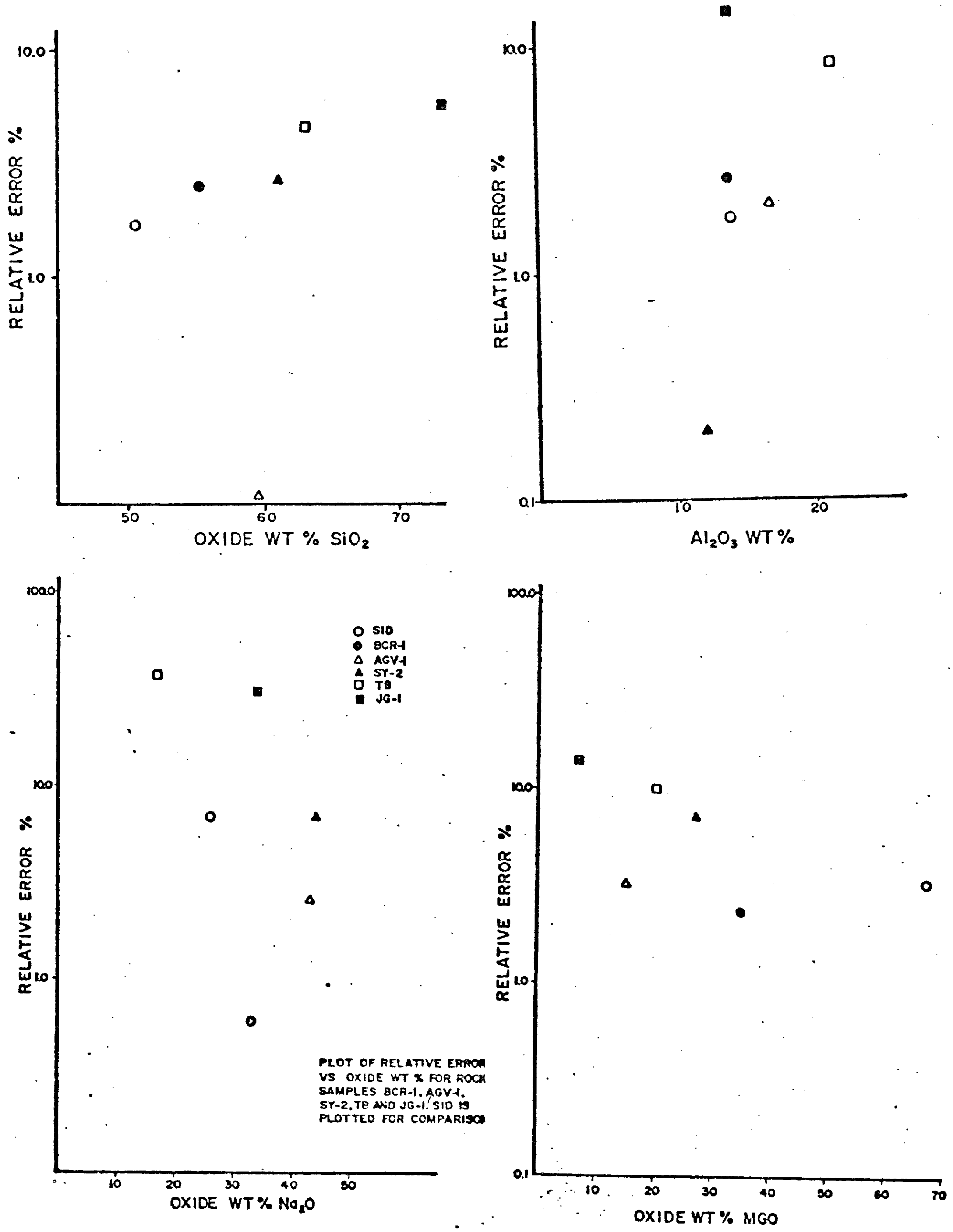
In general for rocks with composition between basalts and andesites the method yields a relative error better than 5%. (For SiO_2 the relative error is better than 2.5%).

All analyses were, however, compared with the Kakanui Kaersutite which is a mineral standard. This standard was used throughout the experiments to ensure uniformity and in order that analyses could be compared to each other. Improved accuracy can be expected if each rock type is compared to a suitable rock standard.

TABLE 5-1: Analyses of samples BCR-1, AGV-1, SY-2, TB and JG-1 - Accuracy test. SID included for comparison.

		Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	TiO_2	MnO	FeO
SID (10 analyses on same spot)											
	$\bar{x}$	2.80	6.93	13.82	49.94	.24	.19	10.95	1.75	.20	11.91
	s	.108	.092	.097	.144	.073	.031	.064	.08	.070	.145
	CV	3.8	1.3	0.70	0.29	30.3	16.5	0.59	4.6	35.1	1.2
Std. Value		2.62	6.71	14.06	50.81	.20	.19	11.12	1.85	.22	11.83
Relative Error %		6.9	3.3	1.7	1.7	20.0	0.0	1.5	5.4	9.1	.68
Absolute Error		.18	.22	.24	.87	.04	0.0	.17	.10	.02	.08
BCR-1 (10 analyses)											
	$\bar{x}$	3.30	3.60	13.47	54.01	.44	1.57	7.34	2.43	.18	12.75
	s	.15	.116	.178	.528	.095	.050	.15	.143	.032	.413
	CV	4.5	3.2	1.32	.98	21.7	3.1	2.0	5.9	17.8	3.2
Std. Value		3.32	3.52	13.82	55.39	.37	1.73	7.03	2.24	.18	12.31
Relative Error %		.60	2.3	2.6	2.5	18.9	9.2	4.4	8.4	0.0	3.6
Absolute Error		.02	.08	.36	1.38	.07	.16	.31	.19	0.0	.44
AGV-1 (10 Analyses)											
	$\bar{x}$	4.19	1.50	17.08	59.58	.59	2.88	5.00	1.09	.07	6.42
	s	.097	.283	.057	.841	.218	.081	.173	.312	.061	.83
	CV	2.3	18.9	2.97	1.41	19.9	2.8	3.5	28.6	86.5	12.3
Std. Value		4.30	1.55	17.43	59.62	.50	2.92	4.95	1.05	.10	6.17
Relative Error %		2.6	3.2	2.0	.07	18.0	1.4	1.0	3.8	23.0	4.1
Absolute Error		.11	.05	.35	.04	.09	.04	.05	.04	.03	.25
SY-2 (10 analyses)											
	$\bar{x}$	4.12	2.54	12.37	59.45	.47	4.28	8.12	.09	.34	5.65
	s	.187	.292	.733	.738	.108	.338	.788	.075	.075	.579
	CV	4.6	11.5	5.9	1.24	23.0	7.9	9.7	83.8	21.9	10.24
Std. Value		4.42	2.73	12.35	61.09	.45	4.59	8.13	.15	.33	5.75
Relative Error %		6.8	7.0	.20	2.7	4.4	6.8	.10	40.0	3.0	1.7
Absolute Error		.30	.19	.02	1.64	.02	.31	.01	.06	.01	.10
TB (10 analyses)											
	$\bar{x}$	.85	2.23	23.28	60.35	.16	3.60	.17	.95	.05	6.79
	s	.078	.24	2.43	3.69	.094	.245	.046	.189	.038	.997
	CV	9.26	10.9	10.4	6.1	58.7	6.8	27.3	19.9	76.0	14.7
Std. Value		1.37	2.02	21.49	63.07	.11	4.02	.35	.97	.05	6.54
Relative Error %		38.0	10.3	8.3	4.3	45.0	10.4	51.0	2.0	0.0	3.8
Absolute Error		.52	.21	1.8	2.7	.05	.42	.18	.02	0.0	.15
JG-1 (11 analyses)											
	$\bar{x}$	2.38	.82	16.32	69.10	.11	4.02	2.57	.28	.07	2.02
	s	.771	.186	3.19	5.58	.081	.664	.825	.095	.049	.465
	CV	32.4	22.8	19.5	8.1	73.7	16.5	33.9	33.9	70.3	23.1
Std. Value		3.42	.77	14.32	72.98	.09	3.99	2.19	.27	.06	1.97
Relative Error %		30.0	14.0	14.0	5.3	22.0	.75	17.4	3.7	16.6	2.5
Absolute Error		1.05	.05	2.0	3.9	.02	.03	.38	.01	.01	.05

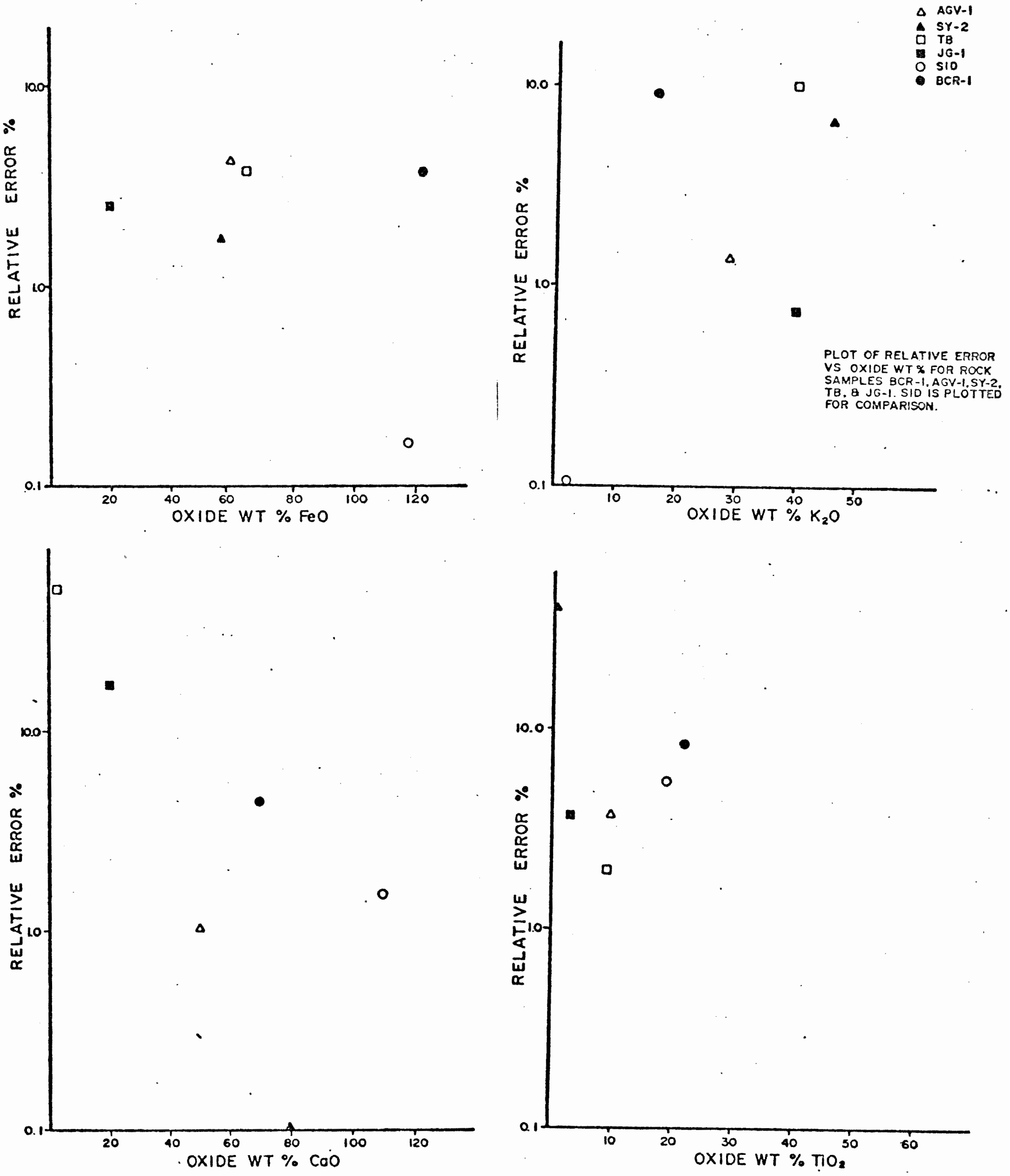
Fig. 5-1, 5-2, 5-3 and 5-4. Plot of relative errors against oxide weight percent for the rock samples. The diagrams show relative errors for SiO_2, Al_2O_3, Na_2O and MgO. Relative errors are higher for minor elements than for major elements.



PLOT OF RELATIVE ERROR VS OXIDE WT % FOR ROCK SAMPLES BCR-1, AGV-1, SY-2, TB AND JG-1. SID IS PLOTTED FOR COMPARISON

FIGS. 5-1, 5-2, 5-3, 5-4

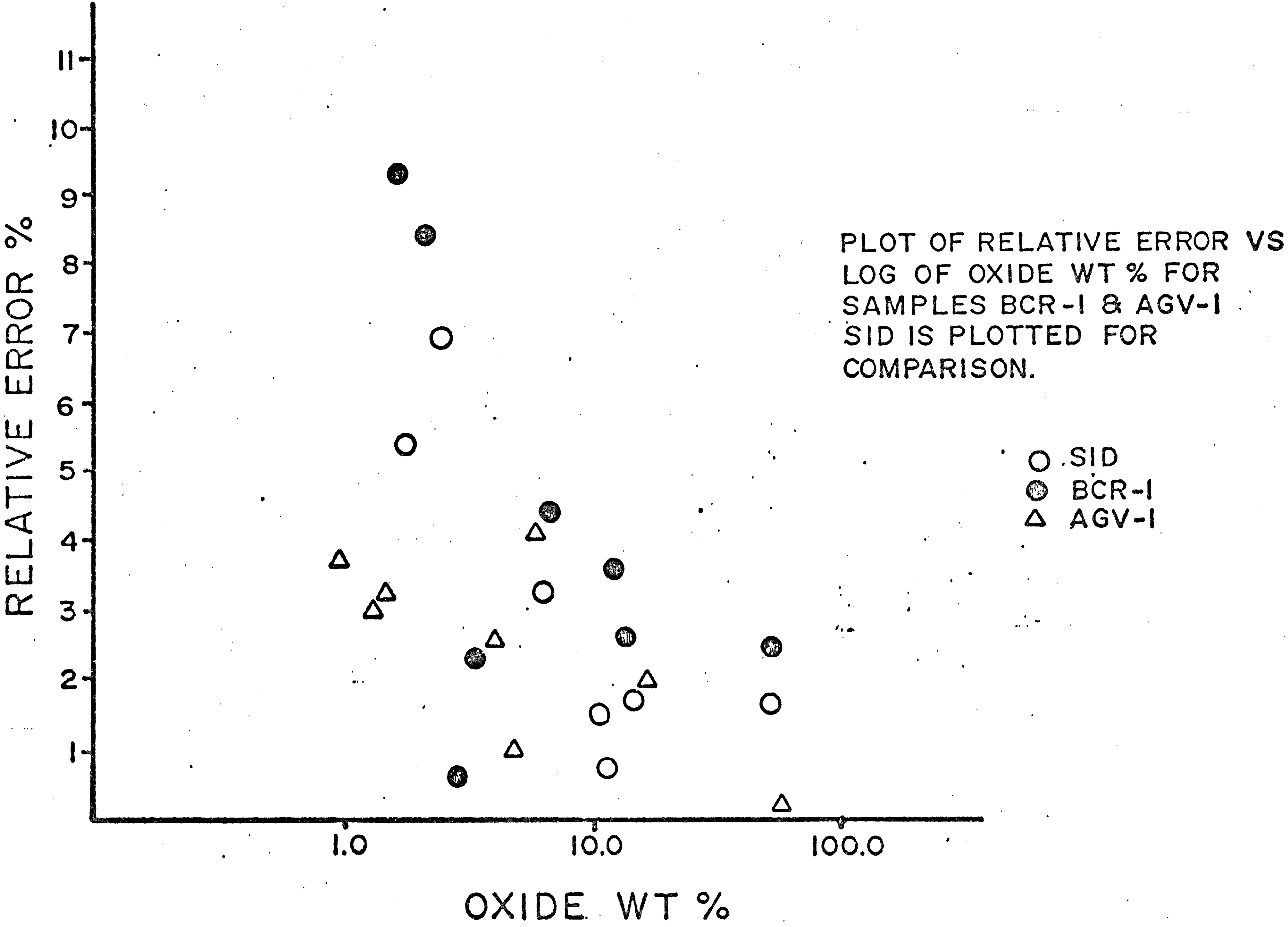
Fig. 5-5, 5-6, 5-7 and 5-8. Plot of relative errors against oxide weight percent for the rock samples. The diagrams show relative errors for FeO, K_2O, CaO and TiO_2. The relative errors are generally high for these minor elements compared to the major elements.



PLOT OF RELATIVE ERROR VS OXIDE WT % FOR ROCK SAMPLES BCR-1, AGV-1, SY-2, TB, & JG-1. SID IS PLOTTED FOR COMPARISON.

FIG. 5-5, 5-6, 5-7, 5-8

Fig. 5-9. Plot of relative error vs the log of oxide weight percent for samples BCR-1 and AGV-1. Greater relative errors exist for minor elements than for major elements. For comparison SiD, a basaltic glass which was not melted, is also shown. Melted samples BCR-1 and AGV-1 compare favorably with SiD.



PLOT OF RELATIVE ERROR VS LOG OF OXIDE WT % FOR SAMPLES BCR-1 & AGV-1 SID IS PLOTTED FOR COMPARISON.

FIG. 5-9

Experiment 6

Purpose

To determine the effects on sodium and potassium, two potentially volatile elements, with increasing melt times for the rock powders.

Apparatus

The same equipment used in this experiment was used in Experiment 2. Sample E2 was used as the rock powder and all melts were made with a Tantalum strip.

Procedure

Generally, the procedure followed was the same as in Experiment 2. Sample E2 was melted on a tantalum strip under two conditions, first under a vacuum and secondly in a nitrogen atmosphere. Several runs were made with each sample held at the melt temperatures for various times. Glasses produced from the melt were then analyzed in the electron microprobe to determine the values of sodium and potassium.

Observations and Discussion

Table 6-1 shows the analyses for sodium and potassium vs melt times. Figure 6-1 and Figure 6-2 show plots of the data from Table 6-1. Except for the measured values of potassium from melts in nitrogen gas, all the plots show a decrease in measured values as melt times are increased.

Conclusion

From the plots it is obvious that extended melt times result in a loss of potassium and sodium. In both nitrogen gas and in a vacuum, the loss of sodium is greater than the loss of potassium. The greatest loss for both sodium and potassium is in a vacuum. Volatile loss is a minimum for both sodium and potassium in a nitrogen atmosphere with fusion times less than 30 seconds.

TABLE 6-1: Observed oxide weight percent of sodium and potassium for sample E2 with melt times.

Run #	Time	Na	K	Melted in a Vacuum
1	10 sec	7.07	6.34	Average of 4 analyses
2	20 "	6.71	6.33	Average of 4 analyses
3	30 "	7.09	6.51	Average of 4 analyses
4	45 "	6.21	6.18	Average of 12 analyses
5	60 "	6.06	5.79	Average of 4 analyses
6	90 "	5.87	6.14	Average of 12 analyses
r^2		0.80	0.35	
y intercept		7.19	6.43	
Slope		-0.02	.0049	
Equation of the line		$y = -0.02x+7.19$	$y = -.0049x + 6.43$	
				Melted in nitrogen gas
7	10 sec	7.13	6.41	Average of 5 analyses
8	20 "	7.01	6.27	Average of 5 analyses
9	30 "	6.59	6.55	Average of 5 analyses
10	45 "	7.14	6.78	Average of 5 analyses
11	60 "	6.52	6.71	Average of 5 analyses
12	90 "	6.30	6.35	Average of 5 analyses
r^2		.60	.03	
y intercept		7.18	6.46	
Slope		-.01	.0011	
Equation of the line		$y = -.01 x + 7.18$	$y = .0011 x +6.46$	

where x = melt time
and y = oxide wt %

Fig. 6-1. Plot of the measured values of Na_2O and K_2O from sample E2 vs melting times in nitrogen gas. There is a loss of Na_2O as melt time is increased. There is no apparent loss of K_2O with increased melt time.

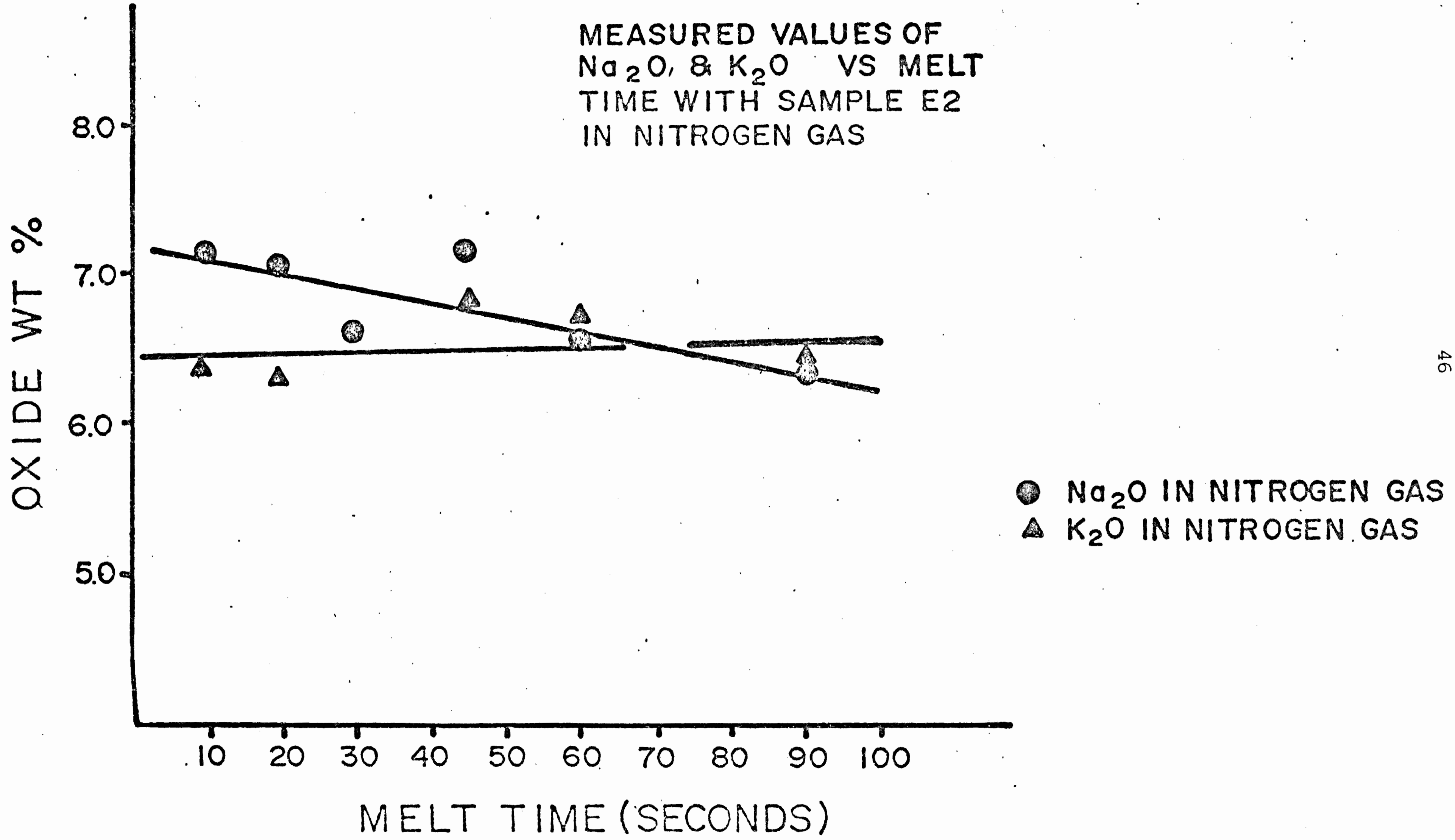

MEASURED VALUES OF
Na_2O, & K_2O VS MELT
TIME WITH SAMPLE E2
IN NITROGEN GAS
OXIDE WT %
8.0
7.0
6.0
5.0
10
20
30
40
50
60
70
80
90
100
MELT TIME (SECONDS)
● Na_2O IN NITROGEN GAS
▲ K_2O IN NITROGEN GAS

FIG. 6-1

Fig. 6-2. Plot of the measured values of Na_2O and K_2O from sample E2 vs melting times in a vacuum. Both Na_2O and K_2O show a loss as the melt time is increased.

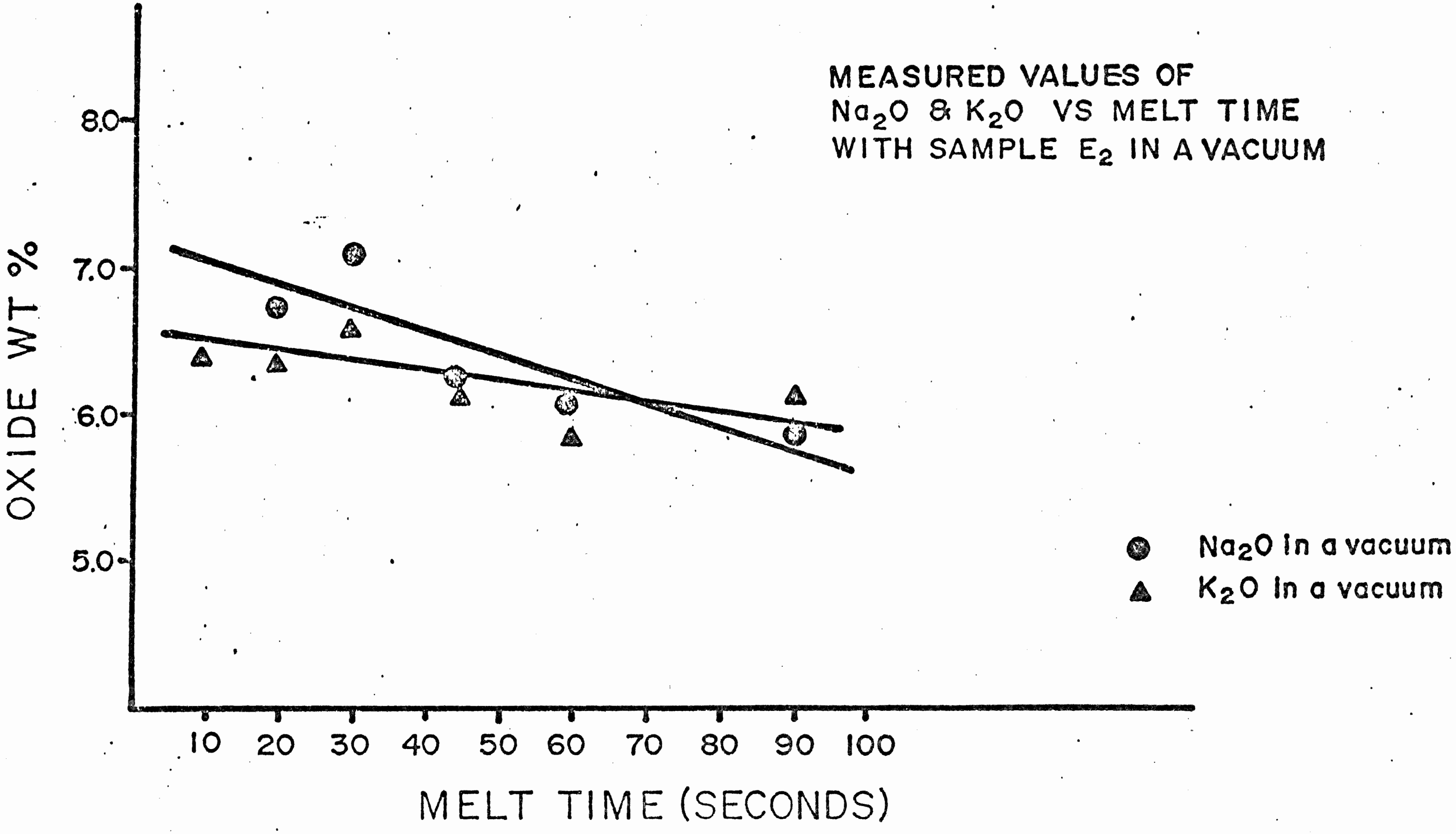

MEASURED VALUES OF
Na_2O & K_2O VS MELT TIME
WITH SAMPLE E_2 IN A VACUUM
OXIDE WT %
8.0
7.0
6.0
5.0
10
20
30
40
50
60
70
80
90
100
MELT TIME (SECONDS)
● Na_2O in a vacuum
▲ K_2O in a vacuum

FIG. 6-2

Experiment 7

Purpose

To determine the effects of adding a flux to the rock powder before melting.

Apparatus

The same equiment was used in this experiment as was used in Experiment 2. The flux used was lithium tetraborate. Sample Z656 and DPL were the rock powders used in this experiment.

Procedure

The same procedure was used in this experiment as was used in Experiment 2 and 4. In this experiment, however, only molybdenum strips were used and all melting was carried out in a nitrogen atmosphere. The DPL was melted five times and sample Z656 melted four times. The flux was added to the rock powder prior to melting.

Observations and Discussion

The analyses were re-calculated in two ways. First, analyses were calculated from the rock powder/flux ratio and secondly analyses were scaled to 100% from the new data. In order to make a uniform comparison the percent flux for the DPL was kept to about 25% whereas the percent flux for sample Z656 was varied from 9 to 33% to see if this would adversely affect the results.

Table 7-1 shows the mean, calculated weight percent and the mean scaled to 100% for five separate melts of DPL. Table 7-2 shows the average of the five melts from Table 7-1, scaled to 100%. Also shown on Table 7-2 is the standard deviation expressed as a percent of the five melts, the mean of twenty-five analyses of DPL when melted without a flux, and the accepted book value of DPL, for comparision.

Table 7-3 again shows the mean, the calculated weight percent and the mean scaled to 100% for sample Z656. Table 7-4 shows the average of the four melts from Table 7-3 along with the standard deviation and twenty-five analyses of Z656 when melted without a flux as well as the accepted book value of Z656 for comparison.

Table 7-5 shows the absolute error for both DPL and Z656 for each melt when compared to the calculated weight percent value and compared to values scaled to 100%. Again absolute errors for DPL and Z656 when melted without a flux are included for comparison. Table 7-6 is a summary of the data on Table 7-5.

Conclusion

For the DPL both methods of recalculating the data produced similar results, except that use of the rock powder/flux ratio produces better values for SiO_2, whereas the scaling method gives slightly better CaO values. The results compare favourable with the DPL analyses without a flux. This suggests that the addition of a flux does not adversely affect the results for major elements although sensitivity is reduced for minor elements. The standard deviations for the five DPL melts were expressed as a percent of the amount of the element present. For most major elements, all melts produced results within 1% of each other.

For sample Z656 recalculation of analyses from the rock powder/ flux ratio produced poor results. Again the standard deviations for the four melts were expressed as a percent age and for major elements the melts produced results within 4% of each other. This may indicate that the rock powder and flux weight measurements were in error or H_2O was lost during fusion or possibly the amount of flux present affected the results. The totals for the four melts using the rock powder/flux ratio recalculation are low.

The scaling method, however, produced excellent results when compared with the book value for sample Z656 (Table 7-4). Also the absolute error for Z656, using the scaling method, compared favorably with the absolute error of DPL without a flux (Table 7-6). This suggests that rocks with a high percent of Al_2O_3 + SiO_2 can successfully be analyzed using a flux.

TABLE 7-1: Five separate analyses of sample DPL with a flux added.

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	TiO_2	MnO	FeO	Total
Melt #1, average of 5 analyses. Percent flux = 26.4%											
$\bar{x}$	1.77	6.69	10.99	37.44	.10	.15	8.20	.80	.10	6.84	73.08
Calculated wt%	2.41	9.10	14.94	50.90	.14	.20	11.15	1.08	.14	9.30	99.36
$\bar{x}$ scaled to 100%	2.42	9.15	15.05	51.23	.13	.20	11.22	1.09	.13	9.36	99.88
Melt #2, average of 5 analyses. Percent flux = 22.85%											
$\bar{x}$	1.86	7.02	11.97	39.34	.09	.16	8.66	.86	.12	7.14	77.11
Calculated wt%	2.41	9.10	14.87	51.00	.12	.21	11.23	1.11	.16	9.26	99.47
$\bar{x}$ scaled to 100%	2.42	9.15	14.95	51.28	.11	.21	11.29	1.12	.16	9.31	100.00
Melt #3, average of 5 analyses. Percent flux = 24.44%											
$\bar{x}$	1.85	6.85	11.28	38.67	.10	.15	8.47	.84	.12	7.04	75.37
Calculated wt%	2.45	9.07	14.93	51.18	.13	.20	11.21	1.11	.16	9.32	99.73
$\bar{x}$ scaled to 100%	2.45	9.09	14.97	51.31	.13	.20	11.24	1.11	.16	9.34	100.00
Melt #4, average of 5 analyses. Percent flux = 23.68%											
$\bar{x}$	1.55	7.15	11.92	40.28	.13	.09	8.84	.86	.11	7.38	70.31
Calculated wt%	2.03	9.37	15.36	52.78	.17	.12	11.58	1.13	.14	9.67	102.35
$\bar{x}$ scaled to 100%	1.98	9.15	15.00	51.57	.16	.15	11.32	1.10	.14	9.44	100.01
Melt #5, average of 5 analyses. Percent flux = 23.81%											
$\bar{x}$	1.84	6.88	11.36	38.85	.11	.15	8.62	.82	.09	7.15	75.87
Calculated wt%	2.42	9.03	14.91	51.00	.14	.20	11.31	1.08	.12	9.38	99.59
$\bar{x}$ scaled to 100%	2.43	9.07	14.97	51.21	.14	.20	11.36	1.08	.12	9.42	100.00

TABLE 7-2: Average of the five DPL melts from Table 7-1 compared to DPL without a flux.

DPL - AVERAGE OF THE FIVE MELTS - scaled to 100%

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	Ca_2O	TiO	MnO	FeO	TOTAL
$\bar{x}$	2.34	9.12	14.99	51.32	.13	.19	11.29	1.10	.14	9.37	99.99
S	.20	.04	.04	.15	.02	.02	.06	.02	.02	.05	

Standard Deviation expressed as a percent of the amount of the element present

	8.5	.44	.27	.29	15.4	10.2	.53	1.8	14.3	.53	

DPL without a flux, average of 25 analyses

$\bar{x}$	2.24	9.09	15.13	51.77	.13	.20	11.46	1.03	.17	9.17	100.39

accepted

DPL Accepted Value

$\bar{x}$	2.32	9.39	14.85	50.81	.12	.35	11.37	1.12	.18	9.54	100.05

TABLE 7-3: Four separate analyses of Z656 with a flux added.

SAMPLE Z 656	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	TiO_2	MnO	FeO	Total
Melt #1, average of 6 analyses, Percent flux = 9.09%											
$\bar{x}$	3.14	.17	12.10	63.74		4.58	.78			.51	85.02
Calculated wt%	3.45	.18	13.31	70.11		5.04	.86			.56	93.51
$\bar{x}$ scaled to 100%	3.69	.20	14.32	74.87		5.39	.92			.60	100.09
Melt #2, average of 6 analyses, Percent flux = 16.67%											
$\bar{x}$	2.99	.16	10.86	59.93		4.30	.73			.49	79.46
Calculated wt%	3.59	.19	13.03	71.92		5.16	.88			.59	95.36
$\bar{x}$ scaled to 100%	3.76	.20	13.68	75.47		5.14	.92			.55	99.99
Melt #3, average of 6 analyses, Percent flux = 24.32%											
$\bar{x}$	2.56	.08	10.03	56.22		3.73	.60			.41	73.63
Calculated wt%	3.38	.11	13.25	74.29		4.92	.79			.54	97.28
$\bar{x}$ scaled to 100%	3.48	.10	13.62	76.35		5.07	.81			.57	100.00
Melt #4, average of 7 analyses, Percent flux = 33.33											
$\bar{x}$	2.28	.10	8.64	47.78		3.17	.48			.27	62.72
Calculated wt%	3.42	.15	12.96	71.67		4.76	.72			.40	94.08
$\bar{x}$ scaled to 100%	3.64	.16	13.78	76.18		5.05	.76			.43	100.00

TABLE 7-4: Average of the four Z656 melts from Table 7-3 compared to Z656 without a flux.

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	TiO_2	MnO	FeO	Total
Z656 average of the four melts, scaled to 100%											
$\bar{x}$	3.64	.17	13.85	75.74		5.23	.85			.54	100.02
S	.12	.04	.32	.64		.19	.08			.07	
Standard Deviation expressed as a percent of the amount of the element present											
	3.3	23.5	2.3	.84		3.6	9.4			12.9	
Z656 without a flux, average of 25 analyses											
$\bar{x}$	1.93	.21	13.74	77.44	.05	5.65	.79			.44	100.25
Z656 Book value											
$\bar{x}$	3.48	.41	13.80	75.57	.06	5.11	.77	.11	.02	.63	99.96

TABLE 7-5: Absolute errors for samples DPL and Z656. Errors shown for analyses scaled to 100% and calculated from weight percent.

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	TiO_2	MnO	FeO
Absolute Error - calculated wt% to book value, DPL										
Melt 1	+ .09	- .29	+ .09	+ .09	- .02	- .15	- .22	- .04	- .04	- .24
Melt 2	+ .09	- .29	.02	.19	0.0	- .14	- .14	- .01	- .02	- .28
Melt 3	.13	- .32	.08	.37	.01	- .15	- .16	- .01	- .02	- .22
Melt 4	- .29	- .02	.51	1.97	.05	- .18	.21	.01	- .04	.13
Melt 5	.10	- .36	.06	.19	.02	- .15	- .06	- .04	- .06	- .16
DPL without a flux	- .08	- .30	.28	.96	.01	- .15	.09	- .09	- .01	- .37
Absolute Error - scaled to 100% value vs book value DPL										
Melt 1	.1	- .24	.20	.42	.01	- .15	- .15	- .03	- .05	- .18
Melt 2	.1	- .24	.10	.47	- .01	- .14	- .08	0.0	- .02	- .23
Melt 3	.13	- .30	.12	.50	.01	- .15	- .13	- .01	- .02	- .20
Melt 4	.32	- .24	.15	.76	.04	- .20	- .05	- .02	- .04	- .10
Melt 5	.11	- .32	.12	.40	.02	- .15	- .01	- .04	- .06	- .12
Absolute Error - calculated wt% to book value, Z656										
Melt 1	.03	- .23	- .49	-5.46		- .07	.01			- .07
Melt 2	.11	- .22	- .77	-3.65		.05	- .04			- .04
Melt 3	- .10	- .30	- .55	-1.28		- .19	- .17			- .09
Melt 4	- .06	- .26	- .84	-3.81		- .35	- .05			- .23
Z656 without a flux	-1.55	- .20	-0.6	-1.13		.54	.02			- .19
Absolute Error - scaled to 100% value vs. book value Z656										
Melt 1	.21	- .21	.52	- .60		.28	.15			- .03
Melt 2	.28	- .21	- .12	- .10		.30	.15			- .08
Melt 3	0.0	- .31	- .18	.78		- .04	.04			- .06
Melt 4	.16	- .25	- .02	.61		- .06	- .01			- .20

TABLE 7-6: Summary of absolute errors from Table 7-5.

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	TiO_2	MnO	FeO
DPL average of five melts - absolute error calculated wt% and compared to book values	.12	.25	.15	.56	.02	.15	.15	.02	.04	.20
DPL average of five melts - absolute error scaled to 100% value and compared to book value	.15	.26	.14	.51	.02	.15	.08	.02	.04	.16
DPL without a flux - absolute error, average of 25 analyses	.08	.30	.28	.96	.01	.15	.09	.09	.01	.37
Z656 average of four melts - absolute error calculated wt% and compared to book value	.05	.25	.66	3.55		.18	.07			.11
Z656 average of four melts - absolute error scaled to 100% and compared to book value	.16	.24	.21	.52		.17	.09			.09
Z656 without a flux - absolute error, average of 25 analyses	1.55	.20	.06	1.13		.54	.02			.19

Experiment 8

Purpose

To determine the accuracy and precision of the method when a flux is added to the rock powder.

Apparatus

The same equipment was used as in Experiment 2. The flux used was lithium tetraborate and the rock powders were samples NIM-D, TB, Z656 G2 and JG-1.

Procedure

The same basic procedure was followed in this experiment as was used in Experiment 7. For the accuracy test, samples NIM-D, TB, Z656, G2 and JG-1 were melted once. For the precision test JG-1 was melted 6 times.

Observations and Discussion

By using a flux, rocks with a high $Al_2O_3 + SiO_2$ content or rocks with a ultramafic composition can more easily be melted with this technique. Sample NIM-D is a dunite and was used in this experiment because peridotites tend to crystallize when quenched rather than forming a glass when melted without a flux. The other samples were used in this experiment because of their high $Al_2O_3 + SiO_2$ content.

Table 8-1 shows the mean, standard deviation, coefficient of variation and absolute error for six melts of sample JG-1. Various rock powder/flux ratios were used to determine if the amount of flux affected the melt in any way.

Optically all glasses appeared to be homogeneous. The coefficient of variation remained about the same as melts for similar rocks when a flux was not used. It therefore appears that the addition of a flux does not necessarily result in a homogeneous melt. Possibly this can be improved by grinding the rock to a finer powder.

The standard deviation as a percent of the element present (Table 8-2) is not as good for JG-1 as it was for DPL. The results however are biased somewhat in favor of DPL because only two elements make up over 87% of the total composition in sample JG-1. When absolute errors are compared between these two samples, the results - except for Al_2O_3 - look compatible.

Figure 8-1 shows the average standard deviation for the six melts of JG-1. For most of the elements the standard deviation compares favorably with the standard deviation for DPL. This suggests that good precision can be achieved using a flux despite inhomogeneous melts.

Table 8-3 shows the mean, corrected value, absolute and relative errors for the five samples. In Figures 8-2 and 8-3 the oxide weight % of the samples is plotted vs the relative percent error. Despite poor homogeneity the accuracy is good for all samples but the absolute error, for K_2O is somewhat high in samples Z656, G2 and JG-1. The relative accuracy is better than 3% for most of the major elements. For SiO_2

the relative accuracy is better than 1.5% for all samples. Figure 8-4 shows the improvement of absolute error for samples TB and JG-1 when compared with unfluxed melts.

Conclusion

Good precision and accuracy can be obtained with this method on ultramafic rocks and rocks with a high $Al_2O_3 + SiO_2$ content when a flux is added to the rock powder. Too much flux tends to reduce sensitivity; therefore the flux added to the rock powder should be in the range of 25 to 40% of the total. In order that a uniform comparison could be made, one standard was used through the experiment. The standard (KK) was a mineral standard. The use of suitable rock standards should improve the accuracy of the minor elements.

TABLE 8-1: Six separate analyses of sample JG-1 with a flux added - Precision Test.

		Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	TiO_2	MnO	FeO	Wt% Flux
Melt #1	$\bar{x}$	3.52	.79	15.42	71.99	.06	3.70	2.23	.29	.04	1.95	19.2
	S	.21	.128	.465	2.14		.232	.113	.109		.251	
	CV	7.2	19.7	3.7	3.6		7.2	6.1	45.0		1.6	
Absolute Error		.10	.02	1.1	.99	.03	.29	.04	.02	.02	.02	
Melt #2	$\bar{x}$	3.09	.85	16.14	72.07	.06	3.82	2.08	.18	.06	1.66	37.5
	S	.207	.073	.508	3.07		.113	.138	.05		.133	
	CV	9.9	12.8	4.7	6.3		4.4	9.9	41.9		11.9	
Absolute Error		.33	.08	1.8	.91	.03	.17	.11	.09	.02	.31	
Melt #3	$\bar{x}$	4.03	.92	15.48	70.47		4.17	2.46	.21		2.23	42.9
	S	.114	.074	.324	1.52		.133	.071			.089	
	CV	5.02	14.2	3.7	3.83		5.6	5.1			7.1	
Absolute Error		.04	.15	1.2	2.5		.18	.27	.06		.26	
Melt #4	$\bar{x}$	3.60	.92	15.35	72.28		3.62	2.16	.19		1.76	50.0
	S	.156	.039	.442	2.28		.091	.086			.047	
	CV	8.3	8.1	5.5	6.0		4.8	7.6			5.1	
Absolute Error		.18	.15	1.0	.70		.37	.03	.08		.21	
Melt #5	$\bar{x}$	3.40	.86	15.19	73.75		2.78	2.14			1.77	55.5
	S	.154	.053	.415	1.57		.139	.039			.05	
	CV	9.2	12.5	5.6	4.3		10.3	3.7			5.8	
Absolute Error		.02	.09	.23	.77		1.2	.05			.20	
Melt #6	$\bar{x}$	3.64	.86	15.28	73.14		3.09	2.18			1.69	66.6
	S	.068	.073	.550	1.92		.175	.018			.055	
	CV	5.3	23.4	9.9	7.3		15.6	2.3			9.0	
Absolute Error		.22	.09	.96	.16		.90	.01			.28	

TABLE 8-2: Average of the six JG-1 melts from Table 8-1 compared to sample DPL without a flux.

	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	TiO_2	MnO	FeO
Average for the 6 melts JG-1										
$\bar{x}$	3.55	.87	15.48	72.28		3.53	2.21			1.84
S	.31	.05	.34	1.1		.51	.13			.21
Standard Deviation as a percent of the element present JG-1										
	8.6	5.7	2.2	1.5		14.4	4.8			11.4
Standard Deviation as a percent of the element present for DPL without a flux										
	4.2	1.5	.87	.46		9.5	1.3			2.1
Average Absolute Error for 6 melts of JG-1										
	.14	.10	1.0	1.0		.52	.08			.21
Absolute Error for DPL without a flux										
	.08	.30	.28	.96		.15	.09			.37

TABLE 8-3: Analyses of samples NIM-D, TB, Z656, G2 and JG-1 melted with a flux - accuracy test.

		Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	K_2O	CaO	TiO_2	MnO	FeO	Wt% Flux
Sample NIM-D	- average of 6 analyses											
	$\bar{x}$	.15	34.23	.30	30.47			.21		.18	11.86	37.5
Corrected to 100%		.19	44.22	.38	39.36			.27		.23	15.32	
	S	.043	.164	.05	.134			.023		.044	.060	
	CV	28.5	.48	17.0	.44			10.7		24.0	.51	
Absolute Error		.14	.07	.06	.05			.01		.01	.16	
Relative Error			.16	18.7	.13						1.0	
Sample TB	- average of 6 analyses											
	$\bar{x}$	.73	1.09	10.83	31.34		7.94	.12	.43		3.19	50%
Corrected to 100%		1.56	2.19	21.76	62.98		3.90	.24	.86		6.41	
	S	.102	.051	.309	2.88		.052	.021	.065		.11	
	CV	13.2	4.69	2.85	9.19		2.67	18.0	15.2		3.48	
Absolute Error		.19	.17	.27	.09		.12	.11	.11		.13	
Relative Error		13.9	8.4	1.3	.14		2.9		11.3		1.9	
Sample Z656	- average of 6 analyses											
	$\bar{x}$	2.00	.22	7.81	43.44		2.65	.41			.28	43.9
Corrected to 100%		3.52	.39	13.74	76.41		4.66	.72			.49	
	S	.137	.032	.243	1.40		.209	.025			.057	
	CV	6.9	14.5	3.1	3.2		7.9	6.1			20.2	
Absolute Error		.04	.02	.06	.84		.45	.05			.14	
Relative Error		1.1	4.8	.43	1.1		8.8	6.5			22.2	
Sample G2	- average of 10 analyses											
	$\bar{x}$	2.48	.54	9.80	44.09	.10	2.36	1.19	.33	.02	1.48	45.0
Corrected to 100%		3.97	.86	15.71	70.67	.16	3.78	1.91	.52	.03	2.37	
	S	.126	.066	.343	1.40	.068	.228	.074	.128		.11	
	CV	5.1	12.2	3.5	3.2	67.6	9.7	6.2	38.9		7.7	
Absolute Error		.14	.09	.15	.85	.02	.78	.05	.01	0.0	.01	
Relative Error		3.4	11.6	.96	1.2	14.2	17.1	2.5	1.9		.42	
Sample JG-1												
	$\bar{x}$	2.91	.65	12.72	59.37		3.05	1.84	.24		1.61	
Corrected to 100%		3.52	.79	15.42	71.99		3.70	2.23	.29		1.95	19.2
	S	.21	.128	.465	2.14		.232	.113	.109		.251	
	CV	7.2	19.7	3.7	3.6		7.6	6.1	45.0		1.6	
Absolute Error		.10	.02	1.1	.99		.29	.04	.02		.02	
Relative Error		2.9	2.5	7.6	1.4		7.2	1.8	7.4		1.0	

Fig. 8-1. Plot of the average standard deviation for six melts of sample JG-1 against the oxide weight percent. For comparison DPL, which was melted with no flux is also shown. The precision for sample JG-1 in general compares favorably with sample DPL.

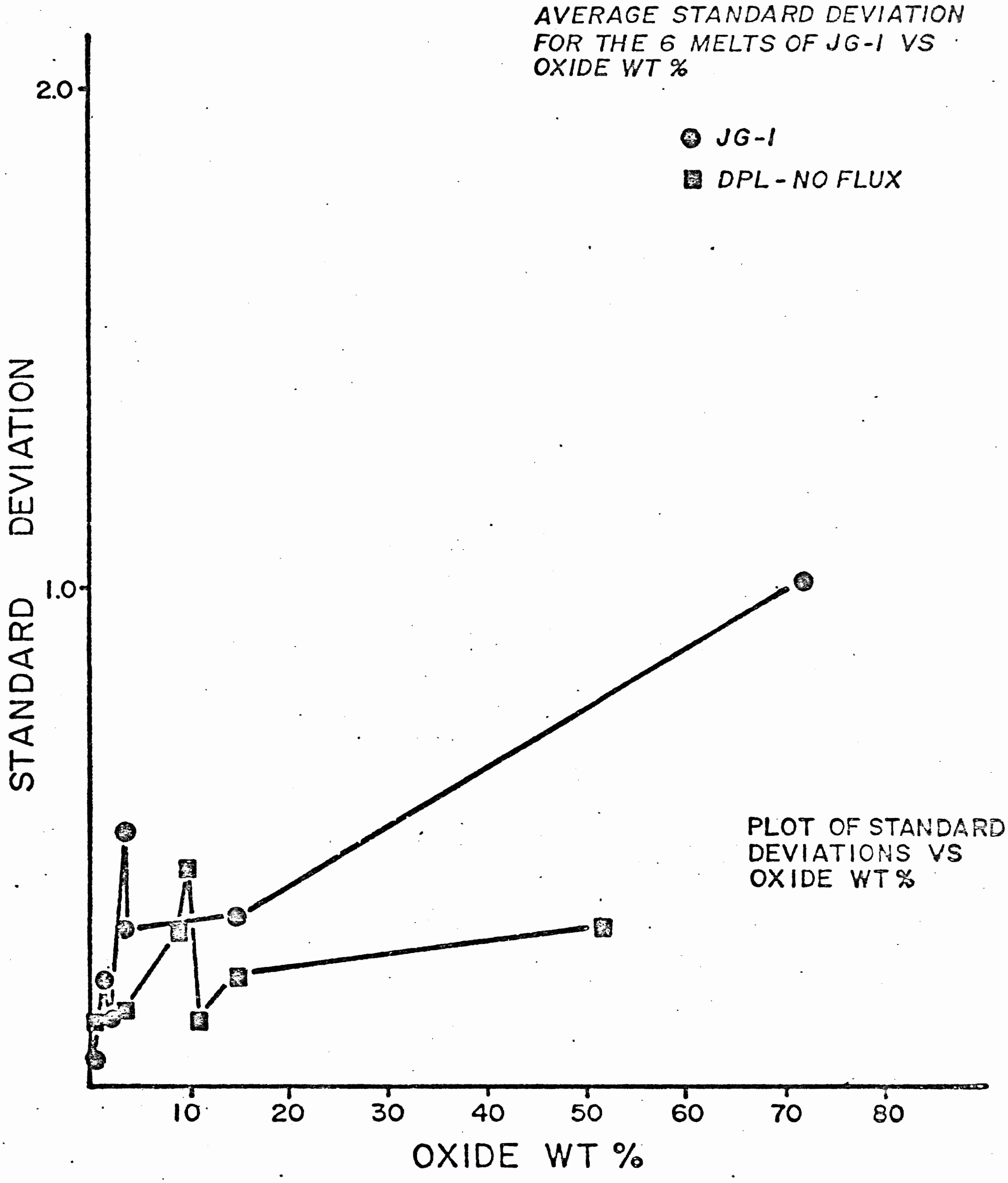

AVERAGE STANDARD DEVIATION FOR THE 6 MELTS OF JG-1 VS OXIDE WT %
● JG-1
■ DPL - NO FLUX
2.0
1.0
STANDARD DEVIATION
PLOT OF STANDARD DEVIATIONS VS OXIDE WT %
10
20
30
40
50
60
70
80
OXIDE WT %

FIG. 8-1

Fig. 8-2. Plot of relative error for samples JG-1, G2, Z656, TB and NIM-D against oxide weight percent. Relative errors are generally poorer for minor elements than for major elements.

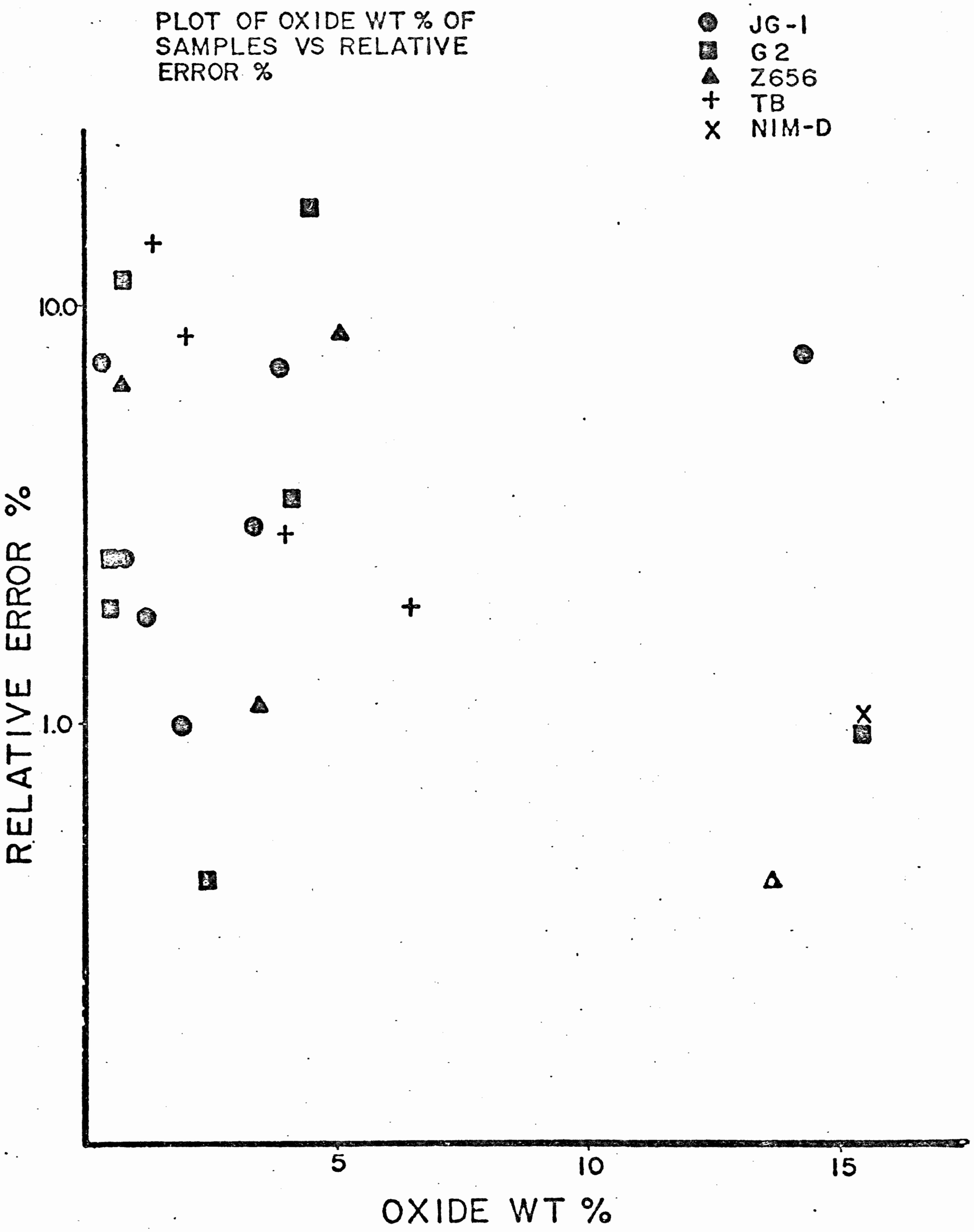

PLOT OF OXIDE WT % OF SAMPLES VS RELATIVE ERROR %
● JG-1
■ G2
▲ Z656
+ TB
X NIM-D
RELATIVE ERROR %
10.0
1.0
5
10
15
OXIDE WT %

FIG. 8-2.

Fig. 8-3. Plot of relative error for samples JG-1, G2, Z656, TB and NIM-D against oxide weight percent. Generally relative errors are better for major elements than for minor elements.

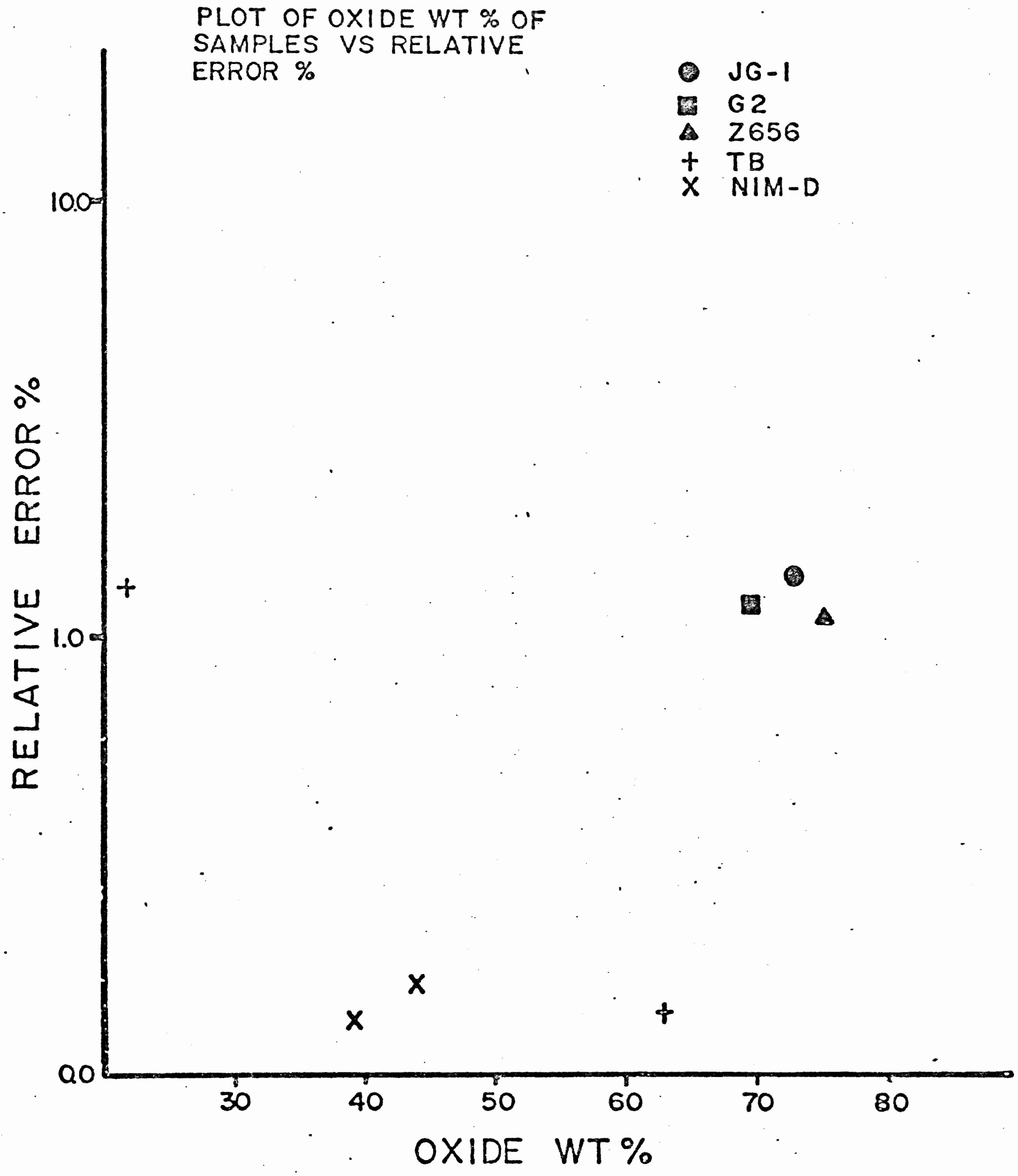

PLOT OF OXIDE WT % OF SAMPLES VS RELATIVE ERROR %
● JG-1
■ G2
▲ Z656
+ TB
X NIM-D
10.0
1.0
0.0
RELATIVE ERROR %
30
40
50
60
70
80
OXIDE WT %

FIG. 8-3

Fig. 8-4. Plot of the absolute error for samples TB and JG-1 against oxide weight percent. Shown are plots for both unfluxed and fluxed melts. The diagram shows improved absolute errors when a flux is added to the samples.

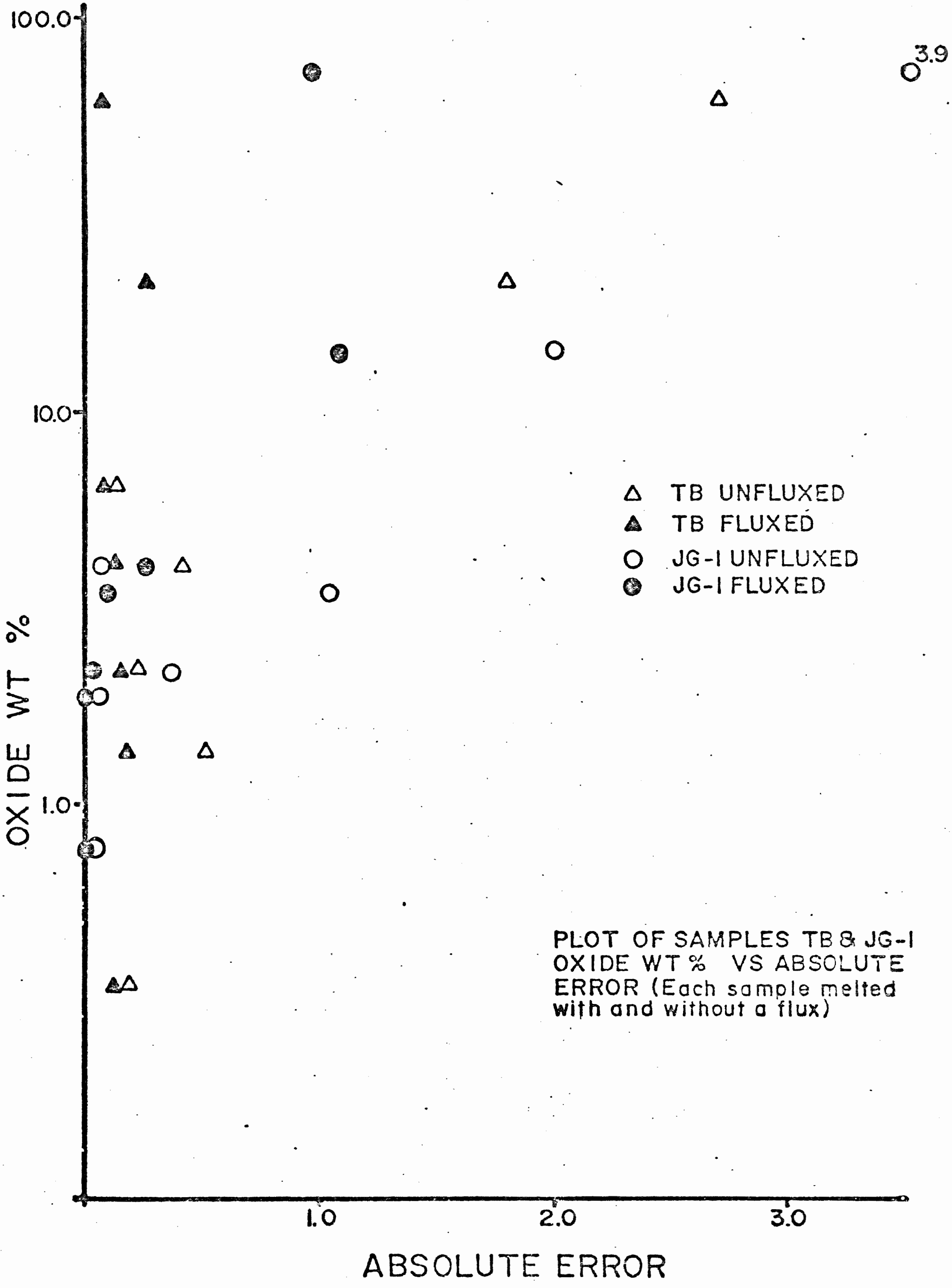

100.0
10.0
1.0
OXIDE WT %
1.0
2.0
3.0
ABSOLUTE ERROR
3.9
△ TB UNFLUXED
▲ TB FLUXED
○ JG-I UNFLUXED
● JG-I FLUXED
PLOT OF SAMPLES TB & JG-I OXIDE WT % VS ABSOLUTE ERROR (Each sample melted with and without a flux)

FIG. 8-4

Experiment 9

Purpose

To investigate the effect of structural H_2O on the scaling correction using a flux.

Apparatus

The same equipment was used as in Experiment 2. For the purpose of the investigation a mica was used.

Procedure

The same procedure was used in this experiment as was used in Experiment 7. The mica was melted seven times, twice without a flux and five times with a flux. Analyses were then carried out on the unmelted mica, on the melted mica without a flux and on the melted mica with a flux.

Observations and Discussion

Table 9-1 shows a normal microprobe analysis of the mica (analysis #1), the same analysis scaled to 100% (analysis #2), two analyses of the mica when melted without a flux (analysis #3 and 4), and five analyses of the mica when melted with a flux (analyses #5, 8, 11, 14 and 17). Also shown are the five fluxed melts corrected for the weight percent flux present (analyses #6, 9, 12, 15 and 18) and the five fluxed melts scaled to 100% volatile free (analyses #7, 10, 13, 16 and 19).

The weight percent flux corrected analyses are erratic which may indicate some retention of structural water in the sample. The addition of a flux lowers the required melting temperature so it is possible that some structural water may be retained. This suggests that it is better to scale fluxed analyses to 100% rather than to correct for the weight percent of flux present.

Conclusions

The analyses in which the mica was melted with a flux and the data scaled to 100% (analyses 7, 10, 13, 16 and 18) shows excellent agreement for major elements with the unmelted mica analysis and the mica melted without a flux (analyses 2, 3 and 4).

The analyses in which the mica was melted with a flux and the data corrected for the flux weight percent (analyses 6, 9, 12, 15 and 18) only sometimes show good agreement with the unmelted mica analyses and the mica melted without a flux (analyses, 2, 3 and 4).

This experiment has demonstrated that:

1) It is reasonable to assume the difference between the element total (of a flux melt) and 100% is due to the presence of the flux.
2) Special care must be taken if sample and flux are to be weighed.

TABLE 9-1; Mica analyses melted with and without a flux.

	1	2	3	4	5	6	7	8	9	10	11	12
SiO_2	47.11	48.73	49.45	48.59	31.77	46.60	48.31	32.82	47.40	48.36	32.37	44.78
TiO_2	.04	.04										
Al_2O_3	35.11	36.32	35.89	36.48	24.09	35.33	36.33	24.81	35.84	36.56	24.76	34.05
FeO	2.88	2.98	3.09	2.88	2.01	2.95	3.07	2.00	2.89	2.95	2.01	2.76
MnO	.02	.02										
MgO	.36	.37	.26	.31	.22	.32	.33	.21	.30	.31	.25	.34
CaO	.09	.09										
Na_2O	.34	.35	.52	.52	.42	.61	.64	.37	.53	.54	.39	.54
K_2O	10.73	11.10	10.88	11.22	7.25	10.63	11.02	7.65	11.05	11.27	7.45	10.24
	96.68	100.00	100.09	100.00	65.67	96.44	100.00	67.86	98.01	99.99	67.43	92.91

	13	14	15	16	17	18	19
SiO_2	48.30	36.24	48.32	48.68	35.54	48.87	48.50
TiO_2							
Al_2O_3	36.72	27.36	36.48	36.75	26.72	36.74	36.46
FeO	2.98	2.23	2.97	3.00	2.14	2.94	2.92
MnO							
MgO	.37	.26	.35	.34	.23	.32	.31
CaO							
Na_2O	.58	.43	.57	.57	.44	.60	.60
K_2O	11.05	7.92	10.56	10.65	8.21	11.29	11.20
	100.00	74.44	99.25	99.99	73.28	100.76	99.99

1. Mica analysis (avg. of 3 analyses)
2. Analysis #1 scaled to 100% volatile free
3. Mica glass analysis, no flux, (avg. of 5 analyses)
4. Mica glass analysis, no flux (avg. of 5 analyses)
5. Mica glass analysis with a flux (avg. of 5 analyses)
6. Analysis #5 corrected for 31.82% wt% flux
7. Analysis #5 scaled to 100% volatile free
8. Mica glass analysis with a flux (avg. of 5 analyses)
9. Analysis #8 corrected for 30.76% wt% flux
10. Analysis #8 scaled to 100% volatile free
11. Mica glass analysis with a flux (avg. of 5 analyses)
12. Analysis #11 corrected for 27.27 wt% flux
13. Analysis #11 scaled to 100% volatile free
14. Mica glass analysis with a flux (avg. of 5 analyses)
15. Analysis #14 corrected for 25.0 wt% flux
16. Analysis #14 scaled to 100% volatile free
17. Mica glass analysis with a flux (avg. of 5 analyses)
18. Analyses #17 corrected for 27.27 wt% flux
19. Analysis #17 scaled to 100% volatile free

CHAPTER 3

Summary of the Conclusions of the Experiments

(1) Little difference in the results of analyses exists when samples are melted on a molybdenum or a tantalum strip. However, if tantalum or molybdenum are present in the quenched glass the Ta $M\alpha_1$ X-ray line will interfere with the Si $K\alpha_1$ X-ray and the $Mo_{L\alpha 1}$ X-ray line will interfere with the S $K\alpha_1$ X-ray. Molybdenum is preferred, hosever, since the quenched glass tends to stick to the tantalum strip more than to the molybdenum strip.

(2) Extended melt times result in a loss of potassium and sodium from the sample. More loss occurs in a vacuum atmosphere and is most pronounced after 30 seconds. There was $\sim$ 17% drop in the measured value of Na_2O and $\sim$ 3% drop in K_2O after 90 seconds. For a nitrogen atmosphere the measured drop in Na_2O after 90 seconds was $\sim$ 10% with no appreciable drop in K_2O observed. Losses are less than 1% if melt times are kept below 30 seconds.

Melting of samples in a nitrogen atmosphere is preferred to melting in a vacuum for the above reason and also because sample powder tends to fly off strip holder when heated in a vacuum.

(3) The precision of the method used in the experiment is generally better than within 2% for all major elements except Na_2O. As the SiO_2 + Al_2O_3 content of the rock increases the temperature required to melt the

rock also increases. High $SiO_2 + Al_2O_3$ content produce inhomogeneous melts.

(4) Good accuracy can be achieved with the method used in the experiments on rocks in which the $SiO_2 + Al_2O_3$ content is between 60 and 80%. Generally the relative accuracy for elements in rocks with this range was better than 5%. The relative accuracy for SiO_2 was better than 2.5%.

(5) For rocks with a low $Al_2O_3 + SiO_2$ content the addition of a flux does not adversely effect the results for major elements whether results are calculated by wt% of flux to rock powder or scaled to 100%. Sensitivity is reduced on minor elements.

For rocks with a high $Al_2O_3 + SiO_2$ content the addition of a flux adversely affects the results if the data are recalculated by wt % of flux to rock powder. Results calculated in this manner often result in totals either too high or too low. If however, the data are scaled to 100% by assuming that the difference between the oxide totals of the elements analyzed and 100% is due to the flux content, then good results can be achieved.

(6) Good precision and accuracy on rocks with a high $SiO_2 + Al_2O_3$ content can be achieved when a flux is added to the rock powder. Best results were obtained when the flux made up ∿ 25 to 40% of the total wt % of the sample. For most major elements the relative accuracy was better than 3%. Accuracy was not good for K_2O but the use of suitable rock-glass standards should improve this. For SiO_2 the relative accuracy was

better than 1.5%.

Comparison of Analyses with other Microprobe Laboratories

Discussion

Two rock samples, BCR-1 and AGV-1 were compared with analyses from other microprobe laboratories. Each laboratory produced glasses from these rock samples using slightly different methods (see Table III-1). Results were compared with published values for BCR-1 and AGV-1 taken from Flanagan (1973) and corrected to 100% volatile free, calculated using KK and rock glasses as standards (Table III-2).

Results

The results are acceptable, being better than some laboratories while somewhat poorer than others. However, a number of variables must be considered.

(1) The type of hardware used. Some laboratories used wavelength spectrometers while others used energy dispersive systems. Better accuracy can be expected with wave length spectrometers for minor elements since the resolution is one order of magnitude better than for semiconductor detectors used in the energy dispersive system.

(2) The powder size. Rock powders ground to a finer size should be more homogeneous and give some what better results.

(3) The number of analyses and counting time. Long counting times and large number of analyses should statistically improve results.

Table III-3 shows the coefficient of variation for a number of laboratories. The coefficient of variation (CV) equals the standard deviation times 100 divided by the mean and reflects the homogeneity of the sample. From this table it can be seen that all laboratories experienced inhomogeneous samples to various degrees.

Checklist to Produce Whole Rock Analyses with the Electron Microprobe

(1) Approximately 200 mg of rock powder required. The rock should be ground to at least 100 μm in size and finer for rocks with a high $SiO_2 + Al_2O_3$ content. All material should be passed through the sieves.

(2) Rocks with a high $SiO_2 + Al_2O_3$ content (> ∿80%) or rocks with a high MgO content should be combined with 25 to 40% of Lithium tetraborate flux and thoroughly mixed by grinding prior to melting.

(3) Sample is then mounted on a molybdenum strip ∿ 5 cm x 1.5 cm in size which is clamped between two electrodes in the bell jar of the vacuum coating unit.

(4) Air is evacuated from the bell jar until a vacuum of better than 10^{-3} torr is reached. At this point nitrogen gas is slowly released into the bell jar and is allowed to flow into and out of the bell jar for the remainder of the melting process.

(5) When the bell jar is completely filled with nitrogen gas, the electric current is turned on and slowly increased through the molybdenum strip by rotating the variac clockwise.

(6) As the current is slowly increased the sample is observed (dark protective glasses are required for viewing purposes) until it just begins to melt. The electric current is then increased slightly above this point and held there for approximately 20 seconds. The sample is quenched by shutting off the electric current.

(7) The resulting glass bead is then crushed and examined optically. Homogeneous pieces located in the centre of the glass bead are then selected for microprobe analyses.

(8) The selected samples are mounted in epoxy mounts. This mount is then ground, polished and carbon coated using standard laboratory procedures for the electron microprobe.

(9) The electron microprobe is operated at 15 KV with a probe current of 5 nanoamperes. The samples are rastered over an area of 50 square microns. Several glass slivers for each sample should be analyzed. It is recommended that a total of 5 separate areas be analyzed for each sample with a counting time of 100 seconds for each analysis. The number of areas analysed should be increased if the sample proves to be very inhomogeneous.

(10) It is recommended that rock standards be chosen for the analyses. With each batch a rock of known composition should also be melted and mounted on the epoxy mount. This control should then be analysed along with the other unknown rock samples.

(11) If a flux is used it should be assumed the difference between the oxide totals and 100% is due to the flux. The analysis should be recalculated to 100% producing a volatile-free analysis. If no flux is used the analysis should not be recalculated to 100%.

TABLE III-1: Comparison of data from several microprobe laboratories.

NAME	HARDWARE	SOFTWARE	METHOD OF FUSION	POWDER SIZE	NUMBER OF ANALYSES	TIME
K. Schimann & D.G.W. Smith (1980)	ARL ORTEC (EDS)	EDATA	Optical Fusion in a partial vacuum	100 µm	2	400 s ea
Jezek, Sinton Jarosewick and Obermeyer (1979)	ALR-SEMO Wavelength Spectrometer	Bence & Albee	W & Mo strip in nitrogen gas	<150 µm	BCR-1 (29) AGV-1 (22)	10 s ea
R. Brown (1976)	ARL Wavelength Spectrometers	PB1, PB2 Bence & Albee	Mo strip in pressurized argon	∿50 µm	20	20 s ea
Rucklidge, Gibb, Fawcett & Gasparrini (1969)	ARL-EMY Wavelength Spectrometers	EMPADR 7	Graphite crucible in a platinum wound quench furnace with hydrogen		10	10 sec ea
T.A. Nicholls (1974)	TPD ORTEC (EDS)	Ware (1973)	Iridium Strip in air	<150 µm	BCR-1 (8) AGU-1 (12)	100 sec ea
R.M. MacKay	Cambridge Mark V ORTEC (EDS)	EDATA	Mo-Ta strip in nitrogen	80 µm	10	100 sec ea

TABLE III-2: Absolute error for samples BCR-1 and AGV-1 from several microprobe laboratories.

	SiO_2	TiO_2	Al_2O_3	FeO	MgO	CaO	Na_2O	K_2O
Sample BCR-1								
1	.09	.11	.08	1.23	.77	.08	.26	.05
2	.02	.14	0.0	.02	.03	.05	.01	.01
3	2.23	.09	.45	.08	.07	.18	.14	.07
4	.63	.13	.21	.32	.09	.29	.29	0.0
5	.49	.18	.04	.47	0.0	.16	.05	.04
Sample AGV-1								
1	1.25	.06	.10	.15	.58	.03	.04	.06
2	.27	.14	.01	.08	.01	.09	.01	.11
3	.45	.18	.13	.37	.12	.18	.09	.03
4	.31	.06	.20	.28	.21	.07	.10	.04
5	.61	.07	.10	.22	.07	.07	.10	.09

1 - Schimann and Smith; 2 - Jezek and et al.; 3 - R. Brown; 4 - I.A. Nicholls; 5 - R. MacKay;

TABLE III-3: Coefficient of variation for samples BCR-1 and AGV-1 from several microprobe laboratories.

	SiO_2	TiO_2	Al_2O_3	FeO	MgO	CaO	Na_2O	K_2O
BCR-1								
1	.78	4.1	2.4	2.4	4.3	1.8	2.3	2.8
2	1.5	19.6	10.0	9.3	9.6	4.1	3.5	5.7
3	.73	2.5	.71	2.4	2.8	1.4	2.8	1.2
4	.95	6.7	1.4	3.2	2.0	1.9	4.9	3.1
AGV-1								
1	.27	6.5	1.7	3.2	4.4	1.7	2.5	2.2
2	5.0	32.5	5.5	16.4	34.5	11.4	4.1	4.7
3	.33	5.4	.56	1.3	2.3	1.6	2.0	1.7
4	.92	24.3	1.1	8.2	20.3	2.7	2.5	2.0
5	2.6	7.0	4.6	6.3	6.7	3.4	11.2	3.0

1) Jazek; 2) Brown; 3) Nicholls; 4) MacKay; 5) Rucklidge

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APPENDICES

APPENDIX I

(Personal communication with Dr. Marcos Zentilli)

AZ-70	Pillow basalt, altered	(Ordovician, Argentina)
AZ-77	Pillow basalt, altered	(Ordovician, Argentina)
Z-668	Glass-rich porphyritic latite-andesite	(Miocene, Chile)
Z-671	Welded ignimbrite, dacite	(Miocene, Chile)
Z-673	Ignimbrite, non-welded, dacite	(Miocene, Chile)
Z-656	Rhyolite, glass rich; rhyodacite	(Eocene, Chile)

All above rocks were crushed first into 1-2 cm chips, using a hammer and/or a pre-contaminated jaw crusher (using part of the same samples as contaminant) and then pulverized to less than 200 mesh in a tungsten carbide shatterbox.

APPENDIX II

Table of accepted values for rock samples used throughout the experiments. All analyses calculated volatile free and for FeO only (Fe_2O_3 recalculated to FeO).

	SiO_2	TiO_2	Al_2O_3	FeO	MnO	MgO	CaO	Na_2O	K_2O	P_2O_5
AZ-77	45.76	1.25	7.67	11.83	.19	24.61	8.18	.41	.07	.08
Z-668	60.29	.79	18.52	4.46	.07	2.45	6.42	4.06	2.13	.33
Z-673	68.59	.39	16.90	2.12	.04	.83	3.61	4.48	2.68	.16
Z-656	75.57	.11	13.80	.63	.02	.41	.77	3.48	5.11	.06
Z-671	63.50	.64	17.55	3.84	.07	1.45	5.73	4.07	2.44	.32
AZ-70	47.58	2.19	14.80	13.36	.24	6.69	11.99	2.55	.27	.22
BCR-1	55.39	2.24	13.83	12.31	.18	3.52	7.03	3.32	1.73	.37
AGV-1	59.62	1.05	17.43	6.17	.10	1.55	4.95	4.30	2.92	.50
JG-1	72.98	.27	14.32	1.97	.06	.77	2.19	3.42	3.99	.09
TB	63.07	.97	21.49	6.54	.05	2.02	.35	1.37	4.02	.11
SY-2	61.09	.15	12.35	5.75	.33	2.73	8.13	4.42	4.59	.45
DPL	50.76	1.12	14.85	9.54	.18	9.39	11.37	2.32	.35	.12
SID	50.81	1.85	14.06	11.83	.22	6.71	11.12	2.62	.19	.20
G2	69.82	.51	15.56	2.38	.03	.77	1.96	4.11	4.56	.14
NIM-D	39.41	.02	.32	15.48	.22	44.15	.28	.05	.01	.02
KK	40.37	4.72	14.90	10.91		12.80	10.30	2.60	2.04	

Sources:

Brown, Roy W. (1977). A sample fusion technique for whole rock analysis with the electron microprobe. Geochimica et Cosmochimia Acta, vol. 41, pp. 435-438.

Flanagan, F.J. (1973). Geochimica et Cosmo. Acta, vol. 39, p. 1129.

Zentilli, M. - Personal communications.

APPENDIX III

Appendix to Experiment 2

	SiO_2	TiO_2	Al_2O_3	FeO	MnO	MgO	CaO	Na_2O	K_2O	P_2O
Z673 Ta in Nitrogen										
1	66.56	.35	17.75	2.39	.05	.65	3.37	5.03	3.12	.13
2	64.67	.26	19.35	2.07	-	.78	3.88	5.02	2.94	.08
3	65.57	.61	18.09	2.15	-	.76	3.88	4.81	3.03	.16
4	64.85	.23	19.43	1.86	-	.57	4.19	4.77	2.81	-
5	66.01	.25	16.58	1.28	-	.42	3.20	4.75	2.99	.15
Z673 Mo in Vacuum										
1	67.12	.24	17.61	2.18	-	.80	3.60	4.74	2.84	.15
2	68.30	.32	16.58	2.14	-	.76	3.16	4.64	2.91	.14
3	65.56	.42	18.31	1.98	.10	.72	3.66	4.97	3.06	-
4	67.33	.38	17.20	2.24	.06	.79	3.68	5.01	3.06	.22
5	65.08	.25	18.19	2.02	-	.67	3.60	4.64	2.88	.12
Z673 Mo in Nitrogen										
1	67.91	.34	17.07	2.03	.05	.60	3.21	4.86	3.08	-
2	60.86	.12	17.16	2.14	.08	1.15	4.74	4.15	2.53	.45
3	62.75	-	19.80	2.45	-	1.05	5.24	4.92	2.54	-
4	67.76	.27	16.24	1.51	-	.56	3.40	4.05	3.43	.11
5	66.86	.21	16.54	1.72		.52	3.16	4.50	3.24	.23
6	68.44	.12	17.32	1.74	.11	.69	3.33	5.02	3.14	.23
Z673 Ta in Vacuum										
1	67.66	.29	17.45	1.93	.11	.93	4.05	4.17	2.76	.28
2	66.37	.38	17.54	1.95	-	.65	3.71	5.05	3.04	-
3	69.23	.35	16.75	1.83	.04	.75	3.50	4.29	2.71	.15
4	67.48	.15	17.28	1.95	.07	.76	3.53	4.39	2.79	.20
5	62.76	.25	17.17	1.93	-	.68	3.81	4.45	2.78	.19
AZ70 Ta in Nitrogen										
	47.66	2.38	14.49	13.84		6.54	12.04	2.51	.22	-
	46.99	2.33	14.22	14.11		6.55	11.98	2.76	.25	-
AZ70 Ta in Vacuum										
	47.22	2.24	14.43	12.79		6.34	12.11	2.40	.19	-
	46.67	2.34	14.67	13.94		6.50	11.97	2.85	.33	-
AZ70 Mo in Nitrogen										
	46.63	2.44	14.74	13.87		6.31	11.72	2.72	.25	-
	47.00	2.05	14.27	14.05		6.48	11.93	2.81	.23	-
AZ70 Mo in Vacuum										
	46.54	2.24	14.59	13.67		6.29	11.65	2.67	.27	-
	46.01	2.38	14.97	13.46		6.12	11.72	2.80	.29	-

Appendix to Experiment 4

SAMPLE	RUN #	TIME	VARIAC SETTING	TEMP.	CORR. TEMP.
Z656	62	17	68	1825	
	63	25	68	1825	
	64	20	67	1815	
	65	20	64	1760	
	66	17	69	1835	
$\bar{x}$		19.8		1812	1892
Z673	67	23	60	1685	
	68	18	57	1635	
	69	21	62	1725	
	70	18	55	1625	
	71	25	58	1650	
$\bar{x}$		21		1664	1744
Z671	72	15	52	1550	
	73	14	50	1525	
	74	13	53	1575	
	75	12	54	1595	
	76	25	54	1595	
$\bar{x}$		15.8		1568	1648
Z668	77	15	44	1425	
	78	20	42	1400	
	79	14	44	1425	
	80	18	44	1425	
	81	14	44	1425	
$\bar{x}$		16.2		1420	1500
Pillow	82	12	42	1400	
	83	12	39	1350	
	84	15	41	1375	
	85	10	39	1350	
	86	13	40	1365	
$\bar{x}$		12.4		1368	1448
AZ70	87	12	38	1325	
	88	20	40	1365	
	89	17	39	1350	
	90	12	40	1365	
	91	12	39	1350	
$\bar{x}$		14.6		1351	1431
AZ77	107	15	35	1275	
	108	20	38	1325	
$\bar{x}$		17.5		1300	1380
Pure Fe			37	1450 (avg. of 3)	
Book				1530	

Pyrometer Reads ∿ 80°C low

SID

Appendix to Experiment 4

	SiO_2	TiO_2	Al_2O_3	FeO	MnO	MgO	CaO	Na_2O	K_2O	P_2O_5
1	51.06	1.42	14.22	11.65	.27	6.92	10.86	2.82	.20	.29
2	51.03	1.83	14.01	11.80	.20	6.83	10.82	2.86	.15	.15
3	50.74	1.80	14.03	11.98	.24	6.74	10.96	2.60	.23	.20
4	50.89	1.93	14.19	11.76	.20	6.83	11.08	2.92	.28	.28
5	50.78	1.46	14.18	11.70	.11	6.85	10.86	2.78	.20	.20
6	50.62	1.72	14.06	12.15	.10	6.80	10.89	2.86	.32	.32
7	50.66	1.67	13.95	11.73	.20	6.65	10.71	2.51	.26	.26
8	51.12	1.15	14.10	11.75	.19	6.92	11.11	2.60	.21	.21
9	50.89	1.70	14.13	11.83	.20	6.90	11.03	2.72	.34	.34
$\bar{x}$	50.87	1.63	14.10	11.82	.19	6.83	10.92	2.74	.19	.25
s	.18	.24	.09	.16	.05	.09	.13	.14	.03	.06
AZ77	45.69	.76	8.55	11.98	.23	23.89	8.66	.56	.05	.19
	45.88	.90	8.41	11.94	.18	24.37	8.54	.26	-	.19
	45.80	.94	8.38	11.77	.11	23.85	8.52	.25	-	.22
	45.48	.88	8.40	11.87	.27	23.42	8.65	.46	.06	.13
	44.65	.85	7.80	11.43	.12	25.01	8.10	.61	.10	.17
	44.92	.86	7.95	12.06	.17	23.51	8.33	.35	-	.16
	44.80	.74	8.14	11.44	.17	23.62	8.37	.53	.09	.19
	45.48	.99	8.94	12.18	.07	19.41	9.51	.28	-	.09
	44.68	.74	7.69	11.10	.07	25.66	7.51	.39	.05	.14
	44.76	.84	8.47	11.93	.27	21.55	9.05	.41	-	.23
$\bar{x}$	45.21	.85	8.27	11.77	.17	23.45	8.52	.41	.04	.17
s	.50	.09	.38	.34	.07	1.72	.53	.13	.04	.04

AZ70

		SiO_2	TiO_2	Al_2O_3	FeO	MnO	MgO	CaO	Na_2O	K_2O	P_2O_5
Sample AZ70											
Melt	1-1	47.11	1.91	14.74	13.90	.28	6.60	11.88	2.79	.29	.28
	1-2	46.93	2.26	14.82	13.45	.31	6.46	11.85	2.78	.27	.28
	1-3	46.30	2.08	14.69	18.85	.16	6.46	12.21	2.50	.19	.30
	1-4	46.36	2.17	14.61	13.81	.14	6.43	11.85	2.42	.22	.12
	1-5	46.82	1.92	14.74	14.02	.37	6.87	12.30	2.71	.34	.46
	2-1	46.82	2.18	14.75	13.70	.24	6.47	11.91	2.69	.28	.31
	2-2	48.14	2.12	14.88	12.33	.19	6.57	12.09	2.71	.21	.25
	2-3	47.07	2.33	15.00	13.43	.30	6.35	12.12	2.63	.21	.20
	2-4	46.75	2.25	14.55	13.89	.30	6.60	12.20	2.60	.25	.30
	2-5	47.26	2.28	15.04	13.20	.38	6.57	12.04	2.66	.26	.42
	3-1	46.97	1.95	14.89	13.33	.28	6.52	11.85	2.70	.23	.32
	3-2	46.63	1.95	14.62	14.36	.15	6.90	12.36	2.54	.22	.31
	3-3	47.12	2.30	14.84	13.59	.12	6.62	12.07	2.53	.21	.20
	3-4	46.51	2.28	14.25	13.99	.17	6.63	12.20	2.58	.18	.29
	3-5	47.60	2.28	14.93	13.34	.30	6.61	12.38	2.79	.28	.27
	4-1	47.34	2.42	14.60	13.39	.22	6.61	12.14	2.69	.24	.19
	4-2	47.05	2.10	14.79	13.37	.20	6.55	11.91	2.57	.25	.16
	4-3	48.04	2.18	15.02	12.46	.21	6.39	11.83	2.84	.24	.24
	4-4	46.87	1.89	14.46	13.78	.16	6.19	11.99	2.50	.25	.21
	4-5	47.14	2.16	15.07	13.26	.24	6.57	12.18	2.75	.21	.22
	5-1	46.43	2.18	14.79	13.96	.19	6.65	12.27	2.47	.22	.24
	5-2	47.09	2.24	14.57	14.10	.28	7.02	12.14	2.78	.28	.34
	5-3	47.11	2.18	14.76	13.52	.20	6.37	12.02	2.74	.25	.24
	5-4	46.84	2.02	14.54	13.65	.17	6.65	12.30	2.91	.21	.25
	5-5	47.12	2.22	14.66	13.31	.15	6.70	12.18	2.80	.22	.19
$\bar{x}$		47.02	2.15	14.74	13.56	.23	6.57	12.09	2.67	.24	.26
s		.45	.14	.17	.46	.07	.18	.17	.13	.04	.08

DPL

	SiO_2	TiO_2	Al_2O_3	FeO	MnO	MgO	CaO	Na_2O	K_2O	P_2O_5
Melt 1-1	51.21	.96	14.62	9.40	.10	9.51	11.34	2.16	.17	.17
1-2	51.15	.95	14.84	9.60	.07	9.59	11.32	2.08	.14	-
1-3	51.54	1.18	15.17	9.14	.16	9.94	11.53	2.27	.20	-
1-4	51.52	.99	15.14	9.20	.14	8.63	11.38	2.36	.18	.14
1-5	51.98	1.04	15.06	9.11	.23	8.77	11.50	2.47	.23	.15
2-1	51.62	1.18	15.22	9.26	.19	9.34	11.31	2.42	.24	.12
2-2	52.10	.95	15.16	9.24	.09	8.60	11.49	2.47	.14	-
2-3	52.05	.90	15.36	9.31	.21	9.07	11.48	2.27	.22	.23
2-4	52.31	1.16	15.45	9.31	.22	8.87	11.54	2.40	.23	.34
2-5	52.35	.91	15.44	9.40	.21	9.04	11.58	2.42	.23	.18
3-1	51.54	1.03	14.95	9.53	.17	9.22	11.20	2.26	.17	.14
3-2	51.93	1.12	15.23	9.06	.21	8.84	11.65	2.20	.22	.10
3-3	51.46	1.15	15.04	9.19	.28	9.75	11.43	2.15	.26	.19
3-4	51.83	1.07	15.00	9.28	.19	8.95	11.26	2.11	.14	.12
3-5	51.45	.91	15.18	8.74	.08	9.49	11.55	2.18	.19	.37
4-1	51.54	1.02	14.73	8.87	.12	9.29	11.46	2.18	.10	.10
4-2	51.83	.73	15.47	9.29	.18	8.91	11.31	2.37	.30	.15
4-3	51.75	.96	15.03	9.09	.21	9.43	11.54	2.09	.22	.17
4-4	51.87	1.12	14.94	9.51	.13	9.14	11.47	2.37	.23	.28
4-5	52.23	1.22	15.50	7.30	.18	9.24	11.65	2.02	.19	-
5-1	51.73	.96	15.21	8.99	.17	9.01	11.60	2.17	.22	.25
5-2	51.32	1.11	14.94	9.04	.17	8.72	11.41	2.29	.22	-
5-3	51.60	1.00	15.29	8.87	.11	8.81	11.42	2.12	.15	.17
5-4	51.56	1.01	15.15	9.19	.17	8.95	11.52	2.19	.23	.19
5-5	51.81	1.07	15.10	9.33	.22	9.10	11.60	2.02	.15	.11
$\bar{x}$	51.73	1.03	15.13	9.17	.17	9.09	11.46	2.24	.20	.13
s	.32	.12	.22	.44	.05	.31	.12	.14	.04	.09

Z668

	SiO_2	TiO_2	Al_2O_3	FeO	MnO	MgO	CaO	Na_2O	K_2O	P_2O_5
Melt 1-1	61.96	.54	19.39	3.48	-	2.12	6.07	4.11	2.28	.19
1-2	58.98	.83	18.43	4.43	.19	2.68	6.59	3.71	2.03	.35
1-3	60.61	.36	20.73	3.92	.05	2.40	7.10	4.12	2.12	.29
1-4	61.76	.45	16.28	4.41	.10	3.19	5.46	3.58	2.34	.36
1-5	59.76	.57	19.02	3.90	.05	2.28	6.31	3.85	2.12	.41
2-1	59.30	.65	18.34	4.15	.07	2.31	5.71	4.43	2.57	.40
2-2	64.91	.29	15.99	3.33	.05	2.83	4.30	3.96	2.60	.29
2-3	58.97	.70	18.18	4.78	.11	2.54	6.23	4.22	2.34	.76
2-4	57.79	.96	17.57	6.10	.06	2.97	6.68	4.02	2.07	.46
2-5	60.51	.95	16.48	5.07	.11	2.43	5.52	3.92	2.33	.33
3-1	58.64	.78	18.80	4.55	-	2.52	6.71	3.76	2.03	.26
3-2	59.02	.89	18.50	4.76	.12	2.55	6.64	3.75	2.02	.33
3-3	61.08	.43	17.95	4.03	-	2.08	5.87	3.82	2.34	.36
3-4	59.85	.84	19.05	5.34	.08	2.88	6.81	3.88	1.99	.47
3-5	59.53	.74	18.43	4.93	.10	2.53	6.29	3.80	2.17	.40
4-1	62.48	.49	17.93	3.81	-	2.12	5.34	4.07	2.47	.20
4-2	59.72	.86	18.34	4.18	.04	2.03	6.24	4.33	2.52	.35
4-3	60.65	.65	17.59	4.65	.07	2.28	6.04	3.83	2.46	.35
4-4	61.98	.65	17.20	4.28	.06	2.24	5.68	3.91	2.39	.37
4-5	57.44	1.05	17.79	5.23	.04	2.96	7.03	3.88	2.22	.38
5-1	55.79	1.24	17.02	6.26	-	4.20	7.51	3.37	1.85	.37
5-2	58.56	.92	17.99	5.14	.14	2.84	6.64	3.97	2.06	.39
5-3	60.76	.38	18.39	3.84	.06	2.21	5.95	4.33	2.47	.24
5-4	62.32	.61	17.34	4.39	.05	3.04	6.19	3.91	2.19	.31
$\bar{x}$	60.10	.70	18.03	4.54	.07	2.59	6.20	3.94	2.25	.36
s	1.90	.24	1.04	.74	.05	.48	.68	.24	.21	.11

Z671

	SiO_2	TiO_2	Al_2O_2	FeO	MnO	MgO	CaO	Na_2O	K_2O	P_2O_5
Melt 1-1	61.80	.34	18.84	3.56	.11	1.49	5.91	4.09	2.55	.34
1-2	64.46	.59	18.68	3.64	.04	1.35	5.36	4.02	2.37	.23
1-3	61.41	.61	18.95	3.23	-	1.31	6.09	3.74	2.33	.21
1-4	64.38	.33	18.16	2.97	.08	1.47	5.58	3.57	2.33	-
1-5	61.14	.76	18.10	3.78	-	1.69	6.48	3.71	2.53	.21
2-1	60.67	.68	18.11	4.62	.05	1.63	6.09	3.86	2.41	.44
2-2	63.92	.77	18.97	2.77	-	1.48	5.84	3.65	2.26	.22
2-3	61.03	.95	18.31	4.12	.20	1.61	6.01	3.75	2.30	.27
2-4	67.04	.77	15.26	2.63	-	1.24	5.17	3.13	2.35	.28
2-5	61.31	.64	18.10	3.32	.07	1.30	5.76	3.79	2.34	.24
3-1	61.99	.53	18.68	3.63	.19	1.50	6.04	3.73	2.34	.23
3-2	62.87	.42	18.05	3.52	.12	1.43	6.09	4.01	2.61	.20
3-3	59.23	.62	18.10	5.41	-	1.71	6.45	3.93	2.27	.46
3-4	61.11	.62	18.18	4.18	.08	1.28	5.71	3.97	2.42	.19
3-5	63.01	.60	18.12	3.60	.08	1.45	5.81	3.59	2.25	.30
4-1	61.49	.47	19.39	3.49	.05	1.93	7.21	3.34	2.49	.35
4-2	68.75	.52	19.20	2.35	-	1.49	5.64	3.21	2.19	.24
4-3	59.26	.68	19.41	4.07	.13	1.98	7.14	3.73	2.15	.33
4-4	62.01	.69	18.56	3.61	.06	1.23	6.03	3.69	2.21	.18
4-5	64.02	.63	16.46	5.15	.11	1.33	5.23	3.22	2.20	.26
5-1	65.92	.82	16.57	3.34	.17	1.36	5.55	2.39	2.11	.22
5-2	63.27	.95	19.46	3.33	-	1.58	6.14	2.66	1.83	.18
5-3	65.91	.59	19.27	2.33	.12	1.81	6.25	2.82	1.97	.20
5-4	64.68	.63	18.75	2.29	-	1.66	5.93	2.97	2.02	.21
5-5	66.36	.82	17.23	2.70	.15	1.56	5.86	2.39	2.05	.28
$\bar{x}$	63.08	.64	18.28	3.51	.07	1.51	5.97	3.48	2.28	.25
s	2.43	.16	1.01	.82	.06	.20	.49	.50	.19	.09

Z673

		SiO_2	TiO_2	Al_2O_3	FeO	MnO	MgO	CaO	Na_2O	K_2O	P_2O_5
Melt	1-1	66.12	.49	18.35	1.89	-	.80	3.79	4.48	3.07	.20
	1-2	64.98	.56	18.45	1.89	.06	.91	3.91	4.42	3.02	.20
	1-3	69.01	.15	16.45	1.45	-	.61	3.20	3.97	2.82	-
	1-4	65.70	.30	17.32	1.93	-	.83	4.14	3.38	2.86	-
	1-5	66.26	.30	17.44	2.26	.07	1.08	4.01	3.94	2.87	.21
	2-1	64.65	.26	18.61	1.98	-	.79	3.80	4.76	3.00	.13
	2-2	66.95	.48	17.74	1.76	.11	.94	3.89	4.07	2.84	.15
	2-3	71.66	.34	19.64	1.88	.07	1.06	4.07	4.46	2.89	.20
	2-4	67.99	.91	18.36	1.87	.10	.91	3.73	4.49	2.92	.15
	2-5	68.99	-	15.65	1.63	-	.62	3.12	4.37	2.90	.22
	3-1	68.36	.46	17.44	2.06	.13	.88	3.78	2.05	2.49	.22
	3-2	67.75	.31	17.56	1.48	.08	.90	3.82	2.74	2.68	.12
	3-3	69.09	.42	18.14	1.80	-	1.00	4.01	2.44	2.34	.14
	3-4	69.71	.10	17.53	1.67	.11	.85	3.44	3.20	3.21	-
	3-5	68.10	.47	18.30	1.62	.08	.99	4.29	3.20	2.88	.25
	4-1	69.98	.36	19.30	1.41	-	1.02	4.01	2.61	2.11	.11
	4-2	69.00	.60	18.67	1.94	.09	1.02	4.04	4.16	3.12	.26
	4-3	64.90	.55	18.20	2.23	-	.87	4.14	3.72	3.08	.15
	4-4	64.62	.50	18.57	2.30	.15	1.00	4.40	3.73	3.11	.33
	4-5	64.62	.14	17.95	1.56	-	.90	4.41	3.96	3.20	-
	5-1	67.06	.59	16.81	1.85	-	.96	4.18	3.95	3.01	.13
	5-2	70.05	.47	17.92	.89	.12	.92	3.57	3.18	2.58	.19
	5-3	67.29	.39	13.83	1.74	.04	.85	3.81	4.61	3.31	.13
	5-4	71.56	.45	17.82	1.41	.05	.92	3.57	3.71	2.68	.20
	5-5	65.39	.17	17.46	1.90	-	.83	4.03	4.11	3.16	.15
$\bar{x}$		67.71	.38	17.95	1.77	.07	.87	3.89	3.75	2.88	.16
s		2.19	.20	.85	.31	.12	.21	.32	.73	.28	.09

Z656

	SiO_2	TiO_2	Al_2O_3	FeO	MnO	MgO	CaO	Na_2O	K_2O	P_2O_5
Melt 1-1	76.01		13.44	.28	.05	.08	.51	1.57	4.97	-
1-2	79.74		12.68	.32		.16	.60	1.66	5.93	-
1-3	75.33		14.03	.57	.07	.24	.88	2.33	5.85	-
1-4	74.17	.16	13.64	.43		.19	.72	2.02	6.10	-
1-5	80.32		13.53	.24		.14	.63	1.98	6.24	-
2-1	73.76	.17	14.15	.67		.16	1.30	1.80	6.40	-
2-2	79.11		9.93	.34		.11	.39	1.29	5.74	-
2-3	75.16	.08	14.21	.45	.06	.29	.85	2.01	6.17	.11
2-4	81.42	.14	13.22	.50	.10	.28	.82	1.56	5.33	.17
2-5	78.92	.08	11.19	.29		.18	.54	1.63	6.10	-
3-1	82.99		10.04	.29		.17	.44	1.62	5.32	-
3-2	71.40	.14	15.18	.97	.11	.27	1.62	1.59	5.79	.15
3-3	78.05	.23	14.65	.42		.30	.99	1.75	5.45	-
3-4	78.66	.17	13.61	.48	.10	.26	.71	2.03	5.91	.21
3-5	80.40		12.86	.44	.06	.21	.69	1.77	5.90	-
4-1	79.94		13.56	.17		.11	.61	.56	3.85	-
4-2	74.55	.07	14.82	.66		.21	1.03	2.82	6.28	.11
4-3	77.75	.11	13.70	.40		.18	.67	1.71	5.49	.09
4-4	77.96		14.68	.43	.10	.27	.80	2.43	5.42	.19
4-5	72.85	.17	16.19	.60		.27	1.06	2.63	6.22	-
5-1	79.83		14.09	.44		.21	.90	1.00	5.61	-
5-2	77.82		13.89	.47	.04	.22	.83	1.59	5.96	-
5-3	76.66	.16	13.28	.19	.11	.14	.57	.89	3.85	.13
6-1	76.88	.14	14.03	.56		.17	.90	2.98	5.57	-
6-2	70.90		17.54	.48	.12	.32	.79	3.45	6.64	.09
6-3	76.63	.10	14.72	.45		.22	.81	2.65	5.47	-
6-4	81.43		13.19	.50		.20	.71	2.76	5.16	.08
6-5	79.58	.12	14.79	.37		.28	.71	2.67	5.35	-
$\bar{x}$	77.44	.007	13.74	.44	.07	.21	.79	1.93	5.65	
s	3.07	.007	1.56	.16		.06	.26	.73	.65	

Appendix to Experiment 5

		SiO_2	TiO_2	Al_2O_3	FeO	MnO	MgO	CaO	Na_2O	K_2O	P_2O_5
JG-1											
	1	71.51	.29	15.45	1.83	-	.71	2.02	2.70	4.55	-
	2	69.34	.32	16.22	1.72	.12	.74	2.11	2.48	4.55	.15
	3	68.58	.34	17.54	1.94	.10	.98	2.66	1.22	3.23	.26
	4	65.66	.23	18.02	2.47	.08	1.03	3.14	2.80	4.36	.16
	5	66.63	.45	18.10	2.22	.12	1.02	3.25	1.77	3.22	.15
	6	60.49	.18	21.00	2.48	.10	.85	4.24	3.94	4.56	.13
	7	68.16	.27	16.63	2.40	.11	1.00	2.84	2.67	4.28	.11
	8	68.14	.27	16.84	1.81	-	.71	2.25	2.48	4.43	-
	9	75.82	.12	12.09	1.53	.09	.63	1.61	2.15	3.64	.13
	10	64.58	.38	18.36	2.65	-	.92	2.80	2.70	4.56	.13
	11	81.18	.19	9.31	1.15	.11	.46	1.32	1.26	2.79	-
	$\bar{x}$	69.10	.28	16.32	2.02	.07	.82	2.57	2.38	4.02	.11
	s	4.48	.10	3.19	.47	.05	.19	.83	.77	.66	.08
TB	1	59.74	1.01	24.22	6.82	.06	2.34	.18	.73	3.52	.32
	2	60.91	1.04	23.12	6.59	.08	2.25	.22	.80	3.71	.13
	3	63.33	.94	20.86	6.26	.09	2.00	.19	.82	3.67	.23
	4	61.35	.98	22.44	7.15	.09	2.16	.22	.88	3.54	.22
	5	57.66	1.13	24.89	7.50	.05	2.34	.19	.84	3.45	.08
	6	66.96	.59	18.63	5.14	.05	1.79	.09	.87	4.03	.14
	7	57.62	.80	24.95	7.25	.08	2.39	.17	.94	3.63	.13
	8	55.35	1.15	26.45	8.27	-	2.58	.19	.80	3.14	.08
	9	56.49	1.11	25.66	7.60	-	2.44	.09	.83	3.43	-
	10	64.06	.71	21.54	5.30	-	1.97	.19	1.01	3.85	.23
	$\bar{x}$	60.35	.95	23.28	6.79	.05	2.23	.17	.85	3.60	.16
	s	3.69	.19	2.43	1.00	.04	.24	.05	.09	.25	.09
BCR-1	1	54.25	2.39	13.58	12.69	.19	3.56	7.32	3.41	1.61	.37
	2	54.11	2.32	13.80	11.92	.18	3.61	7.64	3.18	1.50	.49
	3	53.64	2.41	13.45	12.80	.15	3.33	7.16	3.31	1.54	.24
	4	53.26	2.59	13.25	13.02	.22	3.70	7.38	3.33	1.62	.54
	5	53.74	2.66	13.29	13.26	.20	3.69	7.33	3.36	1.58	.51
	6	54.41	2.18	13.48	12.52	.19	3.61	7.15	3.36	1.61	.47
	7	54.09	2.33	13.46	12.55	.11	3.48	7.34	3.22	1.53	.35
	8	53.26	2.59	13.32	13.37	.21	3.70	7.48	2.99	1.51	.54
	9	54.80	2.24	13.59	12.74	.19	3.65	7.32	3.35	1.64	.45
	10	54.57	2.29	13.68	12.58	.20	3.64	7.27	3.46	1.59	.47
	$\bar{x}$	54.01	2.40	13.49	12.75	.18	3.60	7.34	3.30	1.57	.44
	s	.53	.14	.18	.41	.03	.12	.15	.14	.05	.09

		SiO_2	TiO_2	Al_2O_3	FeO	MnO	MgO	CaO	Na_2O	K_2O	P_2O_5
AGV-1	1	60.06	.94	17.32	6.17	.10	1.42	4.94	4.35	2.93	.55
	2	59.25	1.29	17.09	6.54	.09	1.57	4.99	4.23	2.86	.47
	3	60.25	.98	17.07	5.78	-	1.37	4.89	4.17	2.89	.46
	4	59.76	1.01	17.17	6.17	-	1.46	5.00	4.24	2.91	.52
	5	59.52	1.18	17.66	6.52	.14	1.59	5.23	4.21	2.81	.70
	6	59.54	1.04	17.02	6.37	.14	1.53	5.05	4.15	2.88	.59
	7	60.42	.61	17.13	5.29	-	1.36	4.74	4.27	3.02	.70
	8	58.93	1.47	17.43	7.13	.07	1.12	4.89	4.22	2.85	.42
	9	60.46	.76	17.23	5.93	.16	1.37	4.96	4.06	2.90	.74
	10	57.66	1.64	15.74	8.34	.09	2.21	5.34	4.02	2.71	.70
	$\bar{x}$	59.58	1.09	17.08	6.42	.07	1.50	5.00	4.19	2.88	.59
	s	.84	.31	.51	.83	.06	.28	.17	.10	.08	.12
SY-2	1	59.07	.06	11.73	5.89	.31	2.71	8.97	3.82	3.92	.40
	2	59.40	.14	12.02	5.87	.37	2.66	8.39	3.99	4.07	.51
	3	59.22	.16	11.86	6.19	.42	2.68	8.59	4.19	4.06	.65
	4	59.30	.11	12.24	5.82	.34	2.70	8.45	4.12	4.25	.53
	5	61.47	-	14.01	4.15	.23	1.75	6.18	4.49	5.12	.27
	6	58.93	-	12.26	5.94	.20	2.53	8.18	4.21	4.29	.40
	7	59.13	.08	11.69	6.07	.48	2.74	8.61	3.91	4.03	.42
	8	59.61	-	12.63	5.37	.43	2.55	7.45	4.20	4.47	.51
	9	59.02	.18	12.03	5.63	.36	2.61	8.21	4.20	4.23	.57
	10	59.34	.19	13.20	5.61	.33	2.44	8.04	4.07	4.31	.41
	$\bar{x}$	59.45	.09	12.37	5.65	.34	2.54	8.12	4.12	4.28	.47
	s	.74	.08	.73	.58	.08	.30	.79	.19	.34	.11

Appendix to Experiment 7 FLUX MELTS DPL

	SiO_2	TiO_2	Al_2O_3	FeO	MnO	MgO	CaO	Na_2O	K_2O	P_2O_5	% Flux
Melt 1-1	37.21	.92	10.99	6.88	.19	6.75	8.25	1.85	.19	.15	26.4 %
1-2	37.63	.89	10.90	6.79	.09	6.70	8.22	1.73	.15	-	
1-3	37.28	.63	11.17	6.79	-	6.58	8.14	1.73	.13	-	
1-4	32.27	.70	10.78	6.82	.08	6.76	8.13	1.78	.17	.08	
1-5	37.80	.84	11.12	6.92	.17	6.68	8.28	1.78	.13	.19	
2-1	39.34	.93	11.42	6.98	.05	6.92	8.58	1.96	.18	.13	22.85 %
2-2	39.19	.88	11.57	7.27	.17	7.06	8.62	1.85	.19	.08	
2-3	39.63	.89	11.48	7.17	.18	7.05	8.64	1.76	.16	-	
2-4	39.50	.77	11.50	7.27	.22	7.15	8.73	1.83	.14	.23	
2-5	39.03	.84	11.37	7.00	-	6.94	8.71	1.91	.14	-	
3-1	38.70	.82	11.39	6.87	.06	6.87	8.60	1.82	.13	.09	26.44 %
3-2	38.52	.73	11.29	6.94	.15	6.69	8.48	1.88	.09	.13	
3-3	38.88	.94	11.20	7.04	.17	6.92	8.42	1.84	.20	.19	
3-4	38.62	.80	11.32	7.23	.20	6.87	8.51	1.86	.18	.07	
3-5	38.63	.90	11.18	7.10	-	6.90	8.35	1.86	.13	-	
4-1	40.46	.96	11.76	7.24	.16	7.14	8.84	1.47	.12	.12	23.68 %
4-2	40.44	.81	11.69	7.47	.06	7.01	8.76	1.57	.06	.21	
4-3	40.36	.84	11.80	7.28	.14	7.10	8.83	1.59	.09	.14	
4-4	39.87	.78	11.55	7.52	.09	7.35	8.98	1.51	.10	-	
4-5	40.27	.85	11.79	7.37	.11	7.16	8.77	1.59	.09	.17	
5-1	38.32	.76	11.32	7.31	.07	6.85	8.63	1.91	.17	.23	23.81 %
5-2	38.92	.88	11.23	6.98	.10	6.94	8.65	1.93	.14	-	
5-3	39.18	.88	11.51	7.02	.09	6.85	8.71	1.76	.14	.15	
5-4	39.09	.77	11.40	7.42	.08	6.96	8.50	1.85	.17	.11	
5-5	38.76	.80	11.35	7.03	.10	6.86	8.61	1.76	.11	.08	

Appendix to Experiment 7 FLUX MELTS Z656

	SiO_2	TiO_2	Al_2O_3	FeO	MnO	MgO	CaO	Na_2O	K_2O	P_2O_5	% Flux
Melt 1-1	63.88	-	11.91	.55		.27	.76	3.13	4.54		9.09 %
1-2	63.21	-	11.22	.44		.13	.79	2.88	4.51		
1-3	53.38	.19	13.07	.60		.11	.88	3.16	4.47		
1-4	64.43	-	11.99	.49		.16	.72	3.13	4.69		
1-5	63.27	.12	12.21	.47		.16	.75	3.26	4.73	.08	
1-6	65.27	.10	12.19	.50		.20	.77	3.26	4.52	.10	
2-1	62.56	.15	10.49	.31	.06	.19	.59	2.74	4.25	.10	16.67 %
2-2	61.96	.08	11.12	.43	.08	.15	.62	3.00	4.35	-	
2-3	61.75	.28	10.31	.38	.09	.11	.61	2.79	4.13	-	
2-4	56.07	-	10.80	.45	.07	.23	.86	3.41	4.48	.13	
2-5	57.63	.28	11.24	.55	-	.19	.88	3.11	4.51	-	
2-6	59.58	.25	11.21	.49	.10	.11	.80	2.89	4.13	-	
3-1	56.61	-	10.04	.36		.12	.60	2.68	3.70		24.32 %
3-2	56.46	.12	9.81	.51		.09	.69	2.51	3.75		
3-3	56.33	.13	10.17	.46		.08	.66	2.63	3.79		
3-4	57.01	.08	15.09	.37		.07	.51	2.39	3.76		
3-5	55.41	.07	9.95	.34		.13	.52	2.66	3.59		
3-6	55.50	-	10.11	.39		-	.61	2.50	3.76		
4-1	48.52	.10	8.81	.24		.06	.40	2.39	3.27		33.32 %
4-2	47.34	-	8.55	.25		.15	.51	2.24	3.10		
4-3	48.55	.14	8.79	.31		.12	.45	2.31	3.27		
4-4	47.96	.08	8.55	.34		.07	.52	2.13	3.13		
4-5	46.86	-	8.53	.27		.18	.51	2.33	3.07		
4-6	48.70	-	8.81	.26		.09	.44	2.29	3.30		
4-7	46.54	.14	8.41	.26		.06	.52	2.26	3.06		

Appendix to Experiment 8 FLUX MELTS - JG-1

	SiO_2	TiO_2	Al_2O_3	FeO	MnO	MgO	CaO	Na_2O	K_2O	P_2O_5	% Flux
Melt 1-1	56.89	.40	12.85	2.06	.06	.85	2.02	2.79	2.73	.18	19.2 %
1-2	61.03	.22	12.91	1.62	.07	.71	1.83	3.18	3.05	.12	
1-3	58.66	.13	12.87	1.33	-	.48	1.85	2.73	2.96		
1-4	61.72	.26	12.54	1.55	-	.59	1.70	2.76	2.94		
1-5	60.91	.32	11.90	1.64	.06	.68	1.74	2.83	3.18		
1-6	56.99	.12	13.27	1.44	-	.58	1.88	3.18	3.41		
2-1	48.52	.13	11.54	1.05	-	.48	1.32	2.12	2.57	-	37.5 %
2-2	53.54	.20	10.76	.91	-	.51	1.18	2.57	2.58	.08	
2-3	44.38	.10	11.22	1.24	.06	.62	1.54	2.44	2.44	-	
2-4	49.92	.14	10.05	1.06	-	.53	1.38	2.59	2.66	-	
2-5	47.99	.15	10.59	1.24	.09	.67	1.52	2.44	2.71	.14	
2-6	46.90	.05	10.26	1.21	.08	.60	1.48	2.73	2.43	-	
3-1	37.62	.13	8.42	1.20	.07	.50	1.38	2.29	2.41		42.9 %
3-2	40.26	-	8.89	1.17	-	.44	1.35	2.11	2.12		
3-3	40.38	.15	8.44	1.28	-	.54	1.38	2.26	2.45		
3-4	41.49	.12	9.19	1.25	-	.47	1.33	2.43	2.41		
3-5	38.71	.22	8.67	1.40	.07	.63	1.51	2.25	2.37		
4-1	33.69	.11	7.46	.98	-	.45	1.17	1.90	2.05	-	50.0 %
4-2	39.01	-	8.11	.95	-	.44	1.11	1.82	2.02	.12	
4-3	36.21	-	7.76	.86	-	.43	1.04	1.68	1.85	-	
4-4	39.02	.21	7.98	.97	-	.51	1.20	2.10	1.07	.11	
4-5	37.73	-	8.14	.94	-	.52	1.22	1.88	1.82	-	
4-6	40.75	.18	8.86	.89	-	.48	1.00	2.05	1.86	.15	
4-7	37.91	.22	7.92	.88	.57	.52	1.20	1.72	1.86	-	
5-1	37.30	-	7.65	.85	-	.48	1.09	1.79	1.28	.10	55.5 %
5-2	35.20	.13	7.09	.86	.07	.46	1.09	1.42	1.29	.07	
5-3	36.23	.05	7.59	.80	-	.35	1.00	1.80	1.40	-	
5-4	34.10	.12	6.96	.94	-	.39	1.03	1.70	1.56	-	
5-5	37.99	-	7.96	.88	-	.41	1.04	1.66	1.20	-	
6-1	29.44	-	6.48	.56		.38	.76	1.40	1.32	.07	66.6 ¢
6-2	26.89	.06	5.40	.64		.30	.78	1.29	1.03	-	
6-3	26.32	.06	5.40	.64		.30	.78	1.29	1.03	-	
6-4	24.33	.08	5.05	.67		.20	.80	1.22	.87	-	
6-5	24.33	.08	5.05	.67		.37	.80	1.36	1.22	.12	
6-6	25.38	.06	5.31	.55		.29	.80	1.32	1.16	-	

Appendix to Experiment 8 FLUX MELTS

		SiO_2	TiO_2	Al_2O_3	FeO	MnO	MgO	CaO	Na_2O	K_2O	P_2O_5	% Flux
NIM-D	1	30.65		.36	11.79	.21	34.20	.24	.11			37.5 %
	2	30.63		.32	11.83	.16	34.13	.19	.21			
	3	30.42		.22	11.81	.15	34.13	.21	.11			
	4	30.48		.34	11.86	.25	34.05	.24	.18			
	5	30.29		.27	11.92	.18	34.40	.21	.15			
	6	30.37		.29	11.94	.13	34.46	.19	.11			
TB	1	31.75	.55	11.12	3.16		1.13	.08	.82	1.96	-	50 %
	2	28.81	.40	10.28	3.35		1.06	.13	.71	1.91	-	
	3	33.50	.38	10.95	3.07		1.01	.14	.79	1.92	-	
	4	33.92	.47	11.17	3.08	.07	1.14	.11	.96	2.03	.13	
	5	28.62	.41	10.60	3.28		1.08	.13	.74	1.88	.12	
	6	29.46	.39	10.73	3.21		1.13	.11	.67	1.94	-	
Z656	1	43.14	.09	7.54	.27		.19	.37	1.87	2.69		43.9 %
	2	42.85	-	7.72	.22		.23	.42	2.03	2.79		
	3	45.96	.06	8.10	.28	.04	.24	.44	2.07	2.48		
	4	42.56	-	7.80	.24		.26	.42	1.85	2.31		
	5	44.07	-	8.09	.38		.29	.40	2.22	2.81	.12	
	6	42.06	.06	7.58	.31	.04	.26	.39	1.98	2.82		
JG-1	1	56.89	.40	12.85	2.06	.06	.85	2.02	2.79	2.73	.18	19.2 %
	2	61.03	.22	12.91	1.62	.07	.71	1.83	3.18	3.05	.12	
	3	58.66	.13	12.87	1.33	-	.48	1.85	2.73	2.96		
	4	61.72	.26	12.54	1.55	-	.59	1.70	2.76	2.94		
	5	60.91	.32	11.90	1.64	.06	.68	1.74	2.83	3.18		
	6	56.99	.12	13.27	1.44	-	.58	1.88	3.18	3.41		
G2	1	43.65	.39	9.76	1.52	.08	.46	1.17	2.31	2.22	-	45 %
	2	46.49	.33	10.57	1.54	-	.53	1.20	2.25	1.84	.14	
	3	41.59	.32	9.41	1.36	-	.45	1.11	2.48	2.42	-	
	4	43.62	.27	9.82	1.32	.04	.50	1.14	2.52	2.45	-	
	5	44.38	.38	9.70	1.44	-	.56	1.12	2.44	2.54	.13	
	6	43.72	.50	9.99	1.58	.07	.55	1.27	2.68	2.51	.12	
	7	43.34	.41	9.56	1.62	-	.58	1.27	2.58	2.59	.16	
	8	44.53	.04	9.97	1.62	-	.65	1.28	2.57	2.48	.12	
	9	43.40	.46	9.87	1.42	-	.49	1.23	2.46	2.40	.13	
	10	46.08	.16	9.38	1.35	-	.62	1.08	2.46	2.15	.16	