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A simple method of preparing glasses from rock powders

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Abstract

This note describes a simple and rapid method of preparing glasses suitable for electron microprobe analysis from powdered silicate rock specimens. Rock powder (100–200 mg, less than #200 mesh) is loaded into platinum crucibles fashioned from thin foil (1 mil or less). The powder is then melted in the graphite furnace of a flameless atomic absorption spectrophotometer by heating at 1700°C for one minute. The Pt crucibles filled with glass are conveniently mounted with five or more on a single mount, for subsequent preparation and polishing for analysis. The glasses are analyzed for major elements with an automated electron microprobe. For samples up to at least 60 wt.% SiO_2, homogeneous glasses are obtained. Because of the short heating interval and very short heat-up and cool-down times, no loss of alkalis or iron occurs. The method is rapid, inexpensive, relatively free from possibility of contamination, and is particularly well-suited for small samples.

Introduction

Technical developments in electron probe microanalysis have made rapid multi-element analysis readily available. This capability has been adapted to perform major elements analyses of bulk rocks after a suitable sample was prepared. Several methods of preparing glasses have been described (*e.g.*, Gulson and Lovering, 1968; Rucklidge *et al.*, 1970; Nicholls, 1974; Brown, 1977; and Jezek *et al.*, 1979). Generally, these procedures involve either a flux or direct fusion in a graphite furnace or on a special strip heater. The various advantages of a direct-fusion procedure have been discussed by Brown (1977). Most of the published techniques require fabrication of specialized heating equipment.

We describe a simple direct-fusion technique which we have found to be reliable and rapid over the past two years. The procedure utilizes a standard graphite furnace attachment used for atomic absorption analysis. Because this apparatus is widely used and no other specialized equipment is required, this simple and rapid method of rock analysis is available tc many. Furthermore, the general procedure is readily adapted to the analysis of very small samples.

Fusion procedure

Small "boats" are formed by cutting and folding the ends of 1 cm × 1.25 cm × 0.03 mm (approximately 0.08 g) piece of platinum foil. After thorough cleaning (with acetone, hot nitric acid, and distilled water), the Pt crucibles are filled with rock powder ground to less than #200 mesh. Typically, 100 to 150 mg is loaded, but larger sample sizes can readily be accommodated with tighter packing or larger Pt containers.

A graphite furnace (Perkin–Elmer Model HGA-2200) is used to heat the Pt boats. This furnace is normally used as an attachment on a Perkin–Elmer Atomic Absorption Spectrophotometer Model 372 at the University of Pennsylvania. The use of the furnace for the purpose of glass preparation has no deleterious effect on the spectrophotometer. A variety of similar equipment offered by other manufacturers is suitable. The graphite heating element for this apparatus is a detachable, hollow cylindrical tube (Perkin–Elmer Part #290-1766), 28 mm in length with an 8 mm outside diameter and 6 mm inside diameter. The filled Pt boat is placed upright in the graphite tube which is then clamped into the mount assembly. Graphite tubes previously used but no longer suitable for AA are perfectly adequate.

The sample is heated to approximately 150°C for

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30 seconds to allow drying of adsorbed moisture and then is heated to the fusion temperature of 1700°C which is maintained for 60 seconds. The desired 1700°C temperature is reached within one or two seconds. After the 60 second interval, the heater current is turned off and the sample is quenched to less than 200°C in a few seconds and to about 50°C in about 30 seconds. One advantage of the Perkin–Elmer heater is the rapid quench which occurs partly because the steel connectors holding the graphite tube are water cooled. Another is independent, preset time and temperature selection for up to three heating steps. However, the temperature settings are only nominal and we used an optical pyrometer for initial calibration. A 2 mm diameter hole in the graphite tubes, normally used to load solutions during AA analysis, conveniently allows direct observation and monitoring of the heating cycle.

The glass filled Pt containers can be loaded for electron probe analysis without additional handling. Our procedure is to mount five samples in a single 1.5 cm thick by 2.5 cm diameter Lucite disc with five 0.5 cm holes. The boat-shaped containers are simply dropped into the disc and secured with epoxy. After curing, one end is ground to expose the glasses and is then polished. If desired, the Pt can be recovered before mounting, with some inconvenience and added possibility of contamination. However, the cost of the Pt foil is minor (about $1 per sample) and the Pt can be used for checking beam current during microprobe analysis.

Optical examination of glasses prepared from mafic to intermediate rocks, varing from 45 to 60 wt.% SiO_2, shows them to be holohyaline and free of any quench crystals. Probe analysis shows the glasses to be uniform. Optically clear glasses of basaltic rocks can be produced in less than 30 seconds at 1700°C, but we have used a 60 second heating interval to insure thorough mixing. Furthermore, longer heating significantly reduces the number of bubbles.

Rocks high in hydrous or, particularly, carbonate minerals can pose a minor inconvenience. Occasionally, in such cases, heating directly to 1700°C causes the melt to spurt out of the Pt container and sometimes even out of the graphite tube. This complication is easily avoided by multi-step heating to the fusion temperature. Heating to about 1000°C for 60 seconds before melting seems sufficient to eliminate this problem.

Rocks of high SiO_2 content require longer heating times owing to the high melt viscosity. We have not extensively evaluated the heating requirements of such samples. Cursory examination of glasses prepared from granites indicates a percent or fewer crystals after 60 seconds, and optically uniform glasses after 150 seconds. We feel that an appropriate heating schedule for high silica rocks could be worked out quite easily, as suggested by the work of Brown (1977) and Jezek *et al.* (1979). It should be noted that the maximum heating temperature with this procedure is limited by the melting point of the sample container. Other metals could be substituted for Pt in order to increase the fusion temperature. Furthermore, the gas flow capability of the Perkin-Elmer furnace permits the use of an inert gas atmosphere, if necessary.

Microprobe analyses

For microprobe analysis, we use a defocused beam of 20 μm diameter and a beam current of 0.03 μA at 15kV. We use combined wave length and energy dispersive data with an ARL Electron Probe Microanalyzer with a Princeton Gamma Tech detector and Tracor Northern 880 automation at Lehigh University. For a typical analysis, we take the average of four or more spot analyses.

Because the point of this communication is to describe the principal of the fusion method, we shall not present details of the probe methods. Briefly, we use mineral and glass standards and the Bence–Albee method of data reduction.

The results obtained for two interlaboratory rock standards, BCR-1 and BHVO-1, are given in Table 1 along with the accepted values for comparison. The results show favorable agreement and serve to demonstrate that the fusion procedure is a good one at least for rocks of basaltic composition. Its usefulness for rocks of substantially different composition must be verified.

Two potential problems, high temperature alkali loss and loss of Fe to the Pt container, have been checked closely. Neither of these pose analytical problems. A series of fusions of *BHVO*-1 were made and the glasses were checked for heterogeneities such as low alkalis at the glass-air interface and low iron adjacent to the Pt foil. With one exception, no heterogeneities were found. A low FeO value was observed immediately adjacent to the Pt in a sample heated for three minutes (see Table 1). However, we are not certain if this effect is real, inasmuch as the totals were low, or rather is due to an edge effect or a spectrometer problem. The rapid heating and quench times and the short heating interval minimize iron and alkali loss.

The occurrence of bubbles could pose problems during probe analysis. In our experience, basaltic

Table 1. Analytical results for BCR-1 and BHVO-1

SAMPLE	BCR-1		BHVO-1								
Notation	F35	Ref.*	F34	10	K	MO	R	Q	Q ***	Average	Ref.**
# of Spots	5	-	5	1	2	4	4	3	4	19	-
SiO_2	55.7	55.38	50.8	50.1	50.3	50.3	51.0	51.0	50.4	50.68 ± .45	50.03
Al_2O_3	13.7	13.83	13.9	14.0	13.9	14.3	13.6	14.1	14.1	13.93 ± .28	13.92
TiO_2	2.30	2.24	2.72	2.66	2.74	2.78	2.71	2.64	2.73	2.72 ± .10	2.67
MgO	3.66	3.52	7.45	7.61	7.48	7.35	7.20	6.76	6.93	7.27 ± .34	7.37
FeO	12.1	12.31	10.9	10.7	10.8	10.7	10.5	10.7	9.96	10.72 ± .37	10.77
MnO	0.22	0.18	0.19	0.11	0.22	0.20	0.17	0.25	0.17	0.20 ± .06	0.16
CaO	7.13	7.03	11.4	11.6	11.3	11.3	11.6	11.4	11.4	11.41 ± .17	11.44
Na_2O	3.26	3.32	2.35	2.45	2.54	2.28	2.24	2.50	2.43	2.36 ± .19	2.21
K_2O	1.69	1.73	0.52	0.57	0.56	0.54	0.54	0.58	0.55	0.54 ± .03	0.55
Totals:	99.8	99.5	100.2	99.7	99.8	99.7	99.6	99.9	98.7	99.9	99.1

Heating interval was 60 seconds for all samples except for R (120 sec) and Q (180 sec).

* Flanagan (1973), recalculated on H_2O-free basis.

** Flanagan (1976), recalculated on H_2O-free basis.

*** Analyses immediately adjacent to Pt capsule.

compositions yield glasses with only a few large bubbles but as SiO_2 increases, the number of bubbles increases and their size decreases. A slightly longer heating does reduce the bubble population and is to be recommended for higher silica rocks.

Summary

The widespread availability of automated electron microprobe and graphite furnace AA instruments combine to make rapid major element analyses of bulk rocks available to many researchers without specialized analytical facilities. Furthermore, this direct-fusion technique is particularly useful to samples of small size. Many permutations of the general procedure are possible. Only a few minutes per sample are required to produce suitable probe materials. The potential for contamination is limited and the costs of expendable supplies are minimal.

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References

Brown, R. W. (1977) A sample fusion technique for whole rock analysis with the electron microprobe. Geochimica et Cosmochimica Acta, 41, 435–438.

Flanagan, F. J. (1973) 1972 values for international geochemical reference samples. Geochimica et Cosmochimica Acta, 37, 1189–1200.

Flanagan, F. J. (1976) Descriptions and analysis of eight new USGS standards. United States Geological Survey Professional Paper 840, 33–39.

Gulson, B. L. and Lovering, J. F. (1968) Rock analysis using the electron probe. Geochimica et Cosmochimica Acta, 32, 119–122.

Jezek, P. A., Stinton, J. M., Jarosewich, E., and Obermeyer, C. R. (1979) Fusion of rock and mineral powders for electron microprobe analysis. Smithsonian Contributions to the Earth Sciences, No. 22, 46–52.

Nicholls, I. A. (1974) A direct fusion method of preparing silicate rock glasses for energy-dispersive electron microprobe analysis. Chemical Geology, 14, 151–157.

Rucklidge, J. C., Gibb, F. G. F., Fawcett, H. H., and Gasparrini, E. L. (1970) Rapid rock analysis by electron probe. Geochimica et Cosmochimica Acta, 34, 243–247.

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