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SAMPLE PREPARATION FOR X-RAY FLUORESCENCE ANALYSIS: LI-BORATE GLASS DISKS

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INTRODUCTION

X-ray fluorescence analysis is a widely accepted technique for obtaining rapid chemical analyses of geologic samples. Problems have arisen, however, in sample preparation that is fast, reproducible and inexpensive, and which produces a durable, long-lived standard or sample.

Claisse (1956) pioneerd the flux-fusion technique for preparation of X-ray fluorescence samples. Subsequent modifications by Rose, Adler, and Flanagan (1962) have been widely adopted. However, fluorescence samples prepared by briquetting crushed glass boules are susceptible to contamination, are subject to deterioration, and require finely-machined dies and a laboratory press. A fused glass disk sample preparation has been described by Norrish and Hutton (1968), but the writers experienced difficulty preparing disks that did not crack upon removal from the graphite mold. The writers' proposed technique is similar to that developed by Stephenson (1969), but eliminates the oven annealing process designed to relieve stress within

the glass disk sample. We also reduced the $LiBO_2$-to-sample ratio to approach the eutectic composition in the system LiO_2-B_2O_3-SiO_2.

Sample Preparation

For major element analysis in silicate rocks a lithium- metaborate flux is prepared in the following proportions, and thoroughly mixed:

$LiBO_2$	87.00
La_2O_3	12.20
$LiNO_3$	0.80
	100.00 wt percent

Rock samples are finely ground and mixed with the lithium metaborate flux as follows:

Flux	3.000 g
Powdered rock sample	0.450 g.

For Na and Mg analysis $LiBO_2$ disks are fused without heavy absorber. Here we use a sample-to-Li BO_2 ratio of 1:1.

Crucibles must be prepared from coarse-grained, 1-1/2″ diameter graphite rods (comparable to Grade UF4S, #U618-R, Ultra "F" Purity graphite rods, produced by Ultra Carbon Corp., Mich.). Rods are cut in sections 38 mm long, and one end bored to an ID of 28 mm and a depth of 19 mm. The inside walls of the crucible should be bored with a slight taper, and the floor-walls junction gently chamfered to facilitate release of the sample disk from the crucible. The crucible's thick bottom serves as a thermal reservoir which anneals the glassdisk as it cools. See Figure 1.

An aluminum quenching plunger should be prepared with a flat bottom, an OD of 27 mm and approximate weight of 200 grams.

A flux-sample mixture is placed in a graphite crucible, and fused in

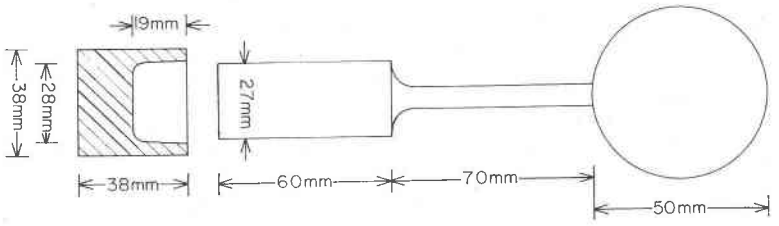

Fig. 1. Dimensional view of graphite crucible and plunger.

an oven at 1,000°C for 4 minutes. When removed from the oven, the crucible and molten sample should be swirled once or twice to ensure homogeneity and set on a horizontal surface to cool. While bright cherry-red, the molten sample is lightly quenched with the aluminum plunger until the boule retains the form of a flat-topped glass disk. Quenching time should be minimized to enhance annealing of the glass disk. When thoroughly cool, the crucible is inverted and tapped lightly to free the disk.

The glass disk may be embedded in plastic by placing it facedown in a metal ring mold and filling the mold with casting resin (comparable to "Cadco Casting Resin," Cadillac Plastic & Chemical Co., Detroit, Michigan). When cured, the disk is ground until the full diameter of the glass disk sample is exposed. The exposed glass is then readily polished to give an optimum surface for irradiation.

Glass disks prepared in this manner are tough and homogeneous. Electron microprobe scans across glass surfaces showed no appreciable variations in composition. Samples embedded in plastic are nearly indestructible. Surfaces for irradiation may be quickly and reproducibly prepared on all samples. Unlike boric acid pellets, glass disks apparently do not deteriorate with time; after six months of constant use, X-ray fluorescence standards made with USGS Standards II consistently generate reproducible calibration curves of exceptional fit. If contaminated, glass disk samples may readily be restored by polishing away any contaminated surface.

With 8 or 10 crucibles, any number of sample disks may be rapidly prepared in assembly-line manner.

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