**DEPARTMENT OF
GEOLOGY AND GEOPHYSICS**

MASTER

REPORT: FINAL: Volume 77-6

CONTRACT: EY-76-S-07-1601

AGENCY: DOE/DGE

TITLE: THE ANALYSIS OF SODIUM AND POTASSIUM IN
SILICATE ROCKS BY A LITHIUM METABORATE
FUSION METHOD

AUTHORS: W. P. Nash and Harrison Crecraft

DATE: December, 1977

UNIVERSITY OF UTAH

SALT LAKE CITY, UTAH 84112

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

MASTER

FINAL REPORT: VOLUME 77-6

DOE/DGE

EY-76-S-07-1601

DISCLAIMER

This book was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED *WDM*

THE ANALYSIS OF SODIUM AND POTASSIUM IN
SILICATE ROCKS BY A LITHIUM
METABORATE FUSION METHOD

By

W. P. Nash and Harrison Crecraft

Introduction

The lithium tetraborate fusion method devised by Ingamells (1964) and used by Suhr and Ingamells (1966) for emission spectroscopy and flame photometry analysis of silicates has been modified slightly to produce rapid and precise analyses of sodium and potassium contents in silicate rocks. The analytical method we previously employed is similar to that of Cooper (1963), and yields precise sodium and potassium values in the hands of a skilled analyst. However, the procedure requires considerable manipulation and is time consuming, taking about three days per batch of 6 to 12 samples. The new method makes it possible for a relatively unskilled analyst to analyze a dozen samples in half a day.

Analytical Method

Reagents

The flux is anhydrous lithium metaborate, LiBO_2 . Suhr and Ingamells (1966) describe several methods of purifying LiBO_2 , although we have found that Baker lithium metaborate (A.R.) yields entirely satisfactory results for sodium and potassium. The flux should be stored in a tightly closed bottle.

Nitric acid. HNO_3 50% V/V.

Standards. The standards used consist of analyzed U.S.G.S. rock samples or in-house analyzed rocks which are carried through the procedure. It is advisable to use standards with similar alkali concentrations as the unknown samples.

Procedure

1. Transfer exactly 0.7457g of 100% pure LiBO_2 to a waxed weighing paper. (Use 0.7554g of 98.8% purity Baker A. R. LiBO_2). The amount of flux must be weighed accurately as it serves as the interval lithium standard for the flame photometer. The amount of flux provides 15 mEq/l of Li (140 ppm) in the solution aspirated in our flame photometer (Instrumentation Laboratories model 343).
2. Weigh and transfer to the flux 0.1000g of finely ground (<200 mesh) sample powder. Carefully mix the powder intimately with the flux by rolling the corners of the weighing paper, and then transfer completely to a graphite crucible.
3. One or more standard samples should be weighed at the same time together with one sample consisting solely of the flux to serve as a blank.
4. Fuse in a muffle furnace at 1000°C for 15 minutes.
5. Remove the crucibles from the muffle furnace, swirl gently to gather up any uncoalesced molten material, and drop the hot bead into a 125 ml plastic bottle to which approximately 50 ml of boiling water has been added. Loss by spattering does not occur and none of the sample should remain in the crucible. The same crucibles may be used for subsequent fusions.
6. Add a Teflon-covered stirring bar and place the bottles on the magnetic stirrers. Turn on the stirrers, and, using a tilting pipet, add 5 ml of 50% HNO_3 to each bottle.
7. Stir rapidly for 60 minutes. The beads usually dissolve within half an hour. Shapiro (1975) notes that carbonate and phosphate rocks may need additional stirring.

8. When the beads have dissolved completely, remove the bottles from the stirrers and add 100 ml of water and 15 ml of 50% HNO_3 to each (a tilting pipet is again convenient for dispensing the HNO_3).
9. Transfer the solutions to in turn to a 1 l. volumetric flask, using a funnel to catch the stirring bar. Rinse the bottle several times with water, transferring the washings to the flask. Make the flask up to the mark with water and mix. Rinse the bottle with the solution several times and pour the solution back into the plastic bottle for storage.
10. Run the samples on the flame photometer. The lithium in the flux serves as the internal standard and the direct concentration readout in ppm is numerically equivalent to the percentage of Na and K in the sample. Alternatively, the concentration readout may be set to read Na_2O and K_2O directly.

Results

Precision data are given in Table 1. The sample run is a homogeneous, phenocryst-poor, obsidian. The relative standard deviations for both the Na and K are considerably less than 1%, and values obtained are comparable to those obtained by a more classical HF digestion method.

Accuracy is difficult to determine, but some measure may be had by comparing our results with preferred values for U.S. Geological Survey rock standards GSP-1, AGV-1, and W-1 (Table 2).

Table 1. Precision of method

MR 74-3	Concentration weight, %	Std. dev.	Rel. std. dev., %	No. of detns.
Na ₂ O	3.85	0.02	0.5	3
K ₂ O	5.29	0.02	0.4	3

A HF digestion method yielded results of

Na₂O = 3.82, K₂O = 5.26

Table 2. Comparative results on U.S. Geological
Survey standard rocks

	Na ₂ O	K ₂ O
<u>GSP-1</u>		
Utah	2.81	5.51
Flanagan (1976)	2.80	5.53
Abbey (1973)	2.80	5.53
<u>AGV-1</u>		
Utah	4.26	2.93
Flanagan (1976)	4.26	2.89
Abbey (1973)	4.31	2.93
<u>W-1</u>		
Utah	2.15	0.61
Flanagan (1976)	2.15	0.64
Abbey (1973)	2.15	0.64

References

- Abbey, S., 1973. Studies in "standard samples" of silicate rocks and minerals part 3: 1973 extension and revision of "usable" values. Geol. Surv. Canada Paper 73-36, 25p.
- Cooper, J. A., 1963. The flame photometric determination of potassium in geological materials used for potassium argon dating. Geochim. Cosmochim. Acta, y. 31, 289-308.
- Flanagan, F. J., 1976. 1972 compilation of data on USGS standards. In Descriptions and analyses of eight new USGS rock standards. U.S. Geol. Surv. Prof. Paper 840, 131-184.
- Ingamells, C. O., 1964. Rapid chemical analysis of silicate rocks. Talanta, v. 11, 665-666.
- Shapiro, L., 1975. Rapid analysis of silicate, carbonate, and phosphate rocks - revised edition. U.S. Geol. Surv. Bull. 1401, 76 p.
- Suhr, N. H., and Ingamells, C. O., 1966. Solution technique for analysis of silicates. Anal. Chem., v. 38, 730-734.