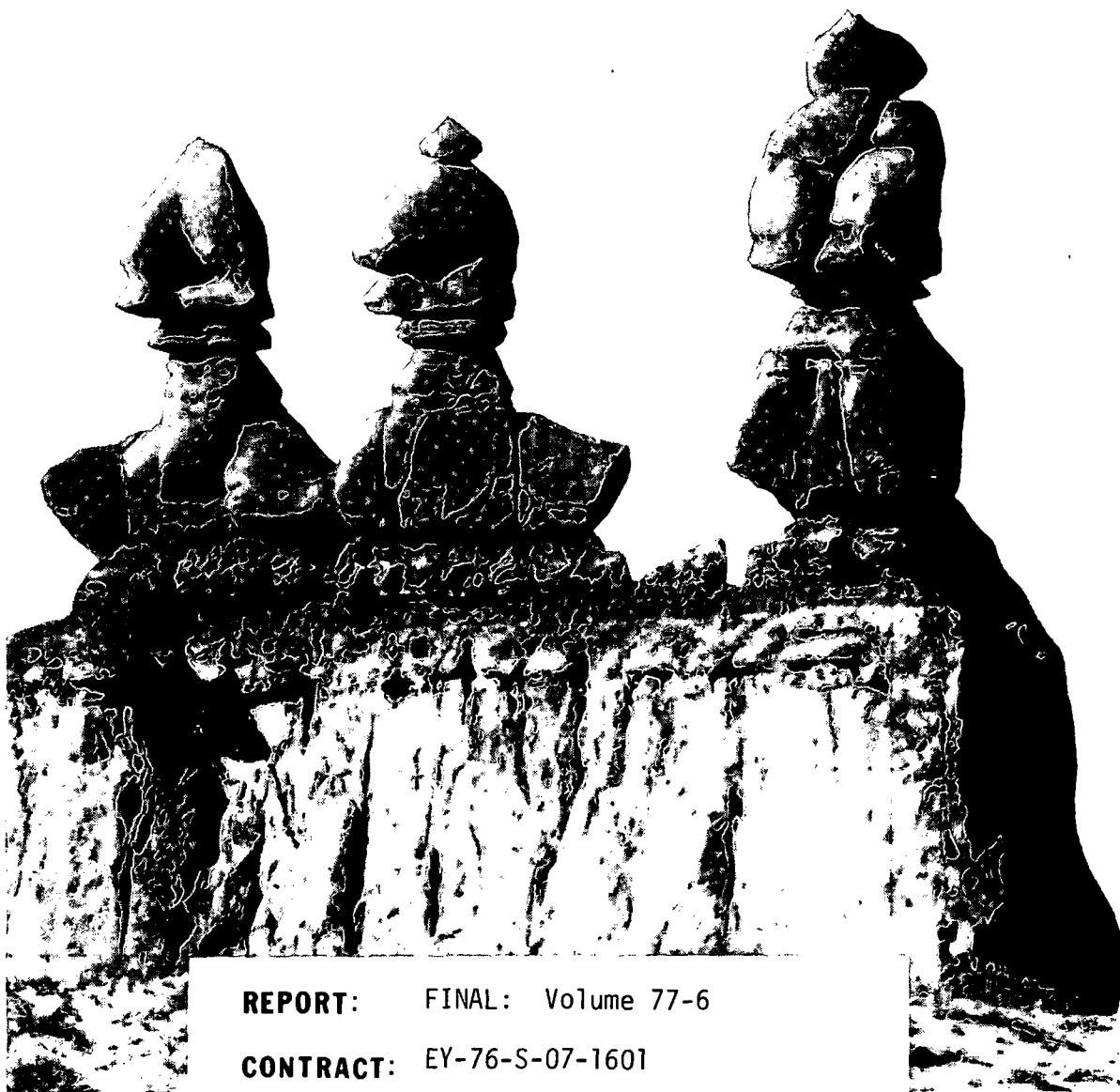


GLD1145

**DEPARTMENT OF  
GEOLOGY AND GEOPHYSICS**



**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**

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,  $\text{LiBO}_2$ . Suhr and Ingamells (1966) describe several methods of purifying  $\text{LiBO}_2$ , 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.  $\text{HNO}_3$  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  $\text{LiBO}_2$  to a waxed weighing paper. (Use 0.7554g of 98.8% purity Baker A. R.  $\text{LiBO}_2$ ). The amount of flux must be weighed accurately as it serves as the internal 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  $1000^\circ\text{C}$  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%  $\text{HNO}_3$  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%  $\text{HNO}_3$  to each (a tilting pipet is again convenient for dispensing the  $\text{HNO}_3$ ).
9. Transfer the solutions 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  $\text{Na}_2\text{O}$  and  $\text{K}_2\text{O}$  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	Std. dev.	Rel. std. dev., %	No. of detns.
	weight, %			
Na <sub>2</sub> O	3.85	0.02	0.5	3
K <sub>2</sub> O	5.29	0.02	0.4	3

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A HF digestion method yielded results of

Na<sub>2</sub>O = 3.82, K<sub>2</sub>O = 5.26

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

	Na <sub>2</sub> O	K <sub>2</sub> 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, v. 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.