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CORRELATION BETWEEN *X*-RAY EMISSION AND FLAME PHOTOMETER DETERMINATION OF THE K_2O CONTENT OF POTASH FELDSPARS

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Using *x*-ray emission spectroscopy in the quantitative determination of chemical composition is still in the developmental stages. As a test of its accuracy the K_2O content of potash feldspars from granitic rocks of the Inyo batholith was determined both by flame photometry and by *x*-ray emission techniques. Comparison of the results shows that the simpler and non-destructive *x*-ray emission technique gives satisfactory estimates of K_2O values.

To prepare each sample, 0.3–0.4 gram of the feldspar separate was powdered in a Wig-l-bug by shaking 0.1- to 0.2-mm. grains for five minutes in a steel capsule with steel balls. These powders were tightly packed into the wells ($3/4 \times 1/2 \times 1/16$ inch) of aluminum slides that fit the sample holder of the *x*-ray spectrophotometer.

A General Electric XRD-5 *x*-ray spectrophotometer with a lithium fluoride analyzing crystal was used. The tungsten tube was operated at 50 kilovolts and 50 milliamperes. Helium was used in the optical path, and a gas-flow proportional counter was the detector. The analytical line used was $K\alpha$. Background intensity variations of the different samples were insignificant in relation to the peak heights. Intensities were measured during a constant counting time of ten seconds. With a constant helium flow of eight cubic feet per hour the flushing time required to clear the system of air after the introduction of each sample was 120 seconds.

The standards used to establish a working curve were the chemically analyzed G-1 granite (Fairbairn *et al.*, 1951) and a potash feldspar megacryst of the Aiken facies of the McAfee adamellite (Emerson, 1959). The use of standards in a similar matrix, together with a narrow range in composition of samples, avoids significant deviations from the proportionality between analytical-line intensity and weight-fraction of the element being determined (Liebhafsky and Winslow, 1958).

Eighty-nine samples of potash feldspar were analyzed by both x-ray emission and flame photometer techniques. The flame photometer values were averaged from duplicate analyses that agreed within four per cent. The values, ranging from 9.5 to 15.2 per cent K_2O, are plotted in Fig. 1. Comparison of the values obtained with both methods show that those obtained by x-ray are unbiased, with an average error of estimate (Snedecor, 1956, p. 491) of only 0.03 per cent K_2O. The standard deviation of the difference between corresponding values from both techniques, or standard error of estimate, is 0.100 per cent. The sample correlation coefficient (Snedecor, 1956, p. 162) is 0.979, a nearly perfect correlation. The sample regression coefficient (Snedecor, 1956, p. 123) is 1.011, using the flame photometer results as the independent variable.

The high correlation, which is also reflected as a small standard error of estimate, shows that the much simpler and non-destructive x-ray
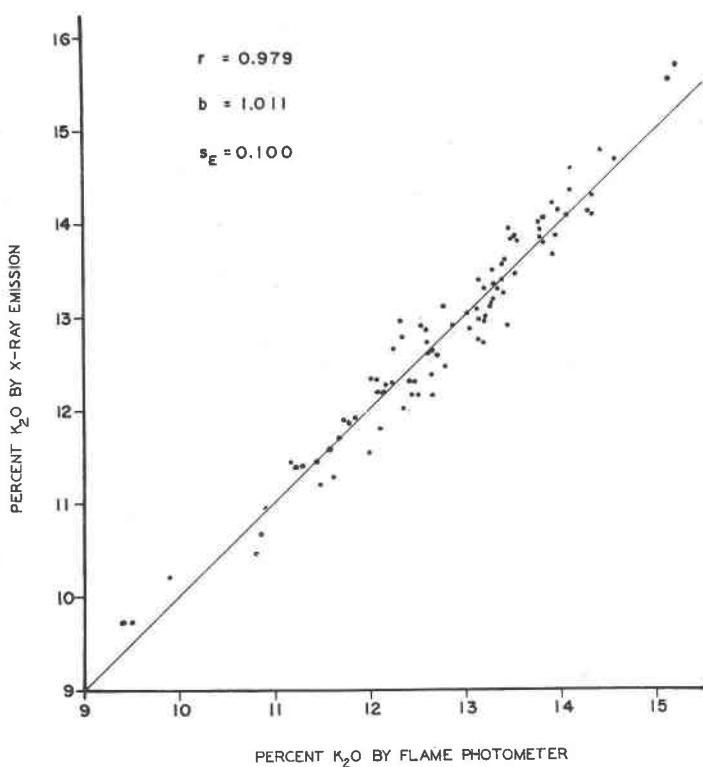

FIG. 1. Comparison of 89 K_2O values from x-ray emission and flame photometer techniques, showing a 1:1 line, correlation coefficient (r), regression coefficient (b) and standard error of estimate (S_E).

technique is as accurate a method of feldspar K_2O determination as is the conventional flame photometer technique.

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THE UNIT CELL OF CARMINITE

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The mineral carminite was described by Sandberger (1958) from Horhausen, Rhine Province, Germany. On the basis of an approximate chemical analysis he gave the composition as $Pb_3Fe_{10}(AsO_4)_{12}$, and the specific gravity as 4.10. Foshag (1937) reported the composition of carminite as approximating $PbFe_2(AsO_4)_2(OH)_2$ for carminite from Mapimi, Durango, Mexico and from an unstated locality in Colorado, but makes no mention of the specific gravity of his material. Le Mesurier (1939) described carminite from the Ashburton District, Western Australia which agreed in composition with Foshag's formula, and for which he reported a specific gravity of 5.22. The original papers of Sandberger and Le Mesurier were not available to the authors, the reported specific gravities having been noted in Dana's System of Mineralogy (1951), page 912.

Samples of carminite from Mapimi, Durango were examined for their suitability for specific gravity determination. These samples consisted of aggregates of minute (<0.5 mm. long) lath-shaped crystals very similar to those described by Foshag. Clusters of carminite crystals were used

TABLE 1. UNIT CELL DATA FOR CARMINITE

$a_0 = 12.25 \pm 0.04$ Å	space group: *Amaa* or *A2aa*
$b_0 = 16.52 \pm 0.04$ Å	cell volume: 1528 Å³
$c_0 = 7.64 \pm 0.04$ Å	cell formula: $Pb_8Fe_{16}(AsO_4)_{16}(OH)_{16}$
$a_0 : b_0 : c_0 = 0.741 : 1 : 0.456$	cell weight: 5046