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**A QUANTITATIVE TECHNIQUE FOR DETERMINING THE MASS-FRACTIONS OF  
AUTHIGENIC AND DETRITAL K-FELDSPAR IN MINERAL SEPARATES**

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**Abstract**

In studies involving radiometric, isotopic, or chemical analyses of authigenic potassium feldspar (K-feldspar), suitable samples are usually available only in the form of authigenic overgrowths on detrital igneous or metamorphic grains. The analysis of these mixtures often requires a measurement of the relative proportion of each component. To address this need, a technique has been developed which utilizes backscattered-electron (BSE) and cathodoluminescence (CL) detectors together with an image analyzer to quantify the relative proportions of authigenic and detrital K-feldspar in monomineralic separates. Authigenic (low temperature) K-feldspar overgrowths are distinguished from high temperature K-feldspar cores by their lack of luminescence. Computer analysis of BSE and CL images of polished grain mounts is used to compute a ratio of the area of luminescing (high-temperature) cores to the area of the total population of grains. Measurements made on synthetic standards indicate that area ratios determined in this manner are equivalent to true volume ratios, and that accuracies on the order of 8 percent (relative error) are obtainable.

**Key Words:** Authigenic K-feldspar, cathodoluminescence, image analysis, sedimentary petrography, geochemistry.

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**Introduction**

Numerous occurrences of abundant authigenic K-feldspar in Lower Paleozoic rocks of the mid-continent region and the Appalachian Basin have been described during the last 50 years (Daly, 1912; Tester and Atwater, 1934; Buyce and Friedman, 1975). For many years, this feldspar was assumed to be either syngenetic or early diagenetic in origin. However, recent  $^{40}\text{Ar}/^{39}\text{Ar}$  and K-Ar analyses, together with petrographic and fluid-inclusion studies strongly suggest that much of the authigenic K-feldspar was formed during the migration of basinal brines mobilized by orogenic activity (Hearn and Sutter, 1985; Hearn et al., 1987). These findings have focused new attention on authigenic K-feldspar as a potential indicator of the timing and chemical character of fluid-migration events in sedimentary basins.

While some monomineralic occurrences have been reported (Woodard, 1972), authigenic K-feldspar occurs more commonly either as overgrowths on detrital grains of K-feldspar or in impure lenses of partially metasomatized clay and silt (Buyce and Friedman, 1975; Kastner and Siever, 1979; Hearn et al., 1987). Due to numerous difficulties encountered in dealing with fine grained mixtures, separates of sand-size (>63 micrometers) detrital K-feldspar grains with overgrowths are often the most convenient form of sample for radiometric, isotopic and chemical analyses. However, because of the difficulty of separating overgrowths from detrital cores, analysts are forced to work with two-phase mixtures, and therefore require some method of quantifying the relative proportion of each component. This paper will describe a relatively simple SEM technique which was developed by the author to facilitate age determinations on authigenic K-feldspar overgrowths by the  $^{40}\text{Ar}/^{39}\text{Ar}$  age-spectrum method (Hearn et al., 1987). The technique employs BSE and CL detectors in conjunction with an image analyzer to determine quantitatively the mass-fraction of authigenic and detrital K-feldspar in purified mineral separates. The analytical procedure should be employable on any SEM equipped with BSE and CL detectors and some type of image analyzer.

### Background

The method described here is based on the difference in the luminescence characteristics of authigenic and detrital K-feldspar. Authigenic feldspar generally does not luminesce when exposed to ionizing radiation (Kastner and Siever, 1979); this property is perhaps the most diagnostic in distinguishing it from high-temperature feldspars, which luminesce in various shades of blue (Nickel, 1978; Kastner and Siever, 1979). While the actual causes of luminescence in feldspars are not well understood, it is most commonly attributed to the presence of trace elements in the crystal lattice which act as activators. Suspected activator ions for plagioclase feldspar include  $Mn^{2+}$ ,  $Ti^{4+}$ ,  $Fe^{3+}$ ,  $Fe^{2+}$ , and  $Cu^{2+}$  (Medlin, 1959, 1961; Garlick, 1955; Klick, 1955; Mariano et al., 1973; Thomas, 1974).

The luminescence characteristics of the different K-feldspar polymorphs have not been extensively studied. Most high temperature K-feldspars appear to luminesce in the range from 400 to 500 nm; there appear to be minor but distinct differences in luminescence wavelength and intensity between various structural types, and between samples of the same polymorph from different locations (Nickel, 1978). Because they precipitate at relatively low temperatures (<200°C), authigenic feldspars generally contain much lower concentrations of impurity elements, which substitute more readily in a high-temperature environment. Authigenic K-feldspar typically contains more than 99 mole percent  $KAlSi_3O_8$  (Kastner, 1971). This high chemical purity, and the corresponding depletion in activator elements is most probably responsible for its failure to luminesce.

### Experimental Procedures

The samples used in this study were extracted from Cambrian-age limestones and dolostones of the central and southern Appalachians. Following removal of carbonates in dilute HCl (2M), purified separates of detrital K-feldspar with authigenic overgrowths were obtained by gravity separation in a mixture of bromoform and dimethyl formamide (specific gravity = 2.60) and sieving to the 63-150  $\mu m$  size fraction. A small cylindrical mold was made by wrapping adhesive tape around a circular SEM sample stub. Approximately 200-300 mg of the K-feldspar separate was then poured into the mold and mixed thoroughly with a suitable petrographic epoxy. After curing, the tape was removed and the hardened epoxy was ground and polished to expose grain cross sections.

The SEM used in this study was an \*ETEC AUTOSCAN, equipped with a Si-diffused junction P-type solid-state BSE detector, a photomultiplier-tube CL detector, and an EDAX 9100 analyzer. Typical operating conditions were as follows: accelerating voltage = 30 kV; working distance = 20 mm; specimen current = 50-100 nanoamps. EDAX PARITD image analysis software was used to locate and measure the area of selected features within a given image based on their relative gray level. After specifying the gray-level range of interest

for a given image, the SEM is put in spot mode and the analyzer computer then drives the beam across the sample in predetermined increments. When the beam encounters a feature having a gray level within the specified range, the computer reduces the increment size, determines the boundaries of the feature, and stores the area relative to the total image in memory. After the beam traverses the entire field of view in this manner, the computer outputs the number of features encountered and their total area.

To make a measurement, a sample mount is placed in the SEM and imaged at low magnification (20-40x), so that the field of view contains several hundred grains. The total area of K-feldspar grains (cores + overgrowths) is first measured using the BSE detector; since their mean atomic weights (Z), and therefore their BSE intensities are virtually identical, cores and overgrowths are measured together. The BSE detector used can discriminate differences in Z on the order of 0.1; the image analysis system is capable of distinguishing differences on the order of 0.5. The gray-level threshold can therefore be adjusted so that minor amounts of quartz (the most common contaminant in K-feldspar separates) are not included in the measurement (Fig. 1). The above procedure is then repeated on a CL image of the same field. The relative mass fraction of cores (luminescing) and overgrowths (non-luminescing) is obtained from the ratio of these two measurements. Representative BSE and CL micrographs of K-feldspar grains with authigenic overgrowths are shown in Figure 2, together with photographs of the corresponding beam-trace images generated by the image analyzer.

As the CL detector used in this study measures luminescence from the entire visible-light spectrum, minor variations in component wavelengths do not affect the measurement of total luminescing area. Using a coarse traverse

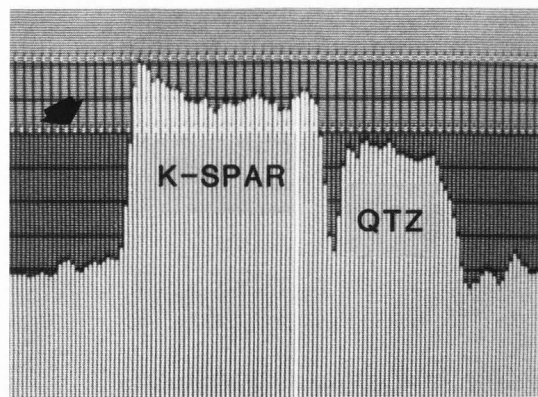


Figure 1. Video line scan across adjacent grains of K-feldspar and quartz (polished surface of epoxy grain mount). The vertical axis represents relative gray level; the horizontal axis represents distance (approximately 300 micrometers from left to right). Lowermost horizontal dotted line (arrow) indicates gray-level threshold used to discriminate these two phases.

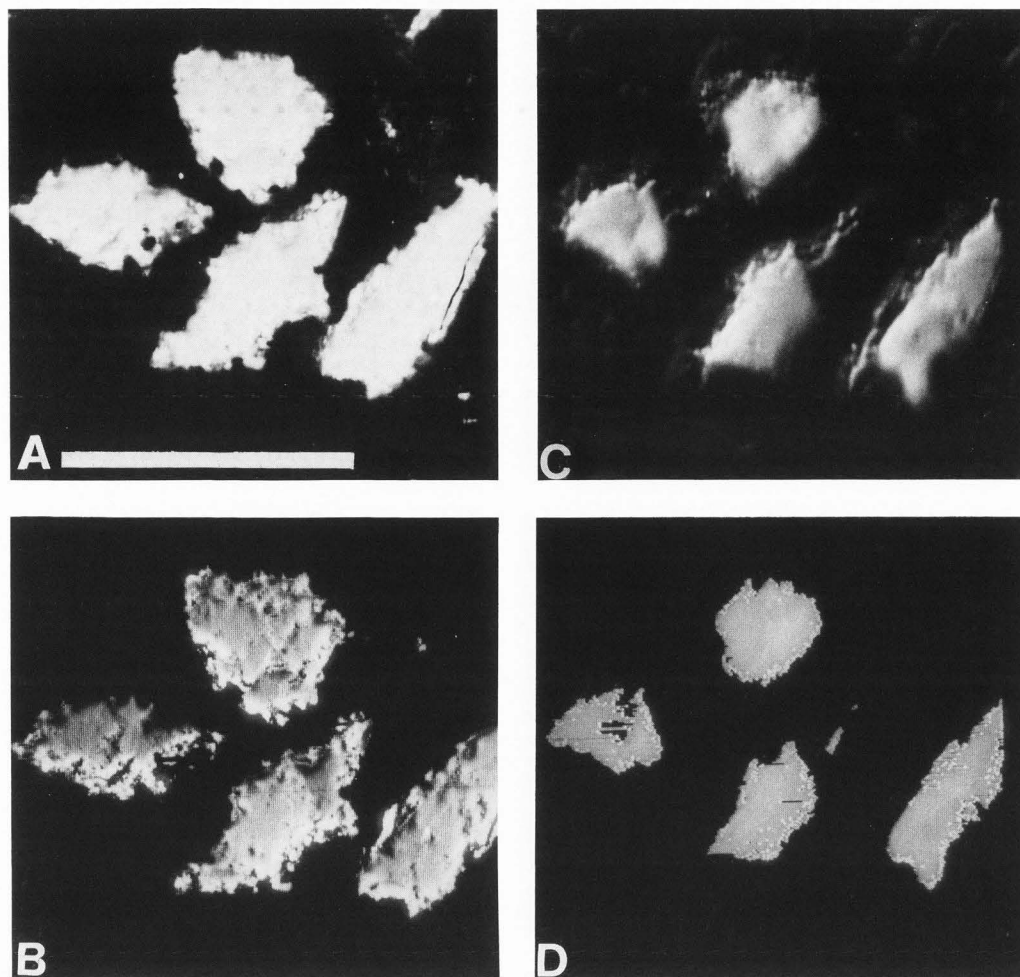


Figure 2. A) BSE micrograph of K-feldspar grains with authigenic overgrowths (polished surface of epoxy grain mount). Scale bar equals 100 micrometers. B) Time-exposure photograph of beam trace of the same field generated by image analyzer in BSE mode. C) CL micrograph of the same field, showing luminescing (high-temperature) cores. D) Time-exposure photograph of the beam trace generated by the image analyzer in CL mode. (Photographs by Bill Reckert)

increment of 25-50  $\mu\text{m}$  and a fine traverse increment of 5-10  $\mu\text{m}$  a field containing 500 to 700 grains requires 30-40 minutes to scan in both BSE and CL mode. The image-analysis software permits minimum particle dimensions to be designated, so that features having dimensions smaller than these values will not be included in the measurement. In this way, small spurious BSE and CL signals (from scratches or from the edges of bubbles in the epoxy) can be discriminated from the signals produced by K-feldspar grains. Also, since the field of analysis is relatively large ( $10\text{ mm}^2$ ), the current density on the surface of a given grain is not excessive. Accordingly, beam damage and reduction in the intensity of luminescence as a function of time are avoided.

#### Results and Discussion

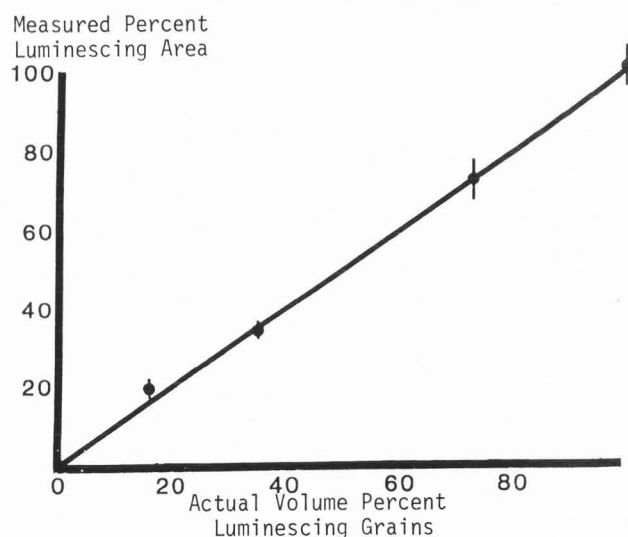
Grain mounts of K-feldspar separates extracted from four samples of Appalachian limestones were prepared and analyzed as described above. Five sets of measurements were made on each sample. The results of these determinations are shown in Table 1. The analytical precision varies from 1 to 7 percent.

The technique described here implicitly assumes that the ratio of areas for a given population of grains is equivalent to their volume ratio. If the population of grains in a given cross section is sufficiently large and is free of any preferred grain orientation, the assumption

**Table 1.** Mass Fraction of Authigenic K-feldspar in Purified K-feldspar Separates from Cambrian Carbonate Rocks of the Central and Southern Appalachians

Source	Location	Percent Authigenic
Gatesburg Limestone	Tyrone, Pennsylvania	60 ( $\pm 4$ )
Conococheague Limestone	Clear Spring, Maryland	55 ( $\pm 4$ )
Shady Dolomite	Austinville, Virginia	92 ( $\pm 1$ )
Maynardsville Limestone	Jefferson City, Tennessee	56 ( $\pm 3$ )

should be valid. In order to test this assumption and assess the accuracy of the method, standards containing known proportions of luminescing and non-luminescing grains were prepared. As no sample of igneous K-feldspar of sufficient purity and luminescent intensity was available, a purified and sized (63-150  $\mu\text{m}$ ) separate of apatite was used as the luminescing component. This material was mixed in varying proportions with a purified and sized separate of non-luminescent hydrothermal K-feldspar (adularia). The area ratios of luminescing and non-luminescing grains in these standards were determined in the manner described above; five replicate measurements were made on each standard. The results show a relatively good agreement between the measured area ratios and the actual volume ratios (Fig. 3). The data indicate an average relative error of 8.0 percent, and an average precision of 5.5 percent.



**Figure 3.** Measured area percent versus actual volume percent of luminescing grains in synthetic standards.

## Conclusions

The results of this study show that the mass fractions of authigenic and detrital K-feldspar in monomineralic separates can be measured quantitatively by processing BSE and CL images of grain mounts with an image analyzer. Although the described technique is somewhat time-consuming (2-3 hours for five measurements), it is one of the most practical and convenient methods of obtaining this type of data currently available.

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\*Trade names are used for descriptive purposes only and do not constitute endorsement by the U.S. Geological Survey.

Discussion With Reviewers

F. Laabs: Our cathodoluminescence studies of rare-earth materials indicate a linear relationship between intensity of a given wavelength vs. concentration down to the part per million range. Why wouldn't the same hold true for authigenic K-feldspar?

Author: The relationship between activator concentration and luminescence intensity probably does apply to authigenic K-feldspar, at least in principle. Unfortunately, there are no data available as to what the specific activator elements are, and what their concentrations are in authigenic K-feldspar. However, the absence of luminescence in most reported occurrences of authigenic K-feldspar suggests that activator concentrations are quite low.

F. Laabs: Does the epoxy mount material luminesce and if so, how is it separated from that of the detrital K-feldspar.

Author: The epoxy used in this work did luminesce weakly, as do most petrographic epoxies I have used. However, the intensity of the luminescence was significantly less than that of the high-temperature K-feldspar, and therefore could easily be distinguished on the basis of gray level.

G. Remond: Most of the luminescent minerals exhibit an intrinsic luminescence which is generally of a low intensity and an intense luminescence emission resulting from defects or impurities acting as luminescent centres (fluorescence emission) or electron traps (phosphorescence emission). Have you observed by means of point X-ray spectrometry analysis a relationship between the luminescent behavior of the different feldspar grains and their content in trace elements?

Author: I have not attempted a study of this nature. Such work clearly needs to be initiated, as there are currently no data as to which activator elements are responsible for luminescence in K-feldspars. However, I strongly suspect that the concentration of the activator ions would be close to or below the detection limits for these elements on the electron microprobe, and that a more sensitive analytical technique will be required.

F. Laabs: Has any attempt been made to utilize the EDS detector to negate counting grains which luminesce but are not in fact detrital K-feldspar?

Author: No. I don't think this is a real problem, however. It is possible to produce high-purity separates (>95% K-feldspar) using heavy-liquid techniques. Invariably, the major contaminant is quartz, which luminesces much less brightly than K-feldspar. If the initial separation is done properly, the concentration of other luminescing minerals (e.g., apatite, plagioclase) will be much less than the analytical error of the method.