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BETA DOSIMETRY OF POTASSIUM FELDSPARS IN SEDIMENT EXTRACTS USING IMAGING MICROPROBE ANALYSIS AND BETA COUNTING

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Abstract: We provide a comparison of two independent methods used to determine the K concentration of potassium feldspar extracts from sand-sized lacustrine, colluvial, and aeolian sediments. The two methods being compared are gas-flow beta counting and imaging microprobe X-ray fluorescence analysis. Imaging analysis demonstrated that the proportion of potassium feldspar grains in a K-feldspar extract varied from a low of 7% to a high of 84%. All extracts included a significant proportion of quartz, and some also contained a few plagioclase feldspar grains. However, the K_2O concentration in individual potassium feldspar grains of all eight extracts examined was within the range of 15.5 ± 0.7 % K_2O by weight. The K_2O concentrations in four of five extracts, measured using gas-flow beta counting, were significantly lower than this value. This reflects the dilution effect of significant amounts of non-K bearing grains in these extracts. This difference can result in up to an 80% underestimate in the internal beta dose rate of a potassium feldspar grain. A 14% underestimate in the total dose rate to potassium feldspar grains, and therefore a 14% overestimate in sediment ages determined by luminescence dating of nominally pure potassium feldspar extracts is thus possible. We suggest that gas-flow beta counting is suitable for the determination of K concentrations of bulk sediments and therefore the external beta dose rate, but that internal beta dosimetry is best performed by imaging microprobe X-ray analysis. An alternative, inexpensive approach would be to assume a 15.5 ± 0.7 % K_2O concentration for all nominal K-feldspar extracts separated using a heavy liquid with specific gravity of 2.58 $g \cdot cm^{-3}$, and use this value to determine the internal beta dose rate. Internal beta dose rate errors incurred with this approach are expected to be significantly less than those incurred by the small sample gas-flow beta counting method.

1. INTRODUCTION

It is well known that quartz is a preferred natural dosimeter for many depositional environments, especially when the total accrued dose does not exceed 50-100 Gy. Feldspar, the second most ubiquitous natural dosimeter, can also be used to advantage, in spite of the fact that it can present a more complex analytical situation. Naturally occurring feldspars show a wide variety of dose response efficiencies which can differ by as much as a factor of 10 for adjacent emission bands (**Fig. 1** and **Table 1**). Typically, the feldspars with the lowest efficiency are the Ca-rich plagioclase end members (**Fig. 2**), which are also known to exhibit strong anomalous fading. In addition, because feldspars form a continuous reaction in igneous melts, as per the Bowen Reaction Series, strong differences in geochemistry and therefore dosimetric properties may be exhibited on a microscopic scale (**Fig. 3**).

For these and other reasons, luminescence dating of potassium feldspar (K-feldspar) extracts from sandy sediments has become quite routine in the past few years (Edwards, 1993; Balescu and Lamothe, 1994; Clarke, 1994; Clarke *et al.*, 1995a). In principle, dating a K-feldspar grain has several advantages:

1. K-feldspars typically have a high luminescence efficiency under IR stimulation.
2. K-feldspars show less anomalous fading than other feldspar species.

3. The dose dependent saturation of the luminescence signal takes place at significantly higher doses in a K-feldspar than it does in quartz.
4. In principle, a high purity K-feldspar extract is easy to obtain by using heavy liquids at a specific gravity of 2.58 g cm^{-3}, after an initial separation at 2.62 g cm^{-3}. This procedure floats K-feldspars but causes quartz and plagioclase feldspar grains to sink.
5. A large proportion of the total dose absorbed by a K-feldspar grain is due to its internal beta dose, thus variations in ambient water content and other external dose rate variables are less significant. One might be tempted to conclude therefore that luminescence ages obtained on K-feldspar extracts are more accurate than those obtained on quartz or plagioclase feldspar dominated extracts.

Table 1. *Sources for feldspar data in* ***Figs 1*** *and* ***2****.*

AFHJ	Anorthite, Fuggope, Hokkaido, Japan
ALAR	Microcline, Magnet Cove, Arkansas
ALBO	Albite, Bancroft, Ontario
ALSD	Albite, Keystone, South Dakota
AMEL	Albite, Amelia Court House, Virginia
ANLN	Anorthoclase, Larvik, Norway
ANNY	Andesine, Essex Co., New York
ARGV	Anorthoclase, Grass Valley,
ARUS	Microcline var. amazonite, USSR
BYCM	Bytownite, Crystal Bay, Minnesota
BYSM	Bytownite, Sonora, Mexico
FCT1	Sanidine, Fish Canyon Tuff, San Juan Volcanic Field, Colorado
FJTC	Microcline, Joshua Tree N. P., California
LADB	Labradorite, Donnelly Butte, Oregon
LBLQ	Labradorite, Lake St. John, Quebec
LBNL	Labradorite, Nain, Labrador, Canada
MARQ	Microcline var. amazonite, Rouyn Distr., Quebec
MCPC	Microcline, Crystal Peak, Colorado
MFDU	Labradorite var. moonstone, Delta, Utah
MIPO	Microcline, Perth, Ontario
MKSD	Microcline, Keystone, South Dakota
MPSO	Microcline, Parry Sound, Ontario
OMGB	Oligoclase, Minas Gerais, Brazil
OMNC	Oligoclase, Mitchell Co., North Carolina
ORGH	Orthoclase, Gothic, Colorado
ORMN	Orthoclase, Montana

Because as much as 1/3rd of a K-feldspar's total dose may be due to its ^{40}K content, the accuracy of luminescence dating of K-feldspar extracts is in large measure dependent on an accurate determination of the internal K concentration in the K-feldspar grains. K concentrations of high-potassium minerals, in which the K_2O concentration exceeds 1% by weight can be measured commercially at low cost by several methods. However, the small size (typically <0.5 g) of K-feldspar extracts prepared for luminescence dating limits the methods to flame photometry, ICP-MS, or small sample gas-flow beta counting. In recent years, several luminescence laboratories have adopted gas-flow beta counting to determine the K content of their K-feldspar extracts. This allows the bulk K concentrations of ~200 mg of grains to be determined in several hours to days of counting. The advantage of this method is that it can be done in subdued light, and is non-destructive. Thus, a K-feldspar extract can first be beta-counted, and later used in luminescence measurements. This approach assumes that a K-feldspar extract is pure, i.e., that it is made up of K-feldspar grains only. The drawback of this assumption has led to several attempts to a more accurate determination of a feldspar extract's K concentration (Godfrey-Smith *et al.*, 1996; Dütsch and Krbetschek, 1997; Huntley and Baril, 1997).

For several K-feldspar extracts, we undertook to compare the bulk K_2O concentration obtained by using the beta counting approach with the K_2O concentrations of individual grains determined using X-ray fluorescence in conjunction with imaging electron microprobe analysis. Our results indicate that there are significant differences in the results obtained by the two methods.

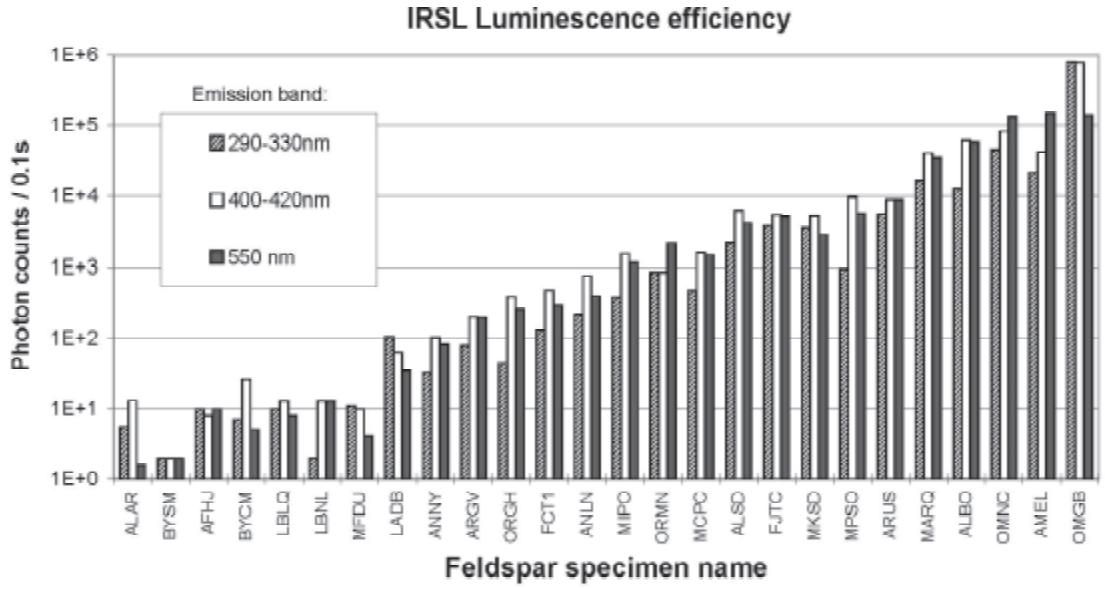

Fig. 1. *Absolute dose response luminescence efficiencies for a suite of feldspars, in the UV, deep blue, and yellow emission bands.*

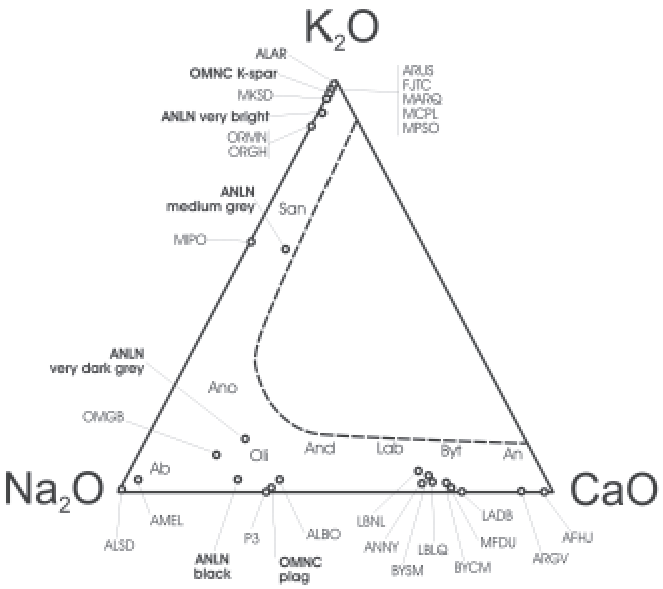

Fig. 2. *Placement of the feldspar suite on the K-Na-Ca ternary diagram, on the basis of X-ray fluorescence major element analysis. The descriptive information for samples OMNC and ANLN (in bold) refer to distinct phases detected by backscattered electron imaging.*

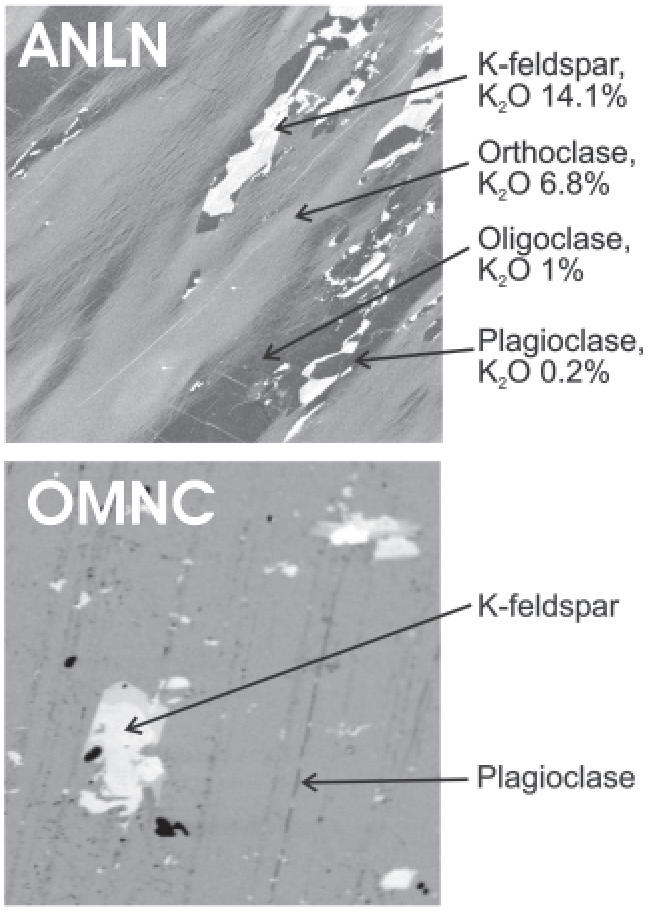

Fig. 3. *Electron backscattered images of two feldspars from the suite. K-feldspar areas are very bright (strongly reflective); feldspars with decreasing K content are progressively darker. Clearly, a comminution of such specimens into strictly mono-minerallic grains is, for all practical purposes, impossible.*

2. METHODS

Beta counting was undertaken in a Risø GM-25-5 multicounter system (Bøtter-Jensen and Mejdahl, 1985; 1988) at the University of Wales in Aberystwyth. This system allows five 100 mg samples to be measured simultaneously, including a K-feldspar standard (NIST 70a, K_20 = 11.8%) and MgO as a background. The counter gas used in the system was 99% argon - 1% isobutane. The cosmic ray contribution was determined and subtracted from the sample counts using anticoincidence counting. Good agreement has been found between this method and flame emission spectrometry for the bulk K concentrations of sand-sized K-feldspar extracts (Clarke *et al.*, 1995b).

A different approach to internal dosimetry has been developed at Dalhousie University. Once a K-feldspar extract is prepared, about 100 grains of the separate are mounted in epoxy and polished. The polished section is then coated with gold in a Minivac vacuum evaporator, and analyzed nondestructively using an imaging X-ray microprobe.

The instrument used is a fully automated JEOL 733 Electron Microprobe X-ray microanalyzer equipped with four wavelength spectrometers and an Oxford Link eXL 131 eV energy dispersive detector. The image analysis system produces backscattered electron images to provide quantitative data on abundance, size, shape, orientation, and association of grains in heterogeneous compounds. Compounds made up of heavier elements appear brighter in a backscattered electron image, thus the K concentration of K-feldspar grains makes them easy to distinguish from quartz. High resolution coloured images are saved as TIFF files for further processing.

For eight K-feldspar extracts prepared by heavy liquid separation, we used backscattered electron imaging to determine if all the grains in a mount (typically 35-45 grains are viewed) are in fact pure K-feldspar. Typically, this is not the case. As seen in **Fig. 4**, a significant proportion of the grains are pure quartz, and the remainder is K-feldspar. In some of the extracts, a few grains of plagioclase feldspar are also present.

Digital analysis of the backscattered electron image of each mount determined the total field area, the K-feldspar area, and the quartz area. Point-by-point X-ray fluorescence analysis for major element compositions was performed on 12-18 individual K-feldspar grains in each mount, and the data averaged to determine an average K_2O concentration of the K-feldspar grains. We also analysed the plagioclase grains when these were observed. This method is non-destructive in the sense that the few grains given over to the polishing process remain mounted indefinitely, and can be viewed at a later date for further analyses if necessary.

For five of the K-feldspar extracts analyzed by the above procedure, we also determined the K concentration using small sample gas-flow beta counting. After conversion of K to K_2O values, we compared these values with the K_2O concentrations of mounted K-feldspar grains. The results of the analyses are presented in **Table 2**.

Table 2. *Potassium concentrations of eight K-feldspar extracts, determined by small sample gas-flow beta counting and imaging microprobe X-ray fluorescence analysis.*

		Beta counting	Microprobe imaging analysis: N of grains	Image area		XRF analysis	N
No.	**Name**	**% K_2O**	**in image**	**% K-spar**	**% Qtz**	**% K_2O in K-spar grains**	
1	SAO	13.37	34	80.0	20.0	14.54 ± 0.56	16
2	MD40	9.88	38	69.8	30.2	14.81 ± 0.49	13
3	LP2	15.90	65	84.0	16.0	15.41 ± 0.56	12
4	MD15	8.08	40	71.9	28.1	14.52 ± 0.61	16
5	LLRS-K	1.69	23	7.0	93.0	15.94 ± 0.48	7
6	LMBS-K	ND		57.2	42.8	16.03 ± 0.39	
7	LISP-K	ND		14.8	85.2	16.16 ± 0.43	
8	LOGS-K	ND		12.9	87.1	15.42 ± 1.07	

1. % K obtained by gas flow beta counting is expressed as % K_2O, where K_2O = % K × 1.204
2. N = number of K-feldspar grains analyzed by XRF for K_2O content.

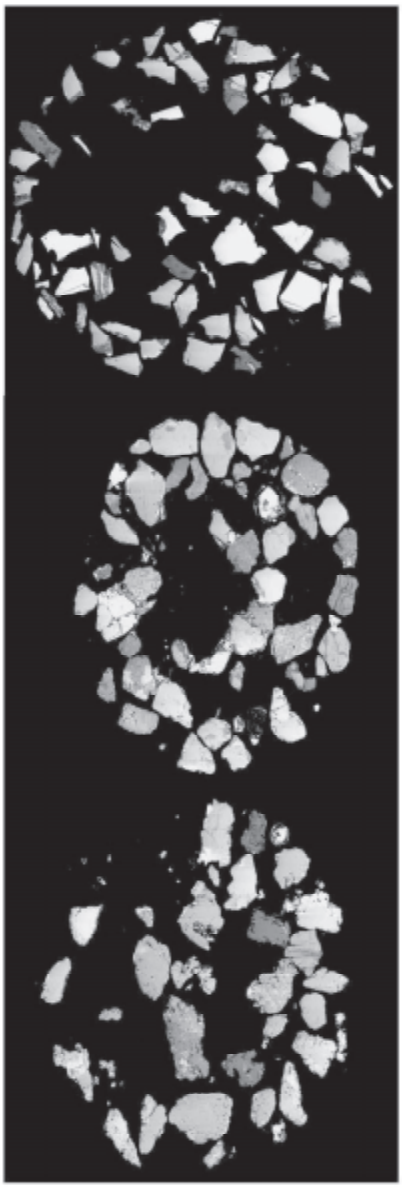

Fig. 4. *Electron backscattered images of three samples. In electron backscattered images, images containing heavier elements appear brighter. Here, the K-feldspar grains are very bright (strongly reflective) while plagioclase and quartz grains are grey to dark grey. Grains composed of more than a single mineral can be readily discerned in each field.*

It is clear from the data that the purity of a K-feldspar extract can vary a great deal. Even in the best case (sample LP2) it does not exceed 84%. This is a factor of several variables: 1. the proportion of K-feldspar grains in the grain size selected for heavy liquid separation; 2. the accuracy with which the specific gravity of the NapT (sodium polytungstate) solution used for heavy liquid separation is prepared; 3. the weight of dry grains immersed per unit volume of NapT solution; and 4. how many of the grains are lithic or polyminerallic, rather than monominerallic. In general, smaller grains are expected to be monominerallic, while larger grains from the same source are more likely to be lithic fragments of still-conjoined quartz and feldspar mineral fragments. In addition, the high viscosity and surface tension of NapT solution may prevent complete dispersion of all grains in the suspension, and allow heavier quartz grains to be dragged upwards by the lighter K-feldspar grains to which they adhere.

To compare the beta counting data with the electron microprobe analyses, we first converted the K content measured by gas-flow beta counting to bulk K_2O content using the relationship: K_2O % = K% × 1.2046. We assumed a 5% standard error in the measured K_2O content. Using this conversion, the K_2O contents determined by beta counting for five extracts range from 1.7 to 15.9% by weight. In contrast, the K-feldspar grains in eight extracts examined by imaging analysis have K_2O contents ranging from 14.5 ± 0.6% to 16.2 ± 0.43%, with an average value of 15.4 ± 0.7%.

To compare the two analytical results, we calculated the bulk K_2O content of each extract from the microprobe image analysis data, using the product of the mean K_2O content of individual K-feldspar grains of that extract and the absolute percentage of K-feldspar grains in the image. The standard error was calculated by adding in quadrature the uncertainty in the percentage of K-feldspar grains in the image and the standard deviation in mean K_2O content of the K-feldspar grains. The comparison is shown in **Table 3** and **Fig. 5**.

The large error in calculated bulk K_2O contents of the extracts is primarily due to the uncertainty in the absolute number of K-feldspar and quartz grains in each bulk extract. The rather small number of imaged grains (N)

results in a large standard counting error of ($N^{-½}$ x 100)%. Naturally the K-feldspar purity of an extract affects its bulk K_2O concentration. This is reflected in the much lower K_2O concentrations calculated for the bulk extracts in **Table 3**.

The K_2O concentrations of five K-feldspar extracts are correlated in **Fig. 5**. The dashed line is provided as a visual guide to a perfect correlation (R^2 = 1.00). An unconstrained linear least squares regression for the data shown yielded a correlation coefficient R^2 = 0.864. Of the five extracts compared, the data agree within ±1σ for four of them, and within ±2σ for the fifth. This agreement is judged to be reasonable, for the number of grains imaged during microprobe analysis.

Table 3. *K_2O concentrations of five potassium feldspar extracts. Beta counting values were obtained by direct measurement; microprobe values were calculated from area analysis of electron backscattered images and K-feldspar XRF analyses for K concentration.*

Sample	Beta counting	Microprobe
	K_2O %	K_2O %
SAO	13.37 ± 0.67	11.64 ± 2.30
MD40	9.88 ± 0.49	10.34 ± 2.07
LP2	15.90 ± 0.80	12.94 ± 1.84
MD15	8.07 ± 0.40	10.44 ± 2.04
LLRS-K	1.69 ± 0.08	1.12 ± 1.00

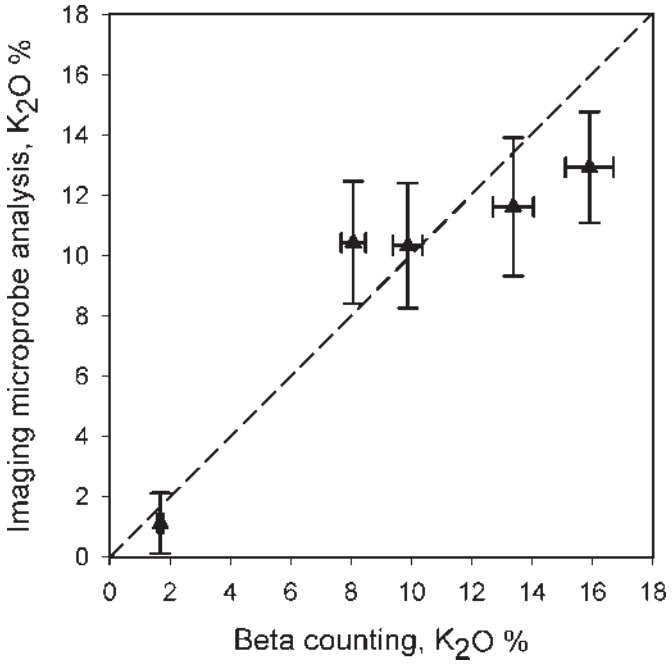

Fig. 5. *K_2O concentrations of five K-feldspar extracts obtained by the two methods show good agreement with each other. The standard deviation associated with beta counting is assumed at ±5% of the measured K_2O value.*

Beta dosimetry

In dosimetry-based dating applications, the K_2O concentration of sediments is used to calculate their beta dose rate. This dose rate is strictly external for quartz and plagioclase feldspar grains, however a significant proportion of the beta dose absorbed by a K-feldspar grain is due to the ^{40}K it contains. Additionally, the beta self-dose in a K-feldspar grain becomes more significant with increasing grain size, due to the attenuation of beta radiation over distances corresponding to sand-sized mineral grains (100-300 μm).

In an effort to relate the differences in K_2O contents determined by the above two methods to differences in total dose rate, we calculated the internal beta, total beta, and total dose rates for a typical test sediment. We assumed the following: depth below surface = 5.0 m, water content = 0.25 ± 0.05, bulk K_2O content = 1.5%, total alpha activity = 0.60 ± 0.01 ks^{-1}, Th alpha activity = 0.30 ± 0.04 ks^{-1}, grain size = 210 mm. We assumed that the grains were briefly etched with 10% HF, a procedure normally followed to render the external alpha dose contribution irrelevant. We also assumed negligible internal U and Th alpha activity. These values reflect those for a typical extract from a coarse sandy sediment such as a dune deposit. The total dose rate to a K-feldspar grain with a 15% K_2O content and the above variables, is 2.52 Gy/ka, of which 0.85 Gy/ka is due to the grain's self beta dose. If, due to bulk dilution effects, the internal K_2O content is underestimated at 10% K_2O, the self dose rate is reduced by 0.283 Gy/ka, to yield a total dose rate of 2.24 Gy/ka. For the 5 samples in **Tables 1** and **2**, we found that adjusting the K_2O content from that measured by gas-flow beta counting to that determined by microprobe XRF analysis increased the internal dose rate by as much as 0.32 Gy/ka, or 14% of the total dose rate.

3. SUMMARY

We determined that gas-flow beta counting tends to underestimate the internal K content of K-feldspar grains in nominally pure potassium feldspar extracts due to dilution effects of quartz and occasional plagioclase grains remaining in the extract. Even in the purest K-feldspar extract, the non-potassium feldspar grains comprised 16% of the extract. Microprobe XRF analysis of 8 extracts leads us to conclude that, on average, the internal K_2O content in K-feldspar grains is 15.4 ± 0.7%. In spite of the fact that we have made our K-feldspar extraction procedure more stringent, now consisting of two successive heavy liquid separations at 2.54 g cm^{-3} we find that a small percentage of the extract is invariably composed of quartz or plagioclase. Thus, a small dilution effect of the non-K grains in gas-flow counting appears to be unavoidable. We suggest that the internal K_2O content of K-feldspar grains should best be measured by imaging microprobe XRF analysis. Barring that, the average K_2O content given here may be used. Since this study was initially reported (Godfrey-Smith *et al.*, 1996) we have confirmed this value for a wide range of samples; the highest mean detected by us is 17.0 ± 0.9 % K_2O (N=11), which agrees with our

suggested value at 1σ. Errors introduced into the dose rate calculation with this value are expected to be less than those resulting from small sample beta counting of quartz-diluted extracts. Finally, we recommend that small sample gas-flow beta counting is a useful as a tool to verify the purity of a K-feldspar extract; if such an extract is observed to yield a K concentration significantly below 12.5 (equivalent to a K_2O concentration of 15%), the extract should be re-purified with further heavy liquid separation steps.

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