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DETERMINATION OF PURITY OF MINERAL SEPARATES USED IN K-Ar DATING — AN INTERPRETIVE REVIEW *

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Abstract

A literature survey of potassium contents of 2700 biotites shows average K_2O values of :

7.99 percent in K-Ar dated samples,
8.57 percent in bulk chemically analyzed samples,
and 9.29 percent in biotites analyzed by electron microprobe.

Comparisons were also made of: 1) CaO and Na_2O in bulk chemical analyses and microprobe analyses, 2) reported percent impurity *vs.* percent K_2O and 3) relative error in repetitive determinations *vs.* percent K_2O. All data indicate that biotites should generally contain between 8.0 and 9.5 percent K_2O, values below 8.0 percent K_2O being due to sample impurities. Impurity levels are difficult to determine and are often underestimated or not reported, even though they can be crucial to interpretation of K-Ar ages. Consideration of percent K_2O and relative error may help geologists to re-evaluate published age data as well as provide guidelines for those currently preparing geochronologic samples and standards.

Introduction

The determination of the purity of separated minerals used in K-Ar dating may be critical to the interpretation of the ages obtained (Engels 1971). Methods for determining the purity of mineral separates are often difficult, time consuming, and inaccurate. X-ray diffraction techniques, though rapid, are limited by a fairly high threshold of detection for one mineral in a separate of another. Grain-counting is time consuming and is severely affected by differences in particle shape and mineral density, as well as by an inability on the part of the investigator to properly weight aggregate grains. Modal counts of polished grain mounts may help eliminate some of the deficiences of grain counting, but, again, these can be time consuming and inaccurate. Additional criteria may be needed to improve control of mineral separates and to evaluate published data for which mineral purity estimates are not provided.

This discussion is limited to the effect of impurities on the measured potassium concentrations in biotites because of the availability of large amounts of data on biotites. Biotite is a relatively easy mineral to separate,

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however, and obtaining a pure sample is often much more difficult in other mineral separates. Analytical problems related to sample impurity will be compounded for minerals of low potassium content, *e.g.* amphiboles, pyroxenes, and plagioclases, which can be contaminated with relatively high potassium-bearing phases. Although this work will be confined mainly to a discussion of the problem for biotite, the reader is reminded that biotite is probably the "best case," and that the principles discussed are of even greater importance for other minerals.

Potassium Content as an Index of Sample Purity

The potassium content of analyzed biotites may be a very good indicator of degree of sample purity (Evernden & Kistler 1970, p. 2-4).

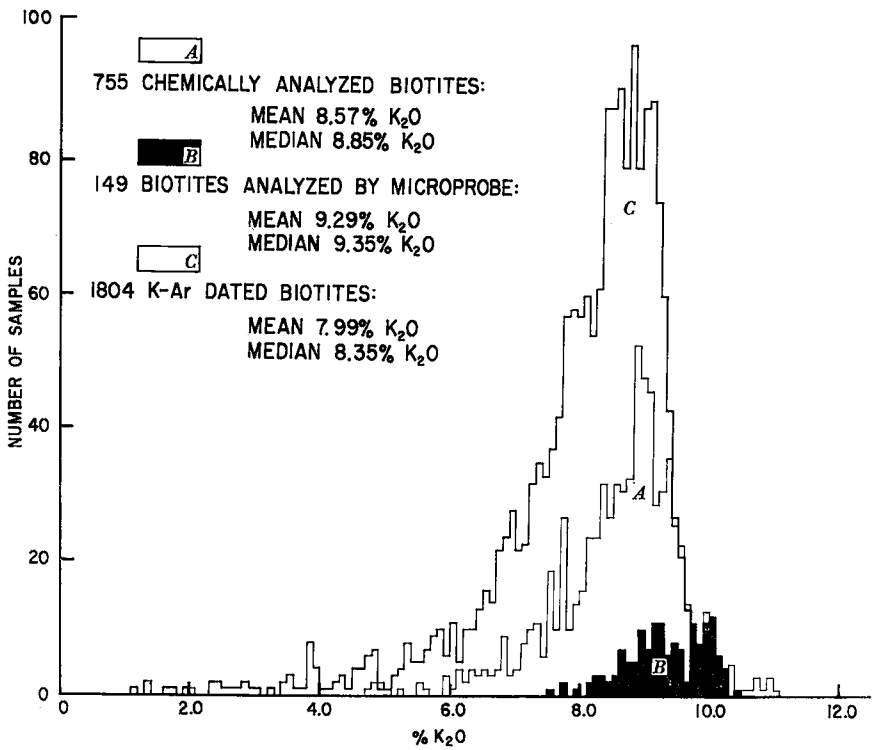


Fig. 1. Percent K_2O in biotites from

A. Papers on the chemistry of biotites.
B. Papers giving microprobe data on biotites.
C. Papers giving K-Ar data on biotites.

K_2O contents reported in 39 papers[1] on the chemistry and structure of biotites are plotted as histogram A of Fig. 1. While no attempt has been made to include every biotite ever analyzed, enough have been included that the addition of data from other papers should not appreciably alter the overall picture. The average K_2O content of 755 samples is 8.57 percent, and although these samples show a fairly regular Gaussian distribution, there is a perceptible skewing to the low-potassium side, undoubtedly due in part to sample impurity.

K_2O values for 149 biotites analyzed by electron microprobe are shown by histogram B of Fig. 1. Twelve papers were available, and although the data are few, the overall average of 9.29 percent K_2O is markedly higher than that for chemically analyzed samples (A). Only a few percent of the samples fall below 8.0 percent K_2O. In a similar study of potassium contents of biotites used for K-Ar dating, the average of 1804 dated biotites from 71 sources, plotted as C of Fig. 1, is 7.99 percent K_2O. The extreme skewing toward the low-potassium side indicates that many of the samples are severely contaminated. Taking the bulk chemical and microprobe data as a criteria, the average dated biotite at 8 percent K_2O could contain between 6 and 15 percent impurity. Those containing less than 8 percent K_2O (about 37 percent of the samples) are more seriously contaminated. While the ages of such biotites might prove to be the same as those of more carefully prepared separates, the chances are good that some of them may be completely erroneous (Engels & Ingamells 1970 ; Engels 1971).

Calculated K_2O values for the pure magnesium and ferrous end members of the biotite group are 11.3 and 9.2 percent, respectively, but vacancies and/or extensive interlayer cation substitution (Ca, Na, Rb, Ba, etc.) have been postulated to account for lower K_2O values. However, CaO or Na_2O in amounts greater than one percent would be necessary to lower the K_2O to less than 8 percent. Since most of the papers used to plot histogram A reported CaO and Na_2O values, these values were plotted in histograms (Fig. 2) to check out this possibility. While many high CaO and Na_2O values are reported, a small collection of electron microprobe data plotted above the bulk chemical data suggests that the high Ca and Na values may be unrelated to biotite. That is, while there may be an occasional mixed-layer biotite-Ca/Na mica, in general the higher

[1] Data used to construct this histogram and those which follow were taken from the literature and from some unpublished sources at random, with the intention of representing as many laboratories and investigators as possible. The entire bibliography of some 120 entries would be unwieldly to include in a paper of this size but is available upon request.

CaO and Na_2O values are contained in apatite, amphibole, feldspar, glass and other impurities in the biotite separates. Three authors of papers in this group indicate that this is indeed the case. (The CaO histogram is probably a more meaningful indicator of impurity than the Na_2O plot,

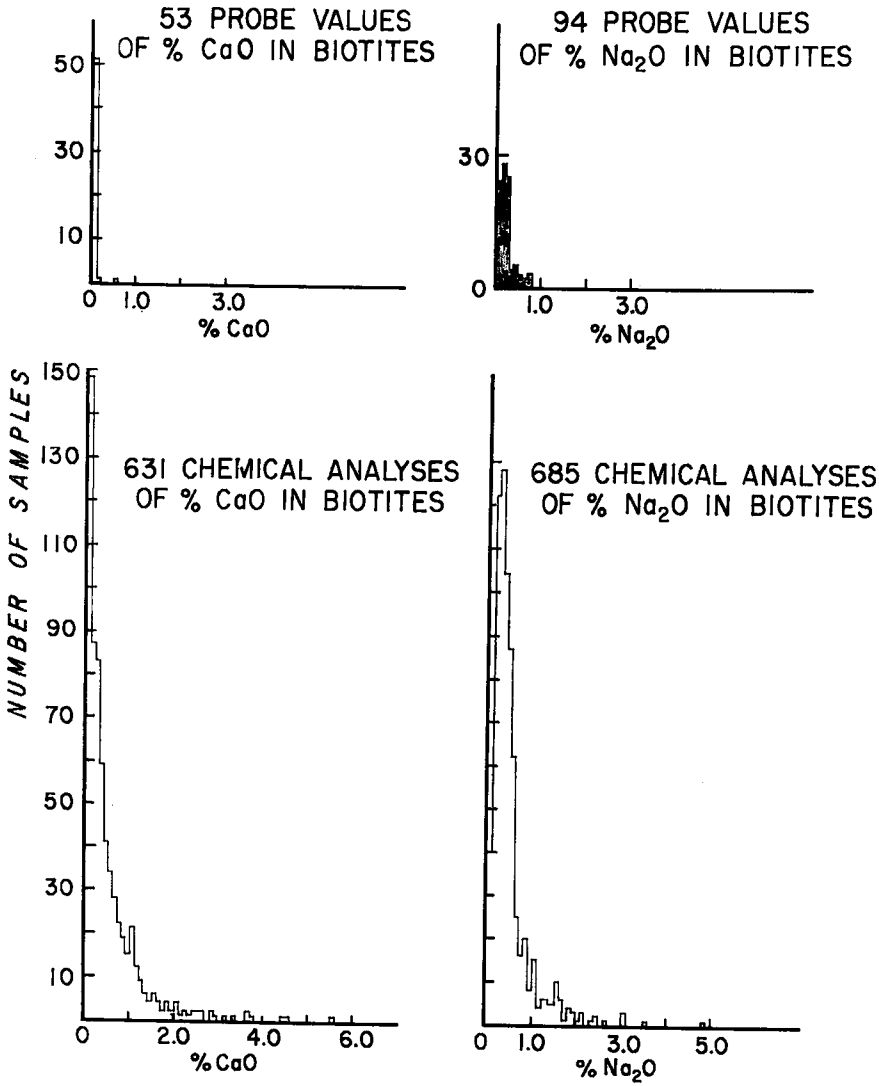


FIG. 2. Comparison of CaO and Na_2O in bulk chemical analyses and microprobe analyses.

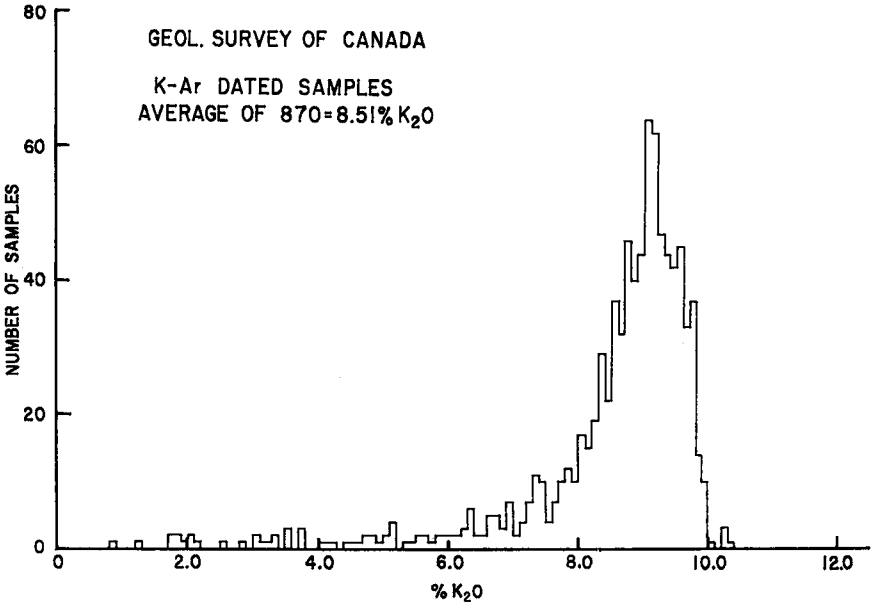


FIG. 3. Histogram of percent K_2O in K-Ar dated samples from the Geological Survey of Canada.

partly because reported Na_2O values may include Li_2O in samples for which the latter was not determined separately.)

A histogram of the K_2O contents of 870 biotites dated by the Geological Survey of Canada (Fig. 3) shows the average K_2O content to be 8.51 percent; only 19 percent of the samples fell below 8.0 percent K_2O. In this case, it can be shown that the skewing of the histogram toward the low K side in Fig. 3 is due to sample impurity, as impurity data are given in the Geological Survey of Canada reports. Thus, there is compelling evidence that pure biotites should, on the average, show at least 8.5 percent K_2O and possibly as much as 9.4 percent.

The reported percent impurity *vs.* percent K_2O for the Canadian samples is shown in Fig. 4. Only eight of the papers used to construct C of Fig. 1 give impurity data; these are plotted *vs.* percent K_2O in Fig. 5. Both of these plots indicate that clean biotites should show a high K_2O content. This kind of plot should indicate a trend toward 100 percent impurity at 0 percent K_2O, or slightly above 0 percent K_2O if there is potassium in the impurities. Fig. 5 shows a trend that would indicate an intersection at 60-75 percent impurity and 0 percent K_2O, and this can be

interpreted to be a measure of the degree to which the impurity has been generally underestimated. Fig. 4, on the other hand, shows more of a trend toward 100 percent at 0 percent K_2O, indicating that the combined optical and *x*-ray technique for impurity determination used by the Canadian Survey (Rimsaite 1967) is probably quite accurate.

It is possible that some of the low-potassium samples in Fig. 4 are actually relatively pure low-potassium biotites, rather than contaminated ones. Indeed, while making the literature search for complete chemical analyses to be used in Fig. 1, the author noted occasional biotites with, say, 6 percent K_2O and 10 percent H_2O. Such samples are probably mixed-

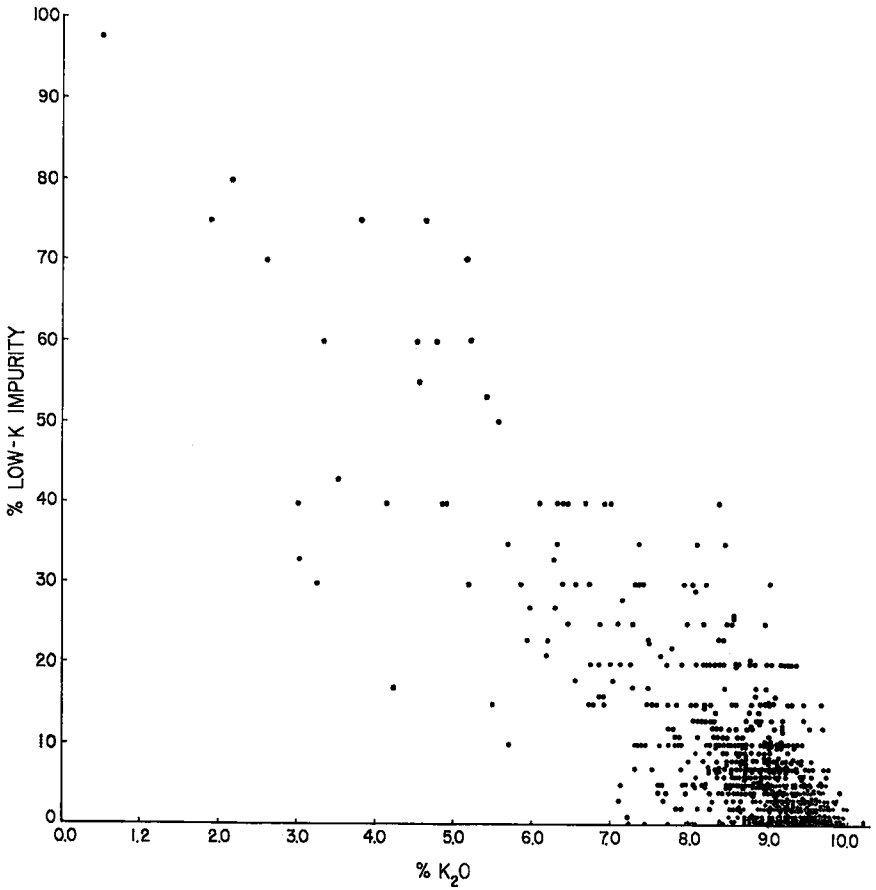


FIG. 4. Percent impurity *vs.* percent K_2O in K-Ar dated samples from the Geological Survey of Canada.

layer vermiculites and hydrobiotites, and are rare. If a line is drawn on Fig. 4 from 100 percent impurity and 0 percent K_2O to 0 percent impurity and 8.0 percent K_2O, one finds that only about 3 percent of the samples fall to the left of the line. This can be interpreted to mean that, in general, there is a less than 3 percent chance that an uncontaminated biotite will show less than about 8.0 percent K_2O. (The same conclusion could be reached from the microprobe data in Fig. 1B.) Considering the fact that the underestimation of impurities is common and that this would account for some of the samples in the 3 percent group, the probability of an uncontaminated biotite containing less than 8.0 percent K_2O may be much less than 3 percent.

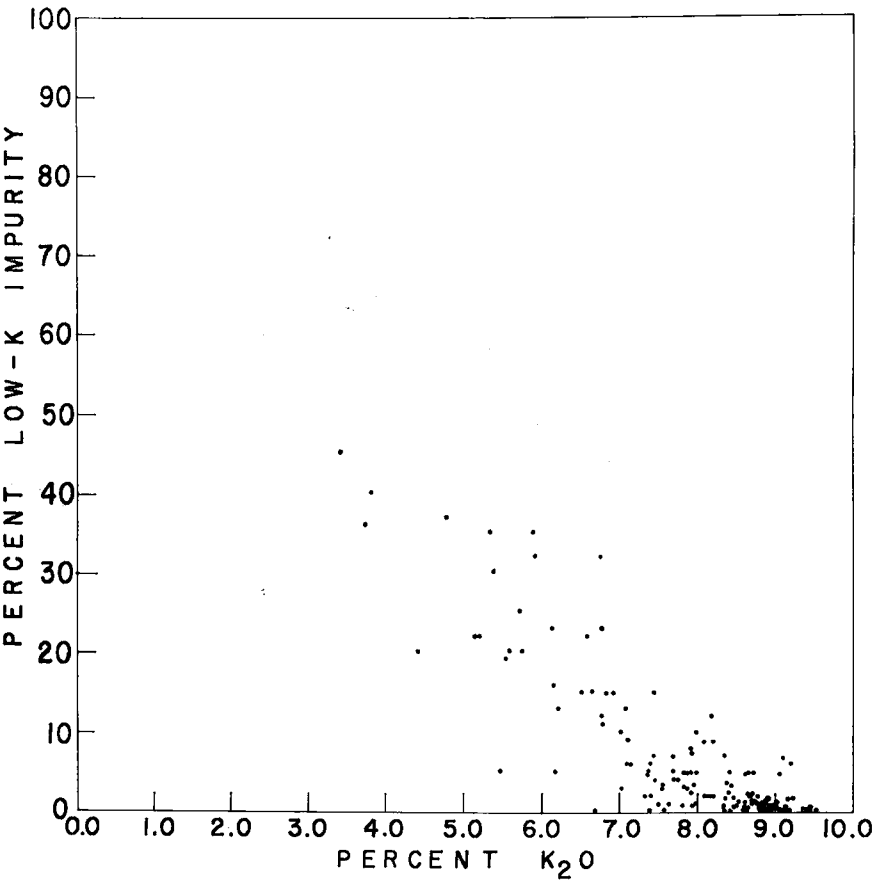


FIG. 5. Percent impurity *vs.* percent K_2O for K-Ar dated samples in the literature.

Relative Error as a Measure of Sample Inhomogeneity

An additional criterion that can be applied in determination of sample purity is the relative error obtained by running several splits of the same sample for potassium. For example, Table 1 shows the results of repetitive K_2O determinations on several minerals, all of which were originally thought to be pure and homogeneous. The first four columns indicate homogeneous mineral concentrates and establish the precision of the method [2] as well. Data in the remaining four columns indicate that these samples are impure and/or inhomogeneous and this has been substantiated by more rigorous examination and further purification of the concentrates. Furthermore, the data show that duplicate analyses may not expose existing sample inhomogeneity. There are numerous examples in the last four columns of agreement between any two analyses and gross disagreement among all the samples in the set.

Table 1. Percent K_2O in Mineral Separates

	Biotite	Phlogopite	Pyroxene	Amphibole	Biotite	White mica	Pyroxene	Amphibole
	8.55	10.20	.0115	.913	8.97	7.59	.0157	.919
	8.56	10.20	.0115	.913	8.83	8.80	.0185	.923
	8.55	10.17	.0115	.913	8.84	8.90	.0190	.918
	8.53	10.17	.0113	.914	8.90	8.78	.0113	.911
	8.55	10.20	.0115	.915	8.87	8.87	.0157	.911
	8.53	10.17	.0113		8.57	7.90	.0180	
		10.17			8.95	7.87	.0203	
		10.18					.0203	
		10.17					.0173	
		10.18					.0203	
		10.20					.0190	
		10.18					.0207	
X	8.55	10.18	.0114	.914	8.85	8.39	.0180	.916
S	.01		.0001	.001	.13	.57	.0026	.005
R	.14		.87	.11	1.50	6.82	14.7	.58

(Relative deviation in percent)

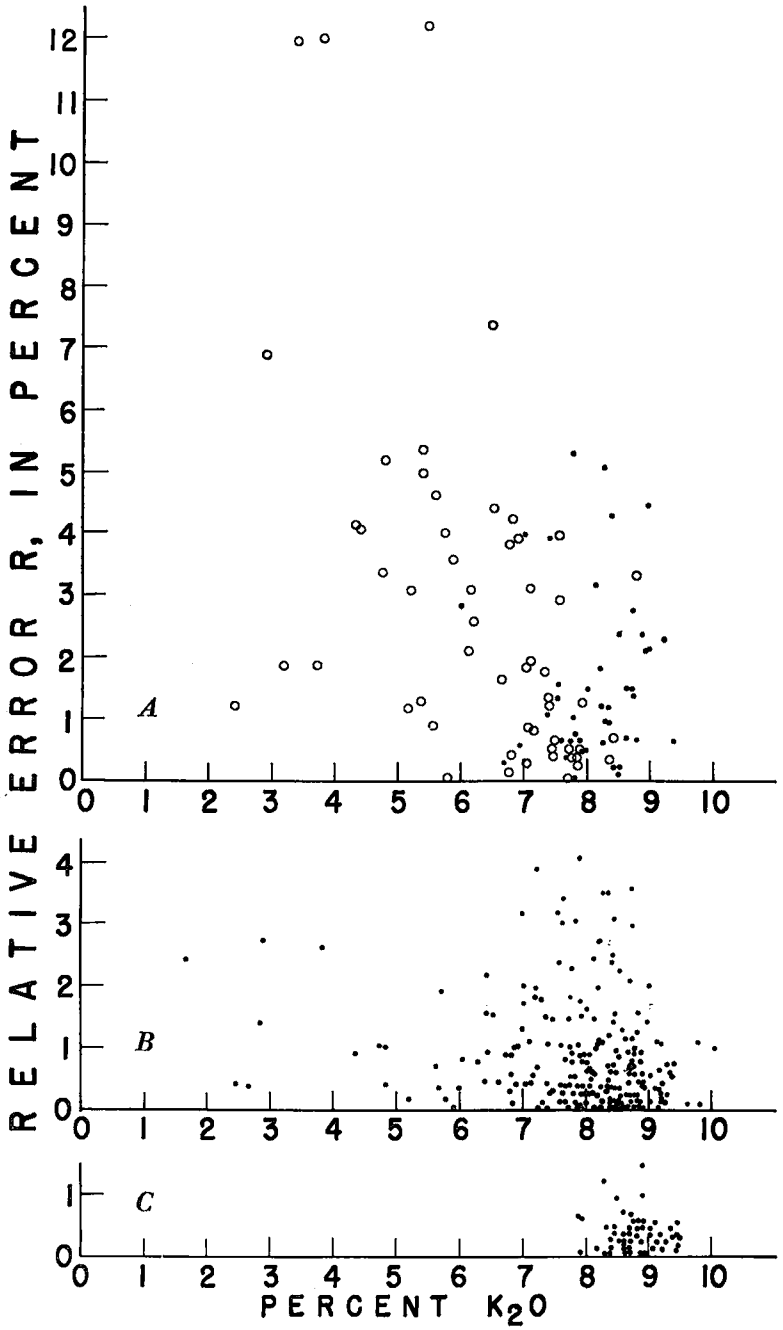
[2] Potassium determinations were made by L. B. Schlocker, on an Instrumentation Laboratories flame photometer with a lithium internal standard using a method described by Engels & Ingamells (1970).

Given a precise method for potassium determination, relative errors from multiple analyses can be ascribed entirely to sample inhomogeneity (Engels & Ingamells 1970; Evernden & Kistler 1970). Figure 6 is a plot of percent K_2O *vs.* relative error on duplicate and multiple potassium determinations for three groups of data. Data in Fig. 6A are from Armstrong (1966, 1970), and Armstrong & Hansen (1966), and Armstrong *et al.* (1970), recalculated to percent K_2O. Data in Figs. 6B and 6C are unpublished data from a number of investigators and geologic areas (Ingamells, personal communication). The methods, chemists, and time span over which the latter two groups of analyses were performed are identical; the two groups differ only by having different mineral separators and apparently a different philosophy on the part of the investigator of what constitutes mineral purity.

All three groups show a "fanning out" at lower K_2O content that is directly correlative with sampling problems due to impurities, as predicted by Engels & Ingamells (1970). Furthermore, the agreement of any two analyses at lower K_2O values may be quite fortuitous. This is shown dramatically in Armstrong's (1970) Table 3, from which some of the data in Fig. 6A were taken. Low-potassium biotites which have been run more than twice often show good agreement between two splits and poor agreement on a third. This has to be borne in mind in K-Ar dating because good agreement between two splits analyzed for potassium does not at all guarantee that the argon split will be representative of the same material.

Again, it might be argued that the low-potassium biotites which *do* appear to be sampleable, *i.e.*, do not show a large relative error, truly reflect interlayered cation substitution. This may be true in rare cases, but does not hold for the 265 samples plotted in Fig. 6. The circled points in Fig. 6A are those samples for which Armstrong reports impurities of > 2 percent, and these samples account almost entirely for the "fan of unsampleability" at less than about 8.5 percent K_2O. Spot checks of low potassium samples in Figs. 6B and 6C show that an appropriate amount of impurity accounts for the low potassium content.

In most cases, then, either low K_2O content, unsampleability, or both, may be a good indication of sample inhomogeneity. A semiquantitative estimate of the amount and type of impurity may be necessary to the interpretation of ages (Engels 1971), as well as to set limits of sampling error on the ages. Engels & Ingamells (1970) evaluated relative errors for an ideal system in which both the mineral studied and its contaminant were essentially equal in particle shape, size, and density. The plots in Fig. 6 contain a variety of grain sizes and indicate that relative errors in non-ideal systems may be very much greater than can be predicted. An


RELATIVE ERROR R, IN PERCENT
A
B
C
PERCENT K_2O

empirical approach may therefore be the only useful one. If impurities exist as free grains, grain counts may be useful, but the difficulty of estimating percentages of aggregate grains can make a purity estimate useless. In many cases, impurities probably exist as very fine inclusions within thick mineral grains and elude detection during a grain count. Zonation and borders of foreign material may be even more difficult to evaluate; as noted by C. O. Hutton (personal communication 1970), a border 1/10 the diameter of a crystal provides about ½ the total volume. It is the author's experience that there is a "threshold of detection" of perhaps several percent of impurities in microscopic grain counting which is analogous to the threshold observed in *x*-ray diffraction techniques. Certainly micas with interlayered chlorite whose presence is masked by the biotite colour may be undetectable at several percent by either *x*-ray diffraction or grain counting techniques. One of the most difficult cases of all involves two generations of the same mineral. Hand picking the samples and then counting in oils might be imperative in this case, as neither *x*-ray nor bulk chemistry would be expected to reveal the "impurity."

Microchemical Techniques

A potential solution to the problem of sample purity estimation for a number of mica samples is illustrated in Table 2. The second column gives the impurities estimated from grain counts, the third the percent K_2O found on the bulk samples by standard analytical methods (Engels & Ingamells 1970). In the fourth column, the percent K_2O for the pure mica was estimated by subtracting out the impurities by grain count only. The next column is a similar recalculation, but where contaminants were nonmicaceous, the grain counts were weighted by a factor of 1.5 to account for their blocky shape. (This figure was arrived at empirically from a comparison of weight of impurities *vs.* grain count on sample L618). The last two columns illustrate the use of microchemical techniques to check the sample purity estimates. The first technique involves the determination of percent K_2O on a very small (0.010 gram) handpicked sample according to a procedure described by Ingamells (1970 in press). The second is the determination by G. K. Czamanske of percent K_2O by electron

← Fig. 6. Relative error, R., *vs.* percent K_2O for:
A. Armstrong (1966, 1970), Armstrong & Hansen (1966), and Armstrong *et al.* (1970).
B. Unpublished data, personal communication, Ingamells (1970).
C. Unpublished data, personal communication, Ingamells (1970) (by a different investigator than data in B).

TABLE 2. MICROCHEMICAL TECHNIQUES

Sample	Approximate impurity (%)	% K_2O on large (0.1g) bulk sample	% K_2O recalculated to 0 impurity		% K_2O on small (0.010g) handpicked sample *	% K_2O on selected grains (No. in parenthesis) by electron microprobe
			No weight factor	Nonmicaceous minerals weighted×1.5		
B3203 (MIT) (standard biotite)	"< 1 garnet, Q, feldspar, kyanite, hornblende"	8.95 9.03	~ 9.1	~ 9.2	9.36 ± .09	9.38 ± .29 (10 grains)
Kaavi biotite (Lamont Std.)	~2 Q, 2.5% chlorite (Est. by author by grain count)	8.49	~ 8.9	~ 9.0	9.15 ± .09	9.55 ± .22 (10 grains)
P207 (USGS) (Std. Muscovite)	≃2 Q, Mag., Bio., Ap., Ep. etc. (Est. by author by grain count)	10.4	~10.6	~10.7	10.52 ± .11	10.94 ± .22 (10 grains)
LP-6 Biotite	< 0.3 (Est. by author by grain count)	10.10	~10.1	~10.2	10.19 ± .10	n.d.
O-425 Biotite	< 0.3 (Est. by author by grain count)	9.46	~ 9.5	~ 9.5	†	9.58 ± .17 (5 grains)
JE-16-67 Biotite	< 0.3 (Est. by author by grain count)	9.44	~ 9.5	~ 9.5	†	9.65 ± .30 (6 grains)
8W 464 Biotite	< 0.3 (Est. by author by grain count)	9.50	~ 9.5	~ 9.5	†	9.73 ± .16 (5 grains)

TABLE 2. (cont'd.)

L618 Biotite	~32 Hb, Q, Ep, by wt. ~22 Hb, Q, Ep by grain count	5.89	~ 7.3	~ 8.3	8.46 ± .08	9.02 ± .71 (11 grains)
L618 Biotite repurified	~5 Hb, Q, Ep (Est. by author by grain count)	7.92	~ 8.3	~ 8.5		
L589 Biotite	~6.4 Hb, Ep, Q, Ap, Chl., Px (Est. by author by grain count)	8.09	~ 8.6	~ 8.9	8.89 ± .09	n.d.
62AKa10 Biotite	~2.6 CHL, 2.6 Q, Ap, etc., 0.4 clay(?)	8.41	~ 8.91	~ 9.03	8.88 ± .09	n.d.
62Ale 1 Biotite	.3 Ap. 1.9 Chl., .4 Hb (Est. by author by grain count)	8.79	~ 9.02	~ 9.06	9.18 ± .09	n.d.
M110159 Biotite	10 chlorite 2 Hb, other (Est. by author by grain count)	6.90	~ 7.8	~ 7.9	n.d.	n.d.
M110507 Biotite	9 Hb 1 other (Est. by author by grain count)	6.97	~ 7.7	~ 8.1	n.d.	n.d.

* Analyses by L. Schlocker.
** Analyses by C. O. Ingamells.
*** Analyses by G. K. Czamanske.
† No impurities noted in > 300 grains, so not handpicked.

microprobe on a few grains of mica which were carefully selected for their clean appearance.

The microchemical techniques again show that the impurities probably are always underestimated. The reasonably good agreement for the last three columns illustrates the necessity for accounting for grain shape in making grain counts. The fact that the probe values are always slightly higher than those of the bulk analyses on small handpicked samples may simply reflect the inability of the operator to handpick a 100 percent pure sample. The large differences between bulk analyses on unpicked samples and microchemical analyses for standard samples shown here indicate that an entirely new approach to standard sample preparation is going to be necessary before microchemical techniques come into wide use. That is, extensive grain counting, *x*-ray analysis, and agreement of bulk analyses with at least one type of microchemical technique should constitute the minimum criteria for a sample to be used as a microprobe standard, for example.

Since accurate purity estimates of dated minerals are essential to interpretation of both the precision and the accuracy of age determinations, it is fortunate that percent K_2O may by itself be a good indicator. Biotites showing 9.0 to 9.5 percent K_2O probably contain from 0 to 5 percent impurities, and biotites containing less than 8.0 percent K_2O are almost certainly contaminated and should be re-examined. The last two samples in Table 2 were chosen to illustrate this point. As they showed both low K and severe unsampleability, they were pulled from a reference set for a rough purity check. The check indicated that both are severely contaminated and that the true K_2O content should be at least 8.0 percent. The first of the two showed so much free chlorite that the estimate of impurity is probably very low owing to interlayered chlorite that simply could not be detected. If a more accurate purity estimate becomes necessary to an adjustment of their age, and if further purification is impossible, the use of either of the two microtechniques shown here should provide an adequate estimate for such samples.

As stated earlier, this discussion was of necessity confined to biotites, but a few extrapolations to other minerals may be suggested. First, while it is generally conceded that minerals under a given set of conditions may have different argon retentivities, the effects of contaminants with different apparent ages are almost never evaluated in the literature. Secondly, while biotite may be relatively unaffected by a moderate amount of impurity, such as hornblende, the same amount of biotite in a hornblende concentrate can have devastating effects on accuracy in age dating (Engels

1971). In fact, if an average hornblende is contaminated with an average biotite, the hornblende must be greater than 95 percent pure before it takes over from the biotite as the major supplier of K (and of Ar, provided the ages are equal). As hornblende is much more difficult to separate, and with the possibility that it has a different age, the likelihood of error is greatly increased. If the procedure used for the separation of biotites in general as evidenced by histogram C of Fig. 1 are indicative of those used for hornblende, some of the published data should no doubt be re-evaluated. For example, ages given in the very recent literature are reported to be "hornblende dates," even though the samples contain enough biotite impurity to account for virtually all the K in the bulk analysis. Thus, the age and the conclusions regarding retentivity cannot be ascribed to the amphibole. In some cases "biotite-hornblende" age pairs can be shown to be biotite-impure biotite pairs, and so on.

The problem is even more intractable when one considers the extremely low-potassium minerals sometimes used in K-Ar dating. For example, a typical plagioclase may contain about 0.1 percent K_2O. If it is contaminated with a 10 percent K_2O potassium feldspar, it will take a plagioclase purity greater than 99 percent to have more K supplied by the plagioclase than the K-feldspar. Pyroxenes, which are typically hard to separate from amphiboles and biotites, pose a still greater problem. In fact, Amaral *et al.* (1967) pointed out that pyroxenes contain almost no K of their own and that the difference between higher reported values and those found by electron microprobe is due to contamination. This may be a situation analogous to that of Ca and Na in biotite discussed above. While complete separation is often impossible, it is necessary to take mineral separations far enough that the effects of contaminants with potentially different ages can be evaluated.

Absolute errors due to the age difference effect of contaminants constitute only one of two major problems, the other being due to errors of unsampleability, as shown in Fig. 6. Biotites have so much potassium relative to the majority of possible contaminants that the age effect should generally be subordinate to the sampling error in all but the most contaminated samples. (See, for example, Evernden & Kistler 1970, who report an age increase in samples containing 90 percent chlorite.) For contaminated low-potassium minerals, the sampling-error effect will still be present, but it may be overwhelmed by a huge error due to the age-difference effect (Engels 1971).

Conclusions

Percent K_2O of a biotite is a good indicator of sample purity. Data presented here suggest that common biotites should generally contain from 8.0 to 9.5 percent K_2O and that those showing less than 8.0 percent K_2O are likely to be contaminated with one or more low-potassium impurities. Standard techniques for detecting impurity are time consuming and often result in low estimates of impurities.

Microchemical techniques suggest that the range in values of other minor constituents such as Ca and Na may be even more severely restricted and substitution far more limited than bulk chemical analyses have indicated. Impurities cannot be totally eliminated in mineral separates, but chemical data should always be adjusted according to amount and composition of impurities. More accurate purity estimates are crucial to the determination of both precision and accuracy of potassium argon ages of biotites and probably are even more critical for low-potassium minerals.

Acknowledgements

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