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Test of plagioclase dispersion method and rapid probe analysis

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Abstract

Refractive-index measurements on cleavage flakes of analyzed intermediate plagioclases from the Kiglapait intrusion provide a satisfactory test of the dispersion chart based on a Smith-Tsuboi determinative curve. Accuracy is somewhat improved by use of a Smith-Slemmons determinative curve, $An = 1944(n - 1.5290)$ for the range An_{31-84}, where n is the low refractive index on basal cleavage flakes. New determinations have a standard deviation of ±0.6 mole percent An (N = 16) about this curve in the region An_{31} to An_{66}. A rapid probe analysis procedure yields comparable precision with 10 grains per sample, but also gives Or. Both methods are powerful generators of useful compositional data unobtainable by bulk analysis.

Dispersion method

The revised dispersion chart (Morse, 1968) was based on refractive indices measured by J. R. Smith (in Hess, 1960) and optic orientations of Tsuboi (1923). The optic orientations of Slemmons (1962) yielded a slightly different determinative curve, and in 1968 I saw little basis for choosing between the two (Morse, 1968, p. 109). The present work furnishes a test of the relative merits of the Smith-Tsuboi (S-T) and the Smith-Slemmons (S-S) curves. The S-S curve appears preferable in the region An_{31}–An_{66}.

The new data were measured on a suite of 18 analyzed plagioclase feldspars from the Kiglapait intrusion. Feldspars sized 80–200 mesh (185–75 μm) were purified by flotation in heavy liquids and by magnetic separation, followed in some cases by leaching for 5 hours in dilute HNO_3 to remove iron hydroxide stains. Wet-chemical analyses were performed by K. Aoki, Tadashi Asari, and H. Engleman. Two analyses by H. B. Wiik have been published (Morse, 1969); the newer ones will be published in a longer paper on Kiglapait feldspars. Triplicate analyses by different laboratories were performed on seven samples, and duplicate analyses on four samples. The unweighted average of replicate analyses is used here for calculation of molar An/(An + Ab). A great many Kiglapait feldspars have also been analyzed for potassium, which ranges from about 1300 ppm K ($\approx Or_1$) near An_{67} to about 6000 ppm K ($\approx Or_5$) near An_{30}. The suite is clearly low in potassium. Feldspars below about An_{35} are antiperthitic.

The refractive index α'_{001} for (001) cleavage flakes was measured in oil immersion using a substage Jena interference monochromator and commercial plagioclase dispersion liquids (Cargille Set 547) made up from NBS-referenced end members. The mean wavelength of match for an ensemble of grains was converted, after temperature correction, to refractive index n_D *via* the plagioclase dispersion curves of the Hartmann dispersion net (Morse, 1968). As to zoning, the mean range of An within samples is 6.3 mole percent, and the maximum observed range is 14 mole percent, as inferred from the wavelength data.

The results (Table 1) are compared with the S-S curve and the curve of Burri *et al.* (BPW curve; 1967, Table 1, Section 438.02) in Figure 1. The two most sodic samples contain 7 and 8 percent Or, and are excluded from further discussion because of their potassic nature and uncertainty about the effects of exsolution. The data for the region An_{31} to An_{66} have also been compared with the S-T curve.

The S-T and BPW curves have more than one segment in the region of interest. Comparison of slopes and intercepts of regressions would therefore be difficult. A test based on mean algebraic deviation (MAD) serves the purpose of comparison more simply. For this purpose, a deviation is defined as An(obs)–An(calc), where An(calc) is the value predicted by the curve to be tested and An(obs) is taken

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Table. 1. Refractive indices for Kiglapait plagioclase

Sample	An	α'_{001}	No. grains
KI 3645	66.1	1.5628	10
KI 2008	65.4	1.5620	53
KI 3223	63.4	1.5615	16
KI 1154	63.3	1.5615	25
KI 3230	58.8	1.5595	16
KI 3276	54.8	1.5572	14
KI 3360	54.5	1.5574	10
KI 3362	51.6	1.5560	15
KI 3363	51.1	1.5555	15
KI 3367	51.0	1.5548	15
KI 3369	48.3	1.5538	15
KI 3345	44.3	1.5521	10
KI 3243	42.6	1.5509	10
KI 3347	39.0	1.5495	10
KI 3377	34.3	1.5469	57
KI 3002	30.9	1.5448	19
KI 3379	28.0	1.5425	15
KI 3009	25.7	1.5415	15

An is mole percent An/(An + Ab).

Refractive index is referred to the D line.

from Table 1. The algebraic mean of such deviations is minimized for the curve most closely resembling the Kiglapait data set itself, as can be seen from the fact that the algebraic mean would be zero if the curve and the regression on the data set were identical. The standard deviation (SD, with deviation defined as above) is useful as an indicator of random error. In the list that follows, the MAD is followed by the SD in parentheses. Data are given for N = 16, in mole percent An: S-S curve, −0.11 (0.62); S-T curve, +0.34 (0.65); BPW curve, −0.45 (1.11). The S-S curve best describes the Kiglapait data, with negligible systematic error and a 1-SD precision of 0.6 mole percent An.

Coordinates for the S-S curve are given on p. 109 of Morse (1968). At high values of An, the S-S and S-T curves are very similar. Retaining the S-T curve for sodic compositions, the determinative equations are summarized for convenience as follows:

$$\begin{array}{lll} \text{S-T curve: } \mathrm{An}_{0-24}, & \mathrm{An} = 1936(n - 1.5287) \\ \qquad\qquad\ \ \mathrm{An}_{24-31}, & \mathrm{An} = 1790(n - 1.5277) \\ \text{S-S curve: } \mathrm{An}_{31-84}, & \mathrm{An} = 1944(n - 1.5290) \\ \qquad\qquad\ \ \mathrm{An}_{84-100}, & \mathrm{An} = 2133(n - 1.5328), \end{array}$$

where n is the low refractive index on basal cleavages, referred to the sodium line. If the old Hartmann net is used for graphical determinations, a maximum error of −1 percent An will result at An_{41}, relative to the S-S curve.

In summary, the dispersion method appears to be capable of high precision in the central third of the plagioclase range, with a standard deviation of ±0.6 percent An if the S-S curve is used for plagioclases of comparably low K.

Rapid probe analysis

Electron probe analysis shares with the dispersion method the potential for rapid accumulation of large amounts of data in grain mounts, yielding information on within-sample variability that cannot be obtained with a bulk analysis. Many analysts have developed rapid analysis procedures for the electron probe (*e.g.*, Mason *et al.*, 1969), and the purpose of this note is not so much to instruct as to illustrate the potential of the method, using 14 analyzed Kiglapait plagioclases and ternary feldspars as a test.

The routine developed here assumes feldspar stoichiometry and iterates the correction procedure of Bence and Albee (1968) with continually reassigned values of Si and Al. Fixed-spectrometer counts are made for 15 seconds on Ca, Na, and K. A beam size of 15–20 μm is used to allow for possible antiperthitic lamellae. Grain mounts in epoxy are contained in bakelite wheels, each containing 20 unknown samples and a well-characterized, homogeneous standard (KI 3363). Three wheels can be mounted with one standard wheel in the ETEC Autoprobe.

The total analysis and printout time for 10 grains per mount is about six minutes (less with a CRT display and remote printer). Each line of printout gives An, Or, K in ppm, the summation, and a response option which allows deletion of a bad analysis. When the response is the letter "A", the sample average and standard deviation are printed for An, An/(An + Ab + Or), Or/(An + Ab + Or), and ppm

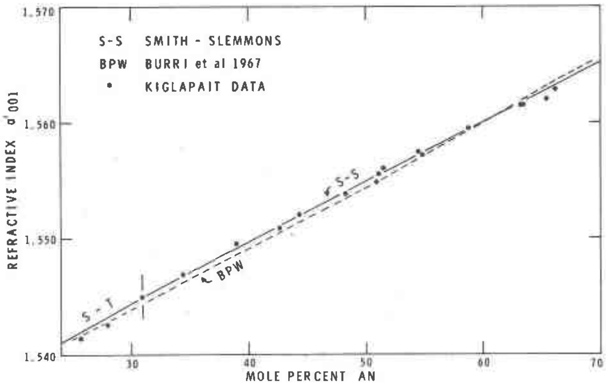

Fig. 1. Refractive index α'_{001} of Kiglapait plagioclase feldspars, compared to the Smith-Slemmons curve (S-S, full line) and the curve of Burri *et al.* (BPW, dashed line).

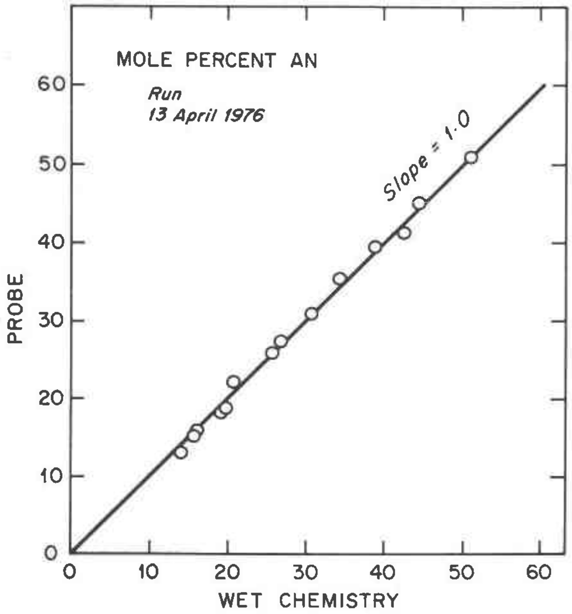

Fig. 2. Results of rapid electron probe analyses (averages of 10 grains per sample) compared to bulk chemical analyses. Circle radius is 0.9 percent An.

K, and the maximum and minimum An's are given. In the first day's operation, seven runs on KI 3363 (An 51.1) gave 51.1, 51.4, 50.7, 50.2, 50.4, 50.9, and 51.1, for a mean (66 grains) of 50.8. In 11.5 hours, including periods for standardization, filament saturation, and beam regulation, 56 unknown samples were run, for a total of about 575 grain analyses. On another day, fourteen analyzed samples were run for information on compositional variation. The group mean An for each of these samples is plotted in Figure 2, relative to the bulk analysis. The standard deviation is 0.84, despite the fact that samples below An_{35} are antiperthites or mesoperthites. The within-sample compositional range for these samples averages 6.3 percent An, but for one of them it is 20 percent An. There is no apparent correlation of error with either composition or compositional range. Ten grains seems an adequate number to count, even for variable samples. Samples with strong local zoning gradients may present worse problems, however.

This procedure forfeits, of course, all knowledge of stoichiometry or of minor elements. These are often best studied in experiments designed for the purpose. The rapid procedure yields large amounts of useful data. The on-line output contains information comparable to a computer run of dispersion method data, but has the great advantage of including values for potassium and Or. The probe procedure is also more objective and less fatiguing than the dispersion method.

Conclusions

Refractive-index measurements by the dispersion method in the range An_{31} to An_{86} favor the use of the Smith-Slemmons determinative curve, with a standard deviation of 0.6 percent An for low-K plagioclase.

Rapid probe analyses of 10 grains per sample in the range An_{13} to An_{51} have a standard deviation of 0.8 percent An. Most of these samples are antiperthites or mesoperthites with inhomogeneities on the scale of one-half to one-tenth the probe beam diameter, so the test is relatively severe.

The grain mount methods have comparable precision and speed. Both yield valuable statistics and for that reason alone may often be preferable to bulk analysis. The conclusions of the earlier paper about the power of the grain-mount methods are reinforced by the new data, and the uncertainties expressed there about the calibration are effectively removed.

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