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QUANTITATIVE PHASE-ANALYSIS OF Rb-ENRICHED MAXIMUM MICROCLINE AND LOW ALBITE BY X-RAY POWDER DIFFRACTOMETRY

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ABSTRACT

Curves derived from intensity data derived by X-ray powder diffractometry are used to determine the proportions of Rb-enriched maximum (or low) microcline (K,Rb)MM and low albite LA in a two-feldspar mixture (*e.g.*, a perthite). Two determinative curves have been deduced, one utilizing a ratio [$^{df}R(\bar{2}01) = R_1$] of diffractogram-derived intensities I of the $\bar{2}01$ peaks of both phases, the other a ratio [$^{cs}R(\text{str}) = R_2$] of intensities derived from a continuous-scan peak-search routine of the strongest peaks of the two phases. Where $R = I_{LA}/(I_{(K,Rb)MM} + I_{LA})$, the (quadratic) equation for the wt.% LA utilizing the first curve is $47.5(2.4)R_1^2 + 52.4(2.4)R_1 - 0.1(0.4)$, and that for the second curve is $36.6(3.3)R_2^2 + 63.5(3.3)R_2 - 0.1(0.6)$ (errors in brackets). The curves are estimated to give the phase composition to within ~1 to ~15 wt.% LA in the powdered sample depending upon LA concentration and whether one or both types of intensity measurement (diffractogram, continuous-scan peak search) is made. The curves make possible the rapid two-phase modal analysis of applicable alkali feldspars and, with a chemical analysis, the determination of the Ab_{ss} in the microcline.

Keywords: Rb-enriched maximum (low) microcline, low albite, perthite, X-ray powder diffractometry, determinative curve, modal analysis.

SOMMAIRE

On présente des courbes dérivées de données de diffraction X sur poudres pour déterminer les proportions de microcline ordonné enrichi en rubidium [désigné (K,Rb)MM] et d'albite ordonné (LA) dans un mélange, une perthite par exemple. On propose deux relations, une qui utilise le rapport R_1 des intensités I de la réflexion $\bar{2}01$ des deux phases [$^{df}R(\bar{2}01)$], et l'autre qui utilise le rapport R_2 des intensités dérivées d'un examen du spectre continu conçu pour définir les pics les plus intenses des deux phases [$^{cs}R(\text{str})$]. R est le rapport de I_{LA} sur $I_{(K,Rb)MM} + I_{LA}$; l'expression quadratique obtenue pour le pourcentage pondéral d'albite ordonnée est $47.5(2.4)R_1^2 + 52.4(2.4)R_1 - 0.1(0.4)$ selon la première courbe et $36.6(3.3)R_2^2 + 63.5(3.3)R_2 - 0.1(0.6)$ selon la deuxième (erreurs entre parenthèses). On considère ces relations aptes à donner un estimé de la composition des phases à de 1 à 15% d'albite près dans un échantillon pulvérisé, dépendant de la concentration d'albite et si seulement une ou les deux méthodes de faire les mesures d'intensité sont employées. Les courbes rendent possibles l'analyse modale rapide des mélanges à deux phases de feldspaths alcalins et, avec les résultats d'une analyse chimique, une détermination de la proportion exacte du pôle Ab dans le microcline.

(Traduit par la Rédaction)

Mots-clés: microcline ordonné rubidique, albite ordonnée, perthite, diffractométrie X sur poudres, courbes déterminatives, analyse modale.

INTRODUCTION

X-ray powder diffraction (XRPD) records of many K-feldspar specimens, especially of microcline, commonly include one or more of the strongest peaks of albite. It is possible to use the intensities of the albite peaks relative to those for the K-feldspar phase to quantify the proportion of the two feldspars in a given powdered sample. Although other types of observation (optical, single-crystal X-ray, transmission-electron microscopy, *etc.*) must also be used if one wishes to know the nature of the twinning and intergrowth of the separate-phase albite in a dominantly microcline sample (albite/pericline twinning, perthitic lamellae, rims, 'blebs', *etc.*), an XRPD record can provide a rapid two-phase modal analysis. For a sample that has been chemically analyzed, the method can quantify both the Na in separate-phase albite and, by difference, that in solid solution in the microcline.

Kuellmer (1959, 1960) published curves for correlating the proportions of an albite (low, high) and a K-feldspar (microcline, orthoclase, sanidine) in a powder sample with the relative intensities of certain corresponding peaks of the two phases in the XRPD record. However, he gave no intensity data, and his curves are inconvenient to use; in any event, improvements in powder diffractometry in the last quarter of a century make it possible to now derive more precise determinative curves of this type.

Described here are curves that correlate the proportions in a 'mixture' (*e.g.*, a perthite) of two alkali feldspar phases, Rb-enriched maximum microcline and low albite, with the relative intensities on the XRPD record of certain pairs of corresponding peaks of the two phases chosen as standards.

The derivation of the determinative curves involved, in essence, taking detailed XRPD records over the required 2θ range of weighed mixtures of a Rb-enriched maximum microcline and a low albite. The intensities of the two corresponding peaks of each phase chosen as standards were carefully measured, and then the phase compositions of all standard mixtures plotted against the intensity ratios albite/(microcline + albite) for the pairs of standard peaks to give the determinative curves.

Standard Samples and their Preparation

Standard feldspars

Table 1 gives the details of the two natural feldspars chosen as standards, a Rb-enriched maximum microcline [designated (K,Rb)MM] from the Cross Lake granitic pegmatite, Manitoba (Anderson 1984), and a low albite (designated LA). Both these feldspar specimens were found, from their X-ray powder-diffraction patterns (Fig. 1), to contain small amounts of the other phase. These small amounts were quantified in a manner described below, and allowed for in the derivation of the determinative curves.

Standard mixtures

We chose intervals for the mixtures of ~5 wt.% LA from 5 to 15% LA, and ~10% from 15 to 85% LA. This resulted in 11 standard mixtures which, with the two 'end-member' standards, provided 13 points for definition of the composition – intensity curves. The 'weighed' compositions were subsequently modified to allow for the small admixtures of the other phase in each standard to give the 'actual' compositions (in wt.% LA). It is these compositions that were used for the determinative curves.

Sample preparation: considerations

The method described next for preparing the actual sample for diffractometry was adopted because it is simple and fast. As such, the accuracy of the intensity values is probably less than it would be were a rotating sample holder or similar devices used. We adopted this faster, simpler method

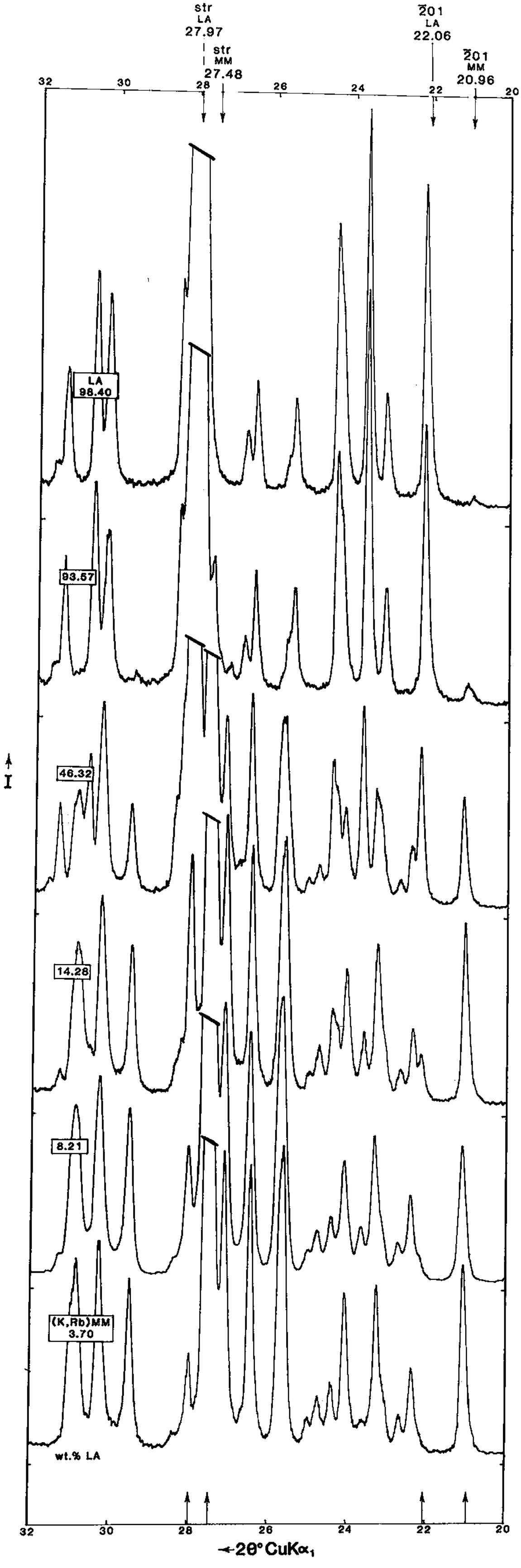

FIG. 1. X-ray diffractograms on an arbitrary scale of intensity I, showing the standard Rb-enriched maximum microcline (K,Rb)MM and the standard low albite LA, and four representative mixtures. Phase compositions are in 'actual' wt.% LA (see text). Across the top are shown the designations for the peaks chosen as standards, $\bar{2}01$ and str (= strongest), for the two phases (K,Rb)MM and LA, with their 2θ values. For operating parameters, see text.

because the phase-analysis curves described in this paper are intended to give moderately accurate yet still meaningful results for fairly large numbers of specimens in a short time. The errors we suggest later for results derived from the use of the method are based on much experimental data, and allow for variations in grain size, preferred orientation, *etc.* in the sample preparation.

Sample preparation: experimental

The microcline and albite standard mineral specimens were coarsely crushed, picked free of visible impurities under the binocular microscope, and then hand-ground using an aluminum oxide mortar and ceramic pestle until no 'grittiness' remained.

To prepare a powder mixture for the diffractometer, the required amounts of the two feldspar samples were weighed out to give a total sample of ~100 mg, placed in a small mortar, and mixed first with a needle and then by grinding with a pestle for ~60 s. To make the actual sample for diffractometry, most of the powder was transferred from the mortar to a 25 × 45 mm petrographic glass slide on which it was worked into a 'smear' ~20×20×0.1 mm in size in an acetone medium using a needle.

DERIVATION OF THE DETERMINATIVE CURVES

Instrumentation, operating parameters and sample scans

The XRPD work was carried out on a Philips automated powder diffractometer (System PW 1710) equipped with a curved-crystal monochromator, broad-focus Cu X-ray tube and automatic divergence (variable) slit. For the present application, a fixed 1° divergence slit can be taken as equivalent to the variable slit. A teleprinter provided control and digital output, the latter supplementing the usual strip-chart recorder.

The following operating parameters were used for most scans (called 'normal', n) (the Philips symbols are shown in brackets): goniometer range 20°–32°

TABLE 1. CHEMICAL AND CRYSTALLOGRAPHIC CHARACTERISTICS OF THE STANDARD SAMPLES Rb-ENRICHED MAXIMUM MICROCLINE (K,Rb)MM AND LOW ALBITE LA

			Rb-enriched maximum microcline (K,Rb)MM			low albite LA		
sample no.			Kf-4-A			Ab1		
locality			Cross Lake, Manitoba			unknown		
rock type			granitic pegmatite			unknown		
Partial analyses, wt. %	K_2O	Na_2O CaO	13.60	0.74	0.06	0.16	11.0	0.337
	Total Fe as Fe_2O_3	BaO SrO	0.02	nd	nd	0.024	0.004	0.003
	Rb_2O	Cs_2O Li_2O	2.43	0.109	0.006	0.003	0.006	0.017
end-member chemical compositions, wt. %[1]			$(Kf_{84.0}Rbf_{8.8}Csf_{0.4})(Naf_{6.5}Caf_{0.3}) = Or_{93.2}Ab_{6.8}$			$(Naf_{96.8}Caf_{1.7})(Kf_{1.2}Lif_{0.3}) = Ab_{98.5}Or_{1.5}$		
two-phase compositions from XRPD, wt. %[2]			$(K,Rb)MM_{96.3}LA_{3.7}$			$LA_{98.4}(K,Rb)MM_{1.6}$		
unit cell parameters	direct	reciprocal						
	a, Å	a*, Å^{-1}	8.605(2)	0.12935(4)		8.134(1)	0.13749(2)	
	b	b*	12.954(3)	0.07726(2)		12.777(2)	0.07849(1)	
	c	c*	7.219(2)	0.15406(6)		7.152(1)	0.15666(2)	
	α, °	α*, °	90.60(2)	90.42(2)		94.27(2)	86.36(2)	
	β	β*	115.96(2)	64.05(2)		116.59(1)	63.50(1)	
	γ	γ*	87.77(2)	92.18(2)		87.73(1)	90.40(1)	
	V, Å^3	V*, Å^{-3}	723.0(2)	1.3832(4)x10^{-3}		662.8(1)	1.5087(2)x10^{-3}	
131, $1\bar{3}1$ indicators	d, Å	2θ(CuKα$_1$) 131	3.031	29.44		2.859	31.26	
		$1\bar{3}1$	2.956	30.21		2.963	30.14	
triclinicity Δ (microcline)[3]			0.93			-		

[1] Or embodies K,Rb,Cs,Li; Ab embodies Na,Ca. [2] See Table 4. [3] $\Delta = 12.5(d_{131}-d_{1\bar{3}1})$.

2θ(Cu$K\alpha_1$), scan speed (SPE) 0.6° 2θ/min.; chart recorder full-scale (RFS) setting 2000 c.p.s., speed (RSP) 20 mm/°2θ; rate meter time-constant (RTC) 2 s; sampling interval-time (INT) 4 s. To record one of the standard peaks that is very strong for some samples, several scans (called 'special', s) were also made with the goniometer range changed to 20°-29° and the RFS to 5000 c.p.s.

To record the XRPD data for each of the 13 standard samples [(K,Rb)MM, LA, 11 mixtures], the following procedure was employed: (i) a smear mount was made on a glass slide (as described above); (ii) the slide was placed in the sample chamber of the diffractometer, and scanned 5 consecutive times using the operating conditions given above; (iii) the slide was then removed from the chamber and the powder scraped from the slide; (iv) this same powder was re-mixed, reground slightly, and then used to prepare a second smear-mount; (v) the second slide was then scanned an additional 5 consecutive times as in (ii) above, and (vi) steps (iv) and (v) above were repeated once again, resulting in 5 scans made on each of 3 different slide mounts of the same powder giving a total of 15 diffractograms with digital output for each of the 13 standard specimens.

Standard peaks and their intensity measurement

Two pairs of corresponding peaks in the microcline and albite were selected as standards, the medium-intensity $\bar{2}01$ (as used by Kuellmer 1959, 1960) at ~21°-22° 2θ, and the strongest ('str') peak at ~28° 2θ. Details about these peaks relevant to our procedure are given in Table 2. The multiple-*hkl* character of the str peaks (Table 2) makes them less desirable as standards than a single peak like $\bar{2}01$, but they are included because of their greater sensitivity to minor amounts of the one phase.

With the intensity of a given peak designated as *I* and with (K,Rb)MM abbreviated to RM, the two pairs of corresponding intensities recorded were ${}_{RM}I(\bar{2}01)$, ${}_{LA}I(\bar{2}01)$ and ${}_{RM}I(\text{str})$, ${}_{LA}I(\text{str})$. The intensity ratio *R* that we adopted as proportional to the amount (in wt.%) of LA in a sample (generalizing RM to MM) is of the type $R(\bar{2}01) = {}_{LA}I(\bar{2}01)/[{}_{MM}I(\bar{2}01) + {}_{LA}I(\bar{2}01)]$ (different from the type ${}_{LA}I(\bar{2}01)/{}_{MM}I(\bar{2}01)$ used by Kuellmer 1959, 1960).

Measurement of peak intensities

The intensities of the two pairs of standard peaks were recorded and measured as peak heights by two different methods: (1) Diffractograms: graphical peak-height-over-background, such intensities being designated by the superscript prefix 'df', *e.g.*, ${}^{df}_{RM}I(\bar{2}01)$. (2) Automated continuous-scan peak-search: peak heights printer-recorded as 'raw' intensities (no subtraction of background), such intensities designated here by 'cs', *e.g.*, ${}^{cs}_{LA}I(\text{str})$.

Initial curves relating phase composition and intensity ratio: dilution of each standard sample by the other phase

From a minimum of 15 scans of each of the standard (K,Rb)MM and LA specimens and the 11 mixtures, the intensities *I* of the four standard peaks were recorded (Fig. 1) and measured as described above, and means taken of all comparable results to provide, along with the 'weighed' compositions of the mixtures, the database (Table 3) for the determinative curves described here. Table 3 is available at nominal charge from the Depository of Unpublished Data, CISTI, National Research Council of Canada, Ottawa, Ontario K1A 0S2. Our procedure results in four curves relating composition and intensity ratio, one for each of ${}^{df}_{RM}R(\bar{2}01)$, ${}^{cs}_{RM}R(\bar{2}01)$, ${}^{df}_{RM}R(\text{str})$ and ${}^{cs}_{RM}R(\text{str})$ [where the subscript RM indicates mixtures of (K,Rb)MM with LA]. All four were found to be described most satisfactorily by quadratic equations (Table 4). Of the four, two were found to be more precise and hence more satisfactory than the others,

TABLE 2. CHARACTERISTICS OF THE MAXIMUM MICROCLINE (MM) AND LOW ALBITE (LA) PEAKS USED AS THE TWO STANDARD PAIRS FOR THE DETERMINATIVE CURVES

pair number	peak designation	maximum microcline (MM)							low albite (LA)						
			d,Å		I	I	peak 2θ(CuKα$_1$)			d,Å		I	I	peak 2θ(CuKα$_1$)	
		hkℓ	B & S	F & B	int	peak	B & S	F & B	hkℓ	B & S	F & B	int	peak	B & S	F & B
1	$\bar{2}01$	$\bar{2}01$	4.213	4.234	76	53	21.08°	20.96°	$\bar{2}01$	4.027	4.026	93	67	22.06°	22.06°
2	strongest (str)	$\bar{2}20$ 002 040	3.253 3.247 3.238	3.264 3.246 3.236	61 100 42	100	27.46°	27.48°	002 040	3.194 3.188	3.192 3.185	100 66	100	27.96°	27.97°

Notes: (1) Terminology (I_{int}, I_{peak}) and data, except where noted, from Borg & Smith (B & S) (1969). (2) F & B designates present authors. (3) Maximum microcline data from B & S are for 'normal' ('Rb-poor')MM, those from F & B are for the Rb-enriched microcline, (K,Rb)MM (Table 1).

TABLE 4. COEFFICIENTS OF THE QUADRATIC EQUATIONS FOR THE DILUTION AND DETERMINATIVE CURVES

Dilution curves[a]: 'wefghed' wt. % LA versus ratio R

ratio R	a	b	y intercept c^b	y for R=1 intercept	y for R=1 intercept −100.0[c]	statistical R^2	Figure No.
${}^{cs}_{RM}R(\bar{2}01)$	63.6(12.0)	51.1(13.9)	-5.4(3.6)	109.3(29.5)	9.3(29.5)	0.9972	-
${}^{cs}_{RM}R(str)$	37.6(8.1)	68.3(7.9)	-4.3(1.6)	101.6(17.6)	1.6(17.6)	0.9975	2
${}^{df}_{RM}R(\bar{2}01)$	51.8(6.9)	53.0(8.0)	-3.2(2.0)	101.6(16.8)	1.6(16.8)	0.9985	-
${}^{df}_{RM}R(str)$	56.7(8.7)	42.7(8.5)	0.0(1.7)	99.4(18.8)	-0.6(18.8)	0.9965	-
|means|= dilutions, wt.%	LA in (K,Rb)MM = 3.7(1.8)[d]			(K,Rb)MM in LA = 1.6(0.2)[d]			

Determinative curves[a]: 'actual' wt.% LA versus ratio R

ratio R	a	b	c	y for R=1	statistical R^2	Figure No.
${}^{df}_{RM}R(\bar{2}01)$	47.5(2.4)	52.4(2.4)	0.1(0.4)	100.0(5.2)	0.9997	3a
${}^{cs}_{RM}R(str)$	36.6(3.3)	68.5(3.3)	-0.1(0.6)	100.0(7.2)	0.9993	3b

[a] Equations are of the form: wt.% LA = $aR^2 + bR + c$; numbers in brackets are standard errors.
[b,c] Magnitudes are the implied dilutions (wt.%) of one standard by the other phase. See text.
[d] See text.

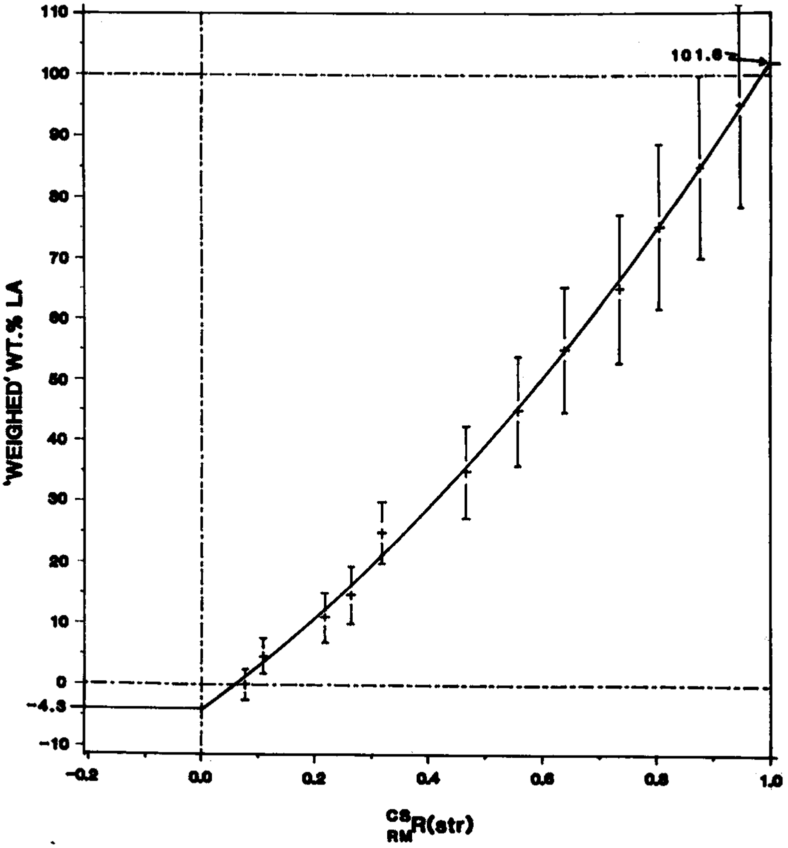

FIG. 2. 'Dilution' curve: plot for all standard specimens of compositions in 'weighed' wt.% LA against ${}^{cs}_{RM}R(str)$, the continuous-scan (cs) intensity ratios for the pair of strongest (str) reflections. The equation for this curve is quadratic (Table 4). Bars show errors of 1σ in the dependent variable. The intercepts at $R = 0$ and $R = 1$ imply 4.3 and 1.6 wt.% of albite and microcline in the standard (K,Rb)MM and LA specimens, respectively.

namely those for ${}^{\mathrm{df}}_{\mathrm{RM}}R(\bar{2}01)$ and ${}^{\mathrm{cs}}_{\mathrm{RM}}R(\mathrm{str})$; the latter is shown as Figure 2.

Because both standard samples were known from their diffractograms to contain small amounts of the other phase, a plot of the nominal or 'weighed' compositions against a given set of intensity ratios R would ideally intersect the composition axis at <0 and >100 for $R = 0$ and 1, respectively; furthermore, the magnitudes of these intersections beyond the ideal limits are a measure of the dilutions of each standard by the other phase. (This is an example of the 'dilution' method of XRPD analysis. See, for example, Klug & Alexander 1974, p.554.) All four curves were found to indeed conform to these types of intersection.

Assuming on crystal-chemical grounds (sizes of the alkali ions) and chemical compositions (*e.g.*, Smith 1974, pp. 68-76) that Rb is present in the microcline rather than the albite, the amounts (wt.%) of (assumed) LA in the microcline standard and of (assumed) (K,Rb)MM in the albite standard implied by the intersections of the two better curves on the composition axes at $R = 0$ and $R = 1$ are, respectively, 3.2, 4.3 and 1.6, 1.6 (Table 4, Fig. 2). We have taken the means of these pairs of values, 3.7 wt.% LA and 1.6 wt.% (K,Rb)MM, as the respective dilutions of the microcline and albite standards. From the coefficient errors in the quadratic equations (Table 4) combined with the chemical compositions of the standards (Table 1), $Ab_{6.8}$ and $Or_{1.5}$, we conclude that reasonable errors in the above two dilution values are 1.8 and 0.2 wt.%, respectively, of the other phase.

THE DETERMINATIVE CURVES AND THEIR APPLICATION

The determinative curves

The dilution values of both standard specimens by the other phase were used to convert the 'weighed' compositions of all 13 standard samples to their 'actual' compositions, both in wt.% LA (Table 3). Plots of these 'actual' compositions against the two R data-sets that give the most satisfactory curves, ${}^{\mathrm{df}}_{\mathrm{RM}}R(\bar{2}01)$ and ${}^{\mathrm{cs}}_{\mathrm{RM}}R(\mathrm{str})$, constitute our two determinative curves. Both curves are quadratic in form, with the coefficients and their errors given in Table 4. The two curves are shown, respectively, as Figures 3a and 3b.

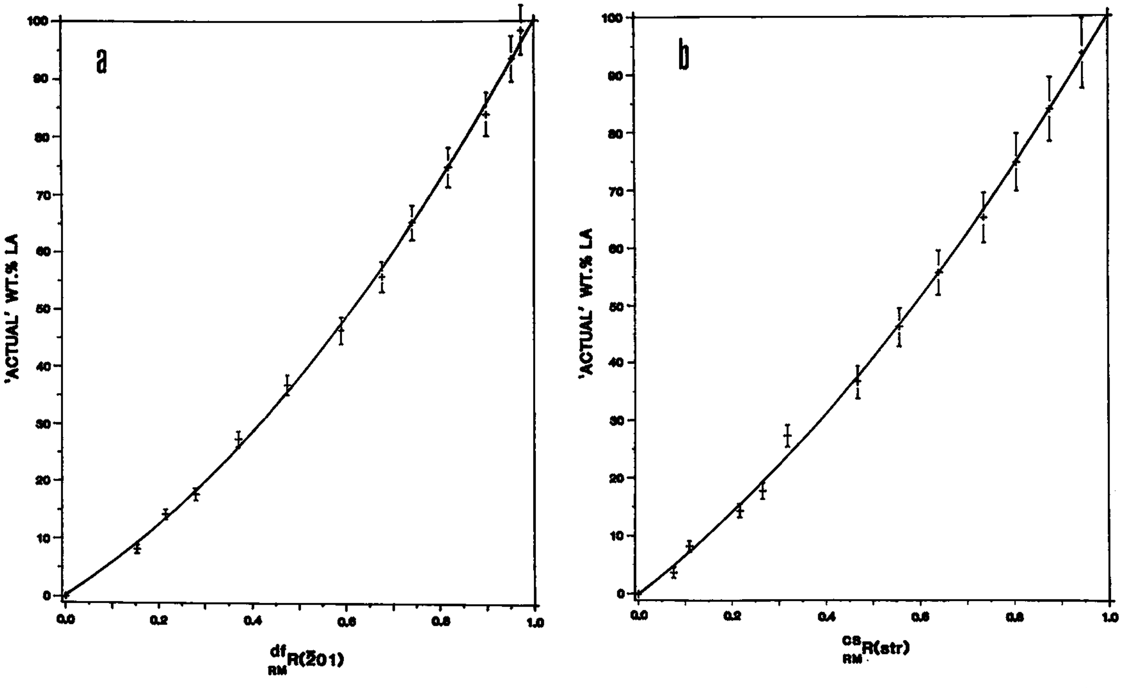

FIG. 3. Determinative curves: plots for all standard specimens of compositions in 'actual' wt.% LA against intensity ratios R. Coefficients and errors for the quadratic equations are given in Table 4. Bars show errors of 1σ in the dependent variable. For 'likely errors' in derived results, see Table 6 and text. a. ${}^{\mathrm{df}}_{\mathrm{RM}}R(\bar{2}01)$, the intensity ratio determined using diffractograms (df), for the pair of $\bar{2}01$ peaks. b. ${}^{\mathrm{cs}}_{\mathrm{RM}}R(\mathrm{str})$, the intensity ratio determined using continuous-scan peak search (cs), for the pair of strongest (str) peaks.

TABLE 5. PHASE COMPOSITIONS (Wt.% LA) AND RELATED CRYSTALLOGRAPHIC AND CHEMICAL DATA FOR THE SEVEN TEST SPECIMENS OF Rb-ENRICHED MAXIMUM MICROCLINE

locality	specimen no.	(K,Rb)MM partial cell data		bulk chemical composition			XRPD phase analysis			Ab_{ss} in (K,Rb)MM	
		a,Å V,Å^3	γ, ° tricl. Δ	end-members, wt.%[1] (Kf Rbf Csf Lif) (Naf Caf)	'molecules' wt.% Or	Ab	cs_Rstr comp'n., wt.% LA	$d_R\bar{2}01$ comp'n., wt.% LA	phase comp'n., wt.% LA[2]	chem. Ab-phase LA = wt.%[3]	mol.%
CROSS LAKE, MANITOBA	Kf-4-F	8.611(5) 723.9(5)	87.78(3) 0.91	(81.8 7.6 - 0.4) (9.1 1.1)	89.8	10.2	0.103 6.8(0.9)	0.157 9.4(0.8)	8.1[1.3]	2.1(1.7)	2.0(1.6)
	Kf-7-C	8.612(6) 724.3(6)	87.75(3) 0.91	(76.5 8.0 0.4 0.3) (12.5 2.4)	85.2	14.9	0.155 10.6(1.1)	0.210 13.1(0.9)	11.9[1.3]	3.0(1.5)	2.8(1.4)
	KF-69-D	8.603(4) 725.8(4)	87.72(3) 0.91	(71.4 5.9 0.4 4.5) (17.8 -)	82.2	17.8	0.246 17.6(1.1)	0.280 18.4(1.1)	18.0[0.4]	(-0.2)0(1.6)	0(1.5)
	Kf-96-DD	8.609(6) 724.9(7)	87.72(5) 0.88	(68.7 7.4 0.4 0.3) (23.2 -)	76.8	23.2	0.260 18.8(1.6)	0.293 19.5(1.2)	19.1[0.4]	4.1(2.1)	3.8(2.0)
TANCO, BERNIC LAKE, MANITOBA	BLM-22	8.617(5) 725.2(4)	87.80(3) 0.92	(86.0 8.4 0.8 -) (4.8 -)	95.2	4.8	0.116 7.7(1.0)	-[4] -(-)	7.7[-]	(-2.9)0(3.0)	0(2.8)
	BLM-24	8.617(4) 725.3(3)	87.83(3) 0.92	(77.6 8.5 0.8 -) (13.1 -)	86.9	13.1	0.157 10.7(1.1)	0.220 13.9(0.9)	12.3[1.6]	0.8(1.5)	0.7(1.4)
	BLM-30	8.606(5) 723.9(4)	87.72(2) 0.99	(74.6 7.0 0.4 -) (16.9 1.1)	82.0	18.0	0.242 17.3(1.5)	0.232 14.8(1.0)	16.1[1.3]	1.9(1.9)	1.8(1.8)

[1] Includes only end-members present in amounts ≥0.1. [2] Means of the compositions in the two preceding columns; numbers in square brackets[] are the departures of the individual values from the means. [3] Numbers in round brackets ( ) are the assumed errors in phase compositions (preceding column) and in the Ab_{ss} values (this column); see text. [4] Incomplete data.

Sample qualifications and preparation

To analyze a feldspar specimen by these curves (Table 4, Figs. 3a, b), it must conform to three conditions: (1) it must consist of a single-phase maximum microcline ($\Delta \geq \sim 0.85$) or low albite (or both); (2) it must be 'free' of quartz and any other mineral(s) whose peaks overlap those chosen as standard for the two feldspars; and (3) for these particular curves, the microcline must be Rb-enriched, with $Rb_2O > \sim 1.0$ wt.%.

To prepare an applicable specimen of alkali feldspar for analysis by these curves, a glass slide or smear sample is prepared following the procedure described earlier for the standards under *Sample preparation: experimental.* Each sample is then scanned through the range of $2\theta(CuK\alpha_1)$ from 20 to 32° at least three times on each of three different samples of the same powder using operating conditions comparable to those described earlier for our standards. Recording would normally be in the form of diffractograms from a strip-chart recorder, but also, for those so equipped, as printouts from an automated continuous-scan peak-search routine equivalent to the Philips system used here.

Derivation of intensities I, ratios R and sample compositions

All of the relevant intensities from the diffractograms, ${}^{\mathrm{df}}_{\mathrm{RM}}I(\bar{2}01)$, *etc.* and, where available, from the continuous-scan peak-search printout, ${}^{\mathrm{cs}}_{\mathrm{RM}}I(\bar{2}01)$, *etc.*, are compiled and averaged, and one or both of the determinative ratios ${}^{\mathrm{df}}_{\mathrm{RM}}R(\bar{2}01)$ and ${}^{\mathrm{cs}}_{\mathrm{RM}}R(\mathrm{str})$ extracted. The *R* value is applied to the appropriate determinative curve (Table 4) to give one or two values of the required phase-composition in terms of wt.% LA. The likely error in such a result is discussed below.

RESULTS FOR SOME TEST SAMPLES OF MICROCLINE, LIKELY ERRORS USING THE METHOD, AND IMPLIED Ab_{ss} IN MICROCLINE

The test samples of microcline and their derived compositions

For a test of the method, we have used seven pegmatitic, Rb-enriched samples of maximum microcline obtained from Petr Černý. Table 5 gives

TABLE 6. ERRORS IN DERIVED PHASE COMPOSITIONS

determinative curve		Compositions and standard errors σ (wt.% LA) for some R values				
	R=	0.1	0.3	0.5	0.7	0.9
${}^{\mathrm{df}}_{\mathrm{RM}}R(\bar{2}01)$	composition	5.8	20.0	38.1	60.0	85.8
	error σ	0.6	1.2	1.9	2.8	3.8
${}^{\mathrm{cs}}_{\mathrm{RM}}R(\mathrm{str})$	composition	6.6	22.1	40.7	62.1	86.6
	error σ	0.9	1.8	2.9	4.2	5.7

Likely standard errors in practice				
range of phase compositions		curve(s) applied		
wt.% LA	wt.% Na_2O	only ${}^{\mathrm{df}}_{\mathrm{RM}}R(\bar{2}01)$	only ${}^{\mathrm{cs}}_{\mathrm{RM}}R(\mathrm{str})$	both
<~10	<~1.2	3σ	3σ	2σ
≥~10	≥~1.2	2σ	2σ	1.5σ

these specimens their localities, relevant crystallographic and chemical data, and the results of our XRPD phase analyses. Both the diffractogram (df) and continuous-scan peak-search (cs) intensity ratios $^{\mathrm{df}}_{\mathrm{RM}}R(\bar{2}01)$ and $^{\mathrm{cs}}_{\mathrm{RM}}R(\mathrm{str})$ were evaluated, and two independent compositions for each specimen derived; the results are shown in Table 5. The assessment of these results must take account of the likely error in a given result, which we consider next.

Likely errors in phase compositions derived

The standard errors in the coefficients of the equations for the two determinative curves (Table 4) enable one to calculate a standard deviation σ for a phase composition corresponding to a given R value. Table 6 gives the σ values for some values of R for both curves. Because the determinative curves are based on at least $(3 \times 5 =)15$ goniometer scans of each standard specimen, whereas our recommended analytical procedure is based on only $(3 \times 3 =)9$ scans, and because the analytical curves were artificially constrained to pass through the 0,0 and 1,100 points by heavily weighting the ideal values for them, we conclude that the actual error in a given result will be greater than 1σ.

We have used comparable results in Table 5 to try to arrive at a realistic or 'likely' error in relation to σ. The individual values of the phase compositions (wt.% LA) in the 5th- and 4th-to-last columns in the table are followed by, in round brackets, their errors as 1σ (*e.g.*, 1st entry: compositions 6.8, 9.4; 1σ values 0.9, 0.8). The means of comparable pairs of derived values in the 3rd-to-last column are followed by, in square brackets, the difference between one extreme value and the mean (*e.g.*, 1st entry: mean composition 8.1, extreme/mean difference 1.3). These figures also suggest that the 'likely' error would indeed be, in general, $> 1\sigma$.

These few test-results indicate that the likely error in a derived composition will be a function of two main factors: (1) whether only one of the df and cs procedures is used to evaluate R, and (2) the proportion of LA in the sample. On the basis of these considerations we propose arbitrary values in terms of σ for the 'likely standard errors in practice'; these are shown for particular conditions in Table 6.

For the test samples, the 'likely errors' in the mean composition of the phases given in the 3rd-to-last column in Table 5 are the round-bracketed figures in the 2nd-to-last column (*e.g.*, 1st entry: mean composition 8.1, likely error 1.7).

Implied Ab_{ss} in the microcline samples

If the amount of LA in a microcline-rich powdered sample is subtracted from the chemically determined 'Ab' (= Ab + An), the difference gives the implied amount of Ab in solid solution (*i.e.*, Ab_{ss}) in the microcline. The Ab_{ss} values for the seven test-specimens are given in Table 5, in wt.% in the 2nd-to-last column, and in mol.% in the last column, with their likely phase-analysis errors in brackets (*e.g.*, 1st entry: Ab_{ss} 2.1, likely error 1.7, wt.%; 2.0, likely error 1.6, mol.%).

If one takes at face value the chemical and phase compositions, their differences, and the assumed errors, then one would conclude (1) that essentially all the Na(+Ca) is present as discrete LA, and 'none' as Ab_{ss} in the MM phase for the three specimens Kf–69–D, BLM–22 and –24, and (2) that the phases in the remaining four specimens Kf–4–F, –7–C, –96–DD and BLM–30 contain respectively 2.0(1.6), 2.8(1.4), 3.8(2.0) and 1.8(1.8) mol.% Ab_{ss} (errors in brackets).

Possible application of the determinative curves to 'Rb-free' maximum microcline

Some preliminary work we have done using a 'pure' maximum microcline (made by alkali-exchange of a low albite) suggests that both the standard peak $\bar{2}01$ and strongest (str) peaks may differ by about 10% in intensity for the 'pure' microcline relative to the Rb-bearing one used here. More work is required to derive modifications, presumably small, in the determinative curves described here to make them applicable to 'Rb-free' ($<< \sim 1$ wt.% Rb_2O) or 'normal' microcline. Until such modified curves become available, we suggest that the curves given here may be applied cautiously to 'normal' microcline samples provided errors of perhaps twice those given in Table 6 are assumed.

CONCLUDING STATEMENT

The determinative curves described here make possible the rapid determination of (1) the modal two-feldspar composition of powdered samples of perthitic or other Na-bearing maximum microcline, and (2), when combined with a chemical analysis, the Ab_{ss} in the microcline. Such a quantitative phase-analysis is not possible by single-crystal XRD, electron microscopy, or other such small-crystal-volume procedures. However, in the case of many samples of microcline, except those that are cryptoperthitic, an electron-microprobe analysis for Na(+Ca) will yield the Ab_{ss} directly. A comparison of comparable Ab_{ss} values obtained by the two different methods could prove valuable in assessing both of them.

In that this phase-analysis procedure can help differentiate the 's.s. solvus' from the 'bulk composition' solvus of applicable natural feldspars, it holds potential for elucidating the nature and possible genesis of microcline and the rocks they constitute.

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