

Quantitative analysis of alkali feldspar minerals using Rietveld refinement of X-ray diffraction data

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ABSTRACT

Microcline + albite and albite + quartz standard mixtures were quantitatively analyzed using Rietveld refinement to investigate the reliability of the method. The method was also applied to the determination of weight percentages of component minerals in a perthite sample. The analysis resulted in a standard deviation of approximately 1 wt% for the microcline + albite mixtures and 4 wt% for the albite + quartz mixtures, indicating that the accuracy of quantitative Rietveld analysis is far better than that of conventional XRD methods in which only a few select peaks are used. The inclusion of the Dollase-March preferred-orientation correction function slightly improved the accuracy of the results. That function also improved the rate of convergence and the accuracy of unit-cell parameters. However, the Rietveld-Toraya correction function did not work well with the quantitative analyses. The result of the quantitative analysis of the albite + quartz mixtures was poorer than that of the microcline + albite mixtures, and the weight fraction of albite tended to be overestimated. The analytical result of the microcline + albite mixtures implies that the Rietveld method is more accurate when component minerals have similar preferred-orientation characteristics. However, if component minerals have different crystal habits, as represented by the albite + quartz mixtures, the deviation is much greater and systematic errors may occur. Quantitative phase analysis of a perthite specimen using the Rietveld method resulted in very accurate values for the mineral proportion and unit-cell parameters of the constituent alkali feldspars, demonstrating the power of the Rietveld method to analyze multiphase samples.

INTRODUCTION

For many decades powder X-ray diffraction (XRD) has been regarded as a standard method for identification and quantitative characterization of minerals and other crystalline materials. However, the powder XRD method was not suitable for crystal-structure analysis because of the loss of information and difficulty in measuring accurate Bragg intensities as a result of peak overlap. Introduction of the Rietveld refinement method (Rietveld 1969) to XRD showed that such problems could be overcome and that serious crystal-structure studies could be performed successfully using all information present in XRD patterns (Post and Bish 1989).

Quantitative analysis of multiphase samples is an important application of Rietveld refinement, and Bish and Howard (1988), Bish and Post (1993), and Hill et al. (1993) illustrated the power of this method for geological samples. The effect of preferred orientation, which is an inherent problem in conventional XRD methods, where only several selected peaks are used for quantification, can be minimized by utilizing the intensity information from all peaks. Because Rietveld refinement can also simultaneously provide structural data and unit-cell pa-

rameters of phases in a mixture, maximum information can be extracted from powder XRD data.

Subsolidus phase diagrams with strain-free or equilibrium solvi for various ordering states have been presented (e.g., Luth et al. 1974; Smith 1974). If a quantitative analysis method can be developed to characterize accurately the feldspar components in perthite, the origin of the phases can be interpreted from the phase relations. Several quantitative XRD analytical methods for characterizing perthite have been proposed (e.g., Kuellmer 1959, 1960; Ferguson and Ball 1987). However, because these methods use only a few reflections from each phase, the precision of the analytical results is poor. Furthermore, they are susceptible to primary extinction caused by relatively defect-free crystallites and to preferred orientation caused by the {010} and {001} cleavages.

The present study investigates the accuracy and reliability of the quantitative Rietveld analysis at various weight fractions of the two end-member alkali feldspars. Several standard binary mixtures of microcline and albite were investigated. In addition, binary mixtures of albite and quartz were studied to observe the effects of nonrandomly oriented crystallites in powder samples. In addition, Rietveld refinement was used in the quantitative

TABLE 1. Synthetic binary standard mixtures used in the Rietveld refinements

Microcline + albite mixtures			Albite + quartz mixtures		
Sample	wt% of microcline	wt% of albite	Sample	wt% of albite	wt% of quartz
Mc ₁₀ Ab ₉₀	9.95	90.05	Ab ₁₀ Qtz ₉₀	10.10	89.90
Mc ₃₀ Ab ₇₀	30.03	69.97	Ab ₃₀ Qtz ₇₀	29.99	70.01
Mc ₅₀ Ab ₅₀	50.06	49.94	Ab ₅₀ Qtz ₅₀	39.66	60.34
Mc ₇₀ Ab ₃₀	69.97	30.03	Ab ₇₀ Qtz ₃₀	50.02	49.98
Mc ₉₀ Ab ₁₀	89.94	10.06	Ab ₉₀ Qtz ₁₀	60.12	39.88
			Ab ₇₀ Qtz ₃₀	70.03	29.97
			Ab ₉₀ Qtz ₁₀	90.12	9.88
			Ab ₉₀ Qtz ₁	99.11	0.89

characterization of the alkali feldspars in a natural perthite sample.

EXPERIMENTAL METHODS

Samples

Both the microcline and albite standard samples are from the Little Three pegmatite, central San Diego County, U.S.A. Both have almost complete Al-Si ordering (Stern et al. 1986; Shim 1993). The natural microcline standard sample contained <10 wt% exsolved albite, which was converted to microcline through the exchange of K for Na at approximately 790 °C in a KCl melt. The process should not have affected the Al-Si ordering (Kroll and Ribbe 1983). The standard albite is nearly end-member albite (Ab₉₉) containing only trace amounts of Sr and Ba (Stern et al. 1986). The quartz standard sample consists of clear crystals that do not contain any inclusions.

The perthite sample is from the Chungsan granite, Ogcheon, Korea. The compositions of the constituent microcline and albite are Or₉₈Ab₂ and Or₁Ab₉₉, respectively (Shim 1993). The sample contains a small amount of quartz inclusions.

X-ray diffraction

Each sample was ground to a particle size of <10 μm to minimize the possible microabsorption effect and to improve accuracy in the measured intensities. Two kinds of binary mineral mixtures were prepared by weighing the required amounts of the two standard feldspars to give a total sample of ~0.9 g (Table 1). Samples were mounted in standard aluminum holders with cavities (20 × 20 × 1.6 mm).

Powder XRD data were obtained by step scanning using a Material Analysis and Characterization (MAC) automated diffractometer system equipped with a curved graphite diffracted-beam monochromator. Divergence and antiscatter slits of 1° were used so that the irradiated area could be confined to the sample at angles >20° 2θ. A normal-focus Cu X-ray tube was operated at 40 kV and 30 mA, using a take-off angle of 6°. Diffraction profiles were obtained using a step interval of 0.02° 2θ for both the microcline + albite mixtures and the perthite sample, and 0.08° 2θ for the albite + quartz mixtures, respectively, with a step-counting time of 1 s.

Rietveld refinement

The Rietveld refinement program DBWS-9006PC (Sakthivel and Young 1990), with slight modification, was used on a Cyber 962-31 computer. Starting structure models of microcline and albite were taken from the single-crystal XRD data of Dal Negro et al. (1978) and Armbruster et al. (1990), respectively. The starting parameters for quartz were those of Young et al. (1977), as determined by Rietveld refinement. A pseudo-Voigt function was employed for the profile shapes. The peak width (FWHM) was varied as a function of 2θ using the expression of Caglioti et al. (1958). Peak asymmetry was corrected using the semiempirical relation of Rietveld (1969) over the 2θ range 20–40°. Profile step intensities were calculated over an interval 20 times the FWHM on both sides of each peak centroid.

The refinements were performed over a range of 20–80° 2θ for the microcline + albite mixtures and the perthite sample, and 20–100° 2θ for the albite + quartz mixtures, to exclude featureless regions of the diffraction patterns. Background was fitted with a simple polynomial function. Overall atomic displacement factors were used for each structure. The positions of all atoms of the feldspars and quartz were fixed, as well as individual isotropic and anisotropic displacement factors. Occupancy factors were constrained to obey the measured compositions. For albite + quartz mixtures, three refinements were performed: one without a preferred-orientation correction, another with the Dollase-March function, and the third with the Rietveld-Toraya function (Dollase 1986; Sakthivel and Young 1990). The [001] and [011] directions were used as the special axes for feldspars and quartz, respectively, for preferred-orientation corrections.

The refinement for Chungsan perthite followed the procedure used for standard mixtures. The refinement was performed with the Dollase-March function, which simulates the preferred-orientation effect of feldspars better than the Rietveld-Toraya function. Only microcline and albite were included in the early stages of refinement, quartz being included at a later stage with its scale-factor and profile parameters.

The weight fractions of the component phases were obtained from the following equation (Bish and Howard 1988):

$$W_i = \frac{S_i \rho_i V_i^2}{\sum_j S_j \rho_j V_j^2}$$

where W_i is the weight fraction, S_i is the scale factor, V_i is the unit-cell volume, ρ_i is the density of phase i , and subscript j includes all phases present.

RESULTS

Microcline + albite standard mixtures

Despite the complexity of the diffraction patterns (Fig. 1), the refined weight fractions of minerals for microcline + albite standard mixtures are quite accurate (Fig. 2);

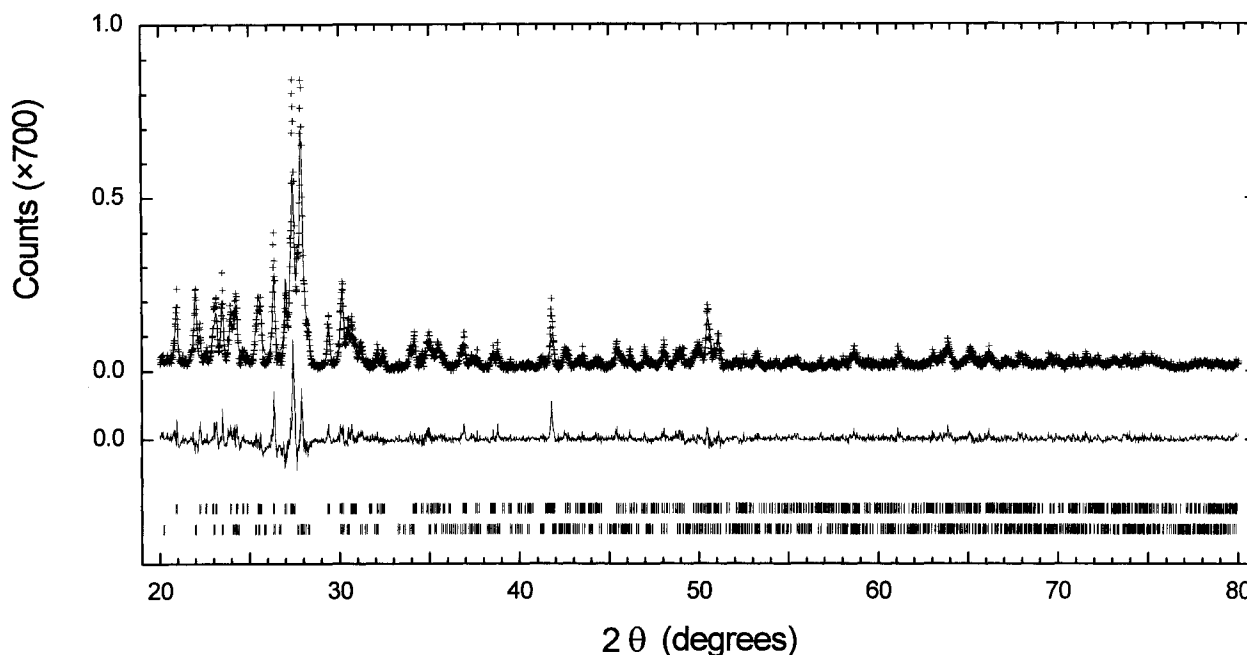


FIGURE 1. Observed and calculated diffraction patterns (plus signs and line, respectively) for $\text{Mc}_{50}\text{Ab}_{50}$ mixture with the Dollase-March correction ($R_p = 20.38$, $R_{wp} = 27.81\%$). The lower curve shows the difference between the observed and calculated patterns. Tick marks indicate positions of allowed reflections of microcline (upper) and albite (lower).

differences between results obtained by direct weighing and Rietveld refinement with the Dollase-March correction are <1 wt%. The values of the refined weight fractions of microcline are generally smaller than those from direct weighing, probably because microcline is slightly more randomly oriented as shown by the G_i parameters in comparison with albite (Fig. 2). The microcline standard sample also may have contained a small amount of perthitic albite as a residue of the K-Na exchange reaction, and the weight fraction of the microcline specimen may therefore have been underestimated. A separate refinement for the K-Na-exchanged microcline showed that there was 0.74 wt% residual albite, but the refined weight fractions of microcline in most of the mixtures are still smaller than the measured values after correction for the weight of the residual albite. The variation trend of differences between refined and measured weight fractions of microcline with the Dollase-March correction (Fig. 2) is similar to that of the Dollase-March preferred-orientation parameters, G_i , of microcline and albite obtained with correction (Fig. 3). This feature suggests that the deviation is apparently related to the preferred orientation of microcline and albite.

The refined unit-cell parameters are consistent with the values of Stern et al. (1986). However, the refined parameters for microcline, especially the unit-cell volume and a , deviate from those of Stern et al. (1986). Unit-cell volume and a of alkali feldspars are highly dependent on the K and Na contents and are nearly independent of the Al, Si distribution (Kroll and Ribbe 1983). The calculated mole fraction of orthoclase in the standard microcline

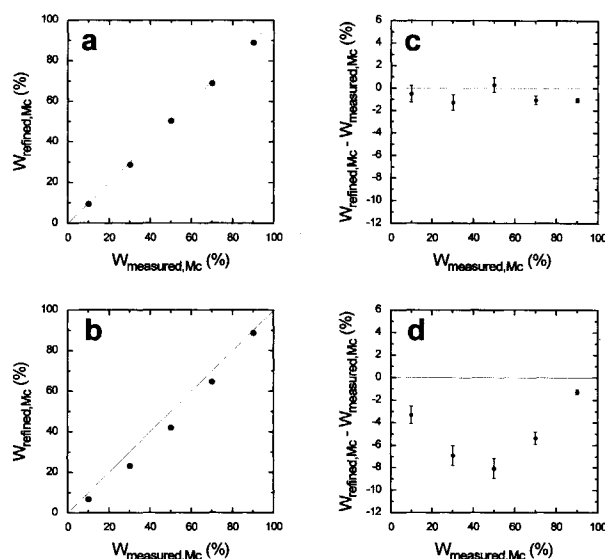


FIGURE 2. Refined weight fraction of microcline with the Dollase-March correction (a) and with the Rietveld-Toraya correction (b), and the difference between the refined and measured weight fraction of microcline for each microcline + albite mixture with the Dollase-March correction (c) and with the Rietveld-Toraya correction (d). Each point is bracketed by an error bar denoting the estimated standard deviation from the refinement.

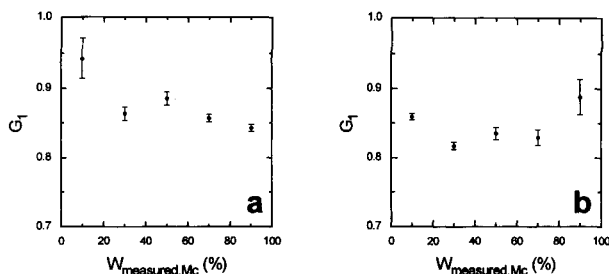


FIGURE 3. Preferred-orientation factor (G_1) of the Dollase-March function for microcline (a) and albite (b) resulting from the refinement of microcline + albite mixtures.

sample using the equation of Kroll et al. (1986) is 100 ± 0.4 mol%, which is greater than the value of 97.7 mol% reported by Stern et al. (1986). That result indicates that minor Na of microcline, as well as Na of albite, were replaced by K during the K-Na exchange experiment, resulting in a slight increase in the K content of the microcline. Therefore, the refined unit-cell parameters of K-exchanged samples can be somewhat different from those of raw samples. The agreement indices for various weight fractions are shown in Table 2.

Albite + quartz standard mixtures

The XRD patterns of albite exhibit the effect of preferred orientation, but those of quartz do not show any distinct orientation effect (Fig. 4). The measured weight fractions of albite are mostly within the estimated stan-

TABLE 2. Agreement indices and other refinement indices resulting from refinements of the microcline + albite standard mixtures

	R_p (%)	R_{wp} (%)	S^2	$R_{B,Mc}$ (%)	$R_{B,Ab}$ (%)	d
With Dollase-March preferred-orientation correction						
MC ₁₀ Ab ₉₀	17.64	24.19	1.32	13.67	8.75	1.39
MC ₃₀ Ab ₇₀	18.50	24.36	1.37	14.01	9.16	1.27
MC ₅₀ Ab ₅₀	20.38	27.81	1.52	12.70	8.90	1.09
MC ₇₀ Ab ₃₀	21.12	27.83	1.42	12.26	15.28	1.25
MC ₉₀ Ab ₁₀	22.78	29.55	1.48	15.28	15.04	1.19
With Rietveld-Toraya preferred-orientation correction						
MC ₁₀ Ab ₉₀	18.74	24.71	1.35	14.58	9.83	1.33
MC ₃₀ Ab ₇₀	20.21	25.86	1.45	15.61	10.39	1.14
MC ₅₀ Ab ₅₀	19.55	25.90	1.42	14.16	8.56	1.19
MC ₇₀ Ab ₃₀	22.65	29.41	1.50	13.61	16.17	1.12
MC ₉₀ Ab ₁₀	24.14	30.90	1.55	16.51	13.31	1.08

dard deviations of refined values with the Dollase-March correction and without a preferred-orientation correction (Fig. 5). The results from both refinements are superior to those of conventional XRD methods, which generally have standard deviations greater than ~ 7 wt% (Pawloski 1985; Maniar and Cooke 1987). However, the deviation from the refinement with the Rietveld-Toraya correction is greater than those from the other refinements, suggesting that the Rietveld-Toraya correction does not accurately compensate for preferred-orientation effects.

The refined weight fractions of albite are greater than the measured ones except in the case of the Ab₁₀Qtz₉₀ mixture. The differences between G_1 values of the Dollase-March function for quartz and albite (Fig. 6) and the

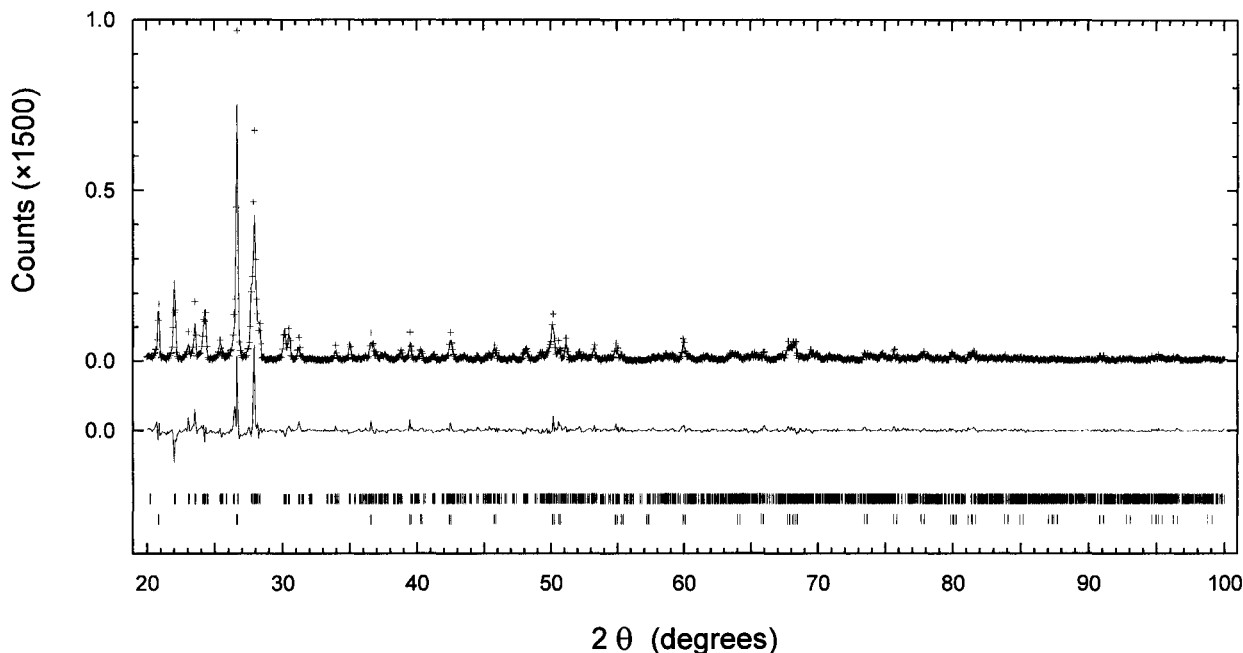


FIGURE 4. Observed and calculated diffraction patterns (plus signs and line, respectively) for the Ab₇₀Qtz₃₀ mixture without preferred-orientation correction. The lower curve shows the difference between the observed and calculated patterns. Tick marks indicate positions of allowed reflections of albite (upper) and quartz (lower) ($R_p = 23.52$, $R_{wp} = 31.40\%$).

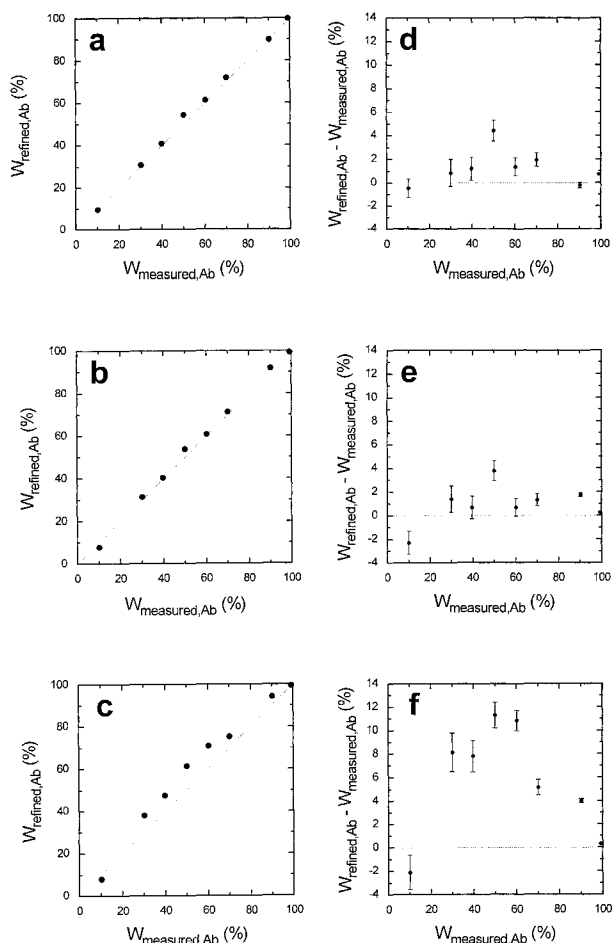


FIGURE 5. Refined weight fractions of albite without preferred-orientation correction (a), with the Dollase-March correction (b), and with the Rietveld-Toraya correction (c), and the difference between the refined and measured weight fraction of albite for each albite + quartz mixture without preferred-orientation correction (d), with the Dollase-March correction (e), and with the Rietveld-Toraya correction (f).

differences between measured and refined weight fractions of albite without a preferred-orientation correction show similar characteristics (Fig. 5); the greater the difference between the degrees of anisotropy in the spatial orientation of component crystallites, the more seriously the refined weight fractions deviate from measured ones. In addition, the values of the refined weight fraction of albite, which is significantly nonrandomly oriented, are generally greater than the measured values.

The refined weight fraction of albite in the $\text{Ab}_{10}\text{Qtz}_{90}$ mixture is smaller than measured. The refined background intensity of this sample was the highest among all mixtures, and the amount of albite was small, so that many of the albite peaks could barely be distinguished from background. The background intensity may have been overestimated in the refinements, and the peak in-

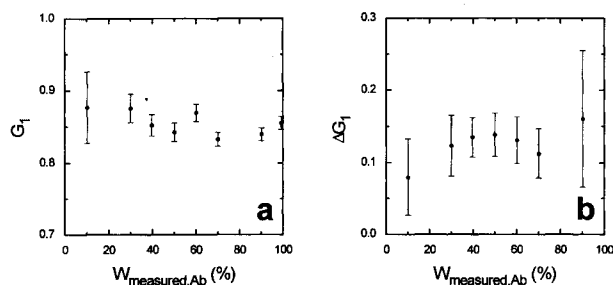


FIGURE 6. (a) Preferred-orientation factor (G_1) of the Dollase-March function for albite resulting from the refinement of albite + quartz mixtures, and (b) the difference between G_1 of quartz and albite (ΔG_1) for each mixture.

tensities of albite may have been underestimated, resulting in low values of the albite weight fraction.

Pattern fitting is improved with the inclusion of preferred-orientation-correction functions (Table 3). The Dollase-March correction improves the pattern fitting better than the Rietveld-Toraya correction. In general, R_p , R_{wp} , and S^2 show a decreasing trend with the increase of the weight fraction of albite. Some of these values deviate from a trend of decreasing values, and such deviations are observed in the mixtures that contain highly oriented albite crystallites (Fig. 6). Such anomalies therefore indicate that the pattern fitting is significantly affected by the preferred-orientation effect of albite.

Perthite sample

The observed and calculated diffraction patterns of the Chungsan perthite sample are shown in Figure 7; >1700 data points were used in the refinement. Quartz was detected using the difference-pattern diagram during an early stage of the refinement process, although the perthite sample contains only about 2 wt% quartz. Results of the Rietveld analysis are presented in Table 4. The G_1 values of the Dollase-March function for microcline (0.905 ± 0.007) and for albite (0.871 ± 0.016) are similar.

The significant overlap of peaks in the pattern of perthite makes determination of unit-cell parameters by conventional XRD methods difficult, but precise parameters can be obtained using the Rietveld refinement method. The estimated unit-cell parameters (Table 5) differ little from other published values for maximum microcline and low albite (Kroll and Ribbe 1983). Determination of Al-Si ordering using the b - c and α^* - γ^* plots of Stewart and Wright (1974) shows that both microcline and albite have nearly maximum ordering (Table 5). Values of mole percent orthoclase determined using unit-cell volumes calculated by the equation of Kroll et al. (1986) are 95.01 ± 0.4 in microcline and 0.49 ± 0.002 in albite. These results agree with microprobe analyses of the perthite sample (Shim 1993).

The low values of the agreement indices (Table 6) such as R_p , R_{wp} , and S^2 suggest that the pattern fitting was accurate. The R_b value of the microcline in the perthite

TABLE 3. Agreement indices and other refinement indices resulting from refinements of the albite + quartz standard mixtures

	R_p (%)	R_{wp} (%)	S^2	$R_{B,Ab}$ (%)	$R_{B,Qtz}$ (%)	d
Without preferred-orientation correction						
Ab ₁₀ Qtz ₉₀	26.19	34.06	1.85	15.19	22.47	1.36
Ab ₃₀ Qtz ₇₀	23.07	30.05	1.61	20.92	8.83	1.65
Ab ₄₀ Qtz ₆₀	21.54	28.66	1.45	16.83	6.76	1.58
Ab ₅₀ Qtz ₅₀	22.77	29.35	1.50	16.74	9.08	1.54
Ab ₆₀ Qtz ₄₀	20.90	28.92	1.45	14.10	8.53	1.65
Ab ₇₀ Qtz ₃₀	23.52	31.40	1.65	15.83	14.25	1.39
Ab ₉₀ Qtz ₁₀	23.08	29.38	1.55	17.14	15.77	1.40
Ab ₉₉ Qtz ₁	22.90	29.35	1.54	15.73	31.98	1.40
With Dollase-March preferred-orientation correction						
Ab ₁₀ Qtz ₉₀	25.03	32.78	1.78	22.36	10.50	1.47
Ab ₃₀ Qtz ₇₀	22.22	29.74	1.57	19.40	8.76	1.69
Ab ₄₀ Qtz ₆₀	20.07	27.51	1.39	15.40	8.05	1.67
Ab ₅₀ Qtz ₅₀	20.12	27.63	1.45	13.90	6.94	1.69
Ab ₆₀ Qtz ₄₀	19.87	27.68	1.39	13.10	7.72	1.72
Ab ₇₀ Qtz ₃₀	19.16	26.25	1.38	12.02	7.86	1.71
Ab ₉₀ Qtz ₁₀	19.20	26.59	1.40	11.25	11.25	1.58
Ab ₉₉ Qtz ₁	18.90	28.37	1.49	10.57	36.32	1.69
With Rietveld-Toraya preferred-orientation correction						
Ab ₁₀ Qtz ₉₀	25.21	33.47	1.81	20.75	10.54	1.45
Ab ₃₀ Qtz ₇₀	22.25	29.77	1.57	20.02	8.77	1.67
Ab ₄₀ Qtz ₆₀	20.48	27.71	1.40	16.12	12.88	1.66
Ab ₅₀ Qtz ₅₀	21.06	28.11	1.47	14.83	7.07	1.66
Ab ₆₀ Qtz ₄₀	20.72	27.83	1.40	14.56	7.70	1.63
Ab ₇₀ Qtz ₃₀	20.00	26.84	1.41	13.35	7.73	1.68
Ab ₉₀ Qtz ₁₀	19.63	27.01	1.56	12.98	21.26	1.56
Ab ₉₉ Qtz ₁	20.32	27.05	1.42	11.93	27.75	1.58

TABLE 4. Results of quantitative Rietveld analysis of the Chungsan perthite

	Scale factor ($\times 10^{-4}$)	V ($\text{\AA}^3$)	ρ (g/cm^3)	Weight fraction (%)
Microcline	0.398(8)	720.85(19)	2.534(112)	81.2(7)
Albite	0.089(6)	664.18(8)	2.635(6)	16.0(8)
Quartz	0.526(23)	112.86*	2.652*	2.8(4)

Note: Calculated standard errors in parentheses refer to the last decimal place.
* Fixed in the refinement.

is much smaller than that of the microcline + albite standard mixtures (Table 3), indicating that the structure of microcline in this sample is similar to that of the starting model.

DISCUSSION AND CONCLUSIONS

The results of the quantitative analysis are quite accurate for the mixtures, which consist of minerals having similar preferred-orientation characteristics such as the microcline + albite mixtures. If the preferred-orientation characteristics of component minerals are not similar, as in the case of the albite + quartz mixtures, systematic errors occur. However, the present study shows that the Rietveld method still minimizes the problem and yields very accurate results.

Correction functions, namely the Dollase-March func-

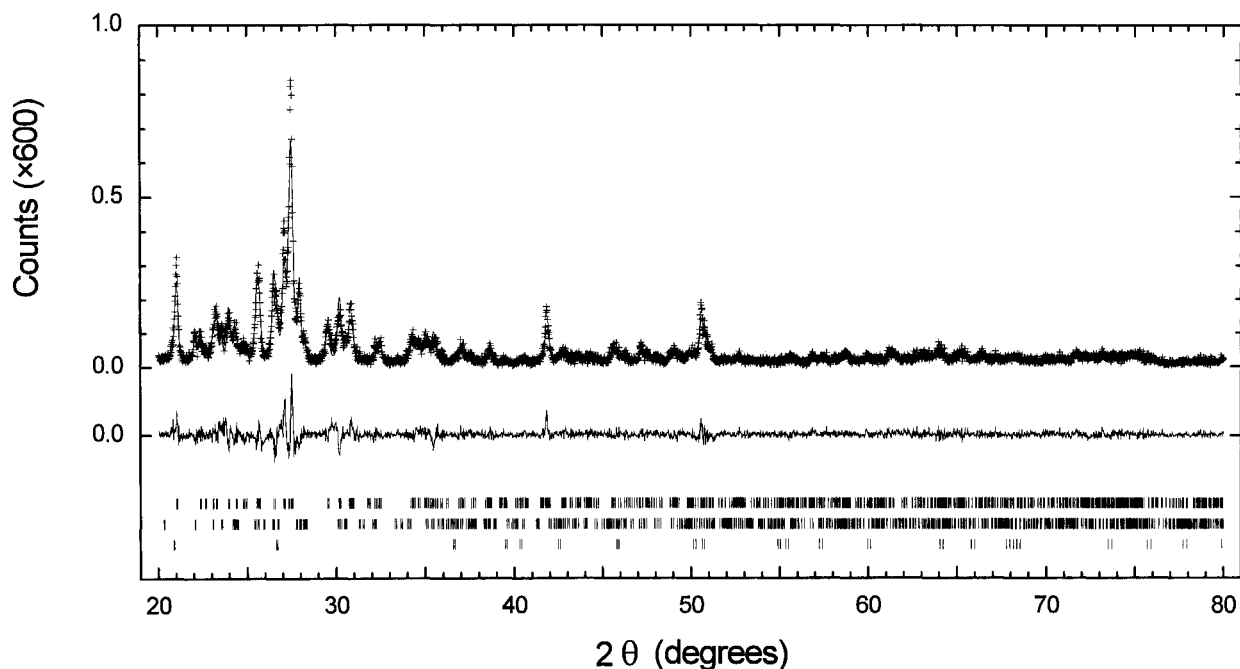


FIGURE 7. Observed and calculated diffraction patterns (plus signs and line, respectively) for Chungsan perthite and quartz ($R_p = 19.34$, $R_{wp} = 25.01\%$). The lower curve shows the difference between the observed and calculated patterns. Tick marks indicate positions of allowed reflections of microcline (upper), albite (middle), and quartz (lower).

TABLE 5. Unit-cell parameters of microcline and albite in the Chungsan perthite

	<i>a</i> (Å)	<i>b</i> (Å)	<i>c</i> (Å)	α (°)	β (°)	γ (°)	T1o + T1m*	T1o - T1m*
Microcline	8.5773(13)	12.9635(14)	7.2178(9)	90.579(12)	116.004(9)	87.985(12)	0.97(4)	0.85(4)
Albite	8.1408(5)	12.7866(8)	7.1534(5)	94.323(6)	116.552(5)	87.801(6)	0.97(4)	0.95(4)

Note: Estimated standard errors in parentheses refer to the last decimal place.

* Using the equation of Kroll and Ribbe (1983).

tion and the Rietveld-Toraya function, were included in the refinements, but errors were still discernible in the quantitative analysis results. The present study indicates that use of the Dollase-March function improves the result of quantitative Rietveld analysis. However, refinement with the Rietveld-Toraya function, which describes the pole-density distribution as a Gaussian distribution, results in worse quantitative results than the refinement without any preferred-orientation correction, as noted by Bish and Post (1993). The Dollase-March function was formulated through a theoretical approach and has been successfully applied to several refinements of nonrandomly oriented minerals (Dollase 1986). It apparently simulates accurately the redistribution of pole density caused by preferred orientation of crystallites. Furthermore, the Dollase-March function is useful for rapid convergence of refinements and accurate estimation of unit-cell parameters.

The standard feldspar samples consist of platy crystals as a result of their pronounced {001} cleavages, whereas quartz crystallites are equant in habit. The anisotropy in the orientation of crystallites in a sample mixture is usually produced during the sample-mounting process, which results, for example, in compaction and settling. In addition, cleavage planes of feldspar crystals may tend to be oriented parallel to the mounting surface during packing on a specimen holder. Therefore, incorrect relative intensities would be measured during the diffraction experiments, and the increased intensities caused by {001} cleavages would result in an overestimation of the feldspar weight fraction.

Unit-cell parameters obtained by the Rietveld method are more accurate than those obtained by conventional powder or single-crystal methods, provided sample displacement and transparency corrections are refined (Post and Bish 1989). In this study, resulting unit-cell parameters of mixtures are essentially the same as those of pure samples, although they were slightly influenced by preferred orientation. If the step width used to obtain the XRD patterns is reduced, more accurate unit-cell parameters for minor phases can be obtained.

It is believed that the result of the quantitative analysis

of the perthite sample is very accurate because the microcline and albite of the perthite have similar morphological characteristics. The method also yields accurate unit-cell parameters of components in mixtures, and structural information, such as Al-Si ordering, of individual mineral components can be obtained during a quantitative analysis. Therefore, the Rietveld method is one of the most useful and promising methods for obtaining accurate quantitative phase information from perthite samples and other multiphase samples.

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TABLE 6. Refinement indices for the Chungsan perthite

R_p (%)	19.34	$R_{B,Mc}$	9.46
R_{wp} (%)	25.01	$R_{B,Ab}$	11.51
S^2	1.28	$R_{B,Qtz}$	8.43
d	1.46		

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