RIETVELD REFINEMENT OF MICACEOUS MATERIALS: MUSCOVITE-2M_1, A COMPARISON WITH SINGLE-CRYSTAL STRUCTURE REFINEMENT

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ABSTRACT

The crystal structure of a 2M_1 muscovite has been refined by the Rietveld method using X-ray powder-diffraction data collected in reflection and transmission geometries, and compared with the results of single-crystal structure-refinement and electron-microprobe analysis of the same material. The Rietveld refinements converged to R_{wp} = 12.4, 8.7% (R_{exp} = 8.1, 5.9%) and R_B = 2.3, 2.5% for transmission- and reflection-geometry data, respectively; the single-crystal structure-refinement converged to an R index of 4.1% for 981 reflections measured with Mo$K\alpha$ X-radiation. Cell dimensions, interatomic distances and angles, and site occupancies obtained by Rietveld refinement agree well with those from the single-crystal structure-refinement. Comparison of the experimental powder-diffraction patterns with the powder pattern calculated from the results of the single-crystal structure-refinement shows preferred-orientation effects in the experimental patterns. However, comparison of the experimental powder-diffraction patterns with the calculated patterns from the Rietveld refinement shows no sign of any residual preferred-orientation effects, indicating that the algorithm for handling preferred orientation in the Rietveld-refinement procedure is effective.

Keywords: Rietveld, single crystal, muscovite, structure refinement.

SOMMAIRE

Nous avons affiné la structure cristalline de la muscovite 2M_1 par la méthode de Rietveld en utilisant des données de diffraction X obtenues sur poudre et prélevées en mode réflexion et transmission, et nous avons fait la comparaison de ces résultats avec ceux d'analyses à la microsonde électronique et d'une ébauche de la structure d'un cristal unique du même échantillon. Les affinements de Rietveld ont donné un résidu R_{wp} de 12.4 et 8.7% (R_{exp} = 8.1, 5.9%) et un R_B de 2.3 et 2.5% pour une géométrie en mode transmission et en mode réflexion, respectivement. Par contre, l'affinement sur cristal unique, portant sur 981 réflexions mesurées avec rayonnement Mo$K\alpha$, a donné sur un résidu R de 4.1%. Les dimensions de la maille, les distances et les angles interatomiques, et l'occupation des sites obtenus par la méthode de Rietveld concordent bien avec les résultats de l'affinement effectué sur cristal unique. Une comparaison des spectres de diffraction X mesurés sur poudre avec le spectre calculé à partir de l'affinement effectué sur cristal unique illustre bien les effets d'une orientation préférentielle des grains dans les spectres mesurés sur poudre. Toutefois, une comparaison de ces mêmes spectres avec les spectres calculés d'après la méthode de Rietveld ne montre pas d'effets résiduels dus à ce facteur. Nous démontrons ainsi que l'algorithme prévu pour compenser l'orientation préférentielle dans le protocole d'affinement de Rietveld a un bon rendement.

(Traduit par la Rédaction)

Mots-clés: Rietveld, cristal unique, muscovite, affinement structural.

INTRODUCTION

The Rietveld method is an important tool in crystal-chemical studies of fine-grained materials. However, in some cases, its effectiveness can be limited by preferred orientation during data collection, particularly where the material has a micaceous habit. Intensities of basal reflections (00*l* reflections) tend to be enhanced (reflection geometry) or diminished (transmission geometry) to a degree that prevents accurate refinement of the structure (Sato *et al.* 1981). There are comprehensive reviews (Bish & Reynolds 1989) on the numerous sample-loading methods. Whereas these methods are generally effective in preparing random mounts of non-micaceous materials, most of them are not totally effective for micaceous materials. However, it has been shown (Bish & Von Dreele 1989, Bish & Johnston 1993, Catti *et al.* 1994) that, with care and certain numerical corrections of the observed intensity data, good-quality Rietveld refinements can be done on naturally occurring fine-grained micaceous materials (*e.g.*, kaolinite and dickite). It is our purpose here to

TABLE 1. CHEMICAL COMPOSITION AND
UNIT FORMULA * OF MUSCOVITE

SiO_2 wt%	45.53	Si	3.068
Al_2O_3	36.34	$^{[IV]}Al$	0.932
FeO	0.52	$^{[VI]}Al$	1.954
MnO	0.22	Fe	0.029
MgO	–	Mn	0.013
CaO	–		
K_2O	10.18	K	0.876
Rb_2O	0.24	Na	0.096
Na_2O	0.74	Rb	0.010
F	1.20	OH	1.744
H_2O**	3.88	F	0.256
O = F	−0.50		
Total	98.35	O	10

* based on 12(O,OH,F) with OH + F = 2.0
** estimated by stoichiometry

examine the accuracy of Rietveld refinement of micaceous materials by comparing the results of Rietveld and single-crystal structure refinements, using muscovite-$2M_1$ as an example.

EXPERIMENTAL

The muscovite used in the present study is from Himalaya mine, Mesa Grande, California. Electron-microprobe analysis using a CAMECA SX–50 operating in wavelength-dispersion mode (Hawthorne *et al.* 1993) shows that it has almost end-member composition, $KAl_2(Si_3Al)O_{10}(OH,F)_2$ (Table 1).

Single-crystal diffraction

A cleavage fragment of muscovite was mounted on a Nicolet *R3m* automated four-circle diffractometer. Cell dimensions (Table 2) were refined from the setting angles of 25 automatically aligned intense reflections.

Intensity data were collected according to the procedure of Hawthorne & Groat (1985). Absorption corrections were done with the psi-scan method, modeling the crystal shape as a thin plate. Intensities were corrected for background, absorption, Lorentz and polarization effects, and reduced to structure factors. Details concerning these procedures are given in Table 2.

Single-crystal structure refinement

Crystal structures were refined using the SHELXTL–PC system of programs; *R* indices are of the form given in Table 2. Scattering curves for neutral atoms, together with anomalous dispersion corrections, were taken from Cromer & Mann (1968) and Cromer & Liberman (1970), respectively. The structure was refined in the space group *C2/c* using the structural parameters of Richardson & Richardson (1982) as the starting model. Full-matrix least-squares refinement of positional and anisotropic-displacement parameters converged to an *R* index of 4.1%. We then introduced a refinable structure-factor weighting scheme and an isotropic extinction correction, but there was no significant improvement in the refinement; here we report the results of the unit-weight refinement.

Powder diffraction

The muscovite crystals were cut as finely as possible with a pair of scissors, and then ground in alcohol to less than 10 μm using an automated grinder. After drying, the powder was worked with a piece of weighing paper such that individual crystallites were disaggregated and randomized as much as possible.

Data collection in reflection geometry: Powders were front-loaded into Al holders, worked with a probe to remove any air pockets, and the surface was then chopped with a razor blade to minimize surface and near-surface preferred orientation of the crystallites. X-ray intensity data were collected on a Philips automated diffraction system PW1710 equipped with a graphite-crystal monochromator for Cu*K*α radiation. Intensities were measured at 0.02°2θ steps with counting times of 3 s per step and a scan range of 8–132°2θ.

Data collection in transmission geometry: A thin film of powder was spread (without solvent) on prolene over a circular area ~10 mm in diameter, whose boundary was confined by a thin wire ring glued onto the prolene using hair spray. The thin film was finely serrated with a razor blade, and then carefully covered by prolene to fix the powder during data collection. Intensity data were collected on a Siemens D5000 X-ray diffractometer in the 2θ range 8–116° in steps of 0.02° 2θ with a step-counting time of 20 s. The

TABLE 2. SINGLE-CRYSTAL X-RAY DIFFRACTION DATA-COLLECTION AND
REFINEMENT INFORMATION FOR MUSCOVITE-$2M_1$

a (Å)	5.180(4)	Crystal size (mm)	0.60 x 0.20 x 0.01
b (Å)	8.993(6)	Radiation/Mono.	Mo*K*α/Gr
c (Å)	20.069(13)	Total no. |*F*|	1376
β (°)	95.69(6)	No. of |*F*|obs	981
V (Å³)	930(1)	*R*(azimuthal) %	2.7
		R(observed) %	4.1
Space group	C2/c	*wR*(observed) %	4.5
Z	4		

$R = \Sigma(|F_o|-|F_c|)/\Sigma|F_o|$
$wR = [\Sigma(w|F_o|-|F_c|)^2/\Sigma wF_o^2]^{1/2}, w = 1$

TABLE 3. DETAILS OF POWDER-DIFFRACTION INTENSITY-DATA COLLECTION AND RIETVELD REFINEMENT FOR MUSCOVITE-2M_1

	Trans.	Refl.		Trans.	Refl.
a (Å)	5.1765(4)	5.1805(7)	Unique reflections	718	718
b (Å)	8.9872(6)	8.994(1)	Structural parameters	37	37
c (Å)	20.072(1)	20.086(2)	Experimental parameters	20	20
β (°)	95.756(6)	95.740(7)	N–P	4888	
V (Å³)	929.08	931.18	R_P	9.5	6.7
Space group	$C2/c$	$C2/c$	R_{WP}	12.4	8.7
2θ scan range (°)	8–116	8–116	R_{EXP}	8.1	5.9
step interval (°2θ)	0.02	0.02	R_{BRG}	2.3	2.5
integration time/step (s)	20	3	DW d statistic	1.42	0.97
maximum intensity (counts)	2496	3509			

instrument operates in transmission geometry and has a curved-Ge crystal incident-beam monochromator that provides monochromatic CuKα_1 radiation. A Kevex Psi-II solid-state (energy-dispersion) detector was used to record the diffracted radiation. Details of the data collection are listed in Table 3.

Rietveld structure refinement

Structure refinement was done using the program LHPM3 (originally written as DBW3.2 by Wiles & Young 1981 and modified by Hill & Howard 1986). A pseudo-Voigt peak-shape was used (with variable-percentage Lorentzian character), the FWHM (full peak-width at half-maximum height) was varied as a function of 2θ using the expression of Caglioti *et al.* (1958), and the peak asymmetry was corrected using the function of Rietveld (1969). Structural variables included atomic coordinates, cation-site occupancies, and an overall isotropic-displacement factor; non-structural variables were scale factor(s) and parameters for background correction, peak shape and asymmetry, and a preferred-orientation correction. Individual isotropic-displacement parameters were fixed at "reasonable" values (*i.e.*, the relative sizes of the parameters were taken from single-crystal work), and an overall displacement parameter was refined to scale the individual values. Those site occupancies taken as variable were refined with no constraints of any sort. Refinement was terminated when the maximum shift or error was less than 0.01. Minor crystalline alumina (Al_2O_3) was introduced into the sample during grinding. This impurity phase was accounted for using simultaneous two-phase Rietveld refinement.

RESULTS AND DISCUSSION

Cell dimensions obtained from single-crystal and Rietveld refinements are given in Tables 2 and 3, respectively. Atomic positions are listed in Table 4,

and interatomic distances and angles are compared in Table 5. Structure-factors and powder-diffraction step-scan intensities may be obtained from The Depository of Unpublished Data, CISTI, National Research Council of Canada, Ottawa, Ontario K1A 0S2.

TABLE 4. FINAL ATOMIC COORDINATES OF MUSCOVITE-2M_1

	x	y	z	Occupancy	U_{iso}
		Rietveld refinement (transmission mode)			
Al	0.251(2)	0.080(1)	−0.0015(6)	0.97(1)	–
T1	0.449(2)	0.257(1)	0.1341(4)	0.98(1)	–
T2	0.033(2)	0.431(1)	0.3650(5)	0.95(1)	–
K	0	0.099(1)	1/4	0.86(2)	–
OH	0.037(3)	0.066(2)	0.4537(7)	1.00	–
O1	0.375(3)	0.245(2)	0.0537(10)	1.00	–
O2	0.042(3)	0.445(2)	0.4469(10)	1.00	–
O3	0.410(3)	0.088(2)	0.1678(10)	1.00	–
O4	0.244(3)	0.368(2)	0.1683(2)	1.00	–
O5	0.249(4)	0.307(2)	0.3440(3)	1.00	–
		Rietveld refinement (reflection mode)			
Al	0.249(2)	0.081(1)	−0.0012(5)	0.98(1)	–
T1	0.447(2)	0.256(1)	0.1346(4)	0.97(1)	–
T2	0.032(2)	0.428(1)	0.3642(4)	0.93(1)	–
K	0	0.104(1)	1/4	0.94(2)	–
OH	0.045(3)	0.068(2)	0.4526(6)	1.00	–
O1	0.379(3)	0.246(2)	0.0540(8)	1.00	–
O2	0.035(2)	0.442(2)	0.4483(8)	1.00	–
O3	0.413(3)	0.090(3)	0.1667(6)	1.00	–
O4	0.267(4)	0.372(2)	0.1691(6)	1.00	–
O5	0.248(4)	0.307(2)	0.3438(7)	1.00	–
		Single-crystal refinement			
Al	0.2510(2)	0.0838(1)	0.0000(1)	0.965(8)	71(3)
T1	0.4514(2)	0.2582(1)	0.1355(1)	0.944(8)	81(3)
T2	0.0345(2)	0.4295(1)	0.3646(1)	0.942(8)	78(3)
K	0	0.0986(2)	1/4	0.894(6)	226(5)
OH	0.0429(6)	0.0617(3)	0.4501(2)	1.00	135(8)
O1	0.3836(6)	0.2511(3)	0.0536(2)	1.00	136(8)
O2	0.0380(6)	0.4447(3)	0.4463(2)	1.00	134(8)
O3	0.4128(6)	0.0925(4)	0.1682(2)	1.00	189(9)
O4	0.2516(6)	0.3726(4)	0.1688(2)	1.00	189(9)
O5	0.2469(7)	0.3083(4)	0.3426(2)	1.00	208(10)

TABLE 5. INTERATOMIC DISTANCES (Å) AND ANGLES (°) FOR MUSCOVITE

		Single–crystal	Trans.	Refl.
T1–O1		1.646(3)	1.62(2)	1.62(2)
T1–O3		1.650(4)	1.68(2)	1.64(2)
T1–O4		1.649(4)	1.66(2)	1.64(2)
T1–O5a		1.643(4)	1.65(2)	1.66(2)
<T1–O>		1.647	1.65	1.64
O1–T1–O3		109.8(2)	108.4(12)	108.4(13)
O1–T1–O4		110.4(2)	110.9(9)	112.4(9)
O1–T1–O5a		112.3(2)	113.9(7)	112.5(8)
O3–T1–O4		107.3(2)	105.7(9)	108.6(9)
O3–T1–O5a		107.0(2)	106.7(10)	106.5(10)
O4–T1–O5a		109.8(2)	110.8(11)	108.2(11)
<O–T1–O>		109.4	109.4	109.4
T2–O2		1.643(4)	1.64(2)	1.69(2)
T2–O3b		1.642(4)	1.60(2)	1.62(2)
T2–O4c		1.646(3)	1.62(2)	1.65(2)
T2–O5		1.641(4)	1.66(2)	1.64(2)
<T2–O>		1.643	1.63	1.65
O2–T2–O3b		109.9(2)	110.6(11)	109.3(11)
O2–T2–O4c		110.6(2)	112.1(8)	110.2(9)
O2–T2–O5		112.5(2)	110.8(9)	111.1(9)
O3b–T2–O4c		107.1(2)	109.1(11)	107.8(10)
O3b–T2–O5		110.1(2)	109.3(10)	110.3(9)
O4c–T2–O5		106.5(2)	104.7(12)	108.1(11)
<O–T2–O>		109.4	109.4	109.5
Al–O1		1.936(3)	1.96(2)	1.93(2)
Al–O1f		1.924(3)	1.93(2)	1.97(2)
Al–O2g		1.920(3)	1.92(2)	1.93(2)
Al–O2h		1.939(3)	1.89(2)	1.89(2)
Al–OHc		1.913(3)	1.89(2)	1.89(2)
Al–OHi		1.913(3)	1.86(2)	1.89(2)
<Al–O>		1.924	1.91	1.92
K–O3	x2	2.824(3)	2.82(1)	2.85(1)
K–O4d	x2	2.833(4)	2.88(1)	2.86(2)
K–O5	x2	2.860(4)	2.87(1)	2.84(2)
<K–O$_{inner}$>		2.839	2.86	2.85
K–O3e	x2	3.308(3)	3.32(1)	3.32(1)
K–O4	x2	3.296(4)	3.25(1)	3.27(2)
K–O5d	x2	3.535(4)	3.55(2)	3.58(2)
<K–O$_{outer}$>		3.380	3.37	3.39

a: 1–x, y, ½–z; b: ½–x, ½+y, ½–z; c: –x, y, ½–z; d: –½+x, –½+y, z;
e: x–1, y, z; f: ½–x, ½–y, –z; g: ½–x, –½+y, ½–z; h: ½+x, ½–y, –½+z;
i: x, –y, –½+z.

The final calculated patterns from the Rietveld structure-refinement are compared to the observed patterns in Figure 1. The fit for the transmission-geometry data is very close (Fig. 1a), with no significant intensity in the difference pattern. The fit is not quite as close for the reflection-geometry data (Fig. 1b). There is some residual intensity associated with the 004, 006 and 00$\overline{10}$ peaks, suggesting that we are not completely accounting for preferred orientation in the sample; nevertheless, the agreement is still quite good.

How significant is the preferred orientation in each sample? We can evaluate this by comparing the observed patterns with the ideal powder pattern calculated from the coordinates and site populations of the refined single-crystal structure (Fig. 2). Both powder patterns in Figure 2 show significant preferred-orientation effects, but these seem to be more severe in the transmission-geometry pattern, as indicated by the intensity difference between the observed and ideal patterns (Fig. 2a). Nevertheless, the difference patterns in Figure 1 indicate that the preferred-orientation correction copes better with this effect in the transmission-geometry sample. Two other effects warrant comment here. First, note the increased resolution in the transmission-geometry pattern compared to that observed in the reflection-geometry pattern. Second, note the difficulty in modeling the shape of the basal reflection 002. This is a common feature in sheet silicates, and is usually accommodated by omitting the lowest-order reflection from the refinement (e.g., Bish & Von Dreele 1989).

Accuracy of the refined structure

Agreement of the observed and calculated patterns is not an indication of accurate results; a model that produces good agreement can still incorporate systematic error and hence be inaccurate. However, in the present case, we can test this possibility for each set of powder intensity-data, as we have results of both an electron-microprobe analysis and a single-crystal structure-refinement on the same material.

The unit formula calculated from the electron-microprobe analysis (Table 1) indicates the following site-occupancies: $Al^* = 1.02$, $T(1) = T(2) = 0.98Si^*$, $K^* = 0.93$; $Al^* = [6]Al + 26Fe/13 + 25Mn/13$, $Si^* = Si + 13[4]Al/14$, $K^* = K + 11Na/19$. These agree closely with the values derived from both single-crystal and Rietveld structure refinement.

The best way to compare two sets of experimental results on the same material is to use half-normal probability-plot analysis (Abrahams & Keve 1971), where the parameter differences are divided by the corresponding pooled standard-deviations, ordered and then plotted against the expected normal distribution for small samples (Hamilton & Abrahams 1972). If there is no error, the plot should have unit slope and should pass through the origin. In the present case, the half-normal probability plots are linear (Fig. 3), with r^2 values of 0.97 and 0.99, and slopes of 1.94(6) and 2.12(5) for the transmission and reflection data, respectively. In each case, the intercept passes though the origin (within the standard error of estimate), and hence there is no systematic error involved in the two sets of results. However, the slopes of the plots should be 1.0 if the assigned standard deviations are correct. For both sets of data, the slopes are 2.0 (within two standard deviations), indicating that the pooled standard deviations are wrong. The standard deviations from the Rietveld refinements (Table 3) are up to an order of magnitude larger than the standard deviations for the single-crystal refinement. The pooled standard deviations used in the half-normal probability-plot analysis are totally dominated by the standard deviations from the Rietveld refinement. Hence, any
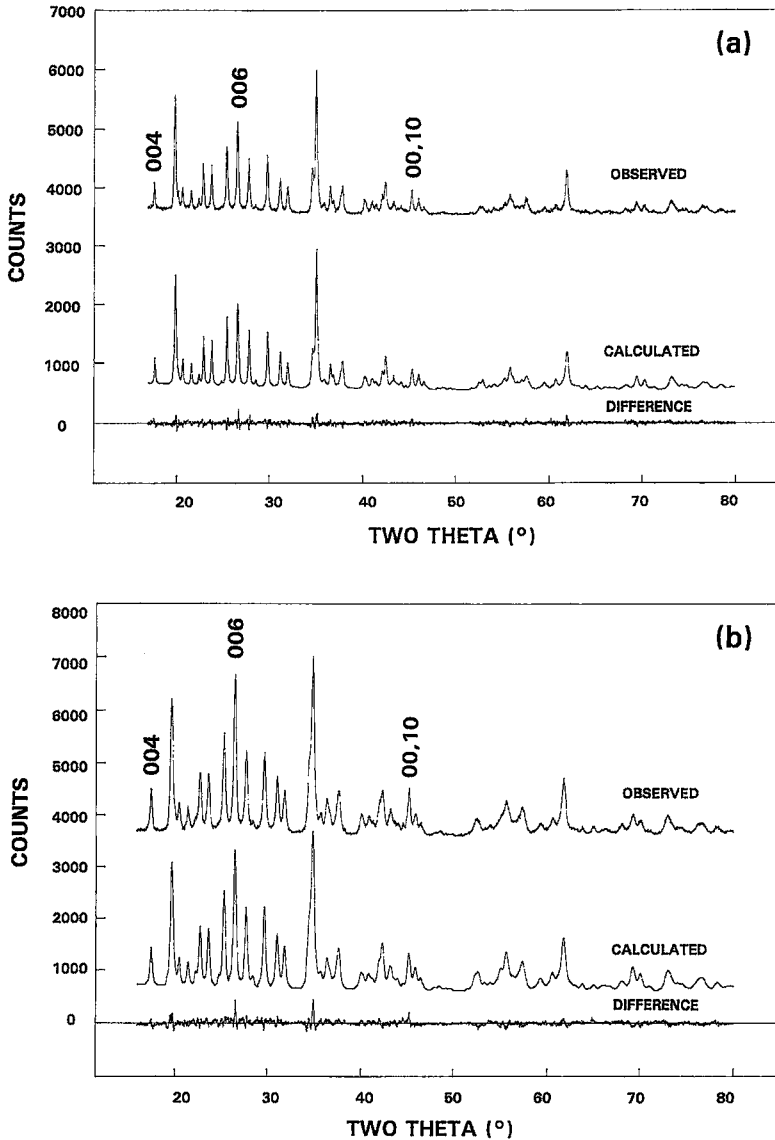
FIG. 1. Observed (upper), Rietveld calculated (middle) and difference (lower) X-ray powder-diffraction patterns of muscovite-2M_1; (a) transmission mode; (b) reflection mode. In each case, constant counts have been added to each pattern to displace them vertically.

reasonable inaccuracy in the single-crystal standard deviations (*i.e.*, by a factor of 1 to 2) will have an insignificant effect on the pooled standard deviations; inaccuracy in the latter must result from inaccuracy in the Rietveld standard deviations.

It is well known (Hill & Flack 1987) that serial correlation in Rietveld structure-refinement results in significant underestimation in the calculated standard deviations; Hill & Flack (1987) have shown that a weighted form of the Durbin–Watson statistic (Durbin & Watson 1971) is sensitive to the amount of serial correlation between least-squares residuals in Rietveld refinement of step-scan powder-diffraction data: a *d* statistic of ~2.0 indicates no serial correlation. The
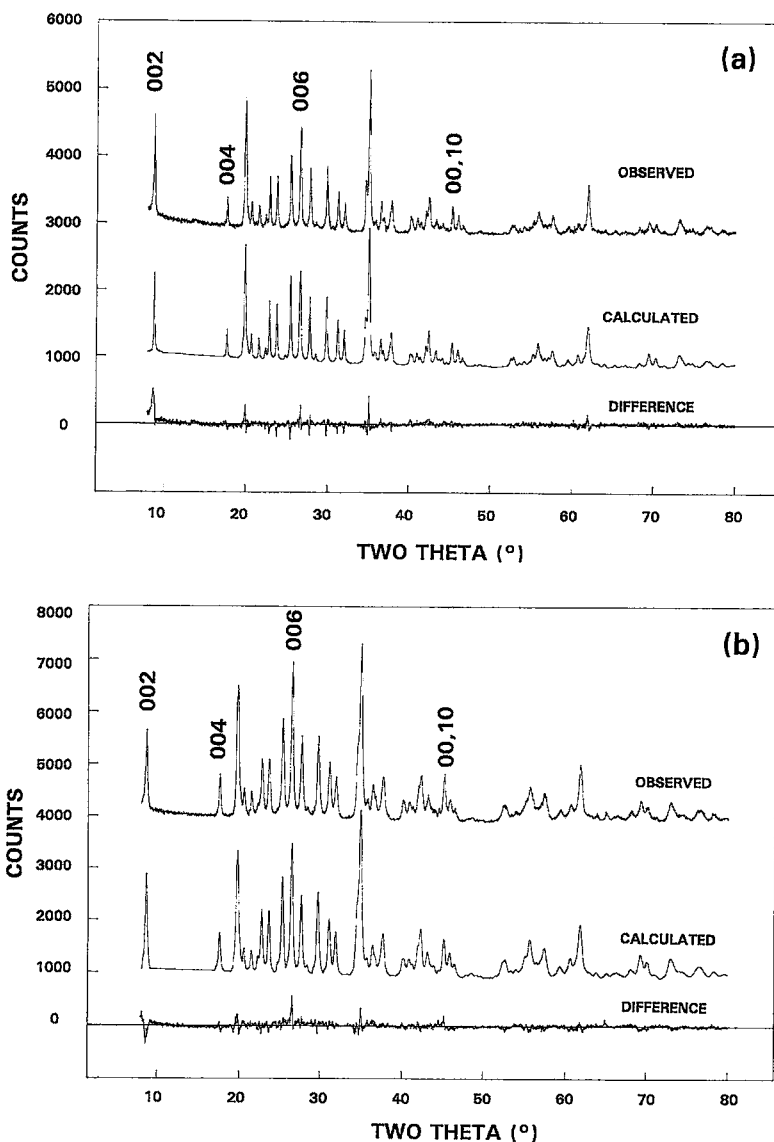
FIG. 2. Observed (upper), calculated from refined single-crystal structure (middle) and difference (lower) X-ray powder-diffraction patterns of muscovite-2M_1; (a) transmission mode; (b) reflection mode.

Durbin–Watson d statistic for the Rietveld refinements reported here do differ significantly from 2.0 (Table 3). Bérar & Lelann (1991) have introduced a method to correct standard deviations for serial correlation, and the standard deviations quoted in Table 4 have been corrected with this algorithm. Nevertheless, the slopes in Figure 3 show that one or both sets of standard deviations are still underestimated. The single-crystal standard deviations are up to an order of magnitude less than the standard deviations from the Rietveld refinements, and hence do not contribute significantly to the pooled standard deviations. Thus the slope of the half-normal probability plot is the factor by which the Rietveld standard deviations are incorrect
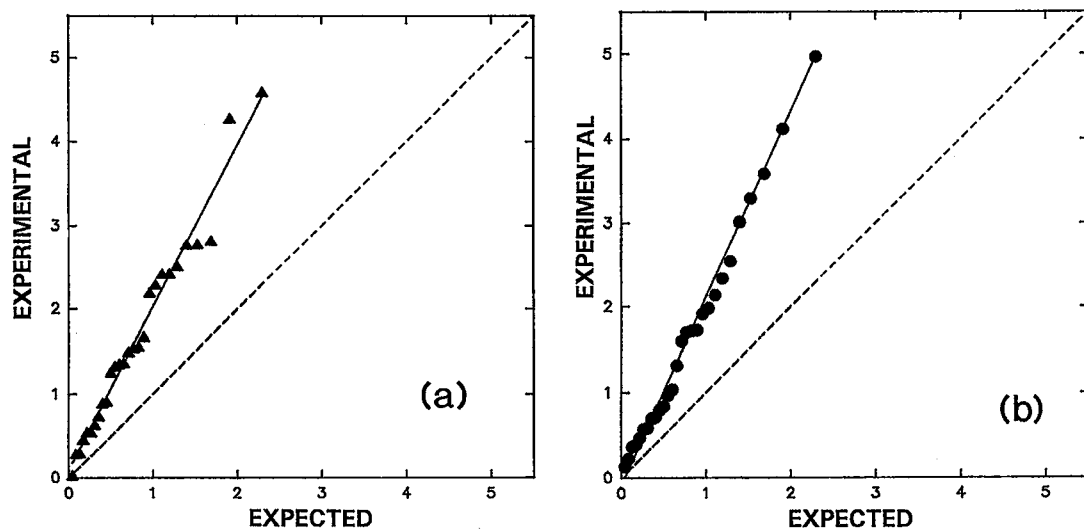
FIG. 3. Half-normal probability plots for refined positional coordinates of muscovite-2M_1; (a) single-crystal and transmission-mode Rietveld refinements; (b) single-crystal and reflection-mode Rietveld refinements.

(*i.e.*, 2.0). Nevertheless, there is good agreement between the refined parameters, indicating that a platy habit and the presence of preferred orientation in the sample do not preclude accurate structure-refinement by the Rietveld method.

CONCLUSIONS

Rietveld refinement of micaceous materials can give accurate structural parameters provided care is taken to minimize preferred orientation for intensity-data collection, and provided a preferred-orientation correction is used in the least-squares refinement. Both reflection and transmission geometries are equally effective for data collection.

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