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STATE-OF-THE-ART HIGH-RESOLUTION POWDER X-RAY DIFFRACTION (HRPXRD) ILLUSTRATED WITH RIETVELD STRUCTURE REFINEMENT OF QUARTZ, SODALITE, TREMOLITE, AND MEIONITE

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Abstract

Synchrotron high-resolution powder X-ray diffraction (HRPXRD) data and Rietveld structure refinements were used to examine the crystal structures of quartz, sodalite, tremolite, and meionite $Me_{79.6}$, and compare them with those obtained by single-crystal diffraction (SXTL). The purpose is to illustrate that crystal structures obtained by HRPXRD are comparable to those obtained by SXTL. The comparisons indicate important and significant differences between the structures obtained by the two methods. The cell parameters obtained by Rietveld refinements using HRPXRD data appear of superior quality to those obtained by SXTL. The <Si–O> distances in pure SiO_4 tetrahedron in quartz, sodalite, and tremolite are 1.6081(3), 1.6100(2), and 1.620(1) Å, respectively. These values are affected by interstitial cations. In meionite $Me_{79.6}$, the average <*T*1–O> and <*T*2–O> distances are 1.647(1) and 1.670(1) Å, respectively, and they indicate that the occupancies are $(Al_{0.28}Si_{0.72})$ for *T*1 where the atoms are partially ordered and $(Al_{0.45}Si_{0.55})$ for *T*2 site where the atoms are nearly disordered, based on sodalite Si–O and Al–O distances of 1.6100(2) and 1.7435(2) Å, respectively.

Keywords: quartz, sodalite, tremolite, meionite, crystal structure, HRPXRD.

Sommaire

Nous nous sommes servis des données diffractométriques sur poudre obtenues en rayonnement synchrotron avec détecteurs à résolution élevée et d'un affinement de Rietveld de ces données pour établir la structure du quartz, de la sodalite, la trémolite et la méionite $Me_{79.6}$, en vue de les comparer avec les résultats d'affinements obtenus sur monocristaux. Notre but était de comparer la qualité des descriptions structurales obtenues par les deux méthodes. Ces comparaisons révèlent des différences importantes. Les paramètres réticulaires obtenus par affinement de Rietveld en utilisant les données obtenues sur poudre à résolution élevée s'avèrent supérieurs en qualité à ceux découlant de l'affinement de données obtenues sur monocristal. Les distances <Si–O> des tétraèdres SiO_4 dans le quartz, la sodalite et la trémolite sont 1.6081(3), 1.6100(2), et 1.620(1) Å, respectivement. Ces valeurs dépendent de la présence de cations interstitiels. Dans la méionite $Me_{79.6}$, la longueur moyenne des liaisons <*T*1–O> et <*T*2–O> est 1.647(1) et 1.670(1) Å, respectivement, distances qui sont conformes à un taux d'occupation de $(Al_{0.28}Si_{0.72})$ pour *T*1, où les atomes seraient partiellement ordonnés, et $(Al_{0.45}Si_{0.55})$ pour *T*2, où les atomes sont presque totalement désordonnés, en nous basant sur les distances Si–O et Al–O de 1.6100(2) et 1.7435(2) Å, respectivement, déterminées pour la sodalite.

(Traduit par la Rédaction)

Mots-clés: quartz, sodalite, trémolite, méionite, structure cristalline, diffraction sur poudre à haute résolution.

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INTRODUCTION

Several techniques are currently in use to determine and refine crystal structures. The commonly used method is based on single-crystal (SXTL) X-ray diffraction using laboratory, synchrotron, or neutron sources. Another method is based on powder X-ray diffraction and Rietveld refinement using the diffraction sources mention above. A dedicated high-resolution powder X-ray diffractometer (HRPXRD) was recently made available at beamline 11-BM at the Advanced Photon Source (Lee *et al.* 2008, Wang *et al.* 2008).

The purpose of this study is to illustrate that crystal structures obtained by HRPXRD are comparable to those obtained by the SXTL method. The HRPXRD technique may be used to study: (1) powdered samples that do not occur as single crystals, (2) many samples rapidly, (3) intergrowths (exsolution and zoning), and (4) superstructures; in addition, the technique may be used as an alternative to the SXTL method.

The minerals selected for comparison of the two methods are quartz, sodalite, tremolite, and meionite; all have a well-known crystal structure. In our comparison, the accuracies of the structures are judged mainly by errors on cell parameters and bond lengths, as they reflect the errors on the atom positions.

EXPERIMENTAL

The quartz sample is part of a euhedral crystal. As quartz usually has a high purity, the formula used in the refinement is SiO_2. The sodalite sample is from Bancroft, Ontario, and the structure of the same sample was refined by Hassan & Grundy (1984). The chemical formula used in the refinement is $Na_8[Al_6Si_6O_{24}]Cl_2$. The sample of tremolite is from the Gouverneur district, New York, and the structure of the same sample was refined by Hawthorne & Grundy (1976). The chemical formula used in the structure refinement is $(Na_{0.38}K_{0.12}Ca_{1.8})Mg_{4.94}Fe_{0.06}(Al_{0.2}Si_{7.8})O_{22}(OH)_{1.34}F_{0.66}$. The meionite sample is from a skarn deposit from Slyudyanka, Siberia, Russia, and the chemical composition is $(Ca_{3.15}Na_{0.77}K_{0.04})[Si_{6.84}Al_{5.16}O_{24}]Cl_{0.01}(CO_3)_{0.67}(SO_4)_{0.24}$ and corresponds to $Me_{79.6}$ (Evans *et al.* 1969).

Crystals were hand-picked under a binocular microscope, and finely ground in an agate mortar and pestle for HRPXRD experiments that were performed at beamline 11-BM, Advanced Photon Source, Argonne National Laboratory. The samples were loaded into kapton capillaries and rotated during the experiment at a rate of 90 rotations per second. Data were collected to a maximum 2θ of about 50 with a step size of 0.0005 and a step time of 0.1 s/step. The beamline optics consist of a platinum-coated collimating mirror, a dual Si (111) monochromator and a platinum-coated vertically focusing mirror. The HRPXRD traces were collected with twelve silicon crystal analyzers, which increase detector efficiency as well as reduce the angular range to be scanned, and therefore, allows rapid acquisition of data. A silicon and alumina NIST standard (ratio of 1/3 Si to 2/3 Al_2O_3) was used to calibrate the detector response, zero offset, and to determine the wavelength used in the experiment (Table 1). Data were merged by interpolating measured counts onto a regularly spaced grid, and by applying corrections for small differences in wavelength (~1 eV). In two recent publications, the technical aspects of the experimental set-up, including schematic drawings, are given (Lee *et al.* 2008, Wang *et al.* 2008).

RIETVELD STRUCTURE REFINEMENTS USING HRPXRD

The HRPXRD traces were modeled using the Rietveld method (Rietveld 1969), as implemented in the GSAS program (Larson & Von Dreele 2000) and using the EXPGUI interface (Toby 2001). The starting coordinates of the atoms, cell parameters, and space groups were taken from Le Page & Donnay (1976) for quartz, Hassan & Grundy (1984) for sodalite, Hawthorne & Grundy (1976) for tremolite, and Teertstra *et al.* (1999) for meionite $Me_{79.6}$.

The background was modeled with a Chebyschev polynomial, and the reflection-peak profiles were fitted using profile type-3 in the GSAS program with the refinable coefficients GU, GV, GW, LX, and LY. All the refinements were carried out in a sequence; we first refined a scale factor, then the background, cell, zero offset, profile variables, atom positions, isotropic and subsequently anisotropic displacement parameters. At the end of the refinement, all parameters were allowed to vary simultaneously, and the refinement proceeded to convergence. HRPXRD patterns for the four structures are shown in Figure 1.

The cell parameters and other information regarding data collection and refinement are given in Table 1. The refined positional coordinates and displacement parameters given in Tables 2a, 3a, 4a, and 5a are for quartz, sodalite, tremolite, and meionite $Me_{79.6}$, respectively. Their corresponding bond distances and angles are given in Tables 2b, 3b, 4b, and 5b.

DISCUSSION

The cell parameters obtained by Rietveld refinements using HRPXRD appear superior to those obtained by SXTL, if judged by errors, σ (Table 1). It may be argued that the SXTL method produces accurate unit-cell parameters if all the reflections are used, but this has not been demonstrated in the literature. The SXTL method typically uses about 25 high-angle reflections to refine the unit-cell parameters. As the HRPXRD cell parameters are different from those obtained by the

TABLE 1. CELL AND RIETVELD PARAMETERS

	Quartz	Sodalite	Tremolite	Meionite $Me_{79.6}$
Space group	*P*3$_2$21	*P*$\overline{4}$3*n*	*C*2/*m*	*I*4/*m*
		HRPXRD		
a (Å)	4.913437(2)	8.880679(3)	9.85145(1)	12.16559(2)
b (Å)			18.02911(2)	7.57446(1)
c (Å)	5.405118(3)		5.273416(5)	
β (°)			104.7566(1)	
V (Å^3)	113.00732(9)	700.3878(4)	905.733(2)	1121.033(3)
* N_{obs}	450	561	2850	1588
ξ R_F^2	0.0539	0.0371	0.0432	0.0651
§ DWd	1.004	1.113	0.938	0.959
λ (Å)	0.40236(2)	0.40166(2)	0.40241(2)	0.40243(2)
		† **SXTL**		
a (Å)	4.9134	8.882(1)	9.863(1)	
b (Å)			18.048(2)	
c (Å)	5.4052		5.285(1)	
β (°)			104.79(1)	
V (Å^3)	113.01	700.70	909.6(2)	
Δ*V* (Å^3)	-0.0027	-0.3122	-3.867	

† Cell parameters and structural data used for comparison are from: quartz (Le Page & Donnay 1976), sodalite (Hassan & Grundy 1984), and tremolite (Hawthorne & Grundy 1976). § Dwd: Durbin–Watson d statistic for the merged data. DWd for the unmerged data is twice as large. * N_{obs} is the number of observed Bragg reflections. ξ R_F^2 = R factor based on observed and calculated structure-amplitudes = $[\Sigma(F_o^2 - F_c^2) / \Sigma(F_o^2)]^{1/2}$.

SXTL method, the bond lengths also are expected to be different. The accuracy of structural parameters can be judged on the basis of the errors, σ, determined by the full-matrix least-squares refinement. It may be argued that these errors are underestimated, but the underestimated errors apply to both experimental methods.

In the case of (low) quartz, our cell parameters are nearly identical to those reported by Le Page & Donnay (1976). However, the cell parameters they used were the mean values of those determined by Cohen & Sumner (1958) on three different samples of synthetic quartz (Table 1). Le Page & Donnay (1976) compared their structure of quartz with results of previous refinements by Young & Post (1962), Smith & Alexander (1963), and Zachariasen & Plettinger (1965), and concluded that their structure is the most accurate one. The structure of quartz by Le Page & Donnay (1976) is well refined to an R factor of 0.0171 for 342 observed reflections, whereas we have obtained an R_F^2 factor of 0.0539 for 450 observed reflections (Table 1). However, they had to deal with the problem of twinning in their sample, which was not encountered in our refinement. The Si–O bond lengths, although similar to those of Le Page & Donnay (1976), differ by as much as 2σ (Table 2b). The differences in individual bond-angles are more significant, with differences of about 3σ. On this basis, the structural parameters obtained in the present study of quartz appear more accurate than those obtained by Le Page & Donnay (1976).

The single-crystal structure of sodalite was refined to an R factor of 0.017 for 157 observed reflections (Hassan & Grundy 1984), whereas we have obtained an R_F^2 factor of 0.0371 for 561 observed reflections. In the case of the sodalite structure, σ occurs in the fifth decimal place for atom positions, and the fourth place for bond lengths (Tables 3a, b). Significant differences in bond lengths occur for the Si–O and Na–O distances, although the difference in cell parameter is not so large, about 1σ (Tables 1, 3b). The difference between the two structures clearly resulted from the number of reflections used in the two studies. Sodalite usually does not occur as good single crystals, and does not scatter well with conventional X-rays. With HRPXRD, in spite of the poor quality of the crystal, sodalite diffracts quite well, and we obtained a better refined structure in the present study.

The crystal structure of tremolite was studied by Warren (1929), Zussman (1959), Papike *et al.* (1969), Sueno *et al.* (1973), Hawthorne & Grundy (1976), and Yang & Evans (1996). The structure of tremolite was refined to an R factor of 0.032 for 1376 observed reflections (Hawthorne & Grundy 1976), whereas we have obtained an R_F^2 factor of 0.0432 for 2850 observed reflections. There are significant differences in the cell parameters; the present structure contains values of σ that occur in the fifth decimal place for many atom positions, and typically in the third decimal place for bond lengths, but the values of σ are smaller than those

TABLE 2a. QUARTZ: ATOM COORDINATES AND DISPLACEMENT PARAMETERS (Å^2)

Atom	x	y	z	U_{eq}	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Si	0.47000(5)	0	⅔	0.0062	0.00733(9)	0.0059(1)	0.00535(8)	0.00294(6)	0.00027(5)	0.00055(9)
O	0.4146(1)	0.2678(1)	0.78543(8)	0.0109	0.0120(3)	0.0105(2)	0.0118(2)	0.0065(2)	-0.0029(2)	-0.0040(1)

TABLE 2b. QUARTZ: BOND DISTANCES [Å] AND ANGLES [°], AND COMPARISON WITH SINGLE-CRYSTAL (SXTL) DATA

		HRXRD	SXTL[†]	Δ[§]
Si-O	× 2	1.6048(5)	1.607(1)	0
Si-O	× 2	1.6113(4)	1.611(1)	0
<Si-O>		1.6081(3)	1.609(1)	0
O-Si-O	×2	110.26(3)	110.39(4)	-0.13
O-Si-O	×2	108.729(9)	108.7(4)	0.03
O-Si-O		109.16(4)	109.07(6)	0.09
O-Si-O		109.69(4)	109.57(6)	0.12
<O-Si-O>		109.47(1)	109.47(10)	0
Si-O-Si		143.84(3)	143.68(9)	0.16

[†] From Le Page & Donnay (1976); [§] Δ is difference between HRP×RD and SXTL in all tables.

obtained by SXTL (Tables 1, 4a, b). Significant differences occur in the important *T*–O distances. Hawthorne & Grundy (1976) observed that the <*T*1–O> is longer than the <*T*2–O> distance and placed a small amount of Al at the *T*1 site, whereas the exact opposite can be deduced from the present study (Table 4b). In addition, longer <*T*2–O> than <*T*1–O> were also observed by Papike *et al.* (1969), Sueno *et al.* (1973), and Yang & Evans (1996).

The average <Si–O> distance in quartz, sodalite, and tremolite are 1.6081(3), 1.6100(2), and 1.620(1) Å, respectively. Interestingly, a longer Si–O distance occurs in sodalite than in quartz because Na atoms bond to O atoms that form the TO_4 tetrahedron in sodalite. As the Na atom attracts electrons from the bridging O atoms, the Si–O bonds are longer in sodalite than in quartz, which is devoid of interstitial cations. The Si–O distance is longer in tremolite, where interstitial cations of higher charge (Ca, Mg) bond to the bridging O atoms. Accurate *T*–O (T = Al, Si) distances are important in mineralogy, as they are commonly used to determine Al, Si occupancy. Because of the similar scattering-factors for Al and Si atoms, there is no possibility of accurate site-refinements. However, problems arise where one has to decide about which *T*–O distances to use for Al, Si occupancy. We have obtained accurate <Si–O> distances for the pure SiO_4 tetrahedron in quartz, sodalite, and tremolite. Which of these distances do we use to determine the *T*-site occupancies in meionite Me$_{79.6}$? We choose the *T*–O distances in sodalite because of the similarity of the chemical composition

TABLE 3a. SODALITE: ATOM COORDINATES AND DISPLACEMENT PARAMETERS (Å^2)

Atom	x	y	z	U_{eq}	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Al	0.25	0	0.5	0.0049	0.0050(3)	0.0049(2)	0.0049(2)	0	0	0
Si	0.25	0.5	0	0.0048	0.0035(2)	0.0054(1)	0.0054(1)	0	0	0
O	0.13780(3)	0.14915(3)	0.43910(3)	0.0106	0.0172(3)	0.0063(3)	0.0082(2)	0.0012(1)	-0.0034(2)	0.0051(2)
Na	0.17772(2)	0.17772(2)	0.17772(2)	0.0181	0.01807(7)	0.01807(7)	0.01807(7)	0.00135(9)	0.00135(9)	0.00135(9)
Cl	0	0	0	0.0247	0.02466(7)	0.02466(7)	0.02466(7)	0	0	0

TABLE 3b. SODALITE: BOND DISTANCES [Å] AND ANGLES [°], AND COMPARISON WITH SXTL DATA

		HRXRD	SXTL[†]	Δ
Al-O	×4	1.7435(2)	1.742(2)	0.002
Si-O	×4	1.6100(2)	1.620(2)	-0.01
Na-O	×3	2.3618(3)	2.353(2)	0.009
Na-O*	×3	3.0885(3)	3.087(2)	0.002
Na-Cl		2.7337(4)	2.736(1)	-0.002
O-Al-O	×4	109.064(8)	108.7(1)	0.364
O-Al-O	×2	110.29(2)	111.0(1)	-0.71
<O-Al-O>		109.472(4)	109.47(4)	0.002
O-Si-O	×4	108.026(8)	107.7(1)	0.326
O-Si-O	×2	112.40(2)	113.0(1)	-0.6
<O-Si-O>		109.484(5)	109.47(4)	0.014
Si-O-Al		138.83(2)	138.2(1)	0.63

[†] From Hassan & Grundy (1984).

TABLE 4a. TREMOLITE: ATOM COORDINATES AND DISPLACEMENT PARAMETERS (Å)

Atom	sof	*x*	*y*	*z*	*U*eq	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O1	1	0.1129(1)	0.08519(7)	0.2219(2)	0.0052	0.0068(10)	0.0037(8)	0.0033(8)	-0.0014(6)	-0.0027(7)	0.0021(6)
O2	1	0.1210(2)	0.17050(6)	0.7278(2)	0.0087	0.0179(11)	0.0061(8)	0.0064(8)	-0.0028(6)	0.0089(8)	-0.0012(6)
O3	0.67O + 0.33F	0.1098(2)	0	0.7158(3)	0.0143	0.0309(16)	0.0054(11)	0.0137(13)	0	0.0156(12)	0
O4	1	0.3638(1)	0.24776(7)	0.7923(3)	0.0051	0.0086(11)	0.0044(7)	0.0020(8)	-0.0024(7)	-0.0002(7)	-0.0008(6)
O5	1	0.3453(2)	0.13409(7)	0.0992(3)	0.0080	0.0062(10)	0.0090(8)	0.0106(8)	0.0115(7)	0.0053(7)	0.0023(7)
O6	1	0.3441(2)	0.11854(7)	0.5906(3)	0.0078	0.0099(10)	0.0081(7)	0.0065(8)	0.0029(7)	0.0032(7)	0.0019(7)
O7	1	0.3367(2)	0	0.2923(3)	0.0127	0.0143(15)	0.0186(12)	0.0071(12)	0	0.0046(11)	0
*T*1	0.95Si + 0.05 Al	0.27914(7)	0.08413(3)	0.2981(1)	0.0048	0.0050(4)	0.0045(3)	0.0047(3)	-0.0012(3)	0.0006(3)	-0.0005(3)
*T*2	1.0 Si	0.28831(8)	0.17138(3)	0.8043(1)	0.0046	0.0070(4)	0.0050(3)	0.0029(3)	-0.0023(3)	0.0025(3)	-0.0014(2)
*M*1	1 Mg	0	0.08840(5)	0.5	0.0062	0.0171(8)	0.0034(6)	0.0017(6)	0	0.0071(5)	0
*M*2	0.97Mg + 0.03Fe	0	0.17720(5)	0	0.0080	0.0091(7)	0.0095(6)	0.0063(6)	0	0.0029(5)	0
*M*3	1Mg	0	0	0	0.0083	0.0087(10)	0.0093(9)	0.0079(8)	0	0.0035(8)	0
*M*4	0.9Ca + 0.1Na	0	0.27779(4)	0.5	0.0080	0.0132(5)	0.0056(4)	0.0085(4)	0	0.0074(4)	0
A	0.12K + 0.18Na	0	0.5	0	0.1424	0.160(10)	0.079(6)	0.263(13)	0	0.185(10)	0

sof: site-occupancy factor.

TABLE 4b. TREMOLITE: BOND DISTANCES [Å] AND ANGLES [°], AND COMPARISON WITH SXTL DATA

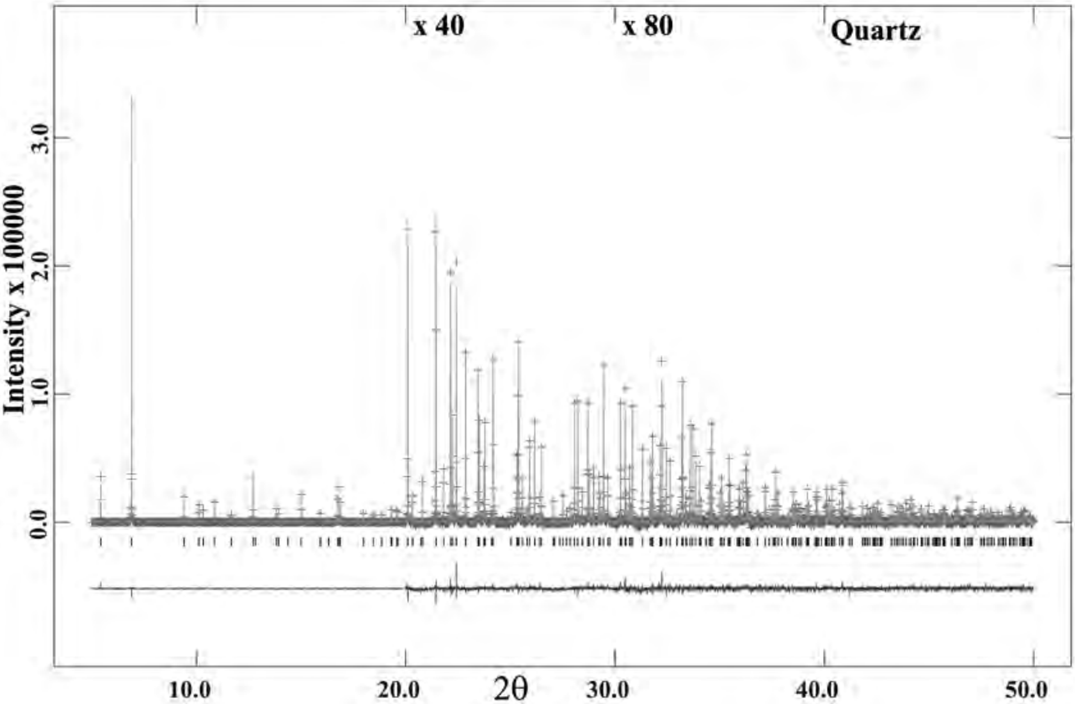
Bonds	HRPXRD	SXTL[†]	Δ	Bonds	HRPXRD	SXTL[†]	Δ
*T*1-O1	1.584(1)	1.611(2)	0	*M*3-O1 ×4	2.074(1)	2.071(2)	0
*T*1-O5	1.638(1)	1.637(2)	0	*M*3-O3 ×2	2.060(2)	2.047(2)	0.01
*T*1-O6	1.634(2)	1.639(2)	0	<*M*3-O>	2.069(1)	2.063(1)	0
*T*1-O7	1.6224(8)	1.629(1)	0				
<*T*1-O>	1.620(1)	1.629(1)	0	*M*4-O2 ×2	2.424(1)	2.415(2)	0
				*M*4-O4 ×2	2.333(1)	2.336(2)	0
*T*2-O2	1.595(2)	1.613(2)	0	*M*4-O5 ×2	2.770(1)	2.766(2)	0
*T*2-O4	1.574(1)	1.589(2)	0	*M*4-O6 ×2	2.541(1)	2.548(1)	0
*T*2-O5	1.655(1)	1.622(2)	0.03	<*M*4-O> [8]	2.517(1)	2.516(1)	0
*T*2-O6	1.672(1)	1.675(2)	0	<*M*4-O> [6]	2.433(1)	2.433(1)	0
<*T*2-O>	1.624(1)	1.625(1)	0				
				A-O5 ×4	2.975(1)	2.973(2)	0
*M*1-O1 ×2	2.055(1)	2.061(2)	0	*A*-O6 ×4	3.146(1)	3.148(2)	0
*M*1-O2 ×2	2.079(1)	2.071(2)	0	*A*-O7 ×2	2.496(2)	2.480(3)	0.02
*M*1-O3 ×2	2.093(1)	2.082(2)	0.01	<*A*-O> [10]	2.9476(4)	2.944(1)	0
<*M*1-O>	2.076(1)	2.071(1)	0				
				*T*1-O5-*T*2	136.9(1)	136.2(1)	0.7
*M*2-O1 ×2	2.167(1)	2.143(2)	0.02	*T*1-O6-*T*2	137.4(1)	137.8(1)	-0.4
*M*2-O2 ×2	2.089(1)	2.091(2)	0	*T*1-O7-*T*1	138.4(1)	137.8(2)	0.6
*M*2-O3 ×2	2.020(2)	2.018(2)	0				
<*M*2-O>	2.092(1)	2.084(1)	0				

[†] From Hawthorne & Grundy (1976).

of sodalite and meionite (both contain large anions), and they are framework aluminosilicates.

The structure of meionite $Me_{79.6}$ was refined to an R_F^2 factor of 0.0651 for 1588 observed reflections (Table 1). The average <*T*1–O> and <*T*2–O> distances are 1.647(1) and 1.670(1) Å, respectively (Table 5b). These distances indicate that the site occupancies are $(Al_{0.28}Si_{0.72})$ for *T*1 and $(Al_{0.45}Si_{0.55})$ for *T*2, based on sodalite Si–O and Al–O distances of 1.6100(2) and 1.7435(2) Å, respectively. Therefore, the Al and Si atoms at the *T*1 site are partially ordered, and nearly disordered at the *T*2 site. Our structure is similar to the structures of $Me_{72.2}$ (Teertstra *et al.* 1999) and Me_{93} (*e.g.*, Lin & Burley 1973, Ulbrich 1973). Our site-occupancy factors (sof) are similar to those indicated by chemical analysis. In particular, the refined sof for Ca is 0.812(4) and gives rise to $Me_{81.2}$ compared to $Me_{79.6}$ from chemical analysis.

The U_{eq} values are similar for the T sites in quartz, sodalite, and tremolite, but they are about twice as large



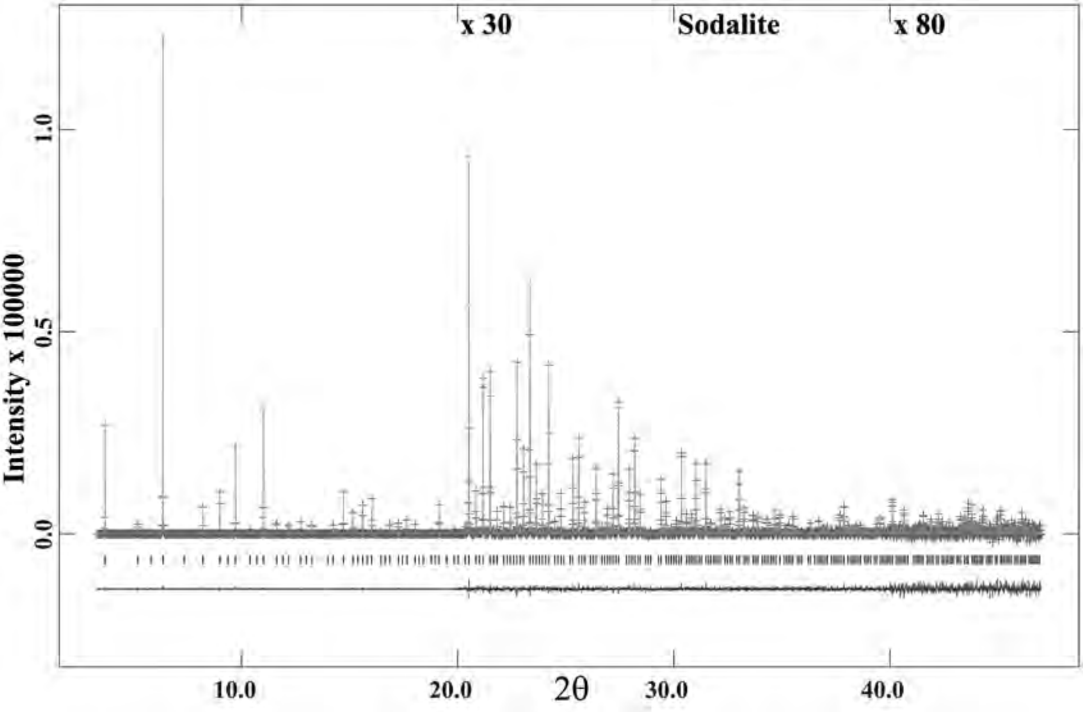
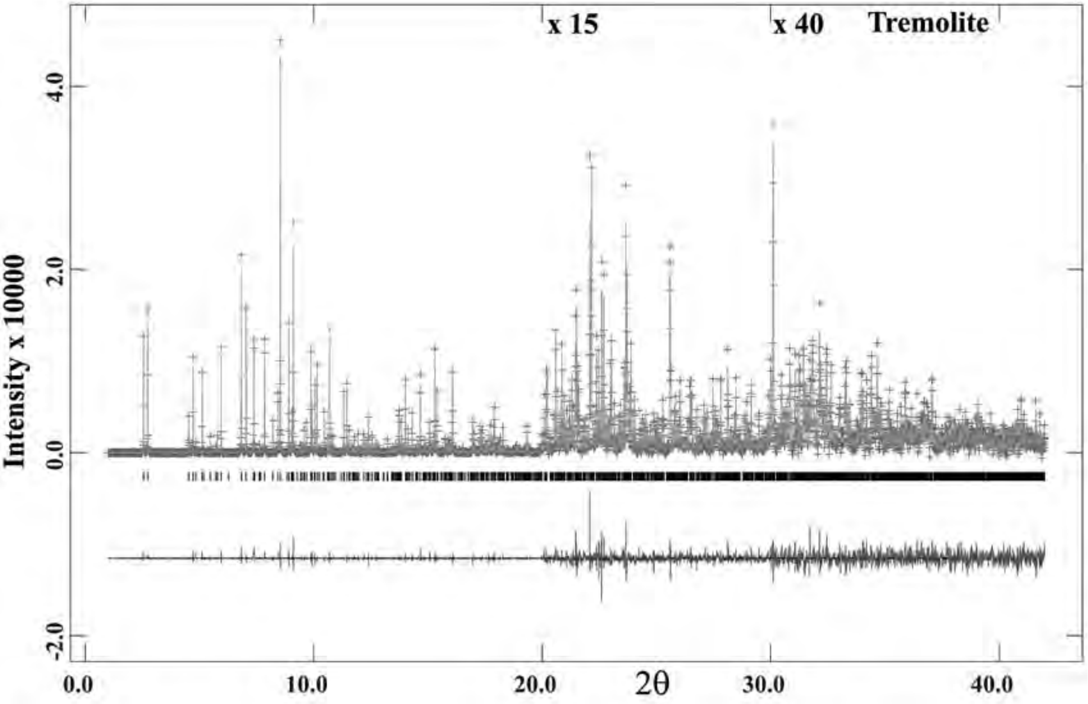
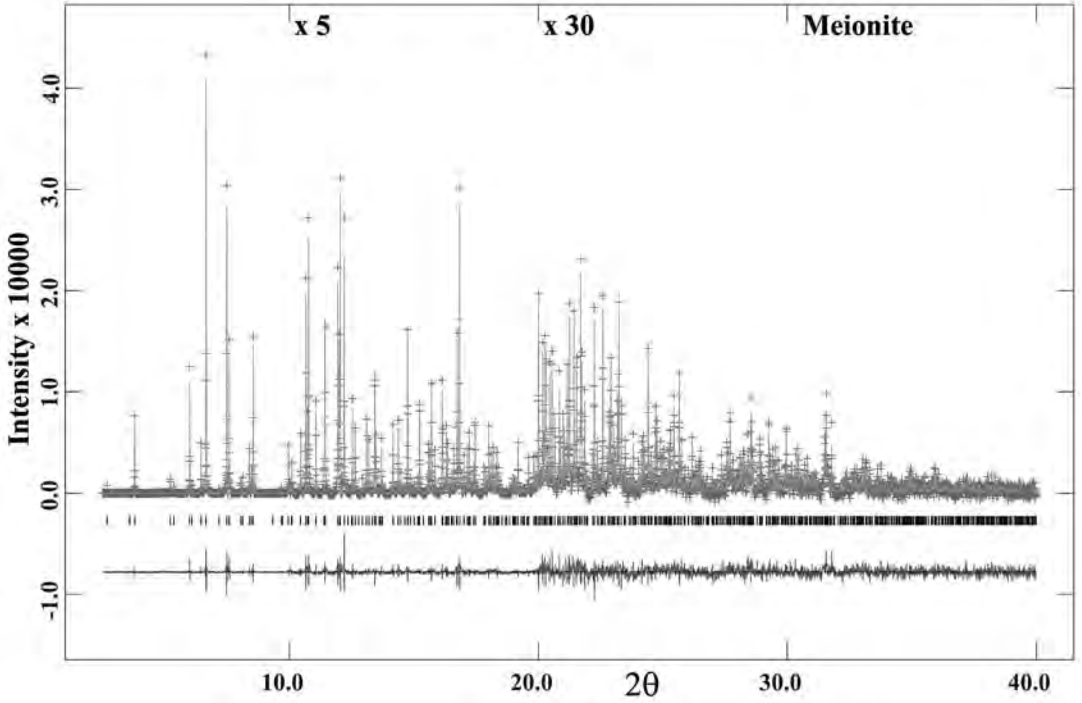


FIG. 1. HRPXRD traces for (a) quartz, (b) sodalite, (c) tremolite, and (d) meionite $Me_{79.6}$, together with the calculated (continuous line) and observed (crosses) profiles. The difference curve ($I_{obs} - I_{calc}$) is shown at the bottom. The short vertical





lines indicate the positions of allowed reflections. Note that the intensity of the high-angle region is scaled by various factors compared to the low-angle region, and this applies to the difference curves as well.

TABLE 5a. MEIONITE $Me_{79.6}$: ATOM COORDINATES AND DISPLACEMENT PARAMETERS (Å)

Atom	sof	*x*	*y*	*z*	*U*eq	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
*T*1	1	0.16039(8)	0.09216(8)	0	0.0117	0.0123(5)	0.0132(5)	0.0096(4)	-0.0001(4)	0	0
*T*2	1	0.16035(5)	0.41298(5)	0.20649(7)	0.0131	0.0101(4)	0.0153(4)	0.0139(3)	-0.0028(3)	-0.0002(3)	-0.0009(3)
Ca1	0.812(4)	0.35870(7)	0.28284(7)	0	0.0237	0.0154(5)	0.0210(5)	0.0346(6)	0.0079(4)	0	0
Na1	0.188(4)	0.35870(7)	0.28284(7)	0	0.0237	0.0154(5)	0.0210(5)	0.0346(6)	0.0079(4)	0	0
S1	0.235	0.5	0.5	0	0.0188	0.024(2)	0.024(2)	0.008(2)	0	0	0
C1	0.668	0.5	0.5	0	0.0188	0.024(2)	0.024(2)	0.008(2)	0	0	0
O1	1	0.0412(2)	0.1518(1)	0	0.0162	0.019(1)	0.008(1)	0.021(2)	0.010(1)	0	0
O2	1	0.1861(2)	0.3735(2)	0	0.0211	0.023(2)	0.019(1)	0.021(1)	0.004(1)	0	0
O3	1	0.4512(1)	0.1493(1)	0.2063(2)	0.0230	0.035(1)	0.022(1)	0.012(1)	0.005(1)	0.0080(9)	0.0011(8)
O4	1	0.2662(1)	0.3673(1)	0.6745(2)	0.0228	0.0260(9)	0.0126(9)	0.030(1)	0.0038(8)	0.0094(8)	-0.0006(7)
O6C	0.501	0.6006(6)	0.5152(9)	0	0.0720	0.089(6)	0.106(6)	0.022(4)	-0.027(6)	0	0
O5S	0.118	0.3931(13)	0.4801(12)	0.0999(15)	0.016(4)						

sof: site-occupancy factor.

TABLE 5b. MEIONITE $Me_{79.6}$: BOND DISTANCES [Å] AND ANGLES [°]

Bonds		HRPXRD	Bonds		HRPXRD
*T*1 -O1		1.622(2)	*M* -O5C		2.51(1)
*T*1 -O1'		1.626(2)	*M* -O5C'		2.37(1)
*T*1 -O4	×2	1.670(1)	*M* -O5S		2.55(1)
<*T*1-O>		1.647(1)	*M* -O5S'		2.49(1)
			M -*A*		3.1519(9)
*T*2 -O2		1.6660(8)			
*T*2 -O3		1.657(1)	C -O5C	×4	1.238(7)
*T*2 -O3'		1.689(1)			
*T*2 -O4		1.667(2)	S -O5S	×4	1.52(1)
<*T*2-O>		1.670(1)			
			*T*1-O1-*T*1		157.1(2)
M -O2	×1	2.372(2)	*T*2-O2-*T*2		139.7(1)
M -O3	×2	2.520(2)	*T*2-O3-*T*2		146.84(9)
M -O4	×2	2.898(1)	*T*1-O4-*T*2		137.94(8)
M -O4'	×2	2.719(1)	<*T*-O-*T*>		145.39(6)
<*M*-O>[7]	×2	2.664(1)			

in meionite $Me_{79.6}$ (Tables 2a, 3a, 4a, 5a). This difference arises from the disordered *T* sites in the meionite structure.

CONCLUSIONS

The cell parameters obtained by Rietveld refinements using HRPXRD data seem to be superior to those obtained by SXTL. The structural parameters obtained by HRPXRD are similar, if not more accurate than those obtained by the SXTL method. The <Si–O> distances for the pure SiO_4 tetrahedron in quartz, sodalite, and tremolite are affected by the surrounding interstitial cations. Therefore, the selection of a T–O distance to determine Al, Si occupancy needs some justification.

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