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MAGNETIC ORDERING AT 4.2 AND 1.3 K IN NONTRONITES OF DIFFERENT IRON CONTENTS: A ^{57}Fe MÖSSBAUER SPECTROSCOPIC STUDY

C. M. CARDILE,[1,3] J. H. JOHNSTON,[1,4] AND D. P. E. DICKSON[2]

[1] Chemistry Department, Victoria University of Wellington, Private Bag Wellington, New Zealand

[2] Department of Physics, The University of Liverpool Liverpool, United Kingdom

Abstract—The ^{57}Fe Mössbauer spectra of six nontronite samples were measured at appropriate temperatures of 4.2 and 1.3 K. Three of the nontronites gave a complex magnetic hyperfine spectrum showing magnetic ordering at 4.2 K, and the other three required a lower temperature of 1.3 K to produce similar magnetic ordering. The spectra were computer-fitted with three closely overlapping sextets which are considered to arise from: (1) Fe^{3+} that is ordered magnetically in the cis-octahedral sites with a greater number of neighboring tetrahedral Fe^{3+} ions (51 T); (2) the cis-octahedral site with the greater number of neighboring Si^{4+} ions (46 T); and (4) the tetrahedral sites (41 T). In an untreated sample a further sextet corresponding to interlayer Fe^{3+} (36 T) was identified. The magnetic ordering was complicated and not directly related to the iron content of these sites. It probably depended also on the overall composition and structural order of the particular nontronite. The ordering appears to have been essentially two-dimensional, consistent with the layer structure of this material.

Key Words—Interlayer site, Iron, Magnetic ordering, Mössbauer spectroscopy, Nontronite, Octahedral site, Tetrahedral site.

INTRODUCTION

Recently Johnston and Cardile (1985) and Cardile and Johnston (1985) reported a detailed ^{57}Fe Mössbauer spectroscopic study of a series of nontronites with different iron contents and interlayer cations. In general, they showed that the Fe^{3+} is located in the two non-equivalent cis-$FeO_4(OH)_2$ octahedral, a tetrahedral, and interlayer sites. The spectrum recorded at room temperature for the untreated (natural) nontronite sample from Spokane County, Washington (SPO) gave a high χ^2 value when computer-fitted with four doublets corresponding to these sites, suggesting some structural disorder, associated mainly with the interlayer cations. The spectrum recorded at 83 K showed a distinguishable broadening at the base of the experimental envelope, indicating partial magnetic relaxation (Cardile and Johnston, 1985). This observation was significant because 83 K is apparently a very high temperature for the onset of magnetic ordering in smectites. Ballet and Coey (1982) and Coey *et al.* (1982, 1984) showed that some 2:1 phyllosilicates order magnetically at 4.2 K, but did not observe such ordering in nontronite at this temperature.

To investigate further the nature of the magnetic hyperfine splitting in the Mössbauer spectrum of nontronite and the magnetic ordering which gives rise to it, the Mössbauer spectra of six nontronites from different locations, having different iron contents and interlayer cations, were measured at temperatures of 4.2 and/or 1.3 K.

EXPERIMENTAL

The following nontronites were investigated: untreated sample A.P.I. H33a from Garfield, Washington (GAR); both untreated and Ca-saturated samples from Spokane County, Washington (SPO); a Li-saturated sample from Panamint Valley, California (CAL); and a Na-saturated sample from Koegas, Republic of South Africa (KOE). The location of all samples and their structural formula calculated in a conventional manner are presented in Table 1.

^{57}Fe Mössbauer spectra were recorded using a conventional constant acceleration spectrometer, with a source of ^{57}Co in a rhodium matrix. Most of the spectra were obtained with the sample in a liquid helium bath cryostat at 4.2 and/or 1.3 K, the latter temperature being achieved by pumping on the liquid helium. The spectra were generally recorded over a velocity range of about ±12 mm/s. The velocity scale was calibrated with reference to natural iron foil, with the centroid of the room-temperature iron metal spectrum being used to define zero velocity.

The Mössbauer spectra were computer-fitted with a number of magnetically split sextet or quadrupole-split

[3] Present address: Chemistry Division, Department of Scientific and Industrial Research, Private Bag, Petone, New Zealand.

[4] To whom all correspondence should be addressed.

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Table 1. Location and composition of nontronite samples used in this study.

Location	% Fe (w/w)	Composition per $O_{10}(OH)_2$
Bingham Canyon, Utah (BIN)[1]	20.1	$(Ca_{0.259}K_{0.009})(Si_{3.491}Al_{0.509})(Fe_{1.455}Al_{0.509}Mg_{0.138}Ti_{0.106})$
Garfield, Washington (A.P.I. H33a) (GAR)[1]	25.1	$(Ca_{0.258}K_{0.004})(Si_{3.443}Al_{0.557})(Fe_{1.827}Al_{0.152}Mg_{0.038}Ti_{0.006})$
Spokane County, Washington (SPO)[1]	29.2	$(Ca_{0.232})(Si_{3.700}Al_{0.034}Fe_{0.266})(Fe_{1.924}Mg_{0.029}Mn_{0.004})$
Koegas, Cape Province, Republic of South Africa (KOE)[2]	33.4	$(Si_{3.31}Al_{0.04}Fe_{0.66})(Fe_{2.03}Mg_{0.05})$
Panamint Valley, California (CAL)[2]	35.0	$(Si_{3.10}Al_{0.07}Fe_{0.83})(Fe_{2.02}Mg_{0.11})$

[1] Supplied, with analyses by K. Norrish, Division of Soils, C.S.I.R.O., Adelaide, Australia. Analyses are for Ca-saturated samples.

[2] Supplied by B. A. Goodman, The Macaulay Institute for Soil Research, Aberdeen, Scotland. Composition from Goodman *et al.* (1976).

doublet components with Lorentzian line-shapes, using a χ^2 minimization procedure. The area of the outer and inner pairs of lines of each sextet component were constrained to be in the ratio of 3:1; the area of the middle pair was free to allow for any preferential orientation in the samples. A visual inspection of the fitted envelope in relation to the experimental data was used in conjunction with the value of χ^2 to obtain the best fit.

RESULTS AND DISCUSSION

The Mössbauer spectrum for nontronite sample SPO which indicated broadening at 83 K (Cardile and Johnston, 1985) was recorded here at 4.2 K (Figure 1). The spectrum is well resolved, complex, and magnetically split, indicative of such magnetic ordering. The spectral envelope comprises three well-separated magnetic hyperfine spectra and a definite indication of a fourth component. Because of this complexity, it was not possible to computer-fit the four sextet components simultaneously; however, by fitting a series of combinations of sextets, the values for the isomer shift (δ), quadrupole interaction (Δ), and magnetic hyperfine field (B) (Table 2) could be determined for each hyperfine resonance. The least well-defined component (not shown fitted in Figure 1) is indicated by the line schematic in the figure. By comparing the relative peak areas of these components with the areas of the corresponding Fe^{3+} doublets in the computer-fitted room temperature spectrum (Cardile and Johnston, 1985), the four sextet components were assigned accordingly to the two cis-octahedral, tetrahedral, and interlayer sites (Table 2). The two sextets with the largest magnetic fields of 50.5 T and 44.7 T were assigned to Fe^{3+} magnetically ordered in the two non-equivalent cis-octahedral sites, the sextet with smaller 41.0 T field to Fe^{3+} similarly ordered in the tetrahedral sites, and the smallest 35.9 T field to interlayer Fe^{3+} (Table 2). In addition a small Fe^{3+} component (line schematic, Figure 1) was noted which suggests that the sample is not fully magnetically ordered at 4.2 K.

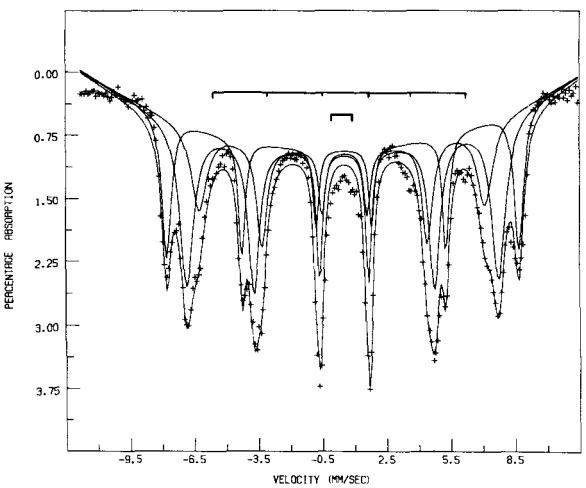

Figure 1. Mössbauer spectrum for nontronite from Spokane County, Washington, at 4.2 K. Peak positions of the interlayer sextet resonance and the central ferric doublet resonance are depicted by the respective schematic line spectra.

Significantly, the respective isomer-shift values, which are usually taken as indicators of covalency, are similar for these two cis-octahedral sites for the 4.2-K magnetically ordered resonances (δ = 0.58, 0.59 mm/s; Table 2) and also the room temperature doublets (δ = 0.376, 0.378 mm/s; Cardile and Johnston, 1985). The magnetic hyperfine splittings, however, which are more sensitive indicators of covalency show that the Fe^{3+} in one of these cis-octahedral sites is slightly more covalently bonded than the other. The sextet with the 50.5-T field arises from the cis-octahedral site in which the iron is least covalently bonded (less covalent cis-octahedral) and is most likely that site which has more tetrahedral Fe^{3+} neighbors than tetrahedral Si^{4+} neighbors (Goodman, 1978). The sextet with the smaller field of 44.7 T suggests that the iron in this cis-octahedral site is slightly more covalently bonded (more covalent cis-octahedral) and therefore has a greater proportion of tetrahedral Si^{4+} neighbors than Fe^{3+} neighbors.

The interlayer cations can also influence the bonding in these octahedral sites (Johnston and Cardile, 1985; Cardile and Johnston, 1985). The magnetic field for the tetrahedral Fe^{3+} is smaller than that for octahedral Fe^{3+} (Table 2), which is also indicative of a greater

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Table 2. Mössbauer parameters for the various nontronite samples.

Sample[1] and temperature	Less covalent cis-octahedral B (T)	δ (mm/s)	Δ (mm/s)	Area (%)	More covalent cis-octahedral B (T)	δ (mm/s)	Δ (mm/s)	Area (%)
Untreated SPO 4.2 K	50.5	0.58	−0.06	24	44.7	0.59	−0.07	45
Untreated SPO[4] Room temperature	—	0.376	0.665	28	—	0.378	0.307	60
Ca-saturated SPO 4.2 K	50.8	0.57	−0.08	29.7	44.6	0.59	−0.07	44.2
Untreated BIN 1.3 K	50.4	0.59	0.00	40.0	44.6	0.58	0.02	23.7
Untreated GAR 1.3 K	50.3	0.58	−0.02	26.5	46.5	0.58	−0.04	53.5
Na-saturated KOE[5] 4.2 K	50.4	—	—	~15	41.6	—	—	~35
Li-saturated CAL 1.3 K	52.3	0.60	−0.03	37.5	47.5	0.60	−0.02	33.8

Sample[1] and temperature	Tetrahedral B (T)	δ (mm/s)	Δ (mm/s)	Area (%)	Interlayer, Fe^{2+} doublet[2] B (T)	δ (mm/s)	Δ (mm/s)	Area (%)	Nonmagnetic[3] component Area (%)
Untreated SPO 4.2K	41.0	0.58	−0.13	28	35.9	0.35	−0.16	3	—
Untreated SPO[4] Room temperature	—	0.147	0.546	8	—	0.42	1.34	4.2	—
Ca-saturated SPO 4.2 K	41.0	0.58	−0.16	21.8	—	—	—	—	4.3
Untreated BIN 1.3 K	40.8	0.60	0.13	30.9	—	1.41	2.62	5.4	—
Untreated GAR 1.3 K	41.5	0.60	−0.01	17.3	—	—	—	—	2.7
Na-saturated KOE[5] 4.2 K	34.7	—	—	~20	—	—	—	—	~30
Li-saturated CAL 1.3 K	42.1	0.60	−0.06	21.2	—	—	—	—	7.5

Approximate errors are ±0.5 T for the hyperfine fields, ±0.05 mm/s for the isomer shifts, ±0.02 mm/s for the quadrupole splitting, and ±0.5% for the area.

[1] Source of samples listed in Table 1.

[2] Nonmagnetic Fe^{2+} component in the BIN nontronite spectrum (Figure 2).

[3] A nonmagnetic Fe^{3+} component, usually a doublet, was also included in the fitting procedure and can be seen in Figure 3. Because this component includes unresolved hyperfine splitting and is generally of low intensity, the fitted parameters are unlikely to be meaningful or reliable and only the area of this component is given.

[4] From Cardile and Johnston (1985).

[5] The spectrum of the Na-saturated Koegas nontronite proved difficult to fit with all the sextet components simultaneously. Values of the hyperfine fields quoted are from different fits and are only approximate. Other hyperfine parameters could not be obtained reliably and only approximate areas are given in the table.

degree of covalency for the tetrahedral Fe^{3+}, as suggested by the δ values for the room temperature spectra (Cardile and Johnston, 1985). The δ value for this magnetically split tetrahedral Fe^{3+} resonance (Table 2) is, however, anomalously high and simply results from the inability to computer-fit four sextets satisfactorily to the experimental spectral envelope, because in a magnetic hyperfine spectrum small changes in the positions of the individual sextet peaks can manifest a significant change in the resulting δ value. The magnetic field splitting is a more sensitive and correct parameter by which to measure the extent of covalency than is the isomer shift. The interlayer Fe^{3+} resonance has the smallest magnetic field, which could be due partly to the extent of covalency and also to the relatively low iron content of these sites.

It therefore seems likely that sufficient Fe^{3+} is located in the different sites to enable the magnetic exchange interactions to extend throughout the structure for each type of site. These interactions presumably couple collectively through all the sites, and a minimum iron content of each may be necessary to observe magnetic ordering.

The spectrum for the Ca-saturated SPO nontronite

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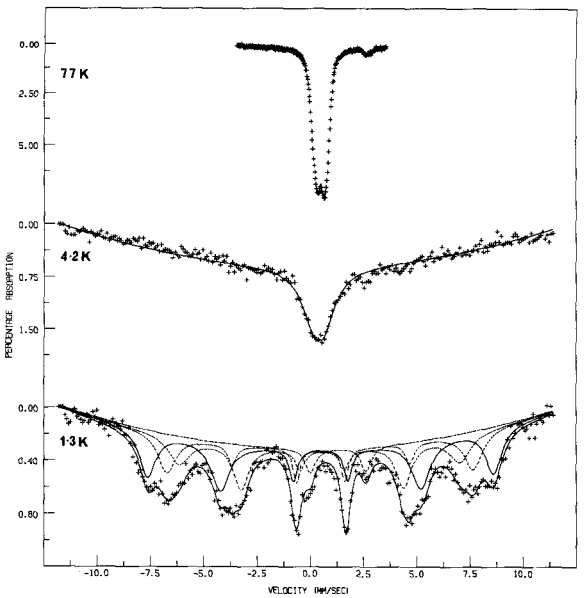

Figure 2. Mössbauer spectra of untreated Bingham Canyon, Utah, nontronite at 77, 4.2, and 1.3 K.

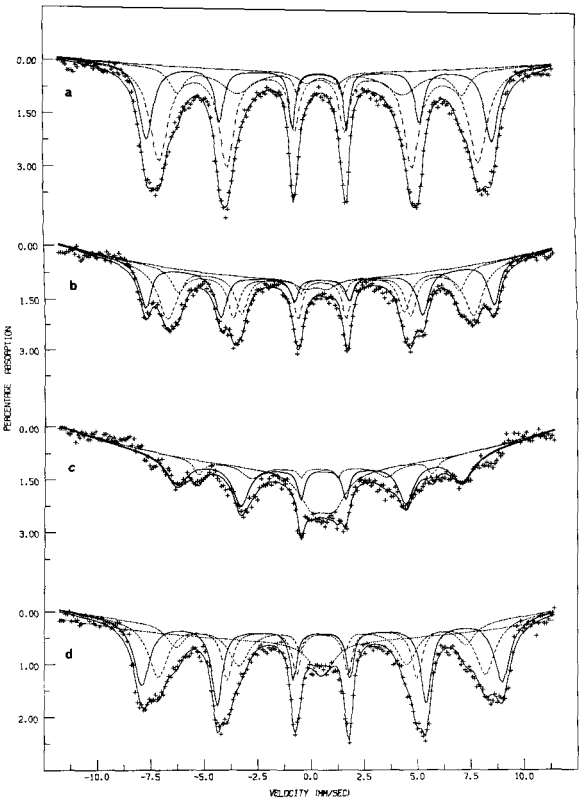

Figure 3. Mössbauer spectra of: (a) untreated nontronite from Garfield, Washington, at 1.3 K; (b) Ca-saturated nontronite from Spokane County, Washington, at 4.2 K; (c) Na-saturated nontronite from Koegas, Cape Province, Republic of South Africa, at 4.2 K; and (d) Li-saturated nontronite from Panamint Valley, California, at 1.3 K.

recorded at 4.2 K (Figure 3b) shows similar magnetic hyperfine resonances for the two octahedral and the tetrahedral sites (Table 2). No interlayer component resonance is present, consistent with the interlayer Fe^{3+} being displaced by Ca-saturation. These data show that the presence of interlayer iron is not a prerequisite for magnetic ordering and that the magnetic ordering of the nontronite layer structure is essentially two dimensional. The small doublet component is present here also, suggesting that this Ca-saturated SPO nontronite is also not completely magnetically ordered at 4.2 K.

The Mössbauer spectra of untreated Bingham Canyon nontronite at 77, 4.2, and 1.3 K are shown in Figure 2, together with the computer fits discussed below. The 77-K spectrum shows a narrow doublet, similar to that observed at room temperature, and a small interlayer ferrous component. The 4.2-K spectrum shows a broad resonance, fitted as a broadened doublet, which results from unresolved magnetic hyperfine splitting and is indicative of the onset of magnetic ordering. The ferrous component, observable in the room-temperature spectrum (Cardile and Johnston, 1985), is also evident at this temperature, but it is partially obscured by the broad band of magnetic absorption. At 1.3 K, the spectrum shows well-resolved magnetic hyperfine splitting with a complex envelope consisting predominantly of three, overlapping magnetic hyperfine spectra. The Néel temperature for this nontronite is between 4.2 and 1.3 K and is therefore very low.

The above observations may mean that the magnetic hyperfine splitting depicted by the Mössbauer spectra at these low temperatures could have arisen either wholly or in part from superparamagnetism, rather than collective long-range magnetic interaction. These three closely overlapping sextets were readily resolved by the computer-fitting; the resulting parameters are given in Table 2. The three magnetic components were similarly assigned to the two cis-octahedral and one tetrahedral iron sites. The δ values for the two cis-octahedral resonances are similar but the different magnetic hyperfine-splitting values show here also that the Fe^{3+} in one of the cis-octahedral sites is more covalently bonded than in the other such site (Table 2).

The low-intensity Fe^{2+} quadrupole-split doublet (Figure 2) shows that Fe^{2+} in the interlayer does not order magnetically even at 1.3 K, whereas interlayer Fe^{3+} can show magnetic ordering at 4.2 K, as demonstrated by the SPO nontronite. In addition, a small area of misfit at about zero velocity is present, consistent with the presence of a small fraction of Fe^{3+} doublet and suggesting that a slightly lower temperature may be required to observe complete magnetic ordering.

The suite of spectra shown in Figure 2 are typical of those measured as a function of temperature for all the

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nontronites studied here. For all samples the doublet spectra observed at higher temperatures gave way to spectra showing the effects of magnetic hyperfine interactions when the temperature was lowered to 4.2 K and below. At a sufficiently low temperature all samples gave spectra with well-resolved magnetic hyperfine splitting. The various samples showed different temperatures for the onset of magnetic ordering and also differences in the details of the observed magnetically split spectra.

The Mössbauer spectra of all the samples investigated, obtained at a temperature sufficiently low that they exhibited well-resolved magnetic hyperfine splitting, are presented in Figure 3. For most samples, the computer-fitting resolved three sextet components with similar isomer shifts and quadrupole splittings and with magnetic hyperfine fields at about 51, 46, and 41 T. The detailed Mössbauer parameters from these computer fits are given in Table 2. The hyperfine fields are consistent with magnetically ordered Fe^{3+} in three sites with differing degrees of covalency, as discussed above.

The 4.2-K spectrum of untreated Garfield (GAR) nontronite shows a Fe^{3+} doublet similar to that of the higher temperature spectra, whereas at 1.3 K (Figure 3a) the spectrum consists of a complex envelope of three overlapping magnetic sextets similar to the spectrum of the untreated BIN nontronite at this temperature. A small central doublet is also present which here also indicates the presence of a few iron atoms that show no magnetic hyperfine splitting. The parameters of this component have considerable uncertainty, but are consistent with those of the interlayer Fe^{3+} observed in the room-temperature spectrum (Johnston and Cardile, 1985). Also, the room-temperature spectra show that this untreated GAR nontronite contains 1% of the iron as Fe^{3+} in the interlayer (Johnston and Cardile, 1985), whereas the untreated SPO nontronite contains 4.2% (Cardile and Johnston, 1985). Thus, the GAR nontronite appears to contain insufficient interlayer Fe^{3+} to order magnetically, but where the Fe^{3+} content of the interlayer is greater, such as in the SPO nontronite, magnetic ordering can be observed for interlayer Fe^{3+} iron at 4.2 K. These data indicate that the iron in the composite layers (tetrahedral and octahedral) in the GAR nontronite is fully magnetically ordered; they also provide further evidence that the interlayer iron is not a prerequisite for such magnetic ordering.

The Mössbauer spectrum of the Na-saturated Koegas nontronite at 4.2 K (Figure 3c) suggests an intermediate stage of magnetic ordering. The somewhat smaller values of the hyperfine fields indicate that the temperature was not sufficiently far below the ordering temperature for the magnetization to have become saturated. The spectrum was therefore less well resolved than the others, and difficulties were encountered with the computer-fitting procedure. The spectrum shows three magnetically-split components, but a satisfactory fit comprising three overlapping sextets could not be obtained. Therefore, several different fits with two magnetically split components were carried out, one of which is shown in Figure 3c. Using this procedure, approximate values of the three hyperfine fields were obtained (Table 2).

The Li-saturated Panamint Valley, California (CAL) nontronite shows, in its 1.3-K spectrum (Figure 3d), behavior more or less intermediate between that of the Na-saturated Koegas nontronite at 4.2 K, discussed above, and that observed for the rest of the samples at the lowest temperatures. This spectrum was also computer-fitted with three magnetic sextet components and a broad central singlet corresponding to the iron atoms for which the magnetic hyperfine splitting is unresolved. As with the other samples, the three sextets correspond to Fe^{3+} in the two cis-octahedral and the tetrahedral sites.

SUMMARY

In summary, the Mössbauer data show that for the nontronite samples studied here, magnetic ordering always occurs at a sufficiently low temperature that depends in an extremely complicated way on such factors as the average separation of the magnetic ions, the nature of the intervening nonmagnetic ions, and the structural arrangement. The dimensionality of the magnetic system is also important. An isotropic, one-dimensional system with only nearest-neighbor interactions cannot order at any finite temperature, whereas a two-dimensional layer structure will order, but at a much lower temperature than an equivalent three-dimensional system (Gupta *et al.,* 1980). The relatively low ordering temperatures of the nontronite samples is therefore consistent with the pseudo two-dimensional layer structure of this mineral.

The above discussions indicate that magnetic ordering does not necessarily require the presence of a magnetic ion in the interlayer to provide three-dimensional magnetic pathways. Indeed, the data suggest that the ordering is not affected by the removal of interlayer iron by saturation treatments. In general, all other things being equal, a higher concentration of magnetic ions will give rise to a higher ordering temperature; however, the positioning of the magnetic ions relative to each other, the nature of the intervening ions, and the degree of covalency are also important factors. It is clear from the present data that simple correlations do not exist between the elemental composition of the samples and their observed ordering temperature. Furthermore, the relaxation processes which affect the observation of the magnetic ordering in the Mössbauer spectrum depend on the detailed electronic structure of each particular site.

The nature of the magnetic ordering and the resulting magnetic structure cannot be deduced from the Möss-

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bauer spectra alone. In most iron-containing minerals the magnetic structure is essentially either antiferromagnetic or ferrimagnetic. For the samples studied here, the magnetic structure is probably highly complex, containing different sets of sublattices that correspond to the different iron sites; further complications arise from structural disorder and the variability in the next nearest-neighbor environments about the Fe^{3+} ions (Johnston and Cardile, 1985; Cardile and Johnston, 1985). As mentioned above, some superparamagnetic contribution is also likely.

Further information on the magnetic structure could have been obtained by Mössbauer measurements in an applied magnetic field or by neutron diffraction measurements, both of which ideally require the use of single crystals. Such single crystals of nontronite were, of course, not available. Also, equipment was not available to determine the temperature dependence of the magnetic susceptibility of the samples in the 1.3–4.2-K temperature range, at which the Mössbauer spectra were determined.

Completely ionic Fe^{3+} exhibits a saturated hyperfine field of more than 60 T, which is reduced by the effects of covalency. The observed hyperfine field values of about 51, 46, and 41 T are entirely consistent with the increasing degree of covalency for the iron atom from the Fe^{3+} in cis-octahedral sites with more tetrahedral Fe^{3+} neighbors, to the Fe^{3+} in cis-octahedral sites with more tetrahedral Si^{4+} neighbors, to the Fe^{3+} in the tetrahedral sites.

An important conclusion from this work is that considerable extra information can be obtained from very low temperature Mössbauer spectra. The results presented here corroborate earlier room-temperature Mössbauer studies of nontronite and indeed confirm the presence of iron in the two non-equivalent cis-octahedral, the tetrahedral, and the interlayer sites. Also, the observation of magnetic ordering provides an additional approach for elucidating the differences between the various iron sites within one sample and between samples with different compositions.

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