Critical behavior in the heat capacity of $Fe[S_2CN(C_2H_5)_2]_2Cl$: Evidence for chiral universality

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The heat capacity of $Fe[S_2CN(C_2H_5)_2]_2Cl$ has been measured in the region of the 2.5 K ferromagnetic transition with much better temperature resolution than in any previous set of data on this material. Analysis of data within 0.1 in reduced temperature $t=(T-T_c)/T_c$ of the transition, both above and below T_c, leads to a value $T_c=2.442_4$ K. This agrees well enough with the previously established value 2.457 K, considering likely uncertainties in absolute temperature among different instruments. Simple power law analysis of the magnetic heat capacity above T_c, i.e., $C(\mathrm{mag})\propto t^{-\alpha}$, yields $\alpha\simeq 0.22$ for reduced temperatures above 0.01. A few data at temperatures yet closer to T_c suggest a larger value for α, and possible crossover; but the probability is substantial that these are transition rounding effects. A global analysis of data above and below the transition, and allowing for additional regular terms in the heat capacity, leads to the T_c given above and $\alpha=0.244\pm 0.005$, along with other parameters. The α is more consistent with the three-dimensional (3D) chiral Heisenberg model value 0.24 ± 0.08 than with the 3D chiral *XY* model value 0.34 ± 0.06. The leading amplitude ratio is $A^+/A^-=0.325\pm 0.005$, also consistent only with chiral model results.

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I. INTRODUCTION

The pentacoordinate, insulating Fe^{3+} compound $Fe[S_2CN(C_2H_5)_2]_2Cl$ was the first molecular ferromagnet to be studied.[1–3] The unusual coordination geometry produces a crystal field ground term $^4A_2$, in which a zero-field splitting of several K develops between $|\pm\frac{3}{2}\rangle$ and $|\pm\frac{1}{2}\rangle$ Kramers doublets. This leads in turn to substantial anisotropy in the single crystal ac magnetic susceptibility,[4] especially at temperatures comparable to the zero-field splitting. One axis, monoclinic [101], displays a ferromagnetic divergence toward a demagnetization limited value;[5] much smaller nondiverging susceptibilities occur along the other two (orthogonal) principal axes.[4]

The critical behavior of $Fe[S_2CN(C_2H_5)_2]_2Cl$ near the 2.457 K ferromagnetic transition has been studied by various methods. Analysis of the initial ac susceptibility yielded a value for the corresponding critical exponent $\gamma=1.16_5\pm 0.03$, while analysis of NMR measurements[6] of relative sublattice magnetization below T_c yielded $\beta=0.24_5\pm 0.02$, in each case with correction to scaling terms included.[7] Scaling analysis of dc magnetization isotherms yielded an independent pair of exponents $\beta=0.24\pm 0.01$ and $\delta=5.65\pm 0.15$ (the latter determining behavior on the critical isotherm).[8] Values of γ, β, and δ satisfy the scaling relation $(\delta-1)\beta=\gamma$ within experimental uncertainty. Yet each differs substantially from accepted values[9] for standard universality classes, in particular from three-dimensional (3D) Ising model values which the macroscopic anisotropy suggests.

The values of γ and β are, however, fairly close to those obtained theoretically by Kawamura for magnets with $Z_2\times S_1$ symmetry, i.e., an Ising chiral symmetry combined with *XY* rotational symmetry.[10] Symmetry of this type may characterize a canted ferromagnet with Ising anisotropy, which $Fe[S_2CN(C_2H_5)_2]_2Cl$ is thought to be.[4,11] Additional neutron work is planned in order to establish the ordered spin arrangement more definitively than it is known at present. The most up to date values of critical exponents for the 3D (stacked triangular) version of the $Z_2\times S_1$ model are $\gamma=1.13\pm 0.05$, $\beta=0.25_3\pm 0.01$, $\alpha=0.34\pm 0.06$ (for the heat capacity), and $\nu=0.54\pm 0.02$ (for the correlation length).[12] The scaling relation given earlier yields from these $\delta=5.47\pm 0.27$, quite close to the experimental value for $Fe[S_2CN(C_2H_5)_2]_2Cl$. Up to now only limited heat capacity data for this compound near T_c have been available;[13] these were analyzed by one of us[7] to yield a provisional estimate $\alpha=0.38\pm 0.06$, also agreeing reasonably well with the $Z_2\times S_1$ model prediction.

It is very desirable to have a denser set of heat capacity data for $Fe[S_2CN(C_2H_5)_2]_2Cl$ near the critical point than those of Ref. 13. This is particularly important for this material because the large canting angles of two ferromagnetic sublattices creates an unusual situation with the respect to the interpretation of susceptibility and magnetization data. The zero field heat capacity represents a thermodynamic quantity which should not be directly sensitive to a microscopic detail like relative spin alignment. We report here the collection and analysis of heat capacity data on $Fe[S_2CN(C_2H_5)_2]_2Cl$ which allows firmer conclusions to be drawn than previously.

II. EXPERIMENTAL

Heat capacity data between 0.615 and 19.99 K, were collected at RIKEN based on a relaxation method using an Oxford Instruments MagLab[HC] microcalorimeter. Three warming/cooling cycles around a given temperature are av-

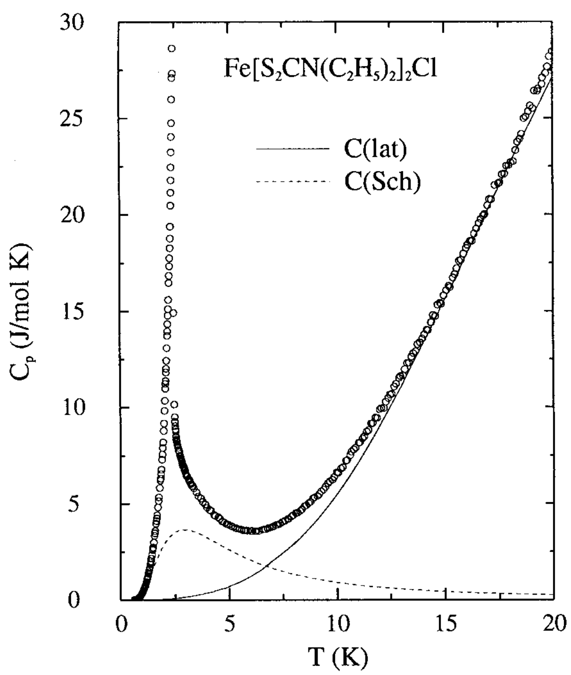

FIG. 1. Molar heat capacity of $Fe[S_2CN(C_2H_5)_2]_2Cl$ as a function of temperature. Also shown are the lattice and Schottky contributions, evaluated as described in the text.

eraged in obtaining the final reported values of heat capacity and temperature. The magnitude of the temperature rise and fall in each cycle is about 0.2% of the measuring temperature, or around 0.005 K near the 2.5 K magnetic transition of the present system. The real precision of relative temperatures in the data set is believed to be some modest fraction of this. The absolute accuracy of the temperature is given by the manufacturer as 2%, hence about 0.05 K near the transition. The sample was a small, platelike single crystal of $Fe[S_2CN(C_2H_5)_2]_2Cl$, of mass 2.00 mg. It was mounted on a sapphire substrate isolated from but connected to the remainder of the apparatus by very thin tungsten wires. For purposes of thermal contact with the substrate, a small amount of Wakefield's compound was employed. Correction is made for the very small contribution of this bonding agent to the total measured heat capacity.

III. RESULTS AND ANALYSIS

The heat capacity of $Fe[S_2CN(C_2H_5)_2]_2Cl$ appears in Fig. 1. Our $C_p(T)$ values generally agree within 1–2 % with those of Arai *et al.*[13] in the 1–20 K range. This is excellent agreement given the imperfect correspondence between the two thermometers used in these studies. A difference in the location of the λ anomaly (giving a first order estimate of T_c) exists: the peak in $C_p(T)$ is at 2.412 K in the Ref. 13 data but is at 2.433_7 K according to the present results. The absolute accuracy of temperatures in the Ref. 13 data is not better than a few 0.01 K, from both thermometric uncertainties and uncertainties resulting from the measurement method (T drift). Similar comments apply to the present data. Hence the apparent difference in T_c of 0.022 K is not unexpected. It is probable that the true T_c is closer to that given in the introduction, 2.457 K within a few 0.001 K, because such emerged from analysis of critical region ac and dc susceptibility and magnetization data from two separate apparatuses with different thermometers. In the present analysis a T_c value consistent with the thermometric characteristics of these heat capacity data will, perforce, emerge.

Subtraction of lattice and Schottky contributions from the total measured heat capacity was made in the same way as previously.[4,7] The agreement of the present higher temperature data with those of Ref. 13 is quite good (1% level with random deviations) and so the same lattice approximation at lower temperatures is employed as before: $C(\text{lat})=5.661\times10^{-3}\ \text{J K}^{-4}\,\text{mol}^{-1}\,T^3$. This contribution below 3 K is less than 2% of $C(\text{obs})$ and generally much less. The T variation of $C(\text{lat})$ between 1.9 and 3 K is more than two orders of magnitude less than the variation of $C(\text{obs})$.

Somewhat larger is the Schottky contribution to the heat capacity associated with the zero-field splitting of the ${}^4A_2$ ground state; the latter has been estimated as 7.01 K, from analysis of single crystal susceptibility data.[4] Between 1.9 and 3 K $C(\text{Sch})$ varies from about 2.8 to 3.7 J/mol K. However, the variations in this contribution in the fitted regions below and above T_c is approximately two orders of magnitude smaller than the variation in $C(\text{obs})$. Thus for this contribution, as for the lattice heat capacity, although correction is made, the effect of such correction is rather small.

As a first step in the analysis, data within 0.10 in reduced temperature $t=(T-T_c)/T_c$ on either side of the apparent T_c of 2.433_7 K [position of maximum $C(\text{obs})$] are considered. Near T_c the temperature dependence of the magnetic specific heat $[C(\text{mag})=C(\text{obs})-C(\text{lat})-C(\text{Sch})]$ is expected to follow the form:

$$C(\text{mag})=(A/\alpha)t^{-\alpha}+B \tag{1}$$

as $T\rightarrow T_c$. Contributions to the parameter B can arise from the phase transition itself, and theoretical predictions are available in some cases.[14,15] The parameter A can take different values above and below T_c; the exponent α should have the same value in these regimes, and T_c itself should not depend upon whether it is approached from above or below. Although different views have been expressed regarding B, the dominant one is that it should be the same above and below T_c.

Initially, we will assume $B=0$. A plot of $\log_{10}C(\text{mag})$ vs $\log_{10}t$ should then be linear with slope$=-\alpha$. Such a plot constructed for data above T_c and assuming $T_c=2.433_7$ K shows acceptable linearity between 2.467_7 K ($t=0.0140$) and 2.685_0 K ($t=0.103$) with $\alpha=0.248\pm0.005$ (standard deviation) and correlation coefficient $r=0.9958$. Restricting the fit to a significantly smaller $t_{\text{max}}=0.061$ gives a very slightly worse fit and a slightly larger α. Also apparent is a large change in slope for the two temperatures closest to T_c, 2.456_4 K ($t=0.00933$) and 2.445_1 K ($t=0.00468$). A fit to these data along with that at 2.467_7 K yields a line with $\alpha=1.18\pm0.05$ and $r=0.9982$. The α value is unphysical, no universality class permitting such a result.

Transition rounding effects tend to shift the apparent T_c [at maximum $C(\text{obs})$] to slightly lower temperature than the

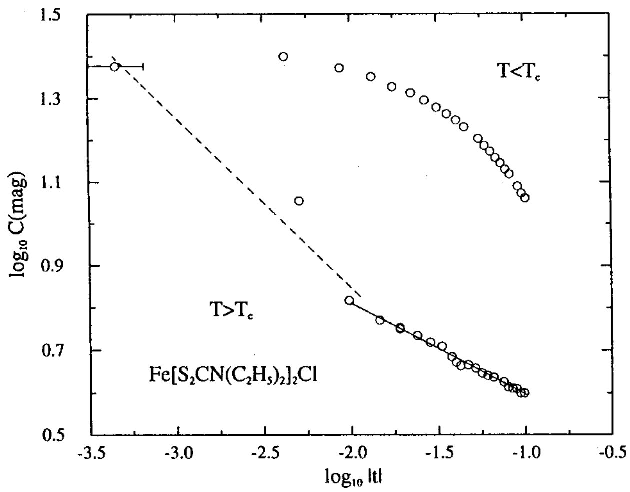

FIG. 2. Molar magnetic heat capacity of $Fe[S_2CN(C_2H_5)_2]_2Cl$ vs reduced temperature ($T_c=2.444_0$ K) in a base-10 logarithmic representation, for data above and below T_c. Linear least squares fits (lines) also appear, as described in the text; the more questionable fit to lower t data is shown dashed.

true T_c.[16] Since T_c is also a parameter which is appropriately varied in fitting critical region data, T_c was allowed to vary here. It was readily established that a small increase in T_c permitted a slightly better fit to be obtained in the 2.467_7 to 2.685_0 K range: $r=0.9973$ with $\alpha=0.217\pm0.004$ for $T_c=2.444_0$ K. Figure 2 shows a log-log plot corresponding to this value of T_c. Error bars along the vertical are within symbol size [precision in $C(\mathrm{obs})$ is at the 1% level], while those along the horizontal (corresponding to ±0.0005 K) are beyond symbol size only for the $T>T_c$ datum closest to T_c.

Two observations concerning Fig. 2 immediately suggest themselves. First, the $T<T_c$ regime appears qualitatively different, with curvature throughout. Second, the $t<0.01$ regime for $T>T_c$ continues to appear distinct; with the T_c estimate of 2.444_0 K the magnitude of the slope in this region is 0.41 ± 0.07. One can even imagine a crossover scenario, since the 3D $Z_2\times S_1$ model has $\alpha=0.34\pm0.06$ while the 3D chiral Heisenberg model has $\alpha=0.24\pm0.08$, which correspond fairly well to the |slope| values in the $t<0.01$ and $t\geq0.01$ regions, respectively. [The chiral Heisenberg model, with symmetry SO(3), has for its other exponents $\gamma=1.17\pm0.05$, $\beta=0.30\pm0.02$, and $\nu=0.59\pm0.02$, with consequent $\delta=4.90\pm0.40$.[12]]

However, the objection can be made that what is seen inside $t=0.01$ are transition rounding effects. These arise from sample inhomogeneities and often appear in heat capacity data near $t=0.001$.[16,17] In a specific system and sample they can occur at somewhat larger t. Such effects can yield an apparent increase in the α value as t decreases, though the opposite is probably more often observed. There are also reasons to believe that any crossover would occur over a wider $\log_{10}t$ interval than is suggested by the data here. Because of the low T_c in our material, and the associated difficulty of obtaining numerous data inside $t=0.01$, it is also safer not to rely on the apparent implications of only a few data points.

The constant B was also allowed to vary, a range of positive and negative values being tried. The quality of the resulting fits to $T>T_c$ data was not very sensitive to the choice of B, though some small systematic worsening occurred as B became quite positive or quite negative. As B became more negative α became smaller ($\alpha=0.155$ for $B=-2.0$), and as B became more positive α became larger ($\alpha=0.365$ for $B=2.0$). There is no basis for preferring any $B<0$ choice here since the fit is marginally worse than for $B=0$. The fit with $B=1.0$, with $\alpha=0.272$, was very slightly best in quality. No choice of B proved capable of changing the qualitative nature (nonlinear) of the $T<T_c$ data in a log-log representation.

A general form for the heat capacity above and below T_c and in the vicinity of a magnetic phase transition is[17]

$$C_p=(A^{\pm}/\alpha^{\pm})|t|^{-\alpha\pm}+B^{\pm}+E^{\pm}t, \tag{2}$$

where the + and − labels refer to the regimes above and below T_c, respectively, and where as before $t=(T-T_c)/T_c$. The parameters A^+ and A^- for the leading singularities on either side of T_c will generally be different; theoretical values have been obtained for certain models, and the ratio of these amplitudes is known for the standard models.[18,19] The parameter B can include nonsingular contributions from the phase transition as well as background contributions; it has been argued that the same value for B^+ and B^- should occur.[18] The last term represents T-dependent regular contributions (which include, in general, lattice and electronic contributions, though these have been explicitly corrected for in this work); to ensure continuity through the transition the constraint $E^+=E^-$ is usually imposed. Scaling theory requires that $\alpha^+=\alpha^-$, though in some analyses of heat capacity data for specific systems different values of α above the below T_c have been reported.[20,21]

Corrections to scaling (CTS) are sometimes incorporated in the analysis of heat capacity data, corresponding to a factor $(1+D^{\pm}|t|^x)$ associated with the first term in Eq. (2).[17] It is usually found that the effects of including CTS on the critical exponent and the leading amplitude ratio A^+/A^- are quite small. In our previous analyses of susceptibility and magnetization data on $Fe[S_2CN(C_2H_5)_2]_2Cl$ it was determined that CTS effects on the other parameters were small. Additional parameters also are introduced with CTS, complicating the overall analysis, yet for chiral models especially there is no theoretical guidance concerning preferred values of D and x. We will present the results obtained without including corrections to scaling and then mention the hardly modified results on including it.

Data above and below T_c were fit simultaneously, employing $|t|$ as high as 0.1; on the assumption that data inside $|t|=0.01$ are influenced by transition rounding effects, these were not included in the fit. The constraints $B^+=B^-$ and $E^+=E^-$ were imposed. In applying Eq. (2) for the total heat capacity the previously described lattice and Schottky heat capacities were introduced as additional contributions on the right hand side. This should provide a more accurate accounting for such background effects, not relying (somewhat artificially) on the B and Et terms to incorporate all nonsingular contributions. The initial value of T_c was 2.444_0 K,

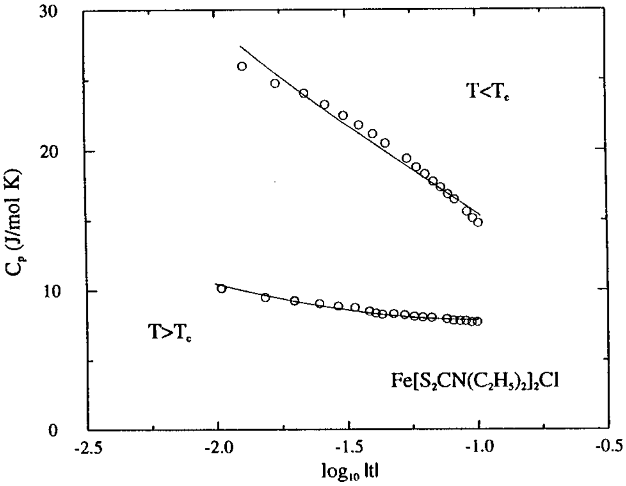

FIG. 3. Molar heat capacity of $Fe[S_2CN(C_2H_5)_2]_2Cl$ vs reduced temperature ($T_c=2.442_4$ K) above and below T_c, and best fit (curves) to data according to Eq. (2) as described in the text. Lattice and Schottky contributions are included in the curves shown.

emerging from linear fits described earlier; this varied slightly in nonlinear fits according to Eq. (2). The $|t|=0.1$ limit was chosen as a plausible bound to the critical region and is similar to values adopted in other heat capacity analyses. Increasing or decreasing this limit by up to a factor of two led to only minor variations in the fitted parameters, with some indication that $|t|$ should not be taken larger than 0.1.

The best fit obtained appears in Fig. 3. Parameter values are $T_c=2.442_4\pm0.002$ K, $\alpha=0.244\pm0.005$, $A^+=0.721\pm0.010$, $A^-=2.22\pm0.02$, $B=-2.35\pm0.10$, and $E=13.39\pm0.50$, with r.m.s. deviation 2.15%. In arriving at this many different sets of initial parameter values were tried in a general purpose nonlinear least squares fitting program. The indicated uncertainties represent parameter variations which yield essentially comparable quality agreement between observed and calculated values overall. A more conservative but still reasonable set of uncertainties would correspond to approximately doubling each of the foregoing; it is not believed that yet larger uncertainties are justified.

The effect of including a CTS term was investigated. Rather than treat the correction exponent x as a fitting variable, the value 0.50 was used. This is quite close to the slightly different theoretical values for various standard models, and will be considered sufficiently accurate especially since x has not been obtained specifically for chiral systems. It was found that in order to obtain a very slight improvement to the fit without CTS, D^+ and D^- needed to be of opposite sign and similar magnitude; for simplicity $D=D^+=-D^-$ was set. A best fit had all the parameters except E (which was 0.90 less than in the fit without CTS given above) shifted only within the uncertainty ranges indicated, with $D=0.030$ and a 2.13% r.m.s. deviation. Any improvement is inconsequential, an outcome often found in previous analyses of heat capacity data.[17,22,23]

Finally, we will mention, without displaying, the outcome on not excluding data inside $|t|=0.01$, assumed in the foregoing to be too influenced by transition rounding. Other constraints remain the same, and corrections to scaling are neglected. Not surprisingly a somewhat poorer quality fit results on extending to data closer to T_c. The best fit had a 3.66% r.m.s. deviation and a modestly larger value of T_c, 2.450_0 K, than before, with a slightly smaller $\alpha=0.227$ as well. However, the other parameters differed relatively little from values given earlier for the best restricted range fit, and were $A^+=0.766$, $A^-=2.27$, $B=-3.00$, and $E=13.61$. The minor variations in A^+ and A^-, in particular, means that the important amplitude ratio (discussed in the next section) is rather robust with respect to fitting range variations.

IV. DISCUSSION

The various fitting attempts give a T_c within 0.01 K of 2.442 K. Considering sources of potential error in absolute temperatures, this is good agreement with the result we take as well established, $T_c=2.457$ K. The heat capacity exponent α is found to be near 0.24, much higher than any value occurring for the standard universality classes (3D Heisenberg: -0.121, 3D *XY*: -0.01, 3D Ising: 0.110, 2D Ising: 0)[9] but near 3D chiral model values, especially for the Heisenberg case.[12]

The ratio A^+/A^- from the simultaneous $T>T_c$, $T<T_c$ fit is an important quantity, as theoretical predictions for this amplitude ratio exist for most models. From the values corresponding to the solid curve in Fig. 3 this ratio is 0.325 ± 0.005. Theoretical values are 0.53 (3D Ising), 1.03 (3D *XY*), and 1.52 (3D Heisenberg), with uncertainties from 4 to 11% judging from some different published values.[18,19] For the two chiral models theoretical estimates are 0.54 ± 0.2 (Heisenberg) and 0.36 ± 0.2 (*XY*).[12] The uncertainties are very large, and we simply observe that our experimental value is closer to the *XY* case. For the standard models only the Ising ratio is remotely near our observation, and the modest experimental uncertainty rules out this correspondence. Hence, not only the exponent value but also the amplitude ratio decidedly favors a chiral model.

A parameter which can be informative is $P=(1-A^+/A^-)/\alpha$.[18] According to ϵ-expansion expressions P assumes the values 4.92, 5.30, and 5.92 for the 3D Ising, *XY* and Heisenberg models, respectively. The P emerging from the present experimental results is 2.77 ± 0.07. This is far below values for standard models, and would clearly be so even if substantially larger uncertainties obtained for α, A^+, and A^- than those previously indicated. Kawamura[12] recently surveyed existing experimental work on chiral model systems, especially those of layered triangular lattice type (though 3D); $CsMnBr_3$ has been particularly well studied.[22,23] Experimental α range from 0.34 to 0.40, with uncertainties of the order 0.06 to 0.09. Similarly, A^+/A^- values range from 0.19 to 0.32, with uncertainties of the order 0.08 to 0.20. Consequent P values range from 1.70 to 2.08, with lower limit uncertainties of order 0.5 and upper of order 0.8. Our $P=2.77$ is somewhat higher than previous experimental values, but consistent with these given the uncertainties.

If one substitutes the previously determined experimental values for β and γ, and their uncertainties, into the scaling relation $\alpha=2-2\beta-\gamma$, there results $\alpha=0.34_5\pm0.05$. The present best estimate of α is 0.244 ± 0.005, below this value by only twice the theoretical uncertainty. Both the original susceptibility measurements and analysis,[4] and the later and somewhat preliminary neutron work,[11] suggested that a canted ferromagnetic sublattice ordered state arrangement occurs in $Fe[S_2CN(C_2H_5)_2]_2Cl$. Noncollinear ordered spin arrangements offer the possibility of chiral degeneracy, and it has been suggested that a canted Ising ferromagnet might belong to the $Z_2\times S_1$ universality class.[10] However, the ordered spin arrangement is not sufficiently well established to say whether or not the symmetry requirements are definitely met. More refined neutron work, on deuterated samples, will soon be pursued.

Neutron data may also shed light on certain ambiguities which exist in the present results. Superficially, the curvature of $T<T_c$ data comport best with a negative α^-. Such a possibility must be rejected in light of scaling requirements and the implications of $T>T_c$ data. One may speculate that perhaps some sort of crossover occurs throughout the t range of the present data (and that data much closer to T_c are needed), in order to rationalize the small but nontrivial deviations in the fit for $T>T_c$ data, and the larger deviations for $T<T_c$ data, in Fig. 3. However, it is difficult to suggest what the crossover scenario might be; moreover, there were no indications of crossover effects in comparable t-range data of earlier susceptibility and magnetization measurements. It does seem clear from the present heat capacity measurements and analysis that the situation does not correspond to one of the standard universality classes. The present system is of special significance, pending the results of more detailed neutron studies, as the first example of a chiral model system which is not of the familiar layered triangular lattice type.

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