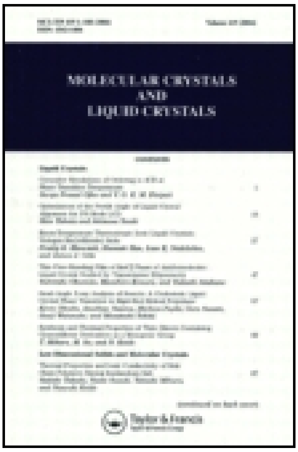

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Magnetic Phenomena in v(TCNE)$_x$y(Solvent): Role of Disorder and Random Anisotropy

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MAGNETIC PHENOMENA IN $V(TCNE)_x \cdot y(SOLVENT)$: ROLE OF DISORDER AND RANDOM ANISOTROPY

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Abstract We report here a summary of recent developments in our magnetization, ac susceptibility, electron spin resonance, charge transport and structural studies of high T_c molecular magnet systems based on $V(TCNE)_x \cdot y(solvent)$. We treat their magnetic behavior within the framework of a correlated spin glass (CSG)/ferrimagnet with wandering axis (FWA) formalism. Quantitative fit to the magnetic field dependent magnetization at low temperature and scaling analysis near T_c supports this model. The central role of the relative magnitudes of magnetic exchange, randomness in magnetic exchange, magnetic anisotropy, and randomness in magnetic anisotropy is proposed. The important role of nonmagnetic solvent in controlling the magnetic properties is described. This perspective provides a guide to the controllable synthesis of molecular based magnets of tunable desired properties including high transition temperatures and/or high magnetic susceptibilities.

INTRODUCTION

The success in developing new magnetic materials[1,2], in particular, those based on the decamethylmetallocene/tectracyanoethanide, $[MCp^*_2][TCNE]$, framework[3] and the achievement[4,5] of higher T_c's through use of S = 1 $[MnCp^*_2]^+$ lead to the attempt at preparing analogous higher spin linear chain electron transfer salts using bis(benzene)vanadium as the donor molecule.[6] Its cation state has the same electronic structure and the same S = 1 as the $[MnCp^{*}_{2}]^{+}$ cation.

SAMPLE PREPARATION, COMPOSITION AND LOCAL ORDER

To prepare the material TCNE and $V(C_6H_6)_2$ were separately dissolved in spinless organic solvents (CH_2Cl_2, CH_3CN, C_4H_8O, C_6H_6, ...). Upon mixing dropwise a precipitate forms immediately[6,7]. The samples then were stored under vacuum to remove excess solvent. The material resulting from preparation in CH_2Cl_2 has the hysteresis loop at room temperature with a coercive field of ~60G, Fig. 1, demonstrating that a room temperature molecular based magnet has been achieved.

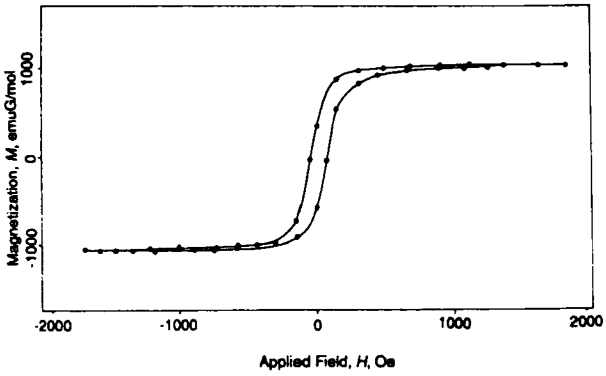

Fig. 1. Hysteresis loop, M vs H, at room temperature for V(TCNE)x.y(CH2Cl2). The line is a guide to the eye. From Ref. 6 (Copyright 1991 by the AAAS).

Chemical analyses determine that the stoichiometry is variable. Representing the materials as $V(TCNE)_x{\cdot}y(\text{solvent})$ yields typical values of x ~ 2, and y ~1/2. There is no indication of any C_6H_6 in the precipitate. In agreement with the variable stoichiometry, powder x-ray diffraction measurement show the presence of several broad diffraction peaks indicating a disordered material with a typical coherence length of ~15Å[8] for material prepared in CH_2Cl_2. Preliminary x-ray diffraction studies indicate that the amount of order decreases when THF (C_4H_8O) is the solvent, while the samples are nearly amorphous when acetonitrile (CH_3CN) is used as the solvent[40].

The C-N stretching frequency points to the TCNE being a monoanion and hence having spin 1/2. From the average stoichiometry and the usual saturation moment (see below) it is assumed that the vanadium is in the V^{+2} oxidation state with spin 3/2. Because V^{+2} usually has octahedral symmetry it is expected to coordinate with up to six TCNE and/or solvent molecules. Similarly the TCNE may coordinate with up to four V^{2+} because of their four C-N groups.

FERRIMAGNETIC ORDERING IN $V(TCNE)_x \cdot y(SOLVENT)$ AND MECHANISM OF EXCHANGE

The temperature dependence of the magnetization of $V(TCNE)_x \cdot y(CH_2Cl_2)$ determined at a number of different applied fields (up to 19.6 kG), Fig. 2, shows a saturation magnetization of ~5300 emuG/mol at 4.2K. The value is substantially lower than expected for ferromagnetic ordering of the S = 3/2 V^{2+} and S = 1/2 $TCNE^-$, but in agreement with the value expected for antiferromagnetic coupling of the V^{2+} spins and the $TCNE^-$ spins. In the latter case a stoichiometry of V:TCNE of 1:2 leads to a ferrimagnet with net spin 1/2 per formula unit and M_s of ~5500 emuG/mol.

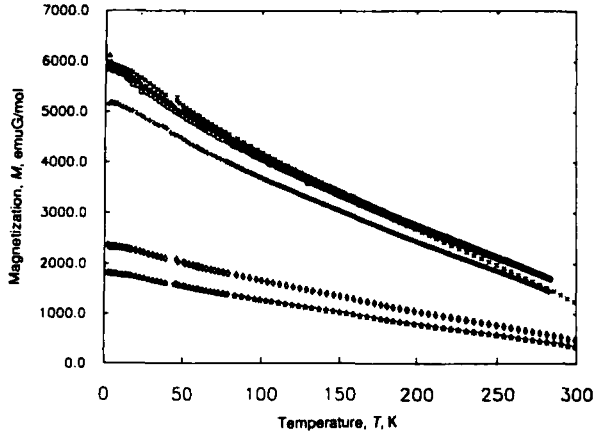

Fig. 2. M vs T at 0.15, 0.5, 2.0, 5.25, 15.8, and 19.5 kG for V(TCNE)x.y(CH2Cl2). From Ref. 6 (Copyright 1991 by the AAAS).

The antiferromagnetic exchange coupling J may have its origin in configuration interaction (virtual charge transfer from V^{2+} to $TCNE^-$ or the reverse). For either direction of charge transfer antiferromagnetic alignment of the V^{2+} and $TCNE^-$ spins is required by the Pauli exclusion principle, Fig. 3. For this scheme the effective exchange would then be given by the sum of the contributions of both forward (V^{2+} to $TCNE^{-}$: the energy cost of an excitation being ΔE_{VT}) and reverse ($TCNE^{-}$ to V^{2+}: the energy cost of an excitation being ΔE_{TV}) charge transfer. Using the Hubbard model,[9] the effective Heisenberg exchange is then $J = 2t^2[(1/\Delta E_{VT}) + (1/\Delta E_{TV})]$ where t is the transfer integral between the $TCNE^{-}$ b_{3g} orbital and the V^{2+} d levels.

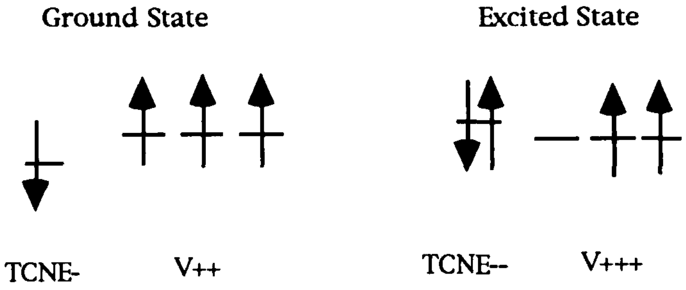

Fig. 3. Schematic illustration of possible charge transfer mechanism for antiferromagnetic exchange among V dications (S=3/2) and TCNE anions (S=1/2). Similarly an antiferromagnetic alignment occurs when forming the [TCNE (neutral); V(+1)] back charge transfer excited state.

$V(TCNE)_x \cdot y(CH_2Cl_2)$: M(T) AND DETERMINATION OF T_c

The temperature dependent remanent magnetization of $V(TCNE)_x \cdot y(CH_2Cl_2)$ was determined by cooling the sample in an applied magnetic field to 2K then reducing the applied magnetic field to zero and monitoring M(T) as the sample warmed to room temperature. The result,[10] Fig. 4, differs substantially from the usually expected behavior for crystalline magnets.[11] The nearly linear decrease of M(T) with increasing T for T>100K is more typical of disordered materials where there are fluctuations in the magnetic exchange values and/or the magnetic anisotropy.[11-14] A linear extrapolation of the M(T) to $M(T_c) = 0$ leads to an estimate[10] of $T_c = 400$K.

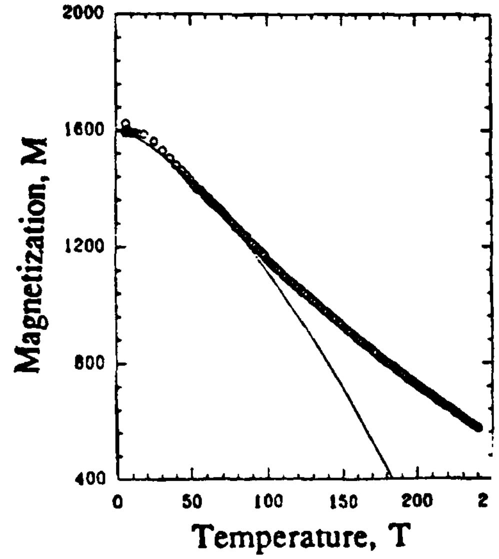

Fig. 4. T-dependent remanent magnetization of V(TCNE)x.y(CH2Cl2). The solid line is a fit to spin wave theory. See text. (from Ref.10)

The low temperature data has been analyzed using spin wave theory, which is usually applicable in the long wave length limit even in the presence of disorder.[11] Assuming

$$M_s - M(T) = BT^{3/2}$$

with the coefficient B related to the spin wave dispersion by $B \sim D^{3/2}$ where $E(k) = Dk^2$ (k is the wavevector for the spin waves), we estimate[10] that D ~ 75 meVÅ^2. Using the correlation between T_c and D developed for disordered magnets,[11] this fit implies $T_c = 400$K, in remarkable agreement with the value determined by linear extrapolation of M(T) discussed above.

These estimates of T_c can be used to determine a value for the average exchange J. Using $T_c = 2JzS(S+1)/3k_B$ for three-dimensionally coupled magnets, and assuming there are an average of five nearest neighbors as well as an effective spin value S(S+1) determined by by the root mean square value $[(1/2)(3/2)(3/2)(5/2)]^{1/2}$, J = 70K. This is only 2.6 times the intrachain exchange determined[9] earlier for

[FeCp$^*{}_2$][TCNE]. The two order of magnitude increase in T_c as compared to T_c of [FeCp$^*{}_2$][TCNE] is in largest part due to the three-dimensional network increasing T_c by a factor of the number of near neighbors, in contrast to a quasi-one-dimensional chain system where T_c is reduced by the root mean square value of the inchain and interchain exchange ($T_c = b(J_{\|} | J_{\perp})^{1/2}$, where b is a constant of value ~1.5).

The sizable coercive field of disordered V(TCNE)$_x$·y(CH$_2$Cl$_2$) suggests significant fluctuations in the local magnetic anisotropy field as might be expected[11] in a system with considerable disorder.

Effect of solvent on magnetism

In order to gain insight into the magnetic state, samples prepared in several different solvents were studied, with particular emphasis on CH_2Cl_2, THF (C_4H_8O), and acetonitrile (CH_3CN). The field cooled magnetization of V(TCNE)$_x$·y(solvent) with CH_2Cl_2, THF, and acetonitrile solvents measured at 2000, 1000 and 1000G applied field respectively are shown in Fig. 5 while their ac susceptibility measured[15] at 400 Hz for typical compositions obtained from these solvents is shown in Fig. 6. All three systems have similar M(T), with T_c in excess of room temperature for the CH_2Cl_2 solvent, Tc ~205K for C_4H_8O, and T_c in the range of 60-150K dependent upon sample composition for CH_3CN. The decrease in χ_{ac} below 100K for CH_2Cl_2 likely reflects the increasing domain wall stiffness of this ferrimagnet as T is decreased. In contrast, χ_{ac} of samples prepared in THF have constant ac susceptibility at lower temperatures consistent with weak or absent coercive field. Acetonitrile prepared samples have an even lower T_c, with a relatively sharp maximum in the χ_{ac} and a decrease in χ_{ac} at lower temperatures) reminiscent of reentrant spin glass like behavior (typically the spin glass freezing temperature, T_f, for acetonitrile prepared samples is in the range of ~7 - ~15K. The broad range of its T_c suggests the critical role of variations in local composition for this system.

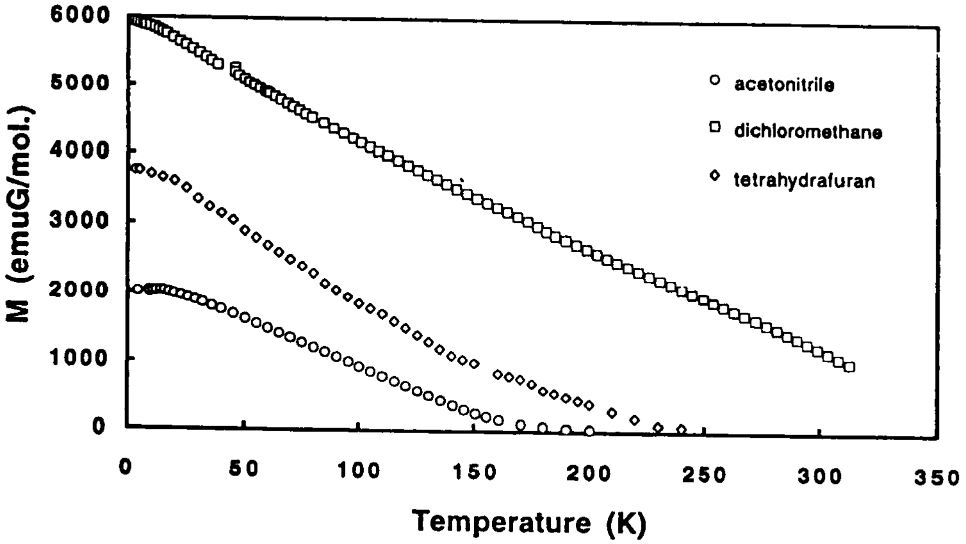

Fig. 5. Field cooled magnetization of V(TCNE)x.y(solvent) at 2000G for CH_2Cl_2 and 1000G for THF and CH_3CN. (from Ref. 15)

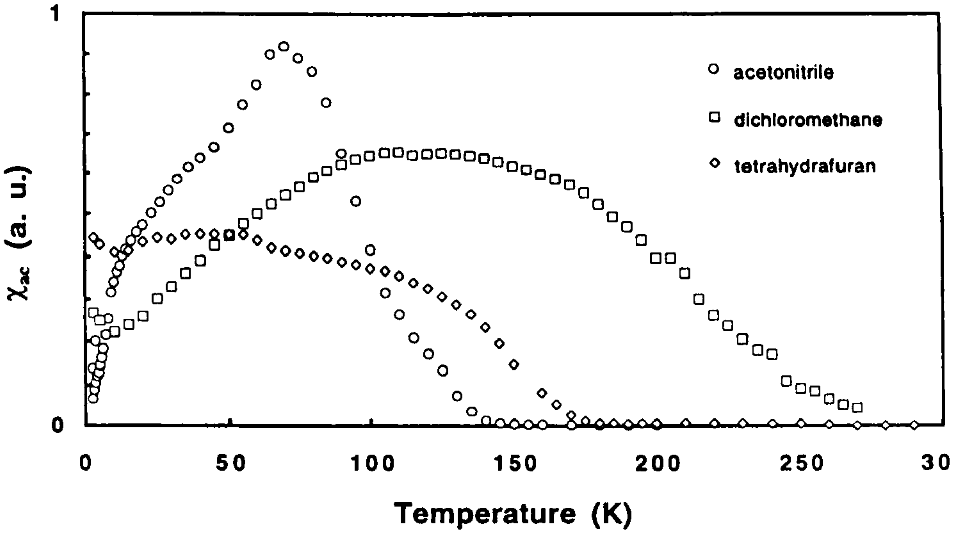

Fig. 6. AC susceptibility of $V(TCNE)_x \cdot y$(solvent) for CH_2Cl_2, THF and CH_3CN. H_{ac} is 220 mG and f = 400 Hz. (from Ref. 15)

$V(TCNE)_x \cdot y(CH_3CN)$: EFFECTS OF FLUCTUATION IN LOCAL ANISOTROPY

We have extensively studied[16] the $V(TCNE)_x \cdot y(CH_3CN)$ acetonitrile samples to gain insight into the origins of the magnetism in these materials. The positive slope of the plot of χ*T vs. T for T>200K, Fig. 7, is typical of ferrimagnetic materials at temperatures greater than the ordering temperature. This positive slope reflects that the primary exchange present in the material is antiferromagnetic, while the rapid increase in the product χ*T below 150K shows the effects of three-dimensional magnetic ordering in this material. The magnetization measured at varying applied fields is plotted in Fig. 8. Similar to the behavior of the CH_2Cl_2 prepared samples, the M(H,T) is very different from that of the usual crystalline magnets with a very strong field dependence and an unusual nearly linear temperature dependence.

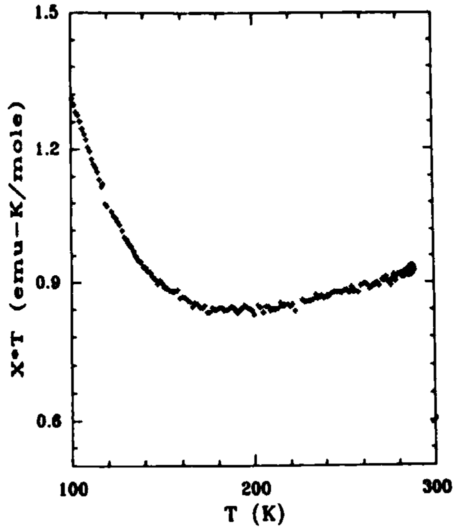

Fig. 7. X*T for V(TCNE)x.y(CH3CN). (from Ref. 16)

Given that there is a substantial disorder in the material, and that the spinless CH_3CN will tend to coordinate with V^{2+} displacing the S=1/2 $[TCNE]^{-\cdot}$, we examined the data in light of available models for the effects of disorder on magnetism. The randomness in coordination and order are expected to lead to some variation in the magnitude of the exchange between the S=3/2 V^{2+} and the S=1/2 $[TCNE]^{-\cdot}$, although the sign of the exchange is expected to remain constant. More importantly, the

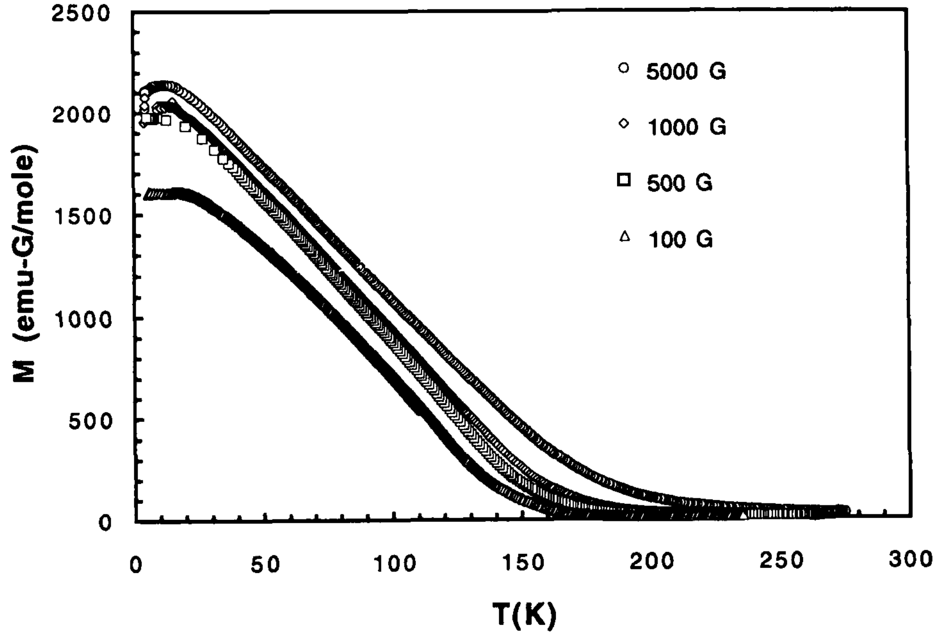

Fig. 8. M vs T at 0.1, 0.5, 1.0 and 5.0 kG for V(TCNE)x.y(CH3CN). (from Ref. 16)

disorder is expected to lead to variation in a small anisotropy as spinless CH3CN molecules displace some of the S=1/2 [TCNE]$^{-\cdot}$. This suggests the application of models based on the effects of random anisotropy being primary.

Real space models for weak random anisotropy magnets (RAM) have recently been developed by Chudnovsky, et al.[12,13] The effects of random anisotropy can be added to the usual isotropic Heisenberg Hamiltonian:

$$H = -\sum_{i,j} J_{i,j}\vec{S}_i \cdot \vec{S}_j - D_r \sum_i \left(\hat{n}_i \cdot \vec{S}_i\right)^2 - D_c \sum_i \left(\hat{N}_i \cdot \vec{S}_i\right)^2 - g\mu_B \sum_i \vec{H} \cdot \vec{S}_i$$

Here the first term represents the average exchange interaction, the second term represents a random magnetic anisotropy (D_r), the third a constant magnetic anisotropy (D_c) and the fourth is the Zeeman term representing interaction with an applied magnetic field. The RAM approach assumes that the magnetic order is determined by the random anisotropy[12,13]. For $D_c < D_r << J_{ij}$ a correlated spin glass occurs, while a ferromagnet or ferrimagnet with wandering axis occurs for large D_c or large Zeeman term. An alternate approach neglects the effects of random anisotropy and assumes that the magnetic order is determined by random exchange only.[14]

Given the behavior of the CH_2Cl_2, THF and CH_3CN materials, we suggest that for the CH_2Cl_2 system the constant magnetic anisotropy term exceeds the random term ($D_c>D_r$) and that its magnetic properties are that of a ferrimagnet with wandering axis (FWA); that for the THF system the constant anisotropy and random anisotropy are of the same order ($D_c \sim D_r$); and that for the CH_3CN system the random anisotropy exceeds that of the constant anisotropy ($D_r>D_c$) and that its magnetic properties are those of a reentrant correlated spin glass. The magnetic properties of the THF system are intermediate between those of the CH_2Cl_2 and the CH_3CN systems. This behavior correlates with the CH_2Cl_2 being the least coordinating of the three solvents with the THF coordinating more easily and the CH_3CN being the most readily coordinating solvent. This also correlates with the CH_2Cl_2 system being the least disordered, with the THF and CH_3CN systems having increasing disorder respectively.[8] In light of this we analyze the low temperature data for the CH_3CN material in the random anisotropy model of Chudnovsky[12,13] with the behavior near the three-dimensional ordering temperature T_c analyzed within the modified equation of state analysis of Aharony and Pytte[17] and also Gehring, et al.[18]

V(TCNE)$_x$·y(CH_3CN): Low Temperature Magnetization

The magnetic field dependent deviation from saturation of the magnetization of V(TCNE)$_x$·y(CH_3CN) measured at 4.2K is shown in Fig. 9. The relatively slow approach to saturation of M(H) is unusual for a magnetic material and suggests the critical role of disorder and random anisotropy. The model of Chudnovsky[12,13] predicts that M(H) increases as $(H+H_c)^{1/2}$ for $D_r>D_c$ and $D_r<<J$, where H_c is the coherent anisotropy

field. The solid line in Fig. 9 is a quantitative fit to the data with $H_C \sim$ 21kG. This value of H_C is only about 2% of the value of J, self-consistent with the model that the system is in an weak anisotropy regime.

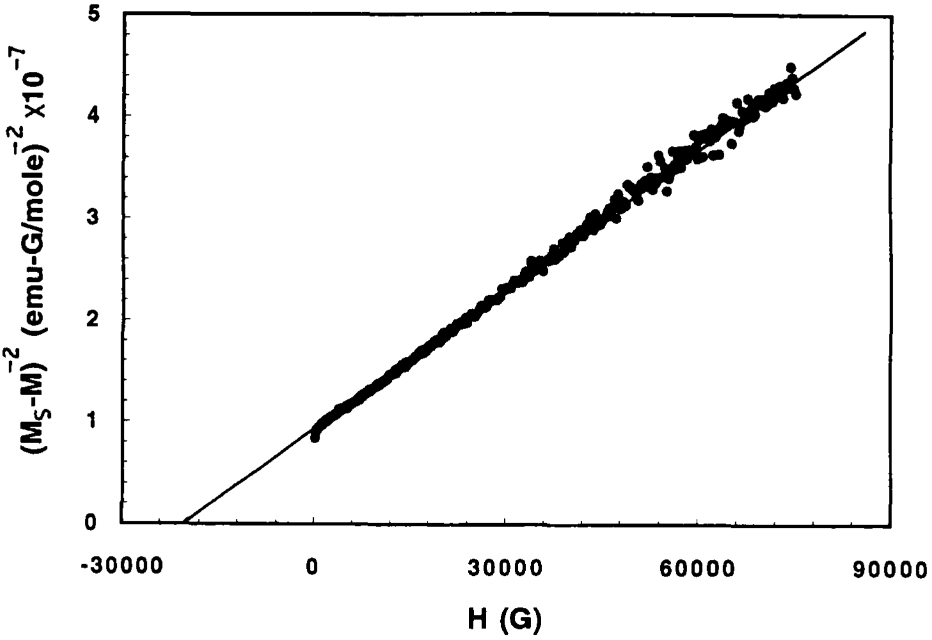

Fig. 9. $(M_S - M)^{-2}$ vs H with saturation magnetization of $M_S = 5250$ emuG/mol for V(TCNE)$_x$·y(CH$_3$CN). (from Ref. 16)

V(TCNE)$_x$·y(CH$_3$CN): Critical exponents near T_C

The behavior near T_C can be analyzed using a modified equation of state approach to obtain effective critical exponents for this disordered magnetic system. A critical isotherm can be determined[16] by plotting M(H) vs H at varying temperatures, with M proportional to $H^{1/\delta}$ at T_C. Using this analysis we determine that $\delta = 4$ and $T_C = 135K$ for a typical sample studied. With T_C and δ determined, exponents β_a and γ_a can be determined directly by analyzing isothermal plots of $(H/M)^{1/\gamma_a}$ vs. M^{1/β_a}. Given β_a and δ all of the M(H,T) data in the vicinity of T_C can be collapsed onto a single set of curves for plots of $\ln(M/|t|^{\beta_a})$ vs. $\ln(H/|t|^{\beta\delta})$ where $t = |T-T_C|$, Fig. 10. The lower curve corresponds to $T>T_C$ and the upper curve corresponds to $T<T_C$.

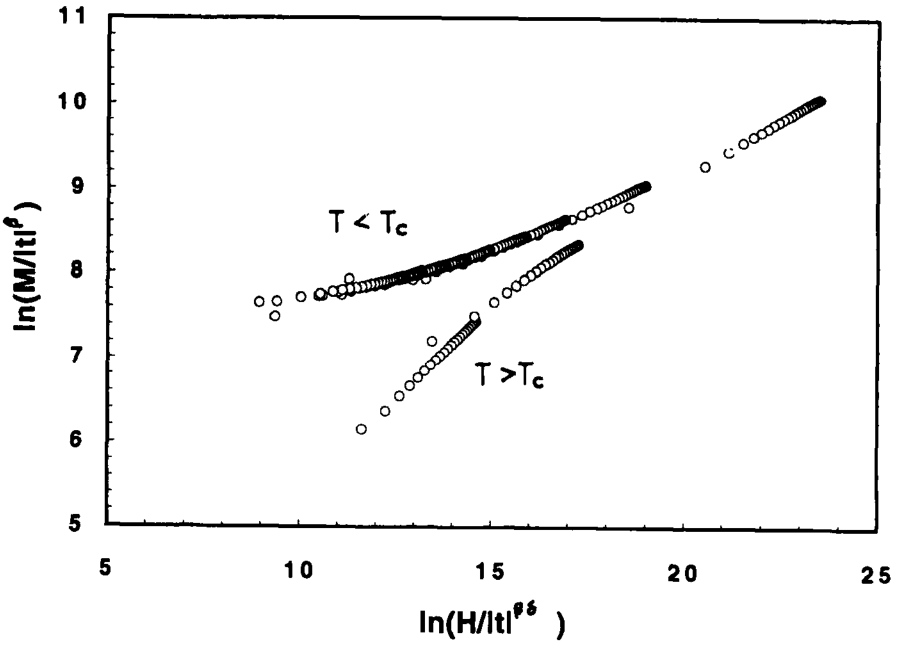

Fig. 10. Scaling plot of for V(TCNE)x.y(CH3CN) with Tc = 138K (see text). (from Ref. 16)

$V(TCNE)_x{\cdot}y(THF)$: EFFECTS OF FLUCTUATION IN LOCAL ANISOTROPY

The magnetic properties of $V(TCNE)_x{\cdot}y(THF)$ are intermediate between those of the CH_2Cl_2 and CH_3CN derived samples. The temperature dependent magnetization at differing magnetic fields, Fig. 11, again shows the quasi-linear behavior below T_c ~ 210K, with an unusually strong magnetic field dependence below T_c indicative of the presence of disorder. There is an upper limit on the coercive field of 10G. The results have been interpreted in terms of the model of dominant effects of random magnetic anisotropy, with comparison to the theory of Chudnovsky[12,13] at low temperatures. The M(T) increases at low temperature (inset of Fig. 15), perhaps indicative of superparamagnetic coupling.[19]

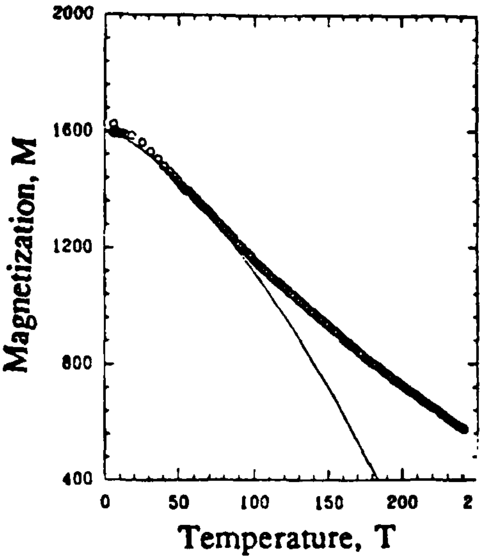

Fig. 11. M vs T at 0.1 and 1.0 kG for V(TCNE)x,y(THF). The inset shows data for 5.0 and 10.0 kG. (from Ref. 19)

Equation of state analyses of isotherms near T_c show a strong fluctuation of local magnetization and yield relatively large values of critical exponent as compared to those expected for the usual three-dimensional systems with long range order. The Arrott-Noakes plot for the critical isotherm, $T_c = 210K$, is in Fig. 12 (β_a=0.95; γ_a=2.38;δ_a=3.5).

ELECTRON PARAMAGNETIC RESONANCE: A FLEXIBLE PROBE OF UNUSUAL PHENOMENA

The electron paramagnetic resonance of V(TCNE)$_x$·y(solvent) has been studied[20] for CH_2Cl_2, THF and CH_3CN solvents. A rich set of temperature dependent spectra give insight into the magnetism. A typical derivative spectrum at room temperature for V(TCNE)$_x$·y(CH_2Cl_2) is shown in Fig. 13.

Four distinct features are observed,[20] (1) the main resonance with g~1.92, (2) an approximately half-field resonance, (3) a broad near

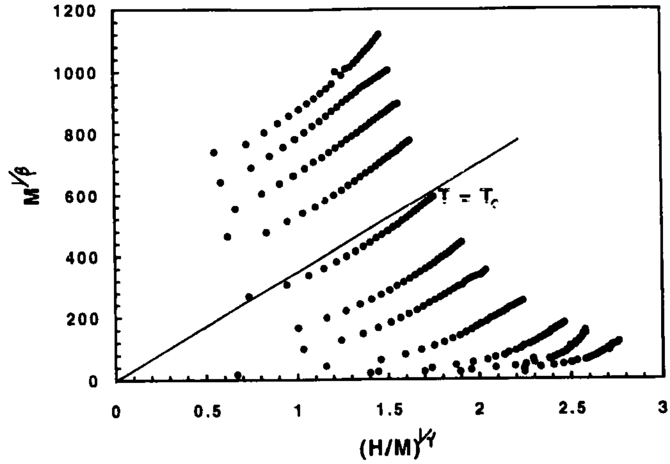

Fig. 12. Arrott-Noakes plot for V(TCNE)x.y(THF) yielding Tc = 210K. (from Ref. 19)

zero field resonance, and (4) a narrow zero field antiresonance. These features vary systematically with samples composition (including differing solvents) and temperature. The latter two features at 200K are more clearly shown in Fig. 14. The integrated intensity of the main resonance for each of the compositions studied scales with the measure

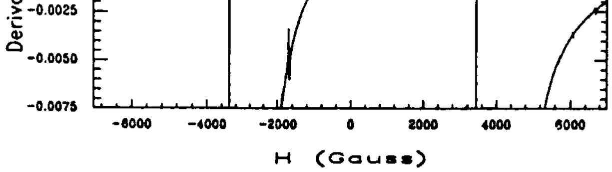

Fig. 13. Derivative EPR signal vs H for V(TCNE)x.y(CH2Cl2) at 295K. (from Ref. 20)

temperature dependent dc magnetization, hence it is associated with the ferrimagnetic resonance. Contrary to the usual magnetic systems, the linewidth reaches a minimum at T_C, with a critical behavior mimicking the critical behavior of M(T), as expected for a material with 'sloppy' spin waves (spin waves with wave vector $q > \xi$, where ξ is the correlation length[21]. The g-shift of the main resonance line scales with the magnetization and agrees in magnitude with expectations of demagnetization effects.

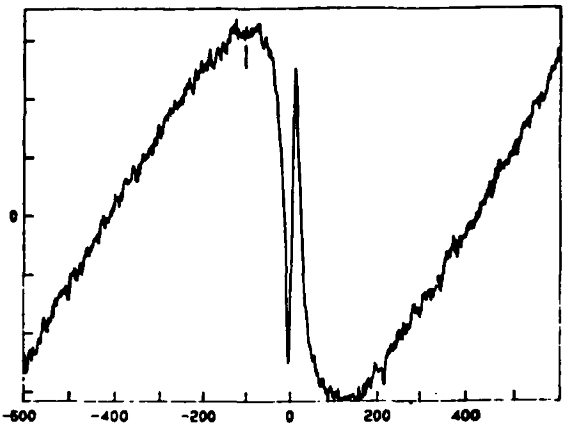

Fig. 14. Near zero field EPR signal of V(TCNE)x.y(CH2C12) at 200K. (from Ref. 20)

The broader low field signal is largest in the CH_2Cl_2 prepared materials, weak in the THF prepared systems and virtually non-existent in the CH_3CN prepared materials. The peak to peak width and its sample variation suggest that it is a domain wall like resonance. The sharp zero field signal is unusual. It is present in each of the compositions below T_C though for the CH_3CN prepared samples it evolves into a resonance from an antiresonance below ~15K, a temperature associated with freezing out of the spins into a spin glass phase. Based on this temperature dependence this antiresonance may be caused by a low field magnetoresistance making it more difficult for electrons to hop among charge carrier sites (presumably TCNE) thereby reducing the sample absorbance of microwaves at higher magnetic fields.

V(TCNE)$_x$·y(SOLVENT) CONDUCTIVITY: CORRELATED HOPPING

The V(TCNE)$_x$·y(solvent) materials have moderate conductivites of order 10^{-5} to 10^{-3} S/cm at room temperature, depending upon the solvent.[22] In general the dc conductivity varies as $\exp[-(T_0/T)^{1/4}]$ reminiscent of Mott variable range hopping,[23], Fig. 15. The frequency (f) dependence, Fig. 16, suggests that the behavior is more complex.[22]. The frequency dependent component of the conductivity varies as f^sT^n with s~0.7 typical of near-neighbor pairwise hopping. However, n~4 instead of 1, the value typical of polymers and amorphous semiconductors.[24]

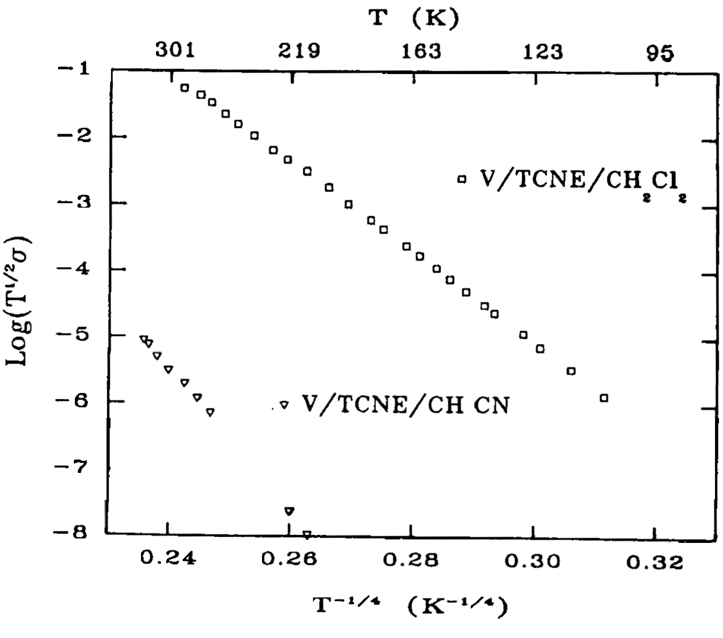

Fig. 15. Temperature dependence of dc conductivity vs $T^{-1/4}$ for V(TCNE)$_x$·y(CH_2Cl_2) and V(TCNE)$_x$·y(CH_3CN). (from Ref. 22)

We suggest that this unusual temperature dependence of the conductivity at higher frequencies may be a reflection of the correlated spin glass behavior of the V(TCNE)$_x$·y(solvent) materials. The charge transport likely involves charge hopping among TCNE sites. (Because of its smaller size the Coulomb repulsion for an extra electron on V^{2+} is expected to be larger than that for an extra electron on the TCNE^{-}·) If neighboring TCNE$^-$ sites have the spins aligned in the same direction, then an electron hop to a near neighbor requires an emission or absorption of a spin flip phonon to conserve total spin (since each of the near neighbor TCNE have their spins aligned in the same direction), while tunneling a distance greater than the magnetic correlation

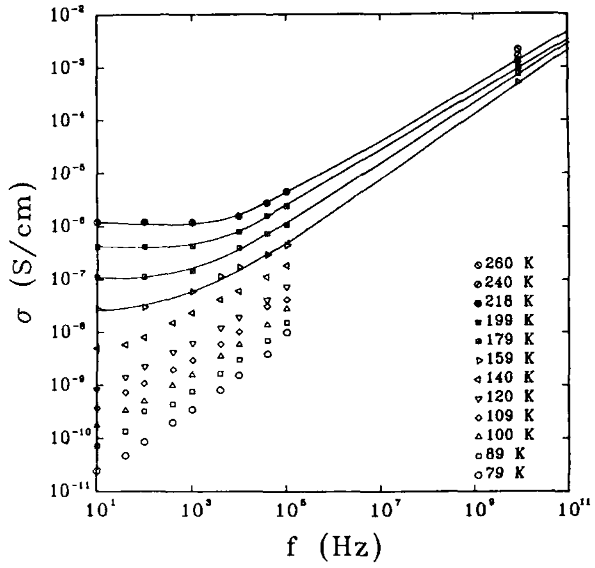

Fig. 16. The temperature and frequency dependence of conductivity of V(TCNE)x.y(CH2Cl2). (from Ref. 22).

length results in an electron transition without the cost of the spin flip process. The probability p_i of electron transition between near and/or far sites may then be modified to reflect the magnetic correlation in the following manner:

$$p_i \sim \exp[-(\Delta E/k_B T - \alpha r - \Delta E_{sf}(r)/k_B T]$$

where ΔE is the typical difference in energy of adjacent sites, α^{-1} the decay length of the electron states involved in the charge transport, and $\Delta E_{sf}(r)$ is the distance r dependent energy required for flipping of spins to allow the electron transition. For sites separated by greater than the magnetic correlation length the spin flip energy term goes to zero.

SUMMARY

Molecular/polymer based magnets provide a framework for study of a wide variety of magnetic phenomena. The advent of the $V(TCNE)_x \cdot y$(solvent) systems demonstrated that room temperature magnetism is achievable in molecular/polymeric materials. The nearly hundred fold increase in T_c compared with that of earlier studied metallocence based systems is attributed to both an increase in J (due to closer approach of the spins) and three-dimensional coordination. Analysis of a variety of data demonstrates that the disorder in the material introduces a weak random anisotropy which in turn determines whether the material is in a three-dimensional magnetic state (even at room temperature), a low temperature magnet, or even a spin glass. Control of the local order, structure, spin, and chemical composition determines the magnetic state achieved and its charge transport and dynamic properties.

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