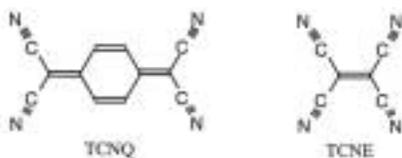


Organic- and Molecule-based Magnets

by Joel S. Miller

Magnets have captivated mankind's imagination for centuries and in the twentieth century their development was essential for the establishment of our present high-tech society. The number, proximity, and consequently the coupling among electron spins, dictate all magnetic behaviors. From time immemorial, materials with strong magnetic behavior were always associated with electron spins with metal atoms or ions. A desire to make a magnet with electron spins residing in p-orbitals on nonmetal sites arose from the realization of metal-like electrical conductivity for some organic materials possessing segregated chains of reduced TCNQ (TCNQ = 7,7,8,8-tetracyano-*p*-quinodimethane). These studies ultimately led to the development of molecule-based organic and polymeric electrical conducting materials.



One phase of $[\text{Fe}^{\text{III}}\text{Cp}^*_2][\text{TCNQ}]$ (Cp^* = pentamethylcyclopentadienide) led to the characterization that this material had an antiferromagnetic ground state, which above a critical applied magnetic field of 1.5 kOe, had a transition to a high-moment ferromagnetic-like state, *i.e.*, a metamagnet. As expected, replacement of $[\text{TCNQ}]^{\bullet-}$ with $[\text{TCNE}]^{\bullet-}$; tetracyanoethylene, with the same number of unpaired electron spins, but importantly delocalized over one-half the size, would have a greater spin density, and being smaller, sufficiently stronger spin coupling to stabilize the ferromagnetic ground state. $[\text{Fe}^{\text{III}}\text{Cp}^*_2][\text{TCNE}]^{\bullet-}$ (Fig. 1), was characterized as a ferromagnet with $T_c = 4.8$ K, and coercivity, H_{cr} , of 1 kOe at 2 K. The latter value exceeds the values needed for magnetic storage, suggesting that these materials might be suitable for magnetic data storage, should T_c be raised above room temperature. The saturation magnetization for single crystals aligned along the chain axis of

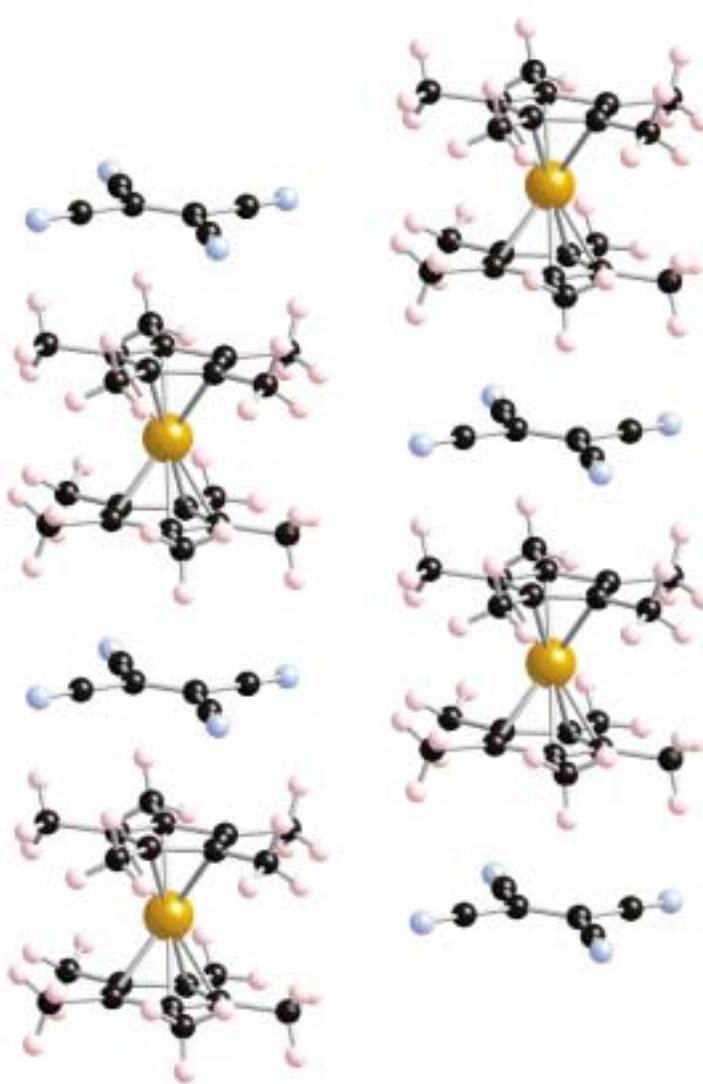


Fig. 1. Crystal structure of $[\text{FeCp}^*_2][\text{TCNE}]$ (Fe^{III} gold; C black; H pink; N blue) showing two parallel chains of alternating $S = 1/2 [\text{Fe}^{\text{III}}\text{Cp}^*_2]^{\bullet+}$ and $S = 1/2 [\text{TCNE}]^{\bullet-}$ unbound to each other in $[\text{Fe}^{\text{III}}\text{Cp}^*_2]^{\bullet+}[\text{TCNE}]^{\bullet-} \cdot \text{MeCN}$.^{2a}

$[\text{Fe}^{\text{III}}\text{Cp}^*_2]^{\bullet+}[\text{TCNE}]^{\bullet-}$ is 16.3 $\mu\text{emu}/\text{mol}$. This is 37% greater than that of iron metal per iron,^{2b} but in accord with the expectation for ferromagnetic coupling among the spins on the organic $[\text{TCNE}]^{\bullet-}$ and the spins on the Fe^{III} sites. Replacement of Fe^{III} with Mn^{III} and Cr^{III} , and $[\text{TCNE}]^{\bullet-}$ with $[\text{TCNQ}]^{\bullet-}$ leads to ferromagnets with the T_c 's of the $[\text{TCNE}]^{\bullet-}$ materials that decrease as $\text{Mn} > \text{Fe} > \text{Cr}$, and exceed those of the $[\text{TCNQ}]^{\bullet-}$ based materials.^{3a}

A few organic nitroxides order as ferromagnets below a T_c of 1.5 K.^{3a,b} These

include a phase of 4-nitrophenyl nitronyl nitroxide reported ($T_c = 0.6$ K).^{3b} The organic salt $(\text{TDAE})(\text{C}_{60})$ ($\text{TDAE} = \text{tetrakis}(\text{dimethylamino})\text{ethane}$) has a more complex magnetic behavior and orders as a ferromagnet ~ 16 K.^{3c}

$\text{V}(\text{TCNE})_x$ Room Temperature Magnets

Reaction of TCNE and $\text{V}(\text{CO})_6$ in dichloromethane rapidly forms $\text{V}[\text{TCNE}]_x \cdot y\text{CH}_2\text{Cl}_2$ ($x \sim 2$; $y \sim 1/2$).⁴ Because of its extreme air/water reactiv-



Fig. 2. CVD-prepared thin film (ca. 5 μm) $\text{V}(\text{TCNE})_x$ magnet on a piece of broken glass being attracted to a SmCo_5 magnet at room temperature in the air.^{5a}

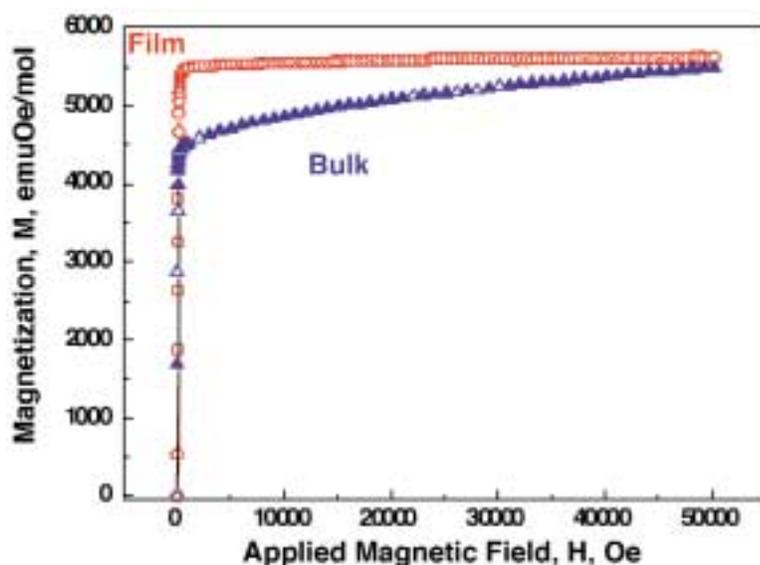


Fig. 3. $M(H)$ for dichloromethane-prepared $\text{V}(\text{TCNE})_x \cdot y\text{CH}_2\text{Cl}_2$ (bulk, blue) and CVD-prepared $\text{V}(\text{TCNE})_x$ (film, red) showing that the latter reaches saturation at a lower applied magnetic field than that the former.^{5b}

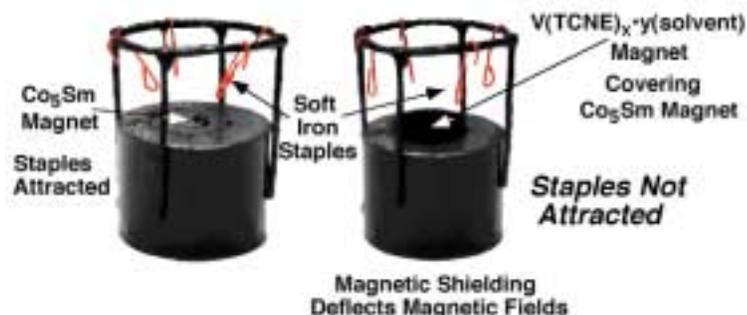


Fig. 4. Illustration of the $\text{V}(\text{TCNE})_x \cdot y(\text{CH}_2\text{Cl}_2)$ magnet being an effective magnetic shield at room temperature; rods (paper clips) of soft-iron (red) are attracted to a Co_5Sm permanent magnet (left); however, when a 1.7 mm pellet of $\text{V}(\text{TCNE})_x \cdot y(\text{CH}_2\text{Cl}_2)$ placed between the Co_5Sm magnet and the rods (red) hang freely (right), demonstrating magnetic shielding.^{6b}

ity, its insolubility, compositional inhomogeneities within and between samples, as well the product being amorphous, the structure remains elusive. Nonetheless, this material is the first ambient temperature organic- or polymer-based magnet ($T_c \sim 400$ K).⁴ The proposed structure has each V being coordinated with up to 6 ligands (N's from six different TCNE's) and each TCNE is reduced and is either planar or twisted and bond as many as four V's. Recently, using a basic chemical vapor deposition (CVD) methodology, solvent-free magnetic thin films of $\text{V}(\text{TCNE})_x$ composition have been prepared on several substrates, e. g., glass, Si, NaCl (Fig. 2).^{5a} While dichloromethane-prepared $\text{V}(\text{TCNE})_x \cdot y\text{CH}_2\text{Cl}_2$ is extremely water/air sensitive, CVD-prepared $\text{V}(\text{TCNE})_x$ exhibit significant air stability (Fig. 2). Figure 3 shows that dichloromethane-prepared $\text{V}(\text{TCNE})_x \cdot y\text{CH}_2\text{Cl}_2$ reaches saturation slower than that of CVD-prepared $\text{V}(\text{TCNE})_x$.^{5b} Due to T_c exceeding room temperature, several uses for this and related magnets can be envisioned.⁶ One identified application is for magnetic shielding, the attenuation of magnetic fields found in many electronic applications, (Fig. 4)^{6b} e.g., high voltage lines, and another is in spintronic application, and the feasibility of using $\text{V}(\text{TCNE})_x \cdot \text{CH}_2\text{Cl}_2$ for these applications have been demonstrated.^{6e}

$\text{V}(\text{TCNE})_x$ prepared in acetonitrile has some bonding of the nitrile to V and consequently has a reduced spin connectivity; this leads to enhanced disorder and a reduced T_c of ~ 135 K.^{7a} Analysis of high temperature magnetization of $\text{V}(\text{TCNE})_x \cdot y\text{CH}_2\text{Cl}_2$ also suggests the key role of random magnet anisotropy.^{7b}

$\text{M}(\text{TCNE})_2$ (M = Mn, Fe, Co, Ni, Gd, Dy) Magnets

Several new members of the $\text{M}(\text{TCNE})_x \cdot y\text{CH}_2\text{Cl}_2$ (M = Mn, Fe, Co, Ni, Gd, Dy) family of high- T_c organic-based magnets have been prepared.⁸ These materials are prepared from the oxidation of iodide in MI_x by TCNE have T_c 's of 97, 75, 44, 44, 3.5, and 8.5 K, respectively. Zero field/field cooled magnetization studies indicate that while the Mn^{9a} system is a reentrant spin glass, the Fe^{9b} system is a random anisotropy system. Both systems exhibit complex behavior below T_c . For example, 5 K $M(H)$ hysteresis curves for $\text{Fe}(\text{TCNE})_2$ are constricted, with a spin-flop shape, indicating ferrimagnetic behavior, and field- and zero-field-

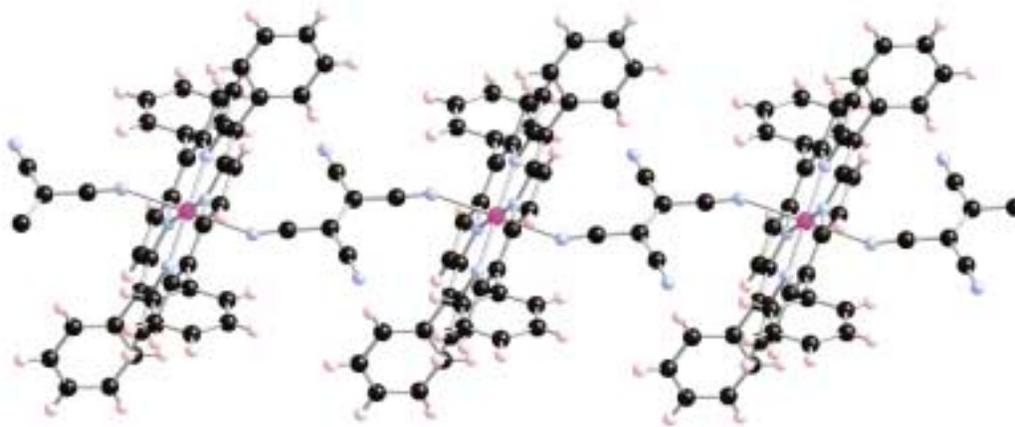


Fig. 5. Segment of a 1-D chain of [MnTPP][TCNE] (Mn^{III} magenta; C black; H pink; N blue) showing [MnTPP]⁺ trans- μ -N- σ -bonding to [TCNE]⁻.¹⁰

cooled magnetization studies reveal magnetic irreversibilities below T_c for both compounds. Static and dynamic scaling analyses of the dc magnetization and ac susceptibility data for the Mn[TCNE]₂ indicate that a transition to a 3-D ferrimagnetic state at T_c , followed by a reentrant transition, to a spin glass state at $T_g = 2.5$ K. In addition, Mn[TCNE]₂^g exhibits photoinduced magnetic behavior that include an enhanced susceptibility up to temperature as high as 75 K that is partially reversible by illumination with lower energy light, in contrast to Fe[TCNE]₂.^{9c}

Mn(porphyrin) (TCNE)-Based Magnets

Electron-transfer salts of TCNE and Mn^{II}(porphyrin), e.g., (MnTPP)(TCNE) (TPP = meso-tetraphenylporphinato) (Fig. 5), form a large family of ferrimagnets ($T_c < 28$ K)¹⁰ demonstrating that metallomacrocycles are viable components in the construction of organic-based magnets. The structure differs from [FeCp₂]⁺[TCNE]⁻ (Fig. 1) as [TCNE]⁻ coordinates to the Mn^{III} in the former system. Due to the 1-D chain structure, the [Mn(porphyrin)]⁺[TCNE]⁻ is an excellent system for studying several unusual magnetic phenomena. As a consequence of the single ion anisotropy for [MnTPP]⁺ and the significant differences in the orbital overlaps within and between chains, this class of materials often undergoes lattice- and spin-dimensionality crossovers as a function of temperature.^{11a} Metamagnetic-like behavior is noted for many members of this family at low temperature with large critical fields, H_c (~25 kOe). These are atypical metamagnetic-like materials as they exhibit hysteresis with very large coercive fields, H_{cr} (~27 kOe), and have substantial remanent magnetizations (~10 kemuOe/mol) (Fig. 6).^{11b}

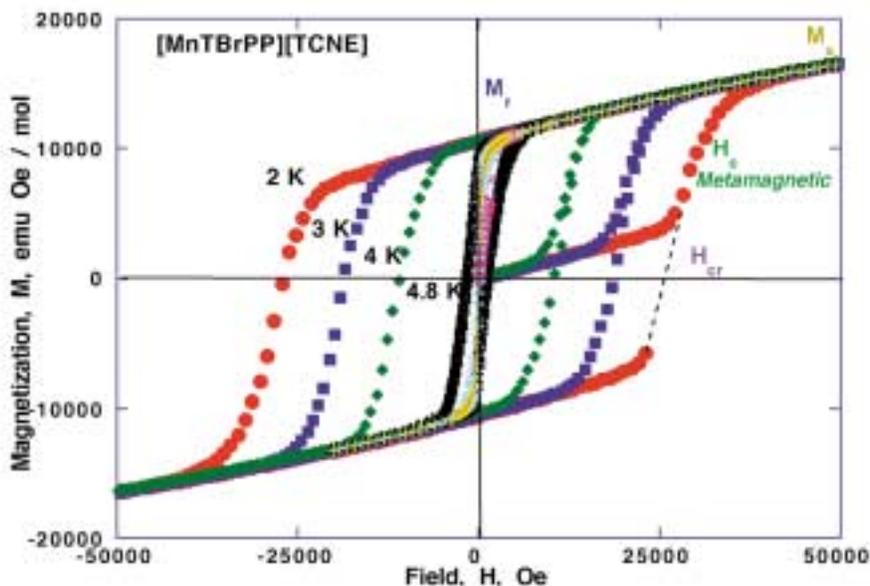


Fig. 6. $M(H)$ for [Mn^{III}TBrPP][TCNE] at 2.0 (o), 3.0 (o), 4.0 (Γ), 4.8 (Δ), 5.2 (x), 5.5 (-), 6.1 (\square), and 6.8 (c) K. Reproduced with permission of VCH-Wiley. Copyright 2000.^{11b}

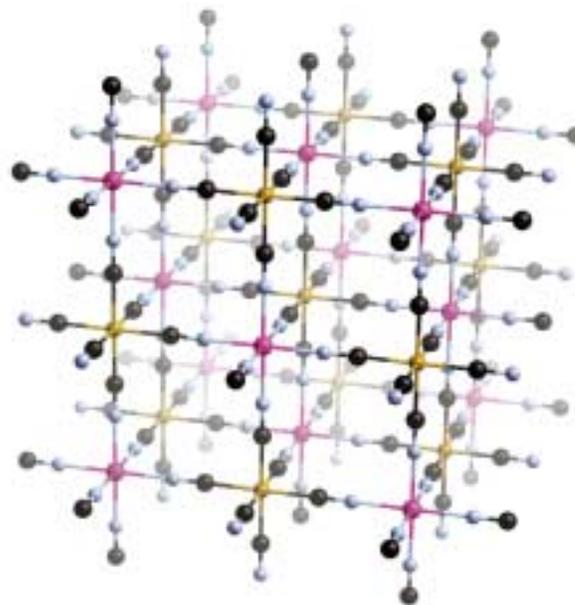


Fig. 7. Idealized structure of Prussian blue, Fe^{III}₄[Fe^{II}(CN)₆]₃ · xH₂O (Fe^{II} gold; Fe^{III} magenta; C gray; and N blue), with \cdots Fe^{III}-N \equiv C-Fe^{II} linkages along each of the three unit cell axes.

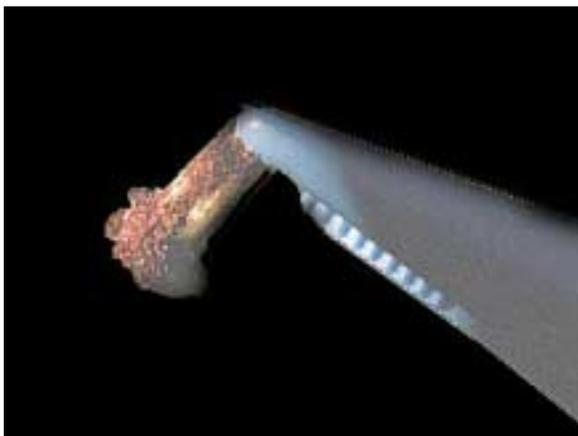


FIG. 8. Sample of a $V[Cr(CN)_6]_y \cdot zH_2O$ magnet ($T_c \sim 100^\circ C$) being attracted to a Teflon coated magnet at room temperature in air.^{14b}

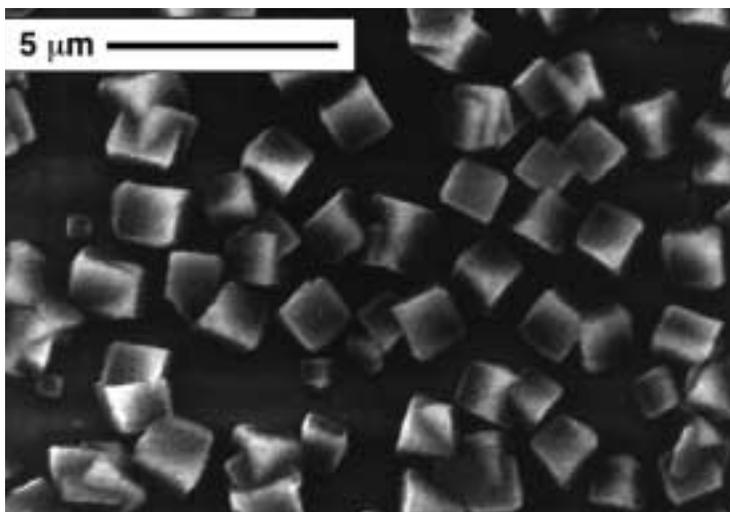
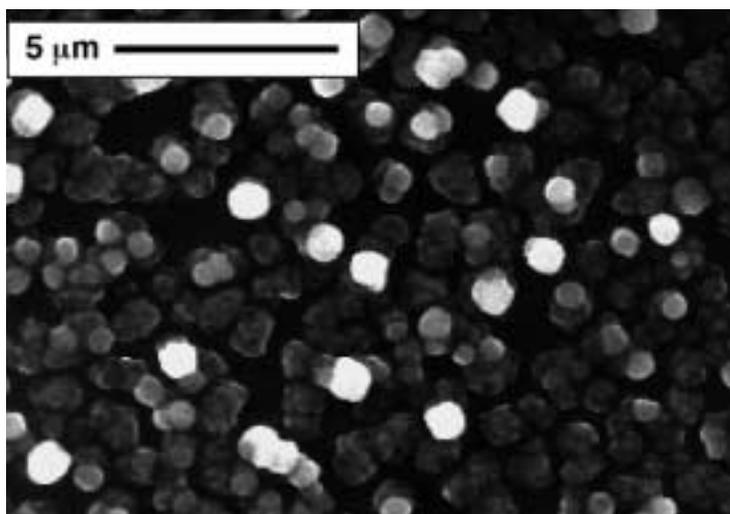


FIG. 9. SEM images of (a) an amorphous film, and (b) crystalline film of $Cr^{III}/Cr^{II}-(CN)_6^{I/0.98} [Cr^{II}(CN)_6]^{0.02}$ composed of $1.0 \pm 0.5 \mu m$ particles.¹⁸

Hexacyanometallate, Prussian Blue Structured Magnets

In addition to organic-based magnets, other molecule-based magnets can

prepared by the synthetic organic chemistry methodologies. A key example is Prussian blue, $Fe^{III}_4[Fe^{II}(CN)_6]_3 \cdot xH_2O$, which has a 3-D network structure with $Fe^{III}-N \equiv C-Fe^{II}$

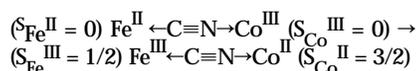
$C \equiv N-Fe^{III}$ linkages along each of the three unit cell axes (Fig. 7), and is a prototype for a large family of fascinating magnets that order as either ferro- or ferrimagnets.^{12,13,14} (Ed. Note: See the following two feature articles in this issue on this class of materials.) Replacement of iron with spin-bearing metal-ions can lead to strong magnetic coupling and magnetic ordering with high T_c 's. For example, $CsNi^{II}[Cr^{III}(CN)_6] \cdot 2H_2O$, due to its unpaired electron spins on adjacent spin sites residing in orthogonal orbitals, is a ferromagnet ($T_c = 90$ K),^{13a} and $CsMn^{II}[Cr^{III}(CN)_6] \cdot H_2O$ is a ferrimagnet ($T_c = 90$ K) as its unpaired electron spins on adjacent spin sites do not reside in orthogonal orbitals.^{13b} Furthermore, $V^{II}_{0.42}V^{III}_{0.58}[Cr^{III}(CN)_6]_{0.86} \cdot 2.8H_2O$ is a ferrimagnet above room temperature ($T_c = 315$ K).^{14a} Further study of this class of materials has led to more complex related compositions containing excess cations and/or excess anions which lead to air stable materials (Fig. 8), with T_c 's as high as $\sim 100^\circ C$ (373 K).^{14b,c}

Thin films magnets are technologically important, and expanding the work using Prussian blues as electrochromic materials,¹⁶ $[Cr^{III}(CN)_6]^{3-}$ -based magnetic films have been electrochemically deposited on tin-doped indium oxide (ITO) and glassy carbon electrodes.^{17,18} Depending on the electrochemical conditions, either oxidized or reduced, amorphous (Fig. 9a) or crystalline (Fig. 9b) films ($1.0 \pm 0.5 \mu m$) can be deposited.¹⁸ The oxidized and reduced films are of $Cr^{III} \{ [Cr^{III}(CN)_6]_{0.98} [Cr^{II}(CN)_6]_{0.02} \}$ and $K_{2.0}Cr^{II}[Cr^{II}(CN)_6]$ composition, respectively. Independent of crystallinity, the T_c varies with the degree of oxidation and ranges between 135 K for the reduced films and 260 K for the oxidized films. Hysteresis with coercive fields as high as 830 Oe at 20 K is observed for the amorphous material, but is substantially less for the crystalline material. This is ascribed to the greater number of defects in the amorphous films.¹⁸ Upon electrochemical oxidation of the films, the magnetic behavior is altered such that for example at 150 K the film becomes ferromagnetic.¹⁷

The M(T) data also reveals a negative or 'freezing-in' or magnetization upon cooling to 20 K in a negative applied dc field. The M(T) obtained upon cooling in an equal, but opposite, field are nearly symmetric about the zero field cooled (ZFC) magnetization (Fig. 10). ZFC measurements result in a positive magnetization reduced by more than

an order of magnitude from the FC data, but both the ZFC and FC magnetization data converge to the same T_c . This negative or reversed magnetization is observed in all $K_nCr_x[Cr(CN)_6]$ films studied. Fields as high as about 100 Oe are necessary at 20 K to reverse the negative magnetization for samples cooled in -5 Oe.¹⁸

Altering the magnetic behavior with light, especially to an ordered magnetic state, is an important area of contemporary research. This effect has been reported for electrochemically deposited thin films (0.05 to 1.0 μm) of $K_{0.4}Co_{1.3}[Fe(CN)_6]\cdot 5H_2O$.^{19a} Upon illumination with visible light, paramagnetic $K_{0.4}Co_{1.3}[Fe(CN)_6]\cdot 5H_2O$ orders magnetically at 26 K. Detailed study of this reaction has led to the characterization of $K_{0.4}Co_{1.3}[Fe(CN)_6]$ as containing mixed-valent Co, *i.e.*, $K_{0.4}Co^{II}_{0.3}Co^{III}_{1.3}[Fe^{II}(CN)_6]$. Here, low-spin Co^{III} , as well as high-spin Co^{II} , are N-bound to the cyanide. The non-stoichiometry suggests a defect-ridden structure. The onset of magnetic ordering is attributed to the photoinduced electron transfer



thus, diamagnetic $Fe^{II} \leftarrow C \equiv N \rightarrow Co^{III}$ ($S_{total} = 0 + 0 = 0$) becomes paramagnetic ($S_{total} = 3/2 - 1/2 = 1$) and can couple other spins in the solid leading to an ordered magnetic state. This state is reversible upon illumination of near infrared light. The magnetization can be further enhanced by 20% upon application of a 5 T applied magnetic field.^{19b} In a related system, the photoinduced magnetism is attributed to cluster glass formation.^{19c} ■

Summary

Numerous molecule-based magnets with a broad array of structures, solubilities, as well as physical and magnetic properties, have been discovered worldwide and a few are summarized in Table I. These new materials offer combinations of properties that may lead to their use in smart materials as well in other devices of the future.

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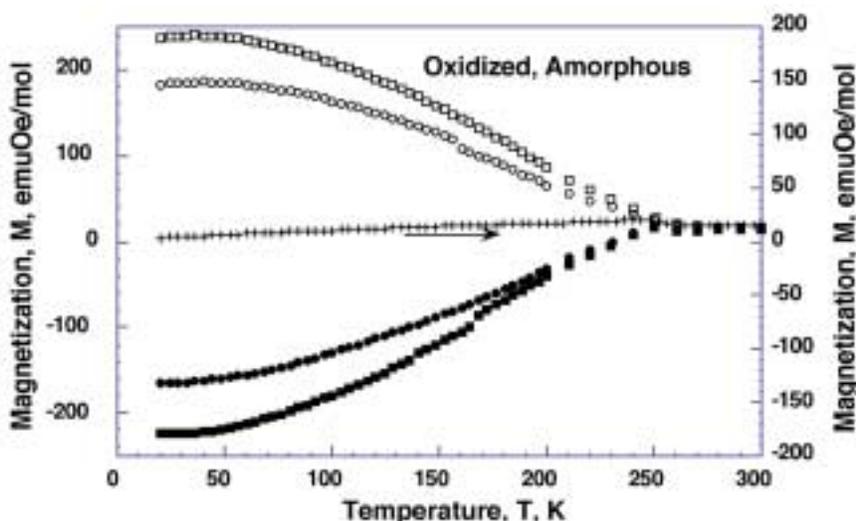


Fig. 10. Temperature dependent magnetization of the amorphous film of $Cr^{III}[Cr^{III}(CN)_6]_{0.98}[Cr^{II}(CN)_6]_{0.02}$ zero field cooled (+) and field cooled in 5 Oe (o), -5 Oe (*), 10 Oe (o), -10 Oe (v) after 2 min oxidation at -0.2 V upon warming in a 5 Oe field.¹⁸

Table I. Summary of the saturation magnetization (M_s), critical temperatures (T_c), and coercive fields (H_{cr}), for representative molecule-based magnets.

Magnet	M_s , emuOe/mol	T_c , K	H_{cr} , Oe	Ref
β -2-(4'-Nitrophenyl)-4,4,5,5-tetramethyl-4,5-dihydro-1H-imidazol-1-oxyl-3-N-oxide	5,600	0.6	8	3b
$[Fe^{III}Cp^*_2][TCNE]$ (Fig. 1)	16,300	4.8	1,000	2,3a
$[TDAE][C_{60}]$	5,585	-16	-0	3c
$[MnTPP][TCNE]\cdot 2(C_6H_5CH_3)$ (Fig. 5)	17,000	13	24,000	10a
$V^{II}_{0.42}V^{III}_{0.58}[Cr^{III}(CN)_6]_{0.86}\cdot 2.8H_2O$	840	315	25	14a
$V[TCNE]_x\cdot yCH_2Cl_2$ ($x \sim 2$; $y \sim 0.5$) (similar to Fig. 7)	10,000	~400	15	5

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About the Author

Joel S. Miller is a Distinguished Professor in the Department of Chemistry at the University of Utah. He is on the advisory boards of Inorganic Chemistry, Advanced Materials, Chemistry - European Journal, Journal of Materials Chemistry, CrystEngComm, and a member of the Inorganic Synthesis Corporation. He was the recipient of the 2000 American Chemical Society's Chemistry of Materials Award for his discoveries in the area of molecule-based magnets. He can be reached at: jsmiller@chem.utah.edu.