and at $\delta = 567$ for the tellurium atoms of the Te_6N_8 core. This is in accord with the idea of a synchronized change of location for the four $TeCl_4$ molecules on the Te_6N_8 surface. Detachment of the $TeCl_4$ molecules does not take place in this process; the ^{125}Te NMR signal of $TeCl_4$ in THF is at $\delta = 1036$.[19] Because of their disorder behavior the change of location of the $TeCl_4$ molecules is also indicated in the crystalline state, even at 223 K. In contrast, the THF molecules which are only loosely bonded are subject to quick exchange according to the ^1H NMR spectrum.

Received: May 20, 1998 [Z11886IE]
German version: *Angew. Chem.* **1998**, *110*, 3008–3010

Keywords: nitride · nitrogen · tellurium

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[13] Crystal structure determination of [**5** · 4THF] · 3.5THF ($C_{30}H_{60}Cl_{16}N_8O_{7.5}Te_{10}$, $M_r = 2496.06$): A colorless crystal (ca. $0.54 \times 0.54 \times 0.45$ mm) was analyzed with an area detector system (IPDS, Stoe) at $-50\,^{\circ}C$ with $Mo_{K\alpha}$ radiation ($\lambda = 71.069$ pm, graphite monochromator). Unit cell monoclinic, $a = 2291.7(2)$, $b = 1222.7(1)$, $c = 5356.0(5)$ pm, $\beta = 101.74(1)^{\circ}$; $V = 14694 \times 10^{-30}$ m^3, space group $I2/a$, $Z = 8$, $\rho_{calcd} = 2.257$ Mg m^{-3}, $\mu(Mo_{K\alpha}) = 4.53$ mm^{-1}, $F(000) = 9184$. Because of a rapid decline in intensity at increasing diffraction angle, data were only collected up to $\theta = 24^{\circ}$, $\Phi = 0-250^{\circ}$, $\Delta\Phi = 1^{\circ}$, $t = 1$ min per record. Of 55355 reflections collected, 11290 were independent ($R_{int} = 0.060$) and 7648 observed ($I \geq 2\sigma(I)$). Measurement at even lower temperature was not possible, since a phase transition apparently occurs at approximately 220 K. Below this temperature diffuse streaks occur along the nevertheless very long c axis with maxima corresponding to a double superstructure. Semiempirical absorption corrections (from equivalent reflections) were carried out, and the structure was solved by direct methods and refined against all F^2 data[14] by full matrix. The $TeCl_4$ molecules bonded to the Te_6N_8 core showed strong disorder. Three of them had to be described by split atom models, which in two cases showed also a shift component in addition to a rotational component. All atoms, just as those of the core, could be refined with individual anisotropic displacement parameters. The four THF molecules coordinated at the core could be refined without using split positions, but with large anisotropic displacement parameters. The 3.5 THF molecules per molecule additionally fitted into gaps of the packing, one of which was situated on a twofold axis, and showed very strong motion and/or disorder. A model refined with split positions for two THF molecules converged at $wR_2 = 0.1182$ (all reflections) and $R = 0.0404$ (observed reflections). Because of the less than satisfactory possibility of describing the electron density blurred over this solvent range, its contribution to the structure factors was calculated by way of Back-Fourier transformation[15] and subtracted from the data set. Thereafter, the main structure [**5** · 4THF] could be refined with clearly improved accuracy ($wR_2 = 0.0901$, $R = 0.0348$, residual electron density $0.95/-0.79$ e Å^{-3}). The geometrical details documented here refer to this refinement. The structure determined at 223 K has the character of a high-temperature form in which the solvate molecules and $TeCl_4$ groups show high mobility or disorder. As documented in the quite physically meaningful shapes of displacement ellipsoids ($U_{eq} = 0.054-0.083$ Å^2) of all atoms of the Te_6N_8 core, its orientation is not noticeably involved. Thus, no significant influence on the geometry is expected. Crystallographic data (excluding structure factors) for the structure reported in this paper (those obtained by Back-Fourier transformation as well as those obtained according to the disorder model) have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-101663 and CCDC-101664. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB21EZ, UK (fax: (+44)1223-336-033; e-mail: deposit@ccdc.cam.ac.uk).

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Langmuir–Blodgett Films of Single-Molecule Nanomagnets**

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The discovery that individual molecules can act as magnets of nanometer size is a very recent one.[1, 2] The most thoroughly studied single-molecule magnets are the mixed-valence manganese clusters $[Mn_{12}O_{12}(carboxylate)_{16}]$ (carboxylate = acetate,[1, 3] propionate,[4] benzoate,[5] and 4-methylbenzoate[6]), referred to here as Mn_{12}. The current excitement for this kind of magnetic cluster is primarily due to

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[**] This work was supported by the Ministeno de Educación y Cultura (Grant No. MAT98-0880) and the EU. M.C.-L. thanks the Generalitat Valenciana for a predoctoral fellowship. We thank the Spanish CICYT and the Generalitat Valenciana for the financial support to purchase the SQUID magnetometer.

the presence of a large magnetic hysteresis comparable to that observed in hard magnets.[1b] This provides the possibility of molecular bistability, opening the way to store information at the molecular level. On the other hand, these nanomagnets provide unique examples for observing the tunneling of an electron through a potential barrier from one orientation to another, which is a fundamental question in physics.[7–9] An elegant approach to arranging molecules into well-organized multilayered films is to use the Langmuir–Blodgett (LB) technique.[10] Although for the most part this method has been used to arrange organic molecules, it was shown very recently that inorganic clusters[11] or even extended layered solids[12] can also be organized as LB films. Here we report the first LB films based on the single-molecule magnets Mn_{12}. These materials contain well-organized magnetic monolayers formed by Mn_{12} clusters that, as expected, exhibit a marked magnetic hysteresis.

In the Mn_{12} clusters the metallic core is formed by an internal tetrahedron of four manganese(IV) ions, each of spin $S = 3/2$, surrounded by eight manganese(III) ions of spin $S = 2$ (Figure 1).[1a] In the crystalline state these neutral clusters exhibit hysteresis with coercive fields as large as 1.5 T at 2 K.[1b] This hysteresis has a molecular origin. It is due to the magnetic relaxation properties of the cluster. In fact, exchange interactions within the cluster results in a ground state with large spin ($S = 10$) that encounters a thermal barrier for reversal of the direction of magnetization along the uniaxial magnetic axis. This leads to extremely slow relaxation times at very low temperature.

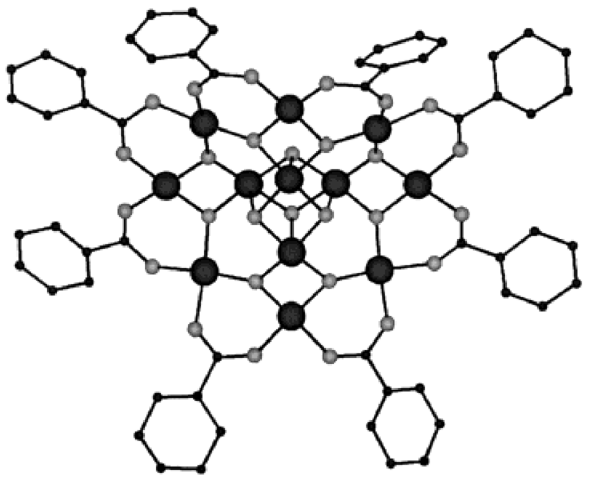

Figure 1. Schematic view of the $[Mn_{12}O_{12}(benzoate)_{16}]$ cluster from the axial viewpoint showing the equatorial phenyl rings. The four water molecules and the eight axial phenyl groups have been omitted for clarity. The larger dark circles represent the manganese ions, the light gray circles the oxygen atoms, and the small black circles the carbon atoms.

In view of the interesting results obtained in the building-up of LB films of molecular metal oxide clusters (i.e., polyoxometalates),[11] extension of the LB technique to the Mn_{12} clusters appears very desirable for fabricating organized films of the aforementioned single-molecule magnets. Two Mn_{12} clusters were used: the acetate and the benzoate derivatives. Upon spreading pure sample at the gas–water interface, the two manganese clusters do not form a stable Langmuir film. Therefore, a mixture of these complexes and a lipid is needed to obtain a monolayer. Behenic acid $CH_3(CH_2)_{20}COOH$ or behenic alcohol $CH_3(CH_2)_{20}CH_2OH$ was used as matrix. In the case of behenic alcohol, Brewster angle microscopy reveals that Mn_{12} clusters form crystallites on the top of the organic layer. In contrast, this optical technique suggests that an homogeneous monolayer is formed upon use of behenic acid as matrix. The resulting molecular system is clearly two-dimensional at the gas–water interface. Furthermore, the Langmuir film is stable over time if the lipid:cluster ratio is high enough (typically 5 or higher). When behenic acid is used as matrix, transfer of the monolayer onto a solid hydrophobic substrate is easily achieved at a surface pressure of 30 $mN\,m^{-1}$. A Y-type LB film is then obtained with a transfer ratio close to 0.90–0.95.

The structure of these brown LB films was investigated by IR spectroscopy and X-ray diffraction. Besides the bands at 2920, 2851, 1472, and 1463 cm^{-1} assigned to the stretching and scissoring modes of the C–H bonds, the IR spectra present a small broad band at about 1550 cm^{-1} which becomes clearly detectable when the concentration of clusters is high enough in the monolayer. This band comes from the carboxylate group and demonstrates that the cluster is indeed diluted within the multilayers. Whatever the complex concentration may be, this band presents no clear dichroism. Such a result is not surprising in view of the quasi-isotropic distribution of the carboxylate groups around the Mn_{12} core. In contrast, the IR bands associated with the alkyl chains are dichroic. The tilt angle of the hydrophobic tails is then easily evaluated: It is close to 22–24° for both the pure matrix and the organic/inorganic mixtures. As a result, the manganese cluster does not strongly modify the matrix organization.

X-ray diffraction shows a continuous evolution as a function of the concentration of the complex within the pristine layer. One to three Bragg peaks are observed for low concentrations of the benzoate cluster (lipid:cluster ratio higher than 20). In this case, the Bragg peaks have the same position as those for the LB films of pure behenic acid and corresponds to a periodicity of about 53.5 Å. However, the intensity of each peak is lower and its width is much larger than in the pristine film. This indicates that the organic layer structure is still maintained, but the coherence length has decreased in the material due to the insertion of the inorganic cluster.[13] Interestingly, when the concentration of the complex is large enough (typically for a lipid:cluster ratio that is lower than or equal to 10), the Bragg peaks associated to the behenic acid periodicity are very weak, but a new peak is clearly observed which corresponds to a new periodicity of 72 ± 2 Å. Taking into account that the disk shape of the benzoate cluster has a van der Waals diameter of about 22.5 Å and a thickness of about 18 Å,[5] such a distance corresponds to the intercalation of a monolayer of clusters in between a bilayer of behenic acid. In view of these distances the clusters should be preferably oriented with the axial direction perpendicular to the plane of the layer. We can conclude that the degree of organization of the clusters within the films is strongly dependent on the lipid:cluster ratio. For the higher ratios isolated clusters or partial monolayers of clusters are obtained, while for ratios in the range of 10:1 to 5:1 lamellar structures with the clusters organized in well-defined monolayers are observed (Figure 2).

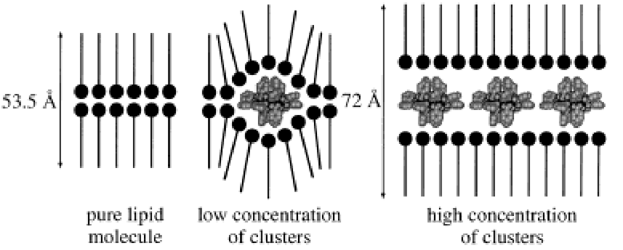

Figure 2. Schemes of the Y-type Langmuir–Blodgett films formed when the lipid:cluster ratio is varied.

The magnetic properties of the LB films were measured after deposition of about 300 layers onto a diamagnetic mylar substrate. Magnetic susceptibility data have provided information on the composition of the LB films. By comparison with the parent crystalline salt, one can calculate how many Mn_{12} clusters are trapped within the LB film. As the area of the substrate and the number of deposited layers is known, it is straightforward to estimate the density of clusters within a layer and the effective diameter per cluster. Thus, in the 10:1 and 5:1 films the estimated size of the benzoate cluster within the layer gives an effective diameter of about 22 ± 2 Å, which closely fits to the real value (see above), indicating that these clusters are close packed. For films with lower concentrations of clusters (ratio 20:1), the effective diameters are larger than 80 Å, which is in agreement with the dispersion of the clusters in between the organic layers, as suggested from X-ray data.

The most remarkable magnetic result comes from the magnetization data obtained as a function of the applied magnetic field below and above the blocking temperature (the temperature below which hysteretic behavior is observed) of the cluster (4 K). As for the parent crystalline salt, the benzoate film shows at 2 K a marked hysteresis loop (Figure 3) with a coercive field of about 0.1 T (0.06 T for the acetate film), which vanishes as the temperature is increased to 4 K (see inset). However, in contrast with the crystals, the magnetization at 5 T is far from saturation. This result is general for all the investigated films. We also observe that the shape of the loop depends on the orientation of the film with respect to the applied magnetic field H (Figure 3). Thus, when H is parallel to the plane of the magnetic monolayer, the loop is softer than when H is perpendicular. Such anisotropy constitutes further support of the preferential orientation of the clusters within the layers (with the uniaxial magnetic axis of the cluster perpendicular to the monolayer). On the other hand, the magnetic features remain completely unchanged when the film is cooled in the presence of a polarizing field of 5 T. This indicates that, in contrast to what has been observed for crystalline powders and frozen glasses,[3] in the LB films the field is completely unable to orient the clusters. Finally, the coercive field at 2 K is reduced by an order of magnitude when we pass from the crystal to the film. Such a difference is surely related to the different spin dynamics of the Mn_{12} cluster in these two media, as it is submitted to different environments and packings. In fact, the magnetic relaxation in the Mn_{12} cluster can be strongly affected by its surroundings. For example, the presence of paramagnetic species around the cluster leads to an enhancement of the relaxation of the magnetization.[14]

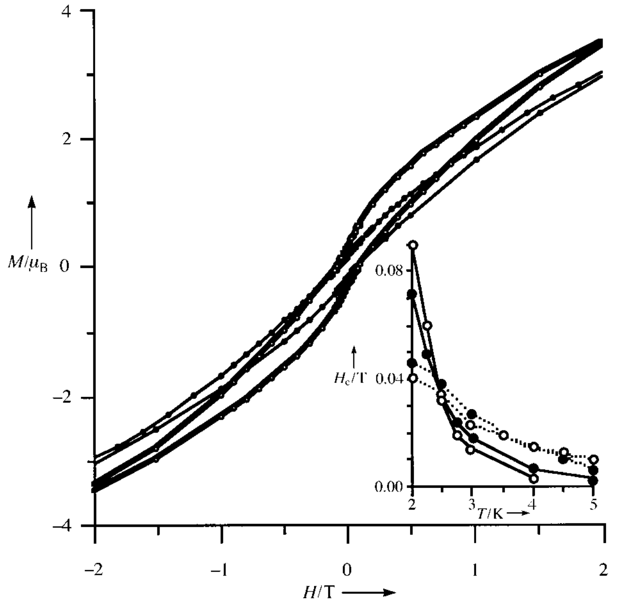

Figure 3. Hysteresis loops at 2 K of the Langmuir–Blodgett film prepared with the $[Mn_{12}O_{12}(benzoate)_{16}]$ cluster and behenic acid with a lipid:cluster ratio of 10:1; magnetic field H perpendicular (thick lines) and parallel (thin lines) to the film layer. Inset: Temperature dependence of the coercive field for the LB films of benzoate (solid lines) and acetate clusters (dotted lines). Filled and open circles refer to the orientation of the LB film with respect the applied field (parallel and perpendicular, respectively).

A precedent for a magnetic LB film showing a weak hysteresis with a coercive field of 0.02 T was recently reported.[12] However, in that case the hysteresis has a cooperative origin. It is a consequence of the magnetic ordering established in a polymeric manganese phosphonate layer, which in the solid state is a weak ferromagnet. Such an approach to magnetic LB films involves the difficult formation in solution of structurally ordered polymeric layers in order to construct films with a "magnetic memory". The molecular origin of the hysteresis in the LB films of single-molecule magnets reported here avoids these difficulties and opens a very efficient and simple way to design well-organized molecular multilayers with useful magnetic properties or combination of properties.

Experimental Section

The manganese complexes were synthesized following the published procedure.[5] Chemical analysis and magnetic measurements confirmed the identity and purity of the manganese complexes. Behenic acid and behenic alcohol were obtained from Fluka (purity > 99%). Chlorofrom (HPLC grade, Prolabo) was used as spreading solvent. Solutions of the manganese complexes (concentration ca. 10^{-4}–10^{-5} M) and the lipids (concentration ca. 10^{-3} M) were kept at −18 °C during the experiments in order to limit solvent evaporation. Built-up films were obtained by the vertical lifting method using an ATEMETA trough working at room temperature under a continuous flow of dried nitrogen. A stepwise compression of the monolayer was performed. After each increase of the surface pressure, a waiting time (10 to 30 minutes) allowed the system to reach equilibrium. Steps of 2 $\mathrm{mN\,m^{-1}}$ were usually chosen. The subphase was water (Q grade, Millipore) with a resistivity higher than 18 $\mathrm{M\Omega\,cm}$. The dipping speed was generally set to 0.5 $\mathrm{cm\,min^{-1}}$. Films were transferred onto optically polished calcium fluoride (precoated with three monolayers of behenic acid) for IR measurements, onto optically polished and silanized glass substrate for low-angle X-ray experiments, and onto a diamagnetic mylar sheet for magnetic SQUID measurements.

IR spectra were recorded on a FTIR 750 Nicolet spectrometer. To determine the orientation of the lipid molecules in the LB films, the linear dichroism in the IR region was used. In a first spectrum, the incident light was set perpendicular to the substrate normal; in a second, the incident IR beam formed an angle of 60° with the substrate normal. The out-of-plane dichroic ratio β for each band is then defined as the ratio of the IR absorption in both spectra. The angle between the substrate normal and the transition dipole moment can then be evaluated from β using a model already described.[15]

X-ray diffraction patterns were obtained using a conventional generator (Kristalloflex Siemens Ltd) delivering non-monochromatized line-focused $\mathrm{Cu_{K\alpha}}$ radiation. This beam passes through the sample, which is mounted vertically and oscillated during exposure. The integrated intensities of the Bragg reflections were collected by an INEL CPS 120 curved position-sensitive detector (with a resolution of 0.1° in 2θ) associated with an IBM computer for peak assignments.

The magnetic measurements were performed with a Quantum Design MPMS-XL SQUID magnetometer between 2 and 300 K. For such experiments, about 300 layers were deposited on a diamagnetic mylar sheet ($0.075 \times 5 \times 15$ mm). The procedure followed for the magnetic susceptibility measurement is described elsewhere.[11] For the magnetization measurements the hysteresis loops were recorded in about 5 hours. Each loop contains 180 points. To stabilize each data point a time of about 2 minutes was required. The errors in the magnetization data are in the range of 1–5%.

Received: May 13, 1998 [Z11851IE]
German version: *Angew. Chem.* **1998**, *110*, 3053–3056

Keywords: clusters · magnetic properties · monolayers · single-molecule magnets · thin films

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Efficient Suzuki-Type Cross-Coupling of Enantiomerically Pure Cyclopropylboronic Acids[**]

Shao-Man Zhou, Min-Zhi Deng,[*] Li-Jun Xia, and Ming-Hua Tang

Transition metal catalyzed Suzuki-type cross-coupling reactions are versatile and powerful methods for the formation of carbon–carbon bonds, because most of these reactions are stereospecific and offer many other advantages.[1] The proposed mechanism of the palladium-catalyzed cross-coupling[1a] contains a transmetalation reaction between organoboron compounds and palladium halide complexes as well as subsequent reductive elimination steps. Undoubtedly, understanding the change in stereochemistry of the chiral carbon atom upon use of a chiral alkylboron compound as reagent is of interest for organic chemists. Bäckvall and Åkermark reported that the configuration of the chiral carbon atom of the alkyl group was retained upon transmetalation from mercury to palladium.[2] Stille found that the transmetalation reaction of a chiral benzyltin compound with a palladium complex in hexamethyl phosporamide (HMPA) proceeded with inversion.[3] Hiyama and co-workers investigated the palladium-catalyzed cross-coupling reaction of chiral alkylsilanes with aryl triflates (triflate = trifluoromethanesulfonate). They determined that the stereochemistry is affected by both the reaction temperature and the nature of the solvent and could be controlled from almost complete retention to inversion by tuning these factors.[4] Recently, we prepared racemic cyclopropylboronic acids and subjected them to Suzuki-type coupling reactions with bromoarenes[5] or bromoacrylates.[6] Herein we report the palladium-catalyzed cross-coupling of optically active cyclopropylboron compounds.

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[**] We are grateful to the Natural Science Foundation of China for financial support.

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