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Communication

Near-Infrared Magnetic Circularly Polarized Luminescence and Slow Magnetic Relaxation in a Tetrazinyl-Bridged Erbium Metallocene

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ABSTRACT: The magnetic and magneto-optical properties of a tetrazinyl radical-bridged Er^{III} metallocene, $[(Cp^*{}_2Er^{III})_2(bpytz^{\bullet-})][BPh_4]$ (**1**; Cp* = pentamethylcyclopentadienyl, bpytz = 3,6-bis(3,5-dimethyl-pyrazolyl)-1,2,4,5-tetrazine), are reported. As confirmed by these studies strong Ln-rad coupling is achieved, with **1** exhibiting slow magnetic relaxation under a 1000 Oe dc field. The optical and magneto-optical profile of **1** is completed by both near-infrared (NIR) luminescence and magnetic circularly polarized luminescence (MCPL), representing the first example of NIR MCPL with Er^{III}.

Magnetic circular dichroism (MCD) is a powerful technique for simultaneous investigation of the ground and excited states of paramagnetic species, providing crucial insights into the polarization of electronic transitions.[1−4] MCD operates on the basis of the differential absorption (ΔA) of left and right circularly polarized light components induced by a magnetic field (H), which can be oriented parallel (positive H) or antiparallel (negative H) to the direction of light propagation. In general, the distinct polarization of electronic transitions results in certain MCD transitions mirroring the bulk magnetization behavior, while others exhibit different dependencies on the magnetic field.[5−7] Similarly to the MCD technique, magnetic circularly polarized luminescence (MCPL) relies on the differential emission (ΔI) of left and right circularly polarized light. This phenomenon arises from the distinct polarization of the Zeeman sublevels in the emitter state.[4,8−13] While MCPL was introduced in the 1970s, with only a few reported examples with lanthanides (Ln^{III}),[14−24] it remains significantly less explored than MCD. The lack of exploration in MCPL presents an opportunity for further research and development, particularly in investigating its utility in probing and understanding electronic and magnetic properties, as well as its potential for applications in fields such as optoelectronics and materials science.

To target materials capable of exhibiting magneto-optical properties of molecular origin, we have directed our focus toward lanthanide-based single-molecule magnets (SMMs). These molecule-based magnets have been highly sought after due to their potential applications in molecular electronics and spin-based devices.[25] The inherent physical properties arising from the 4*f* orbitals make Ln^{III} ions ideally suited for creating optically and magnetically active materials.[26−28] Recently, our group has investigated the magneto-optical properties of tetrazinyl radical-bridged Dy^{III}- and Tb^{III}-based SMMs *via* MCD, as a complementary technique to understand the magnetic properties of these strongly magnetically coupled entities.[29,30] However, the use of Dy^{III} and Tb^{III} ions did not allow us to investigate the magneto-optical properties of transitions connecting the coupled ground state to a "pure" *f* excited state, since all the accessible excited states for these ions overlap with the radical excited states. To overcome this, we have opted to study an Er^{III}-based system where the presence of *f*-*f* transitions in the near-infrared (NIR) region will not overlap with the radical excited states. Additionally, MCPL has never been employed to probe the NIR emission of Er^{III} ions, indicating untapped potential for its application in this spectral range. Thus, herein, we extend our studies on the magneto-optical properties of radical-bridged SMMs to the $[(Cp^*{}_2Er^{III})_2(bpytz^{\bullet-})][BPh_4]$ (**1**) complex (Figure 1; Cp* = pentamethylcyclopentadienyl, bpytz = 3,6-bis(3,5-dimethyl-pyrazolyl)-1,2,4,5-tetrazine). A combination of SQUID mag-

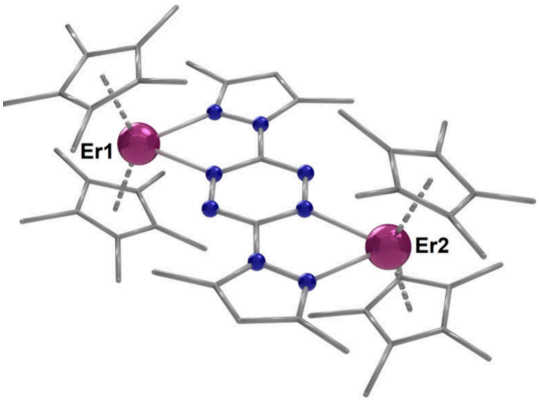

Figure 1. Molecular structure of **1**. For clarity, partial labeling and omission of the H atoms and the BPh_4^- moiety were employed. Color code: Er (magenta), C (gray), N (blue).

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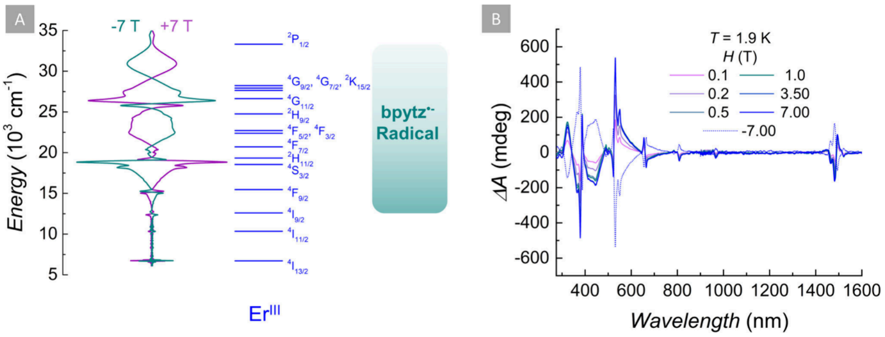

Figure 2. (a) Energy-level diagrams highlighting Er^{III} and radical transitions observed in the MCD spectra at 1.9 K. (b) MCD spectra of **1** with different applied magnetic fields.

netometry as well as MCD and MCPL is used to complete the intricate magneto-optical profile of **1**.

The dinuclear complex **1** was synthesized similarly to the previously reported family of "Ln_2-bpytz$^{\bullet-}$" complexes (Ln = Gd^{III}, Tb^{III}, Dy^{III}, Y^{III}),[31] to which it is isostructural as confirmed by single-crystal X-ray diffraction analysis. IR spectrum (Figure S1) and key structural features are given in the ESI (Figure S2 and S3; Tables S1 and S2).

To probe the magnetic behavior of **1**, direct current (dc) and alternating current (ac) magnetic susceptibility studies were undertaken (see ESI for the dc studies; Figures S4 and S5). To unravel the relaxation dynamics of **1**, ac in-phase (χ') and out-of-phase (χ'') susceptibility measurements were conducted in the 0.1−1500 Hz frequency range with varying magnetic fields (H_{dc} = 0−3000 Oe) at 3 K. When H_{dc} = 0 Oe, no peak of the χ'' susceptibility was observed for **1**, indicating the dominance of quantum tunneling of the magnetization (QTM). However, when $H_{dc} \geq 200$ Oe, a peak of the χ'' was observed (Figure S6), which was fitted to afford the relaxation times (τ, Tables S3 and S4). In the high frequency (HF) region, an increase of the τ was observed, reaching a maximum at 1000 Oe (Figure S7), while above this field the τ start to decrease. Consequently, frequency-dependent ac measurements at varying temperatures were undertaken at 1000 Oe (Figure S8). Between 5.4 and 2.6 K a frequency-dependent signal of the χ" was observed and fitted using a generalized Debye model. Below 2.4 K, a second process appeared in the low frequency (LF) region, which required a double generalized Debye model (Tables S5 and S6). Subsequently, a combination of direct and Raman was used to accurately fit the τ^{-1} vs T plot of the HF process, yielding the following best-fit parameters: $A = 8.39 \times 10^{-11}$ s^{-1} Oe^{-4} K^{-1}, $C = 3.39 \times 10^{-2}$ s^{-1} K^{-n} and n = 7.26 (Figure S9A and Table S7). For the LF region, despite the application of a static dc field, QTM is still present at lower temperatures, which is further supported by the fit of the τ^{-1} vs T data (Figure S9B; $\tau_{QTM} = 1.68 \times 10^{-1}$ s). This can be explained by the short intermolecular Er···Er distances (8.743(10) − 9.078(11) Å; Figure S3) which introduce dipole−dipole interactions at lower temperatures.[31,32] Given the structural similarities of **1** to the Tb_2- and Dy_2-bpytz$^{\bullet-}$ congeners,[31] it is reasonable to attribute any difference in their magnetic behavior to the intricate electronic structures of the individual Ln^{III} ions.

To understand the electronic structure and magneto-optical properties of **1**, MCD spectra at 1.9 K and various applied magnetic fields were obtained (Figure 2). A broad component, assigned to the radical excited states, is observed all over the visible spectral range, overlapping with some Er^{III} transitions, in agreement with the absorption spectrum (Figure S10). Additionally, some Er^{III} *f-f* transition bands with no overlap to radical components are observed at the NIR spectral range. It should be noted that all the spectral features arise from a combination of the radical and Er^{III} $^4I_{15/2}$ coupled ground states. The strong temperature dependence of the MCD spectra under a magnetic field of 0.1 T, confirms the predominance of the *C*-term character of the bands, as expected at low temperatures (Figure S11).[1,2]

The MCD spectra of **1** (Figure 2B) shows an increase of the signal with the applied magnetic field and its mirror-image when applying an antiparallel magnetic field. Aiming to further investigate the magneto-optical properties of **1**, we selected four different integrated areas connecting the ground state with the following excited states (Figures 3 and S12):

$$I_1\text{: bpytz}^{\bullet-} + Er^{III}\,{}^2K_{15/2},\ {}^4G_{7/2},\ {}^4G_{9/2},\ {}^4G_{11/2}(342 - 385\text{nm});$$

$$I_2\text{: bpytz}^{\bullet-}(570 - 620\text{nm});$$

$$I_3\text{: bpytz}^{\bullet-}(675 - 720\text{nm});$$

$$I_4\text{: }Er^{III}\,{}^4I_{13/2}(1420 - 1550\text{nm});$$

The field-dependence of the selected transitions was compared with the bulk magnetization curve obtained *via* SQUID magnetometry (Figure 3). By closely examining the

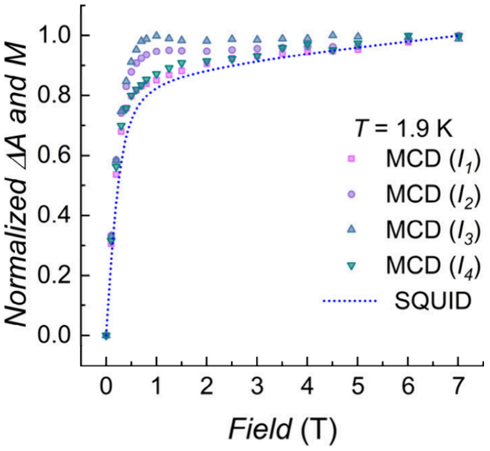

Figure 3. Comparison between the SQUID magnetization data (blue dotted line) and the MCD magnetization data (symbols) obtained at 1.9 K.

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obtained data, it is clear that whether the excited state includes radical contributions (I_1) or not (I_4), transitions connecting the coupled ground state to the Er^{III} excited states are essential to replicate the bulk magnetization. Transitions from the ground state to the radical states that do not overlap with the Er^{III} excited states (I_2 and I_3), possess different polarizations and, therefore, exhibit a distinct magnetization behavior, consistent with our previous observations for a Tb^{III} radical-bridged complex.[30] Our observations qualitatively suggest that I_1 and I_4 possess components within the Er manifolds with magnetic polarization in the same axis as the anisotropy axis (z axis magnetic polarization) and these components are the ones with stronger contributions to the MCD magnetization behavior.

To further understand the magneto-optical profile of radical-bridged complexes, we aimed to investigate the luminescence of **1**. The Er^{III} $^4I_{13/2} \rightarrow {}^4I_{15/2}$ emission occurs around 1550 nm, an area within the C-band telecom transparency windows. Therefore, Er^{III} complexes exhibiting NIR emission can find promising applications for free-space long-distance optical transmission and for light detection and ranging technologies.

Attempts to determine the optimal wavelength to excite **1** were unsuccessful, and we were not able to obtain an excitation spectrum when monitoring the $^4I_{13/2} \rightarrow {}^4I_{15/2}$ emission. However, when exciting **1** with a broadband source, restricted to the 400−900 nm range (see experimental details, Supporting Information), i.e., by completely exciting the radical excited states, we were able to obtain an emission at 1.9 K (Figure 4A). Due to the weak intensity, it was necessary to open the spectrograph emission slit, and thus, it was impossible to fully resolve the crystal field (CF) sublevels, even at 1.9 K. The weak intensity and the necessity to use a broadband excitation to detect any emission suggest that the energy is not efficiently transferred or that most of the transferred energy is dissipated by nonradiative mechanisms, in agreement with the strong overlap of the radical and Er^{III} excited states. It should be noted that the employed spectral resolution was around 10 nm, which does not allow for the resolution of the CF sublevels, but allows for the observation of spectral features on the MCPL spectra, as demonstrated by Guy and co-workers for Er^{III} CPL.[33]

Additionally, the presence of two crystallographically independent Er^{III} ions results in 16 CF sublevels at the ground state, contributing to the peaks broadening, hindering the solving of the ground state sublevel structure.

Intrigued by the emission at 1.9 K, the circularly polarized luminescence of **1** was probed in the presences of a magnetic field. Compared to other Ln^{III} ions with NIR emission,[34,35] Er^{III}-based NIR CPL emitters are rare, being first reported in 2022.[33,36−40] Additionally, there are no previous reports of Er^{III}-based NIR MCPL. Details about the experiment and calculations of ΔI and dissymmetry factors (g_{MCPL}) are discussed on the ESI. Upon application of a 1 T magnetic field, we were able to distinguish between the right and left circularly polarized light (rcp and lcp, respectively) (Figure S13), as well as record the MCPL spectra (Figure 4B) and calculate the g_{MCPL} (Figure 4C). The dominance of the positive signaled components indicates a lcp character for the emission when the magnetic field is parallel to the excitation beam. Reversing the magnetic field results in the mirror-image of the spectrum and confirms the MCPL nature of this emission. A maximum g_{MCPL} of 0.035 is observed for the component centered at 1575 nm. Although the g_{MCPL} values are much lower than the chiroptical g_{CPL} of the few reported Er^{III} complexes,[33,36−40] they are similar to g_{MCPL} for other Ln^{III} complexes ($10^{-3} - 10^{-2}$ T^{-1}).[21−23] Furthermore, the different nature of the CPL and MCPL phenomena does not allow for a direct comparison. Increasing the magnetic field to 5 T (Figure S14 and S15) results in a slight increase of ΔI and the maximum g_{MCPL} to 0.052 for the component centered at 1480.7 nm. It should be noted that some band components show a reduction in g_{MCPL} when increasing the applied magnetic field, suggesting that some of the overlapped components present a right circular polarization, resulting in

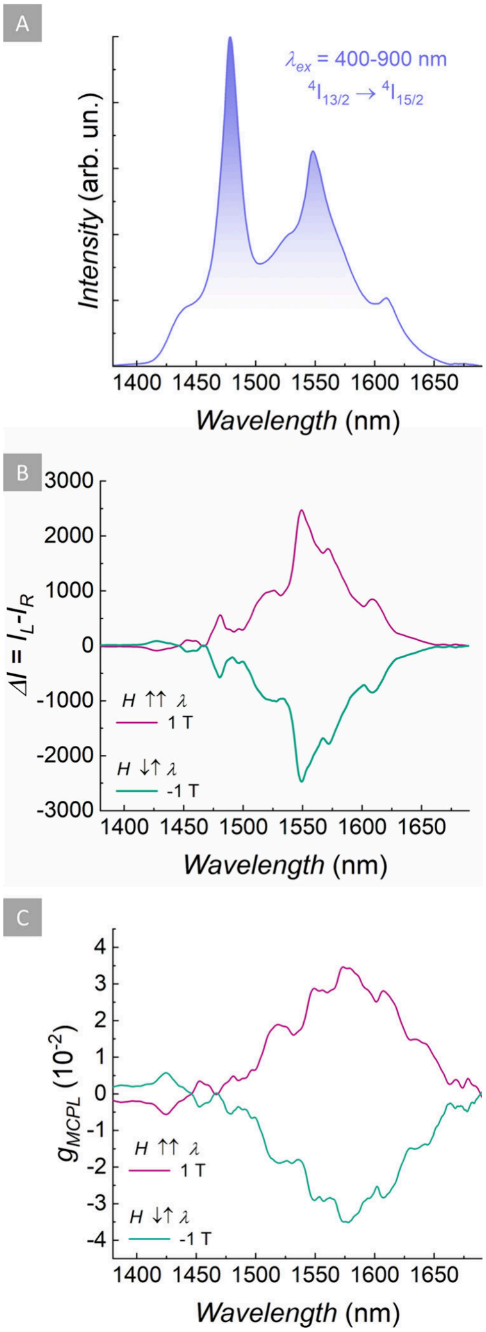

Figure 4. (a) Unpolarized emission spectrum of **1** obtained in the absence of an external magnetic field. (b) MCPL spectra of **1** under a broadband excitation. The purple and green lines represent the spectrum obtained with a +1 and a −1 T magnetic field, respectively. (c) g_{MCPL} calculated from (b). All spectra were obtained at 1.9 K.

a reversion of the direction trend. Future studies with stronger emitters could reveal the nature of the MCPL field dependence for different CF sublevels.

In summary, we have extended the series of “$\text{Ln}_2\text{-bpytz}^{\bullet -}$” complexes to include an Er^{III} analogue. Upon application of a static dc field, **1** exhibits slow magnetic relaxation, mediated by a combination of direct and Raman processes. MCD spectroscopy reveals that a transition connecting the coupled ground state to the Er^{III} excited states is necessary to reproduce the bulk magnetization in **1**, an inaccessible information for our previously studied Tb^{III} and Dy^{III} systems. Furthermore, **1** shows a weak emission in the NIR region at 1.9 K and represents the first Er^{III} complex exhibiting NIR MCPL, showing a maximum g_{MCPL} of 0.035 for the component centered at 1575 nm (1 T) and 0.052 for the component centered at 1480.7 nm (5 T). The presented results open new possibilities for the use of MCD and MCPL as means to provide new insights into the magneto-optical behavior of radical-bridged SMMs, as well as to facilitate the development of innovative materials with tailored optical and magnetic properties.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/jacs.4c10954.

> Experimental details, crystallographic data, as well as additional magnetic, MCD and MCPL data (PDF)

Accession Codes

Deposition Number 2333357 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via the joint Cambridge Crystallographic Data Centre (CCDC) and Fachinformationszentrum Karlsruhe Access Structures service.

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Notes

The authors declare no competing financial interest.

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