A Family of Lanthanide-Nitronyl Nitroxide Complexes: Syntheses, Crystal Structures and Magnetic Properties

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Table S. Selected bond lengths (Å) and angles (deg) for NITNapOMe

N(1)-O(1)	1.285(3)	N(2)-O(2)	1.284(3)
N(3)-O(3)	1.281(3)	N(4)-O(4)	1.282(3)
N(5)-O(5)	1.291(3)	N(6)-O(6)	1.284(3)
N(7)-O(7)	1.288(3)	N(8)-O(8)	1.287(3)
N(2)-C(7)-N(1)	107.3(2)	N(2)-C(7)-C(8)	126.8(2)
N(1)-C(7)-C(8)	125.8(2)	N(3)-C(25)-N(4)	109.4(2)
N(3)-C(25)-C(26)	125.5(3)	N(4)-C(25)-C(26)	125.1(2)
N(4)-C(22)-C(23)	108.4(2)	N(4)-C(22)-C(24)	106.5(2)
N(5)-C(43)-N(6)	107.7(2)	N(5)-C(43)-C(44)	126.6(2)
N(6)-C(43)-C(44)	125.7(2)	N(6)-C(40)-C(41)	109.6(2)
N(7)-C(61)-N(8)	107.8(2)	N(7)-C(61)-C(62)	125.5(2)

Symmetry transformations used to generate equivalent atoms:
#1 -x, y-1/2,-z+1/2 #2 -x, y+1/2,-z+1/2

Table S1. Selected bond lengths (Å) and angles (deg) for **1**

Pr-O(1)	2.418(4)	Pr-O(9)	2.487(4)
Pr-O(10)	2.424(4)	Pr-O(8)	2.490(4)
Pr-O(7)	2.428(4)	O(3)-N(3)	1.306(5)
Pr-O(3)	2.432(4)	N(1)-O(1)	1.317(6)
Pr-O(11)	2.450(4)	N(2)-O(2)	1.271(6)
Pr-O(12)	2.464(4)	O(4)-N(4)	1.267(6)
O(1)-Pr-O(10)	93.62(14)	O(11)-Pr-O(12)	70.68(13)
O(1)-Pr-O(7)	102.22(14)	O(1)-Pr-O(9)	145.01(14)
O(10)-Pr-O(7)	134.01(14)	O(10)-Pr-O(9)	69.92(13)
O(1)-Pr-O(3)	140.14(13)	O(7)-Pr-O(9)	72.68(14)
O(10)-Pr-O(3)	103.74(14)	O(3)-Pr-O(9)	74.79(13)
O(7)-Pr-O(3)	91.11(13)	O(11)-Pr-O(9)	124.28(13)
O(1)-Pr-O(11)	75.81(14)	O(12)-Pr-O(9)	140.52(13)
O(10)-Pr-O(11)	72.88(14)	O(1)-Pr-O(8)	72.49(13)
O(7)-Pr-O(11)	152.81(14)	O(10)-Pr-O(8)	73.75(14)
O(3)-Pr-O(11)	75.56(13)	O(7)-Pr-O(8)	70.59(13)
O(1)-Pr-O(12)	69.68(13)	O(3)-Pr-O(8)	146.70(13)
O(10)-Pr-O(12)	142.61(13)	O(11)-Pr-O(8)	131.67(13)
O(7)-Pr-O(12)	83.10(13)	O(12)-Pr-O(8)	127.34(13)
O(3)-Pr-O(12)	75.01(12)	O(9)-Pr-O(8)	73.27(13)

Symmetry transformations used to generate equivalent atoms:
#1 -x, y-1/2,-z+1/2 #2 -x, y+1/2,-z+1/2

Table S2. Selected bond lengths (Å) and angles (deg) for **2**

Gd(1)-O(5)	2.352(3)	Gd(1)-O(4)	2.410(3)
Gd(1)-O(10)	2.357(3)	Gd(1)-O(8)	2.411(3)
Gd(1)-O(9)	2.363(3)	N(1)-O(11)	1.265(5)
Gd(1)-O(2)	2.365(3)	N(2)-O(10)	1.297(4)
Gd(1)-O(6)	2.387(3)	N(3)-O(1)	1.264(4)
Gd(1)-O(7)	2.402(3)	N(4)-O(2)	1.300(4)
O(5)-Gd(1)-O(10)	93.30(10)	O(6)-Gd(1)-O(4)	124.05(10)
O(5)-Gd(1)-O(9)	136.70(9)	O(7)-Gd(1)-O(4)	138.51(9)
O(10)-Gd(1)-O(9)	101.81(11)	O(5)-Gd(1)-O(8)	74.02(10)
O(5)-Gd(1)-O(2)	103.82(10)	O(10)-Gd(1)-O(8)	72.54(10)
O(10)-Gd(1)-O(2)	139.04(9)	O(9)-Gd(1)-O(8)	72.41(10)
O(9)-Gd(1)-O(2)	90.82(10)	O(2)-Gd(1)-O(8)	147.82(9)
O(5)-Gd(1)-O(6)	71.53(10)	O(6)-Gd(1)-O(8)	131.31(9)

Symmetry transformations used to generate equivalent atoms:

#1 -x, y-1/2,-z+1/2 #2 -x, y+1/2,-z+1/2

Table S3. Selected bond lengths (Å) and angles (deg) for **3**

Tb(1)-O(17)#1	2.342(2)	Tb(2)-O(7)	2.316(2)
Tb(1)-O(17)	2.342(2)	Tb(2)-O(4)	2.336(2)
Tb(1)-O(14)	2.344(2)	Tb(2)-O(9)	2.348(2)
Tb(1)-O(14)#1	2.344(2)	Tb(2)-O(1)	2.349(2)
Tb(1)-O(16)#1	2.373(2)	Tb(2)-O(6)	2.362(2)
Tb(1)-O(16)	2.373(2)	Tb(2)-O(5)	2.372(2)
Tb(1)-O(15)#1	2.388(2)	Tb(2)-O(3)	2.373(2)
Tb(1)-O(15)	2.388(2)	Tb(2)-O(2)	2.394(2)
O(17)#1-Tb(1)-O(17)	136.50(11)	O(7)-Tb(2)-O(9)	135.69(8)
O(17)#1-Tb(1)-O(14)	94.21(8)	O(4)-Tb(2)-O(9)	91.82(8)
O(17)-Tb(1)-O(14)	102.40(8)	O(7)-Tb(2)-O(1)	72.37(8)
O(17)#1-Tb(1)-O(14)#1	102.40(8)	O(4)-Tb(2)-O(1)	74.12(7)
O(17)-Tb(1)-O(14)#1	94.21(8)	O(9)-Tb(2)-O(1)	75.40(8)
O(14)-Tb(1)-O(14)#1	134.24(11)	O(7)-Tb(2)-O(6)	89.47(8)
O(17)#1-Tb(1)-O(16)#1	71.43(8)	O(4)-Tb(2)-O(6)	137.65(8)
O(17)-Tb(1)-O(16)#1	74.08(8)	O(9)-Tb(2)-O(6)	102.78(8)
O(14)-Tb(1)-O(16)#1	149.32(8)	O(1)-Tb(2)-O(6)	147.91(8)
O(14)#1-Tb(1)-O(16)#1	76.22(8)	O(7)-Tb(2)-O(5)	149.99(8)
O(17)#1-Tb(1)-O(16)	74.08(8)	O(4)-Tb(2)-O(5)	74.20(8)
O(17)-Tb(1)-O(16)	71.44(8)	O(9)-Tb(2)-O(5)	73.06(8)
O(14)-Tb(1)-O(16)	76.22(8)	O(1)-Tb(2)-O(5)	133.91(8)
O(14)#1-Tb(1)-O(16)	149.32(8)	O(6)-Tb(2)-O(5)	72.59(8)
O(16)#1-Tb(1)-O(16)	73.82(11)	O(7)-Tb(2)-O(3)	75.38(8)
O(17)#1-Tb(1)-O(15)#1	72.84(8)	O(4)-Tb(2)-O(3)	72.58(8)
O(17)-Tb(1)-O(15)#1	150.57(8)	O(9)-Tb(2)-O(3)	148.86(8)
O(14)-Tb(1)-O(15)#1	71.99(8)	O(1)-Tb(2)-O(3)	123.09(7)
O(14)#1-Tb(1)-O(15)#1	72.83(8)	O(6)-Tb(2)-O(3)	74.81(8)
O(16)#1-Tb(1)-O(15)#1	125.45(8)	O(5)-Tb(2)-O(3)	76.76(8)
O(16)-Tb(1)-O(15)#1	131.52(8)	O(7)-Tb(2)-O(2)	73.19(8)
O(17)#1-Tb(1)-O(15)	150.57(8)	O(4)-Tb(2)-O(2)	146.09(8)
O(17)-Tb(1)-O(15)	72.84(8)	O(9)-Tb(2)-O(2)	69.22(8)
O(14)-Tb(1)-O(15)	72.83(8)	O(1)-Tb(2)-O(2)	73.98(8)
O(14)#1-Tb(1)-O(15)	71.99(8)	O(6)-Tb(2)-O(2)	75.53(8)
O(16)#1-Tb(1)-O(15)	131.52(8)	O(5)-Tb(2)-O(2)	122.65(8)
O(16)-Tb(1)-O(15)	125.45(8)	O(3)-Tb(2)-O(2)	136.46(8)
O(15)#1-Tb(1)-O(15)	77.99(12)	O(7)-Tb(2)-O(4)	107.39(8)

Symmetry transformations used to generate equivalent atoms:

#1 -x+2,y,-z+3/2

Table S4. Selected bond lengths (Å) and angles (deg) for **4**

Dy-O(12)	2.317(4)	Dy-O(11)	2.372(4)
Dy-O(2)	2.322(4)	Dy-O(8)	2.376(4)
Dy-O(7)	2.320(4)	N(1)-O(1)	1.259(7)
Dy-O(3)	2.327(4)	N(2)-O(2)	1.286(6)
Dy-O(10)	2.342(4)	N(3)-O(3)	1.293(6)
Dy-O(9)	2.366(4)	N(4)-O(4)	1.254(7)
O(12)-Dy-O(2)	93.36(16)	O(10)-Dy-O(9)	73.14(15)
O(12)-Dy-O(7)	137.41(15)	O(12)-Dy-O(11)	72.44(15)
O(2)-Dy-O(7)	101.65(16)	O(2)-Dy-O(11)	146.36(15)
O(12)-Dy-O(3)	103.63(15)	O(7)-Dy-O(11)	73.20(15)
O(2)-Dy-O(3)	138.70(15)	O(3)-Dy-O(11)	74.93(14)
O(7)-Dy-O(3)	90.87(15)	O(10)-Dy-O(11)	124.23(15)
O(12)-Dy-O(10)	71.32(15)	O(9)-Dy-O(11)	137.80(14)
O(2)-Dy-O(10)	76.23(16)	O(12)-Dy-O(8)	73.88(15)
O(7)-Dy-O(10)	150.92(15)	O(2)-Dy-O(8)	72.87(15)
O(3)-Dy-O(10)	74.20(15)	O(7)-Dy-O(8)	73.01(14)
O(12)-Dy-O(9)	143.39(15)	O(3)-Dy-O(8)	147.91(15)
O(2)-Dy-O(9)	69.74(15)	O(10)-Dy-O(8)	131.26(15)
O(7)-Dy-O(9)	78.92(15)	O(9)-Dy-O(8)	126.74(15)
O(3)-Dy-O(9)	74.48(14)	O(11)-Dy-O(8)	73.90(15)

Symmetry transformations used to generate equivalent atoms:

#1 -x, y-1/2,-z+1/2 #2 -x, y+1/2,-z+1/2

Table S5. Selected bond lengths (Å) and angles (deg) for **5**

Ho(1)-O(7)	2.308(3)	Ho(1)-O(2)	2.361(3)
Ho(1)-O(9)	2.319(3)	Ho(1)-O(5)	2.372(3)
Ho(1)-O(3)	2.327(4)	Ho(1)-O(4)	2.372(3)
Ho(1)-O(6)	2.341(4)	Ho(1)-O(1)	2.385(3)
O(7)-Ho(1)-O(9)	140.19(10)	O(2)-Ho(1)-O(4)	135.71(12)
O(7)-Ho(1)-O(3)	92.03(12)	O(5)-Ho(1)-O(4)	72.64(11)
O(9)-Ho(1)-O(3)	97.04(12)	O(7)-Ho(1)-O(1)	74.10(10)
O(7)-Ho(1)-O(6)	103.80(11)	O(9)-Ho(1)-O(1)	72.09(11)
O(9)-Ho(1)-O(6)	92.85(12)	O(3)-Ho(1)-O(1)	72.16(11)
O(3)-Ho(1)-O(6)	141.40(11)	O(6)-Ho(1)-O(1)	145.87(11)
O(7)-Ho(1)-O(2)	73.61(11)	O(2)-Ho(1)-O(1)	73.11(11)
O(9)-Ho(1)-O(2)	76.95(11)	O(5)-Ho(1)-O(1)	134.47(11)
O(3)-Ho(1)-O(2)	144.89(11)	O(4)-Ho(1)-O(1)	127.62(11)
O(6)-Ho(1)-O(2)	73.71(11)	O(2)-Ho(1)-O(5)	124.07(11)
O(7)-Ho(1)-O(5)	72.55(11)	O(7)-Ho(1)-O(4)	144.03(11)
O(9)-Ho(1)-O(5)	147.25(10)	O(9)-Ho(1)-O(4)	75.14(11)
O(3)-Ho(1)-O(5)	78.96(12)	O(3)-Ho(1)-O(4)	72.65(12)
O(6)-Ho(1)-O(5)	72.87(12)	O(6)-Ho(1)-O(4)	74.08(12)

Symmetry transformations used to generate equivalent atoms:

#1 -x, y-1/2,-z+1/2 #2 -x, y+1/2,-z+1/2

Table S6. Selected bond lengths (Å) and angles (deg) for **6**

Er(1)-O(4)	2.288(5)	Er(1)-O(5)	2.357(5)
Er(1)-O(8)	2.306(5)	Er(1)-O(11)	2.361(4)
Er(1)-O(3)	2.314(4)	N(1)-O(2)	1.269(9)
Er(1)-O(6)	2.321(5)	N(2)-O(3)	1.297(7)
Er(1)-O(12)	2.336(5)	N(3)-O(6)	1.284(7)
Er(1)-O(9)	2.354(5)	N(4)-O(7)	1.264(8)
O(4)-Er(1)-O(8)	141.56(18)	O(4)-Er(1)-O(5)	72.58(18)
O(4)-Er(1)-O(3)	97.55(18)	O(8)-Er(1)-O(5)	74.37(19)
O(8)-Er(1)-O(3)	92.42(17)	O(3)-Er(1)-O(5)	75.15(18)
O(4)-Er(1)-O(6)	92.28(18)	O(6)-Er(1)-O(5)	144.07(18)
O(8)-Er(1)-O(6)	103.36(18)	O(12)-Er(1)-O(5)	136.10(18)
O(3)-Er(1)-O(6)	140.27(18)	O(9)-Er(1)-O(5)	72.90(18)
O(4)-Er(1)-O(12)	144.70(18)	O(4)-Er(1)-O(11)	71.86(17)
O(8)-Er(1)-O(12)	73.73(18)	O(8)-Er(1)-O(11)	146.05(18)
O(3)-Er(1)-O(12)	76.90(18)	O(3)-Er(1)-O(11)	72.55(17)
O(6)-Er(1)-O(12)	73.10(19)	O(6)-Er(1)-O(11)	74.26(17)
O(4)-Er(1)-O(9)	78.62(18)	O(12)-Er(1)-O(11)	73.27(17)
O(8)-Er(1)-O(9)	73.34(17)	O(9)-Er(1)-O(11)	133.79(17)
O(3)-Er(1)-O(9)	147.47(19)	O(5)-Er(1)-O(11)	127.38(17)
O(6)-Er(1)-O(9)	72.22(18)	O(4)-Er(1)-O(5)	72.58(18)

Symmetry transformations used to generate equivalent atoms:

#1 -x, y-1/2,-z+1/2 #2 -x, y+1/2,-z+1/2

Table S7. Selected bond lengths (Å) and angles (deg) for **7**

Tm(1)-O(7)	2.276(3)	Tm(1)-O(4)	2.335(4)
Tm(1)-O(5)	2.294(4)	Tm(1)-O(6)	2.344(4)
Tm(1)-O(3)	2.299(3)	O(11)-N(4)	1.279(6)
Tm(1)-O(10)	2.306(3)	N(1)-O(2)	1.242(6)
Tm(1)-O(9)	2.325(4)	N(2)-O(3)	1.305(5)
Tm(1)-O(8)	2.335(4)	N(3)-O(10)	1.307(5)
O(7)-Tm(1)-O(5)	141.72(14)	O(7)-Tm(1)-O(4)	78.35(14)
O(7)-Tm(1)-O(3)	92.23(13)	O(5)-Tm(1)-O(4)	73.75(13)
O(5)-Tm(1)-O(3)	103.74(12)	O(3)-Tm(1)-O(4)	72.77(13)
O(7)-Tm(1)-O(10)	97.42(13)	O(10)-Tm(1)-O(4)	147.38(13)
O(5)-Tm(1)-O(10)	92.36(12)	O(9)-Tm(1)-O(4)	124.08(13)
O(3)-Tm(1)-O(10)	139.84(13)	O(8)-Tm(1)-O(4)	133.52(12)
O(7)-Tm(1)-O(9)	145.12(13)	O(7)-Tm(1)-O(6)	73.40(13)
O(5)-Tm(1)-O(9)	73.16(13)	O(5)-Tm(1)-O(6)	73.69(13)
O(3)-Tm(1)-O(9)	73.01(13)	O(3)-Tm(1)-O(6)	144.83(14)
O(10)-Tm(1)-O(9)	77.08(12)	O(10)-Tm(1)-O(6)	74.86(13)
O(7)-Tm(1)-O(8)	71.93(13)	O(9)-Tm(1)-O(6)	135.05(12)
O(5)-Tm(1)-O(8)	145.83(13)	O(8)-Tm(1)-O(6)	128.09(13)
O(3)-Tm(1)-O(8)	73.54(12)	O(4)-Tm(1)-O(6)	72.94(13)
O(10)-Tm(1)-O(8)	72.71(12)	O(9)-Tm(1)-O(8)	73.59(12)

Symmetry transformations used to generate equivalent atoms:
#1 -x, y-1/2,-z+1/2 #2 -x, y+1/2,-z+1/2

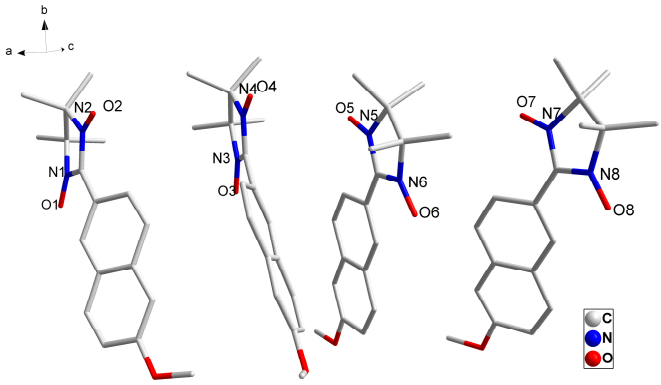

Figure S1. The asymmetric unit of the nitronyl-nitroxide radical (NITNapOMe). All hydrogen atoms are omitted for clarity.

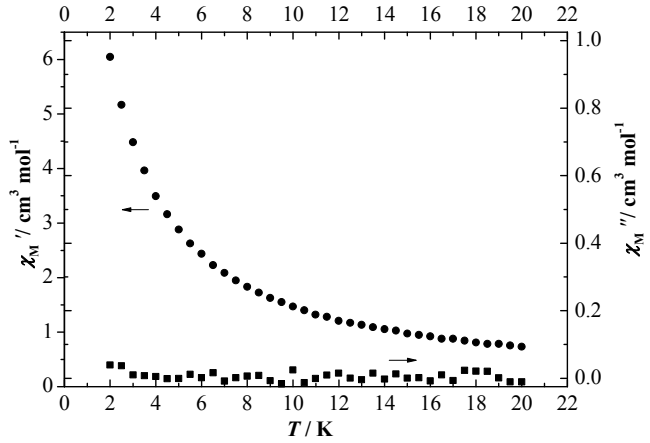

Figure S2. Temperature dependence of the in-phase (χ' round points) and out-of-phase (χ'' square points) components of the alternating-current susceptibility for complex Tb(**3**) under 1000 *dc* field at the frequency of 997 Hz.

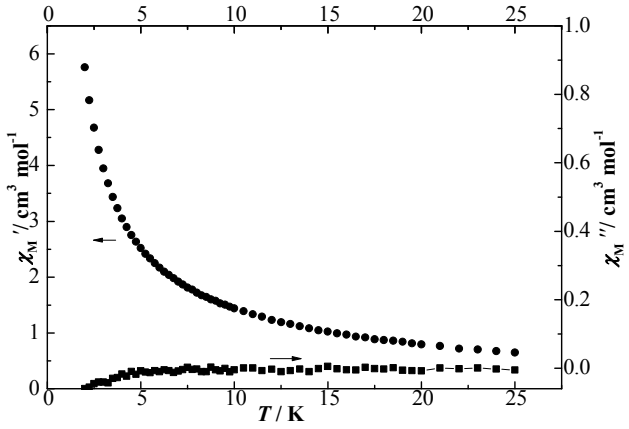

Figure S3. Temperature dependence of the in-phase (χ' round points) and out-of-phase (χ'' square points) components of the alternating-current susceptibility for complex Dy(**4**) under zero *dc* field at the frequency of 997 Hz.

Obviously, a strictly theoretical treatment of magnetic properties for such the system can not be carried out because of the large anisotropy of the Ln(III) ions. However, to obtain a rough quantitative estimate of the magnetic interaction parameters between paramagnetic species (Ln(III) and radical) in the mononuclear tri-spin system, we assumed that the total magnetic susceptibility χ_{total} is given by the sum of the isolated Ln(III) ion and two radicals (Eq 1).

The Ln(III) ion may be assumed to exhibit a splitting of the m_j energy levels ($\hat{H} = \Delta \hat{J}_z^2$) in an axial crystal field [1]. Thus χ_{Tb}, χ_{Dy}, χ_{Tm} can be described as Eqs (3)-(5), respectively.

$$\chi_{total} = \chi_{Ln} + 2\chi_{Rad} \quad (1)$$

$$\chi_{Rad} = \frac{Ng_{Rad}^2\beta^2}{3kT}\frac{1}{2}\left(\frac{1}{2}+1\right)(g_{Rad}=2) \quad (2)$$

$$\chi_{Tb} = \frac{2Ng^2\beta^2}{kT}\left[\frac{36\exp\left(\frac{-36\Delta}{kT}\right)+25\exp\left(\frac{-25\Delta}{kT}\right)+16\exp\left(\frac{-16\Delta}{kT}\right)+9\exp\left(\frac{-9\Delta}{kT}\right)+4\exp\left(\frac{-4\Delta}{kT}\right)+\exp\left(\frac{-\Delta}{kT}\right)}{2\exp\left(\frac{-36\Delta}{kT}\right)+2\exp\left(\frac{-25\Delta}{kT}\right)+2\exp\left(\frac{-16\Delta}{kT}\right)+2\exp\left(\frac{-9\Delta}{kT}\right)+2\exp\left(\frac{-4\Delta}{kT}\right)+2\exp\left(\frac{-\Delta}{kT}\right)+1}\right] \quad (3)$$

$$\chi_{Dy} = \frac{Ng^2\beta^2}{4kT}\left[\frac{225\exp(-225\Delta/4kT)+169\exp(-169\Delta/4kT)+121\exp(-121\Delta/4kT)+81\exp(-81\Delta/4kT)+49\exp(-49\Delta/4kT)+25\exp(-25\Delta/4kT)+9\exp(-9\Delta/4kT)+\exp(-\Delta/4kT)}{\exp(-225\Delta/4kT)+\exp(-169\Delta/4kT)+\exp(-121\Delta/4kT)+\exp(-81\Delta/4kT)+\exp(-49\Delta/4kT)+\exp(-25\Delta/4kT)+\exp(-9\Delta/4kT)+\exp(-\Delta/4kT)}\right] \quad (4)$$

$$\chi_{Tm} = \frac{2Ng^2\beta^2}{kT}\left[\frac{36\exp\left(\frac{-36\Delta}{kT}\right)+25\exp\left(\frac{-25\Delta}{kT}\right)+16\exp\left(\frac{-16\Delta}{kT}\right)+9\exp\left(\frac{-9\Delta}{kT}\right)+4\exp\left(\frac{-4\Delta}{kT}\right)+\exp\left(\frac{-\Delta}{kT}\right)}{2\exp\left(\frac{-36\Delta}{kT}\right)+2\exp\left(\frac{-25\Delta}{kT}\right)+2\exp\left(\frac{-16\Delta}{kT}\right)+2\exp\left(\frac{-9\Delta}{kT}\right)+2\exp\left(\frac{-4\Delta}{kT}\right)+2\exp\left(\frac{-\Delta}{kT}\right)+1}\right] \quad (5)$$

In the expression, Δ is the zero-field-splitting parameter. The Zeeman splitting was treated isotropically for the sake of simplicity [2]. Then the zJ' parameter based on the molecular field approximation is introduced (Eq 6) to roughly simulate the magnetic interactions between the paramagnetic species.

$$\chi_M = \frac{\chi_{total}}{1-\left(2zJ'/Ng^2\beta^2\right)\chi_{total}} \quad (6)$$

Giving the best fitting parameters of g = 1.57, Δ = 0.55 cm^{-1}, zJ' = 0.26 cm^{-1} for Tb(III) complex **3** in the range of 30–300K. For Dy(III) complex **4**, the parameters are g = 1.38, Δ = -0.03 cm^{-1}, zJ' = 0.04 cm^{-1} in the range of 80–300K, for Tm(III) complex **7**, the parameters are g = 1.17, Δ = 3.57 cm^{-1}, zJ' = 2.01 cm^{-1} in the range of 50–300K. The positive zJ' values are indicative of the ferromagnetic interaction between the paramagenetic ions (Ln(III) and radicals) in the two mononuclear tri-spin systems.

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