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Magnetic Properties of Group 8 Metal−Metal-Bonded Porphyrin and Tetraazaporphyrin Dimers

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Solid state magnetic susceptibility data (2−350 K) are presented for the metal−metal doubly bonded dimers $[Ru(OEP)]_2$ (OEP = octaethylporphyrin), $[Ru(OETAP)]_2$ (OETAP = octaethyltetraazaporphyrin), and $[Os(OEP)]_2$. The data are consistent with strong zero-field splitting of the triplet ground state ($D \approx 240-630$ cm^{-1}). Variable temperature (200−300 K) ^{1}H NMR data are presented for $[Os(OEP)]_2$ and $[Ru(OETAP)]_2$ and for two heterodimers, [(OEP)RuRu(OETAP)] and [(OEP)OsRu(OETAP)].

Introduction

Relatively few magnetic studies have been reported for Ru and Os compounds, compared to the vast number of studies reported on iron compounds. Even fewer studies have been reported on Ru compounds containing metal−metal multiple bonds,[1,2] and no detailed studies on the magnetic susceptibility of osmium compounds containing metal−metal multiple bonds have been reported to date.

As shown in several previous papers,[2−4] the Cotton molecular orbital scheme,[5] which governs metal−metal-bonded complexes, correctly predicts that group 8 porphyrin dimers ($[M(POR)]_2$, M = Ru or Os, d^{12})[6] should have a ground state electronic configuration of $\sigma^2\pi^4\delta^{nb4}\pi^{*2}$. This configuration results in a metal−metal double bond and a triplet ground state ($^3A_{2g}$). In turn, the $S = 1$ ground state implies zero-field splitting effects.[7] This bonding scheme is consistent with the crystal structure,[4c] resonance Raman spectra,[8] ^{1}H NMR spectra,[4,9] and electrochemistry[9,10] reported for group 8 homodimers and heterodimers.

Here, we report and model the temperature-dependent solid state magnetic susceptibility data for three group 8 metal−metal-bonded dimers: $[Ru(OEP)]_2$, $[Ru(OETAP)]_2$, and $[Os(OEP)]_2$. We demonstrate that the temperature dependence of the magnetic data is adequately described by the zero-field splitting (ZFS) model which was proposed earlier[1] to account for the magnetic properties of carboxylate-bridged Ru_2^{4+} systems.

We also present variable temperature ^{1}H NMR data for the group 8 homometallic dimers, $[Os(OEP)]_2$ and $[Ru(OETAP)]_2$, and for two heterodimers, [(OEP)RuRu(OETAP)] and [(OEP)-OsRu(OETAP)]. The resonances of the heterodimers are assigned on the basis of the two-dimensional COSY ^{1}H NMR spectra. The NMR spectra observed for these dimers are consistent with the predictions of the ZFS model.

Experimental Procedure

Preparation of Dimers. The dimers were synthesized according to methods described in the literature.[4,9,11] All samples were handled under inert atmosphere. Elemental analysis was used to confirm the purity of the ruthenium homodimers. Although no other species were visible in the ^{1}H NMR spectrum of the osmium dimer, the susceptibility data revealed the presence of a small amount of ferromagnetic impurity (*vide infra*).

Mass Spectra and Elemental Analyses. Mass spectrometry of dimeric porphyrins was performed using inert atmosphere techniques at the Mass Spectrometry Facility of the University of California at Berkeley. Elemental analyses were performed by Midwest Microlab.

Magnetic Susceptibility Studies. Magnetic susceptibility measurements were performed under helium using either a Quantum Design MPMS5 SQUID susceptometer or an SHE 905 SQUID susceptometer.

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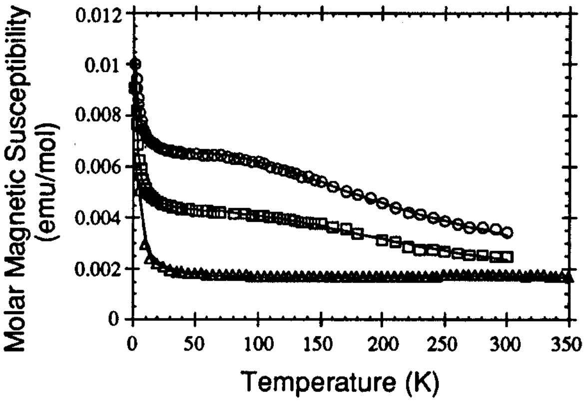

Figure 1. Fits (curves) to data (points) for the molar magnetic susceptibilities of $[Ru(OEP)]_2$ (○), $[Ru(OETAP)]_2$ (□), and $[Os(OEP)]_2$ (△).

Table 1. Diamagnetic Corrections Factors

compound	diamagnetic correction factor, ×10^6 emu/mol
H_2OEP	−481
H_2OETAP	−431
$[Ru(OEP)]_2$	−988
$[Ru(OETAP)]_2$	−888
$[Os(OEP)]_2$	−1027

For each compound, measurements were taken using a field strength of 5000 G and were collected over a temperature range of 2−300 K for the ruthenium dimers or 5−350 K for the osmium dimer.[12] Each raw data file was corrected for the diamagnetic contribution of both the sample holder and the compound to the susceptibility. The values for the diamagnetic susceptibilities of the free-base porphyrins,[13] along with diamagnetic corrections for the metals given in the literature,[7] were used to calculate the diamagnetic correction factors for the porphyrin and tetraazaporphyrin dimers (Table 1). The solid state magnetic susceptibilities of H_2OEP (-481×10^{-6} emu/mol) and H_2OETAP (-431×10^{-6} emu/mol) were determined over the same temperature range as were those of the dimers. The value for the diamagnetic correction factor obtained for H_2OEP is consistent with previous reports in the literature.[13] In addition, the susceptibility data for $[Os(OEP)]_2$ were corrected for the presence of a ferromagnetic impurity; the amount of impurity (64 ppm) was determined from the field dependence of the room temperature magnetic moment of $[Os(OEP)]_2$ by assuming that the field dependence was due to the presence of saturated iron.

The data were fit using a least-squares regression program. The following parameters were varied: the zero-field splitting (D/k), the parallel and perpendicular g values ($g_\parallel$ and $g_\perp$), and the fraction of impurity. The fits to three independent data sets were used to estimate the error bars for $[Os(OEP)]_2$.

Proton NMR Spectra. ^1H NMR spectra were recorded on either a Nicolet NT-300 or a Varian XL-400 Fourier transform spectrometer using benzene-d_6 or toluene-d_8 as solvent. Resonances in the ^1H NMR were referenced versus the residual ^1H signal of the deuterated NMR solvent and are given versus TMS. Variable temperature and two-dimensional COSY ^1H NMR spectra were recorded on a Varian XL-400 Fourier transform spectrometer using toluene-d_8 as solvent. For the variable temperature studies, each temperature was calibrated using an external MeOH sample; the COSY data were obtained at room temperature.[14]

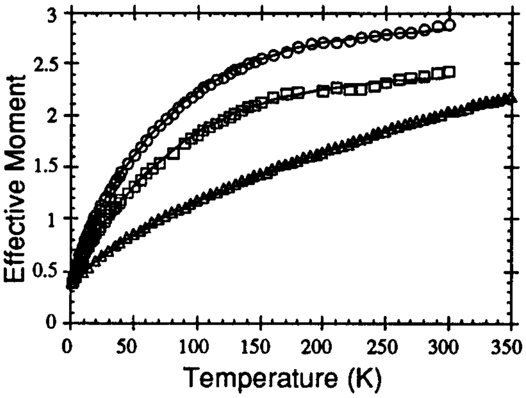

Figure 2. Fits (curves) to data (points) for the effective magnetic moment of $[Ru(OEP)]_2$ (○), $[Ru(OETAP)]_2$ (□), and $[Os(OEP)]_2$ (△).

Results and Discussion

Magnetic Susceptibility Studies. The solid state effective magnetic moment for each of the homodimers decreases and approaches zero as the temperature approaches absolute zero (Figure 2). This immediately reveals a zero-field splitting of the $S = 1$ ground state similar to that of the carboxylate-bridged ruthenium dimers, in which the $m_s = 0$ level lies 200−300 cm^{-1} below the $m_s = \pm 1$ states.[1] Fits were performed on the magnetic moment data (Figure 2). The zero-field splitting (ZFS) model[1,7] provides an acceptable fit to the magnetic data for $[Ru(OEP)]_2$, $[Ru(OETAP)]_2$, and $[Os(OEP)]_2$. This model gives a particularly good fit to the experimental magnetic susceptibility data when the presence of a small amount (∼2%) of a paramagnetic impurity is taken into account (eq 1, Figure 1).[15]

$$\langle\chi\rangle = \left(\frac{2N\mu_{\mathrm{B}}^{2}}{3kT}\right) \times \left[\frac{(g_{\parallel}^{2})\exp\left(\frac{-D}{kT}\right) + (g_{\perp}^{2})\left(\frac{2kT}{D}\right)\left(1 - \exp\left(\frac{-D}{kT}\right)\right)}{1 + 2\exp\left(\frac{-D}{kT}\right)}\right](1 - p) + p\chi_{\mathrm{imp}} \qquad (1)$$

where p = fraction of impurity

and $$\chi_{\mathrm{imp}} = \frac{g_{\mathrm{imp}}^{2}S'(S' + 1)}{8T}$$

That the ZFS model is an adequate model of the magnetic properties of the Ru and Os dimers is not unexpected. Indeed, the large spin−orbit coupling constants of the 4d and 5d metals, and the distortion from cubic symmetry of the crystal field due to the presence of the metal−metal double bond, should all result in large zero-field splitting effects. Furthermore, as predicted by Cotton,[16] the adequacy of the ZFS description for the Ru_2^{4+}

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(12) Preliminary solid state magnetic susceptibility studies were conducted on $[Ru(OEP)]_2$, $[Os(OEP)]_2$, and $[Ru(OETAP)]_2$ by R. S. McLean, J. S. Miller, and G.T.Y. at DuPont in collaboration with C. E. Barnes, J.P.C., and H.A.G. (see ref 2). Plots of these data were used to confirm the reproducibility of the results reported herein.

(13) Sutter, T. P. G.; Hambright, P.; Thorpe, A. N.; Quoc, N. *Inorg. Chim. Acta* **1992**, *195*, 131−132.

(14) See Supporting Information.

(15) The fits at low temperatures to the susceptibility data for the group 8 dimers are significantly improved if we account for the presence of an $S = {}^1/_2$ paramagnetic impurity. The percentage of impurity is small in all cases (≤2%), and the g_{imp} obtained from the fit is ∼2 for all three compounds. This impurity could be due to the presence of monomeric porphyrins or the presence of other metal ion impurities within the sample. However, neither of these impurities was detected in the ^1H NMR spectra or elemental analyses. We also attempted to improve the fit to the data by including a contribution to the susceptibility by temperature-independent paramagnetism (TIP). However, this model led to unreasonable values for the other parameters.

(16) Cotton, F. A., private communication to J.P.C., January 1989.

Table 2. Results of Fits to Experimental Data for the Magnetic Susceptibility of $[Ru(OEP)]_2$, $[Ru(OETAP)]_2$, and $[Os(OEP)]_2$, Assuming Zero-Field Splitting of a Triplet Ground State and Presence of Impurity with $S = {}^1/_2$ and $g_{imp} = 2.0$

	$[Ru(OEP)]_2$	$[Ru(OETAP)]_2$	$[Os(OEP)]_2$
zero-field splitting, D (cm^{-1})	240 ± 45	280 ± 65	630 ± 210
$g_{\parallel}$	2.1 ± 0.7	1.5 ± 0.6	2.9 ± 0.5
$g_{\perp}$	1.6 ± 0.2	1.4 ± 0.3	1.4 ± 0.4
fraction of impurity	0.01	0.01	0.02
sum of squares (fit)	2.6×10^{-7}	2.2×10^{-7}	2.1×10^{-7}
standard deviation (fit)	6.4×10^{-5}	6.0×10^{-5}	5.7×10^{-5}

systems supported by bridging ligands such as carboxylates[1] implies *a fortiori* its validity for metal−metal-bonded Ru(II) or Os(II) dimers that are devoid of bridging ligands.

The ZFS model also provides reasonable values for the parameters that were varied (D, $g_{\parallel}$, $g_{\perp}$, fraction of impurity) (Table 2). The values obtained for the ZFS in the ruthenium dimers ($D = 240 \pm 45$ cm^{-1} for $[Ru(OEP)]_2$, 280 ± 65 cm^{-1} for $[Ru(OETAP)]_2$) are close to those previously observed for other compounds containing ruthenium−ruthenium multiple bonds. As was observed in previous studies, the homologous porphyrin ($[Ru(OEP)]_2$) and tetraazaporphyrin ($[Ru(OETAP)]_2$) dimers exhibit similar behavior.[9,17] The ZFS observed for $[Os(OEP)]_2$ is somewhat larger (630 ± 210 cm^{-1}). The larger ZFS observed for $[Os(OEP)]_2$ relative to $[Ru(OEP)]_2$ presumably reflects the fact that osmium (5d) typically has a larger spin−orbit coupling parameter than does ruthenium (4d).

Previous attempts to detect an EPR signal for $[Ru(OEP)]_2$ were unsuccessful.[4c] As a result, the g values obtained for these dimers in the magnetic fits could not be verified using EPR spectroscopy. However, the average g values ($g_{av} = [(1/3)(g_{\parallel}^2 + g_{\perp}^2)]^{1/2} = 1.9$ for $[Os(OEP)]_2$, 1.8 for $[Ru(OEP)]_2$, and 1.4 for $[Ru(OETAP)]_2$) can be compared to those for related metal−metal-bonded compounds. Using a type of analysis similar to the one discussed herein, Cotton and co-workers obtained similar values of 1.55−1.86 for the g_{eff} of a series of metal−metal-bonded ruthenium 2-hydroxypyridinate complexes.[1]

NMR Studies. The NMR spectra of the group 8 dimers all exhibit three salient features that are readily predicted by the ZFS model.[18] (1) At room temperature, each compound exhibits narrow-line, paramagnetically shifted ^{1}H NMR resonances (Table 3).[4] (2) For any given resonance, a plot of isotropic shift versus inverse temperature exhibits small deviations from linearity.[14,19] (3) For most of these plots, a linear regression of the experimental points yields a line which does not intercept the chemical shift axis at zero.[14]

Table 3. ^{1}H NMR (300 or 400 MHz) Data of OEP and OETAP Dimers in C_6D_6 at Room Temperature under Nitrogen Atmosphere[4,9]

	CH_2CH_3	CH_2CH_3	H_{meso}
$[Ru(OETAP)]_2$	30.29 (m, 16H) 15.69 (m, 16H)	3.44 (t, 48H)	
$[Ru(OEP)]_2$	25.98 (m, 16H) 11.10 (m, 16H)	3.42 (t, 48H)	10.12 (s, 8H)
$[Os(OEP)]_2$	11.50 (m, 16H) 7.77 (m, 16H)	1.92 (t, 48H)	−1.02 (s, 8H)
[(OETAP)RuRu(OEP)]	34.66 (m, 8H) 24.80 (m, 8H) 18.26 (m, 8H) 10.98 (m, 8H)	3.75 (t, 24H) 3.39 (t, 24H)	12.73 (s, 4H)
[(OETAP)RuOs(OEP)]	14.32 (m. 8H) 13.70 (m, 8H) 9.37 (m, 8H) 8.53 (m, 8H)	2.59 (t, 24H) 2.20 (t, 24 H)	6.68 (s, 4H)

The solid state magnetic studies can also offer insight into the differences observed in the ^{1}H NMR spectra of the group 8 dimers. For instance, the larger ZFS of $[Os(OEP)]_2$ results in a smaller magnetic susceptibility for this dimer than for $[Ru(OEP)]_2$; as a result, smaller paramagnetic shifts are observed for $[Os(OEP)]_2$. In the case of the heterodimers, two sets of signals are observed,[14] each of which is distinct from the signals of the corresponding homodimers (Table 3). The sets of signals for the heterodimers are assigned on the basis of the COSY spectra.[14,20] Some of the heterodimer plots of isotropic shift as a function of inverse temperature also show deviations from Curie behavior.[14] These deviations reflect the strong ZFS predicted for these dimers on the basis of their similarity to the homodimers.

Concluding Remarks

Both the solid state magnetic behavior and the variable temperature solution ^{1}H NMR spectra for this series of group 8 dimers can be explained by the zero-field splitting of a triplet ground electronic state. The value of this zero-field splitting, D, is on the order of 240−280 cm^{-1} for $[Ru(OETAP)]_2$ and $[Ru(OEP)]_2$, which exhibit qualitatively and quantitatively similar behavior. The osmium dimer exhibits a larger zero-field splitting (630 cm^{-1}) than do the ruthenium dimers. This is probably due to a larger spin−orbit coupling constant for osmium, which results from the larger effective nuclear charge of the 5d metal.

The ^{1}H NMR spectra of these dimers and two group 8 heterodimers, [(OEP)RuRu(OETAP)] and [(OEP)OsRu(OETAP)], are paramagnetically shifted, and these shifts increase with decreasing temperature. The intercepts of the Curie plots of the variable temperature ^{1}H NMR data for these compounds deviate significantly from zero; this behavior reflects the effect of zero-field splitting on the temperature dependence of the molar susceptibility.

Taken together, these magnetic studies on ruthenium and osmium porphyrin and tetraazaporphyrin dimers indicate that the entire series of group 8 dimers display qualitatively similar behavior. Furthermore, the magnetic properties of these unbridged porphyrin dimers are strikingly similar to those previously reported for other metal−metal-bonded Ru_2^{4+} systems that contain bridging ligands.[1] This similarity suggests that, although the ligand system can have a significant effect on the properties of the system, the qualitative magnetic behavior is dictated by the presence of the metal−metal bond.

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(19) The isotropic shift is defined as the observed chemical shift minus the diamagnetic shift. The diamagnetic shifts are obtained from the ^{1}H NMR spectra of the analogous diamagnetic dimers, $[Rh(OEP)]_2$ and $[Rh(OETAP)]_2$ (ref 17). $[Rh(OEP)]_2$: -CH_2CH_3, δ 4.50 (m, 16H), 4.00 (m, 16H); -CH_2CH_3, δ 1.75 (t, 48H); H_{meso}, δ 9.17 (s, 8H). $[Rh(OETAP)]_2$: -CH_2CH_3, δ 4.43 (m, 16H), 3.80 (m, 16H); -CH_2CH_3, δ 1.87 (t, 48H).

(20) In the ^{1}H NMR spectra of the heterodimers, each set of resonances cannot be unambiguously assigned to a specific macrocycle (i.e., OEP versus OETAP). As a result, the chemical shift values for $[Rh(OEP)]_2$ were used as the diamagnetic shifts for both sets of resonances for each heterodimer.

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Supporting Information Available: Two-dimensional COSY ^{1}H NMR spectra of [(OEP)RuRu(OETAP)] and [(OEP)OsRu(OETAP)]; variable temperature ^{1}H NMR data for [Ru(OETAP)]$_2$, [Os(OEP)]$_2$, [(OEP)RuRu(OETAP)], and [(OEP)OsRu(OETAP)]; and room temperature one-dimensional ^{1}H NMR data for [(OEP)RuRu(OETAP)] and [(OEP)OsRu(OETAP)] (9 pages). Ordering information is given on any current masthead page.

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