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# SYNTHESIS AND NMR CHARACTERIZATION OF COBALT(III) COMPLEXES WITH TRIETHYLENETETRAMINE, 2,2-BIPYRIDINE AND 1,10-PHENANTHROLINE

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The compounds  $\alpha$ -*cis*[Co(trien)(bipy)]Cl<sub>3</sub> and  $\alpha$ -*cis*[Co(trien)(phen)]Cl<sub>3</sub> were synthesized and characterized by one- and two-dimensional NMR spectroscopy. Compared to  $\alpha$ -*cis*[Co(trien)(NO<sub>2</sub>)<sub>2</sub>]Cl, the proton spectra of these two complexes were spread to a wider spectral width. With the aid of two-dimensional experiments, it was possible to assign three multiplets to specific protons, and the remaining multiplet was found to arise from overlap of three separate resonances.

**Keywords:** Cobalt(III); Tetradentate ligand; Triethylenetetramine; NMR spectroscopy

## INTRODUCTION

Sargeson and Searle first studied cobalt complexes containing the tetradentate ligand triethylenetetramine nearly three decades ago [1,2]. Triethylenetetramine can be considered the prototype linear tetradentate ligand, and in the years that followed this initial work many complexes were isolated that contained tetradentate derivatives of triethylenetetramine [3–27]. Approaches include ligands with attached lengthened carbon chains joining the donor atoms [11–13], ligands with attached methyl groups [14–18] and ligands containing both nitrogen and sulfur donor atoms [19–27].

While NMR is a powerful spectroscopic tool, there are obstacles associated with the use of proton NMR spectroscopy to study these systems. Restricted rotation about the carbon–carbon bonds arising from coordination often removes the equivalence of protons attached to the same carbon. This is exacerbated in systems containing three-carbon linkages joining the donor atoms or complexes in which the ligand is coordinated asymmetrically. Furthermore, the proton chemical shifts are often very similar, causing the spectrum to be crowded into a very narrow spectral width.

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Modern NMR spectroscopy [28–35] offers not only higher-resolution spectra but also a wealth of two-dimensional experiments to establish proton connectivities and proton–carbon correlations. In previous work, high-field two-dimensional NMR spectroscopy was applied to a series of cobalt complexes containing tetradentate [36,37,39,40] and pentadentate ligands [36,38]. It was recently discovered [37,40] that using the bidentate ligands 2,2-bipyridine (bipy) and 1,10-phenanthroline (phen) to occupy the remaining two coordination sites in cobalt(III) complexes containing tetradentate ligands resulted in NMR spectra that were dramatically different than those previously observed. Proton spectra with separate multiplets were observed to occur, leading to far greater success in interpretation of the proton NMR spectra.

Based upon this information it was decided that the synthesis of analogous complexes with triethylenetetramine (trien) would be performed to determine if the same effects were observed. Since the complexes  $[\text{Co}(\text{trien})\text{Cl}_2]\text{Cl}$  and  $[\text{Co}(\text{trien})(\text{NO}_2)_2]\text{Cl}$  were originally studied by Sargeson and Searle [1,2], no two-dimensional NMR studies have been performed with triethylenetetramine complexes, and the complexes  $[\text{Co}(\text{trien})(\text{bipy})]\text{Cl}_3$  and  $[\text{Co}(\text{trien})(\text{phen})]\text{Cl}_3$  have not previously appeared in the literature.

## EXPERIMENTAL

All NMR experiments were performed on a Varian Mercury series 250 MHz NMR spectrometer.  $\text{D}_2\text{O}$  (99.9%) was obtained from Aldrich and used as the solvent for all NMR experiments. For  $^1\text{H}$  experiments, DSS was used as the chemical shift reference. For  $^{13}\text{C}$  experiments, 1,4-dioxane was used as the chemical shift reference (67.40 ppm). All experiments were carried out at room temperature and a concentration of 0.10 M.

The compound  $\alpha\text{-cis}[\text{Co}(\text{trien})(\text{NO}_2)_2]\text{Cl}$  was synthesized according to the procedure outlined by Sargeson and Searle [1]. The compound  $\alpha\text{-cis}[\text{Co}(\text{trien})(\text{phen})]\text{Cl}_3$  was synthesized as follows. 1,10-Phenanthroline monohydrate (2.00 g, 0.010 mol) was added to approximately 75 mL of absolute ethanol in a 250 mL sidearm flask, and 25 mL of deionized water was added. Triethylenetetramine (1.47 g, 0.010 mol) was then added to the solution, followed by 2.45 g (0.010 mol) of  $[\text{Co}(\text{H}_2\text{O})_6]\text{Cl}_2$  and 0.85 mL of concentrated HCl. The solution was placed in an ice bath and air was drawn vigorously through the solution for a period of 2 h. The solution was transferred to a 250 mL beaker and slowly concentrated on a hotplate to a volume of approximately 25 mL, at which time orange crystals began to form. The solution was returned to the ice bath, allowed to cool, and filtered through a sintered glass funnel. The product was washed with a few mL of 50% water/50% ethanol, followed by pure ethanol, and finally diethyl ether. Yield: 2.2 g.

The synthesis of  $\alpha\text{-cis}[\text{Co}(\text{trien})(\text{bipy})]\text{Cl}_3$  was carried out in a completely analogous way. 2,2-Bipyridine (2.00 g, 0.0128 mol) was added to a solution of 75 mL of ethyl alcohol and 25 mL of deionized water in a 250 mL sidearm flask. 3.04 g of  $[\text{Co}(\text{H}_2\text{O})_6]\text{Cl}_2$  (0.0128 mol) was then added to the solution, followed by 1.87 g of triethylenetetramine (0.0128 mol) and 1.00 mL of concentrated HCl. Air was drawn vigorously through the solution for a period of 2 h. The volume was reduced slowly to approximately 20 mL on a hotplate, at which time crystals began to form. The solution was then placed in an ice bath, allowed to cool, and filtered through a sintered glass funnel. The orange crystals

were washed with 50% ethanol/50% water, followed by pure ethanol, and finally diethyl ether. Yield: 1.60 g.

## RESULTS AND DISCUSSION

The complexes  $[\text{Co}(\text{trien})(\text{NO}_2)_2]\text{Cl}$ ,  $[\text{Co}(\text{trien})(\text{bipy})]\text{Cl}_3$  and  $[\text{Co}(\text{trien})(\text{phen})]\text{Cl}_3$  each yielded three carbon signals arising from the triethylenetetramine ligand, and the chemical shifts are summarized in Table I. Structural illustrations of  $[\text{Co}(\text{trien})(\text{bipy})]\text{Cl}_3$  and  $[\text{Co}(\text{trien})(\text{phen})]\text{Cl}_3$ , indicating the labeling scheme that will be used, are shown in Fig. 1.

TABLE I Summary of carbon data

Compound	C1	C2	C3
$[\text{Co}(\text{trien})(\text{NO}_2)_2]\text{Cl}$	44.7	59.9	57.4
$[\text{Co}(\text{trien})(\text{bipy})]\text{Cl}_3$	44.2	57.8	56.4
$[\text{Co}(\text{trien})(\text{phen})]\text{Cl}_3$	44.2	58.3	56.8

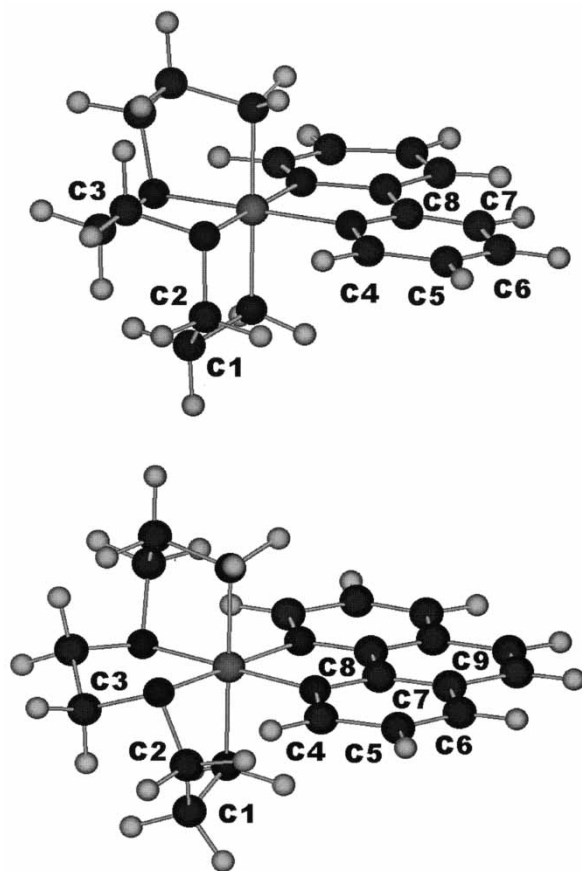


FIGURE 1 Structural illustrations of  $[\text{Co}(\text{trien})(\text{bipy})]\text{Cl}_3$  (top) and  $[\text{Co}(\text{trien})(\text{phen})]\text{Cl}_3$  (bottom).

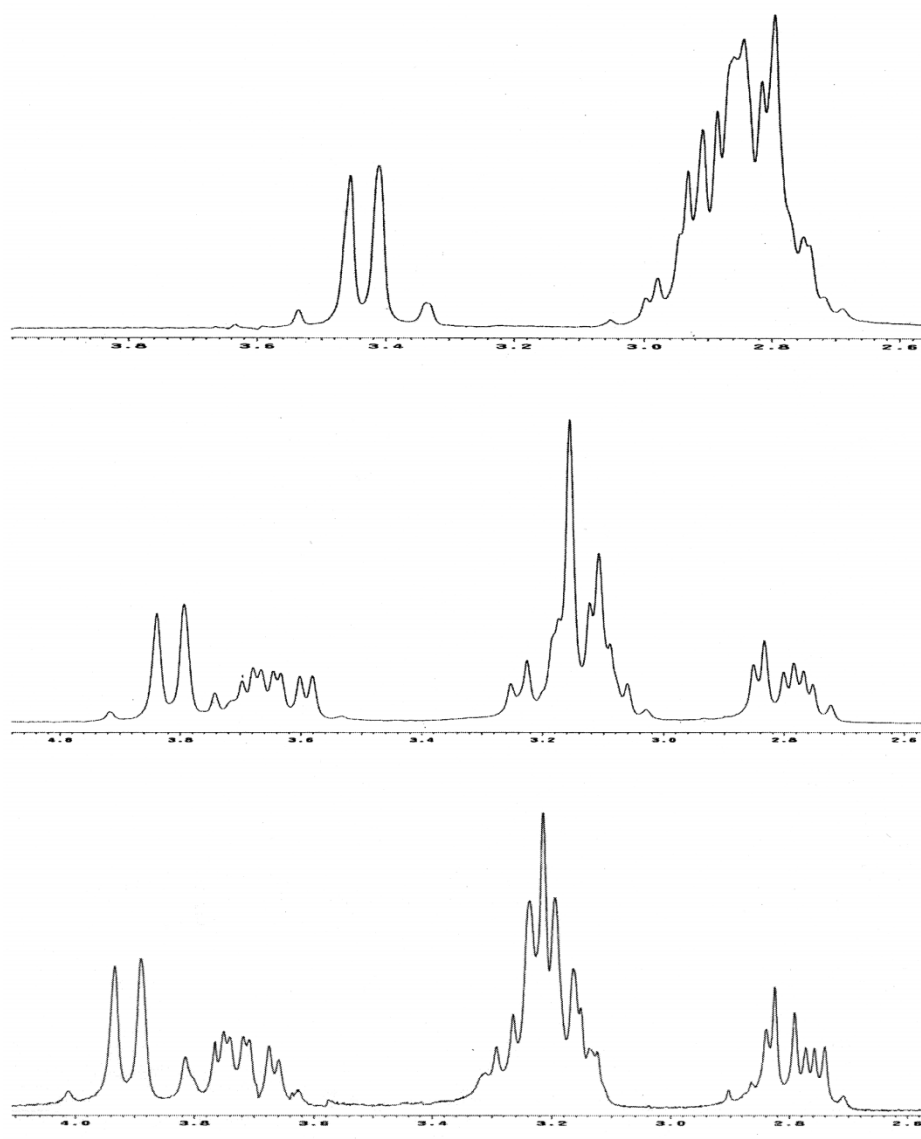


FIGURE 2 Proton spectra of  $[\text{Co}(\text{trien})(\text{NO}_2)_2]\text{Cl}$  (top),  $[\text{Co}(\text{trien})(\text{bipy})]\text{Cl}_3$  (middle) and  $[\text{Co}(\text{trien})(\text{bipy})]\text{Cl}_3$  (bottom).

The proton spectra of  $[\text{Co}(\text{trien})(\text{NO}_2)_2]\text{Cl}$ ,  $[\text{Co}(\text{trien})(\text{bipy})]\text{Cl}_3$  and  $[\text{Co}(\text{trien})(\text{phen})]\text{Cl}_3$  are shown in Fig. 2. Only two general regions can be defined in the proton spectrum of  $[\text{Co}(\text{trien})(\text{NO}_2)_2]\text{Cl}$ ; a doublet centered at approximately 3.42 ppm and a large multiplet centered at approximately 2.88 ppm. The doublet clearly belongs to the center ethylene linkage based upon multiplicity and symmetry considerations, but integration reveals this to correspond to only two protons. The remaining center ring protons, as well as the protons in the terminal ethylene linkages, must overlap to form the complex multiplet at 2.88 ppm.

TABLE II Summary of aliphatic proton data for [Co(trien)(bipy)]Cl<sub>3</sub>

<i>Chemical shift (ppm)</i>	<i>Integration</i>	<i>Assignment</i>
2.72–2.85	2.12	H1
3.00–3.25	6.28	H1, H2, H3
3.58–3.70	1.91	H2
3.79–3.83	2.00	H3

TABLE III Summary of aliphatic proton signals for [Co(trien)(phen)]Cl<sub>3</sub>

<i>Chemical shift (ppm)</i>	<i>Integration</i>	<i>Assignment</i>
2.74–2.84	1.97	H1
3.12–3.29	5.78	H1, H2, H3
3.66–3.81	1.91	H2
3.89–3.93	2.00	H3

The proton spectra of [Co(trien)(bipy)]Cl<sub>3</sub> and [Co(trien)(phen)]Cl<sub>3</sub> are very similar and will be discussed together. The chemical shift and integration data for these two complexes are summarized in Tables II and III, respectively. Both spectra can be divided into four general regions. First, both spectra contain a proton doublet that is centered at approximately 3.82 ppm in the case of [Co(trien)(bipy)]Cl<sub>3</sub> and 3.91 ppm in the case of [Co(trien)(phen)]Cl<sub>3</sub>. Due to chemical shift and symmetry considerations these were assigned to the center ring protons. However, as was the case for [Co(trien)(NO<sub>2</sub>)<sub>2</sub>]Cl, these doublets integrate to only two protons. Second, both spectra contain a multiplet located at 3.58–3.70 ppm for [Co(trien)(bipy)]Cl<sub>3</sub> and 3.66–3.81 ppm for [Co(trien)(phen)]Cl<sub>3</sub>. In both cases the multiplets integrate to approximately two protons. Both spectra contain a third multiplet, integrating to approximately six protons, located at 3.00–3.25 ppm for [Co(trien)(bipy)]Cl<sub>3</sub> and 3.12–3.29 ppm for [Co(trien)(phen)]Cl<sub>3</sub>. Finally, both spectra contain a fourth multiplet, integrating to approximately two protons, at 2.72–2.85 ppm for [Co(trien)(bipy)]Cl<sub>3</sub> and 2.74–2.84 ppm for [Co(trien)(phen)]Cl<sub>3</sub>.

The proton spectra need additional information for interpretation. Several key pieces of information were obtained from a heteronuclear correlation spectrum. The heteronuclear correlation spectrum for *α-cis* [Co(trien)(bipy)]Cl<sub>3</sub> is shown in Fig. 3. The carbon resonance at 44.2 ppm, which was assigned to the C1 carbon on the basis of chemical shift, showed correlations with the proton multiplets at 2.72–2.85 and 3.00–3.25 ppm, indicating that these multiplets arise, at least in part, from the H1 protons. Next, the carbon resonance at 56.4 ppm showed two correlations with the proton multiplets at 3.00–3.25 and 3.79–3.83 ppm. As the doublet at 3.79–3.83 ppm clearly belongs to the center ethylene linkage, the carbon resonance at 56.4 ppm is assigned to the C3 carbon. Finally, the carbon resonance at 57.8 ppm, assigned to the C2 carbon by a process of elimination, showed two proton correlations at approximately 3.00–3.25 and 3.58–3.70 ppm, indicating that these arise from the H2 protons.

The fact that each carbon resonance shows two separate and distinct proton correlations clearly demonstrates the nonequivalence of geminally related protons. This is not surprising due to the restricted rotation about the carbon–carbon bonds resulting

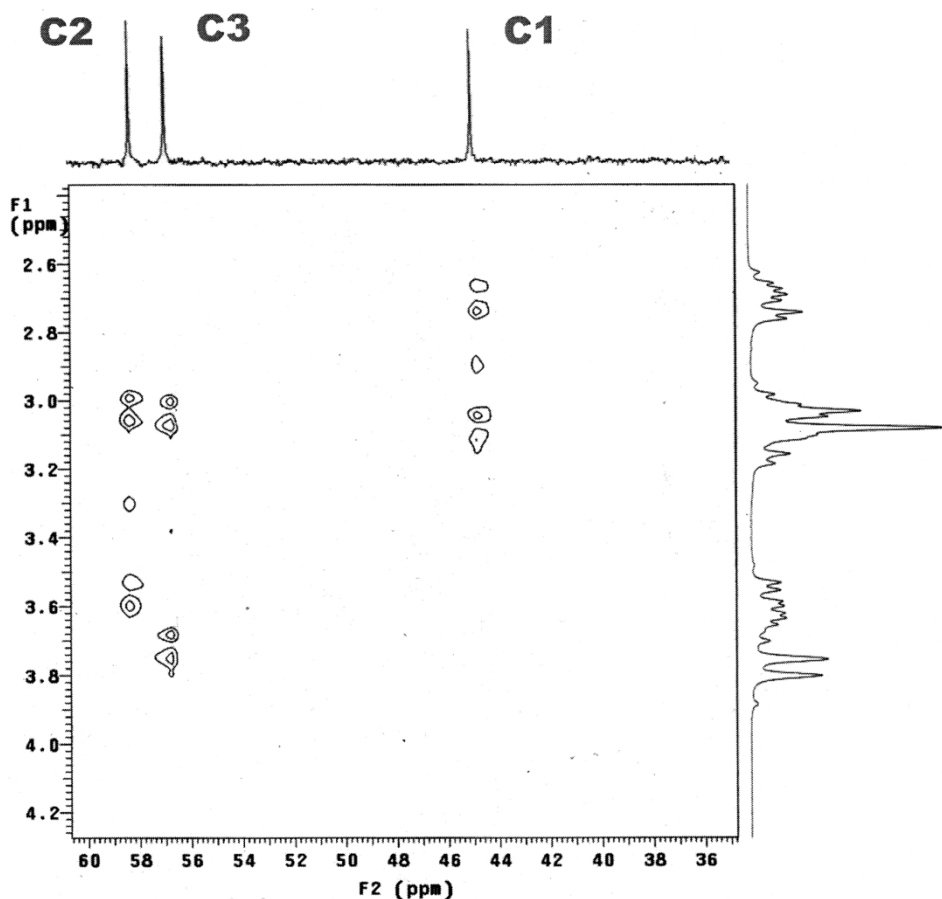


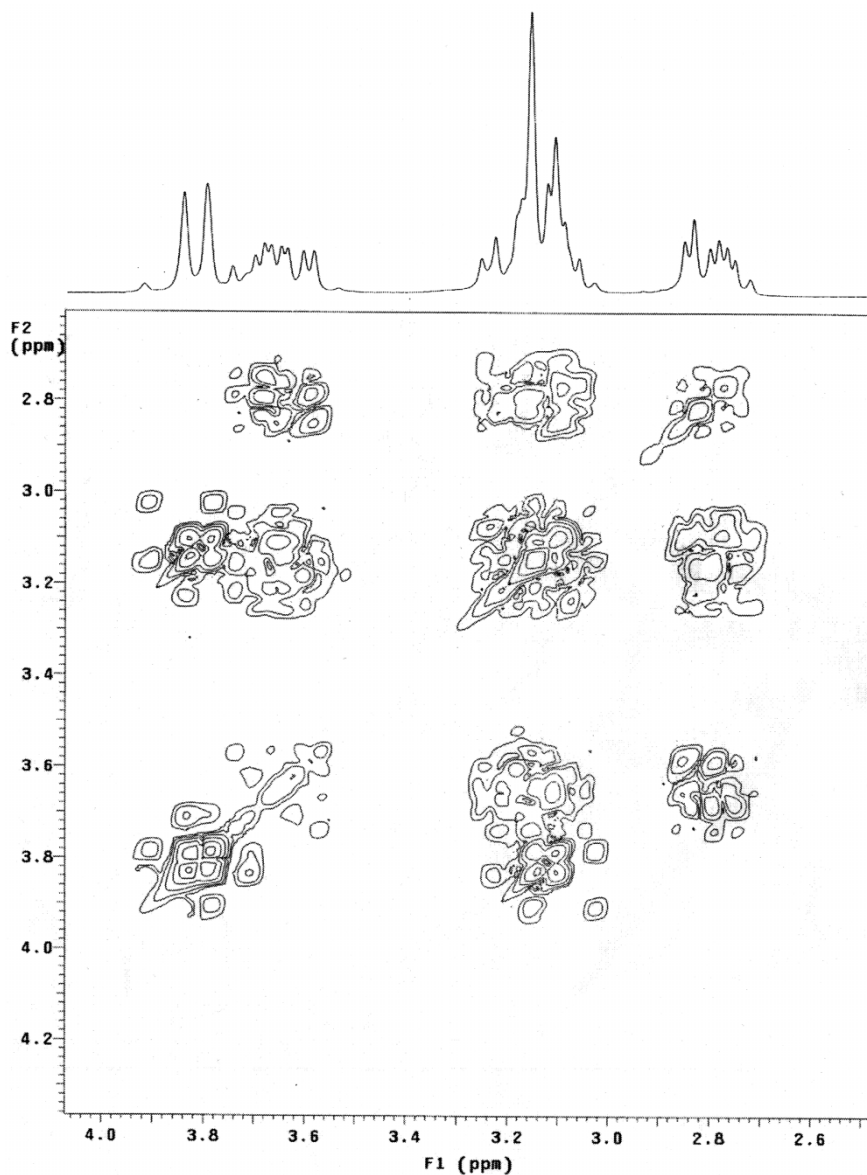
FIGURE 3 Heteronuclear correlation spectrum of  $[\text{Co}(\text{trien})(\text{bipy})]\text{Cl}_3$ .

from coordination of the ligand. Curious, however, is the fact that within each set of geminally related protons, one proton occurs as a separate multiplet and the other is lost to overlap. The large multiplets at 3.00–3.25 ppm for  $[\text{Co}(\text{trien})(\text{bipy})]\text{Cl}_3$  and 3.12–3.29 ppm for  $[\text{Co}(\text{trien})(\text{phen})]\text{Cl}_3$  arise from overlap of one H1 proton, one H2 proton and one H3 proton.

This is further supported by the COSY spectrum for  $\alpha$ -*cis*  $[\text{Co}(\text{trien})(\text{bipy})]\text{Cl}_3$ , shown in Fig. 4. The large multiplet at 3.00–3.25 ppm shows cross-peaks with the multiplet at 2.72–2.85 ppm, the multiplet at 3.58–3.70 ppm, and the doublet at 3.79–3.83 ppm. Cross-peaks between the H1 and H2 protons are expected, whereas cross-peaks between H2 and H3 protons are not expected. As the doublet at 3.79–3.83 ppm, assigned to one set of center ethylene ring protons, shows a cross-peak with the large multiplet at 3.00–3.25 ppm, this indicates that the second set of center ethylene ring protons can be found within the large multiplet at 3.00–3.25 ppm. This particular cross-peak is clearly resolved in the COSY spectrum and has a chemical shift of approximately 3.14 ppm.

While the aliphatic portions of the spectra are very similar for  $[\text{Co}(\text{trien})(\text{bipy})]\text{Cl}_3$  and  $[\text{Co}(\text{trien})(\text{bipy})]\text{Cl}_3$ , the aromatic regions of the spectra are quite different and



FIGURE 4 Magnitude COSY spectrum of [Co(trien)(bipy)]Cl<sub>3</sub>.

are summarized briefly here. The aromatic region of [Co(trien)(bipy)]Cl<sub>3</sub> was observed to contain four proton resonances at approximately 8.10, 8.50, 8.64 and 8.82 ppm. The resonances at 8.64 and 8.82 ppm were observed to be doublets and were assigned to the H7 and H4 protons, respectively, on the basis of multiplicity and chemical shift. An NOESY was then used to establish connectivities within the ring. From the resulting data, the resonance at 8.10 was assigned to the H5 proton and the resonance at 8.50 was assigned to the H6 proton. Finally, a heteronuclear correlation was used to



TABLE IV Summary of aromatic data for [Co(trien)(bipy)]Cl<sub>3</sub>

<i>Assignment</i>	<sup>1</sup> H chemical shift	<sup>1</sup> H multiplicity	<sup>13</sup> C chemical shift
C4/H4	8.82	Doublet	153.4
C5/H5	8.10	Triplet	130.5
C6/H6	8.50	Triplet	143.8
C7/H7	8.64	Doublet	126.4
C8/H8	N/A	N/A	157.7

TABLE V Summary of aromatic data for [Co(trien)(phen)]Cl<sub>3</sub>

<i>Assignment</i>	<sup>1</sup> H chemical shift	<sup>1</sup> H multiplicity	<sup>13</sup> C chemical shift
C4/H4	9.20	Doublet	154.5
C5/H5	8.30	Quartet	128.5
C6/H6	9.05	Doublet	142.5
C7/H7	N/A	N/A	132.5
C8/H8	N/A	N/A	147.9
C9/H9	8.35	Singlet	129.5

assign the C4, C5, C6 and C7 carbons. This left only the C8 carbon, which was assigned by a process of elimination. The proton and carbon data are summarized in Table IV.

The aromatic region of [Co(trien)(phen)]Cl<sub>3</sub> was assigned in a completely analogous fashion. The proton spectrum contained four resonances at approximately 9.20, 9.05, 8.35 and 8.30 ppm. The peak resonances at 9.20 and 9.05 were doublets, and were assigned to the H4 and H6 protons, respectively, based upon chemical shift and multiplicity. The resonance at 8.30 ppm was a quartet, and assigned to H5 on the basis of multiplicity. Finally, the resonance at 8.35 ppm was a singlet and was assigned to the H9 proton on the basis of multiplicity. These initial proton assignments based upon multiplicity and chemical shift were confirmed through the use of an NOESY experiment, which was used to establish the connectivities of the protons in the ring. The carbon spectrum for [Co(trien)(phen)]Cl<sub>3</sub> contained six aromatic signals at 154.5, 147.9, 142.6, 132.5, 129.2 and 128.5 ppm. The C4, C5, C6 and C9 carbons were assigned through the use of a heteronuclear correlation. This left only the C7 and C8 carbons unassigned. Based upon chemical shift, the resonance at 132.5 was assigned to C7 and the resonance at 147.9 was assigned to C8. The proton and carbon data are summarized in Table V.

## CONCLUSIONS

As for [Co(eee)(bipy)]Cl<sub>3</sub> and [Co(eee)(phen)]Cl<sub>3</sub> [40], the presence of 2,2-bipyridine and 1,10-phenanthroline in the complexes [Co(trien)(bipy)]Cl<sub>3</sub> and [Co(trien)(phen)]Cl<sub>3</sub> caused the proton resonances to be spread into a wider spectral width and reduced the overlap in the proton spectra. Within each set of geminally related protons, one was found to give rise to a separate multiplet, and the other was lost in a complex multiplet arising from three separate protons. Overall, the degree of separation was not as great as observed previously for [Co(eee)(bipy)]Cl<sub>3</sub> and [Co(eee)(phen)]Cl<sub>3</sub>.

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