measures of enantiomeric selectivity for these small chiral metal complexes provide a unique probe for local DNA structure in solution. *The size and shape of the metal complex may be tuned to the DNA helical groove size.*

It is interesting, finally, to compare binding parameters of the complexes with nucleic acids differing markedly in structure. Binding to double-stranded RNA is extremely poor as is binding to a DNA with the major groove filled by glucosylation. Binding, therefore, must be preferred through the major groove. This observation is understandable structurally; the minor groove of B DNA is apparently too small to accommodate either isomer. For double-stranded RNA, which has an A-like conformation, what was the B-form major groove is now smaller and deeper while the minor groove is more shallow and extended.[37] Perhaps based upon electronic considerations, intercalation of the phenanthroline ligand is favored from the major groove. Since this groove is narrowed for a RNA helix, binding to the RNA helix is not high. It is interesting to notice that, for both these polymers, glucosylated T4 and double-stranded RNAs, some nonintercalative surface binding is still apparent. Indeed the enantiomeric preference for Λ-Ru(phen)$_3^{2+}$ may be somewhat enhanced. An explanation may be in the fact that both of these polynucleotides contain shallow surfaces along the helical groove. Perhaps the polar glucose units filling the major groove or the shallow base pair surface of the RNA minor groove facilitate binding of the Λ-Ru(phen)$_3^{2+}$ cations by the surface-bound mode, optimizing hydrophobic interactions between the phenanthroline ligands and shallow helical surfaces. This binding interaction could potentially provide the basis for probes of the A conformation.

In summary, the interaction of the chiral metal complexes with polynucleotides may be described in detail by taking advantage of the spectroscopic properties of the ruthenium center combined with measurements of enantiomeric preferences. Two binding modes, one based upon intercalation and one upon surface binding, have been characterized, and both display chiral discrimination. Given these binding models, photophysical experiments coupled with measurements of enantiomeric selectivity may be used to examine subtle features of the polynucleotide structure. Small molecules and chiral complexes in particular offer unique structural probes in solution for the local conformation of sites along the polymer.

Acknowledgment. We thank the National Institute of General Medical Sciences, the National Science Foundation, and the Army Office of Research for their generous support of this work. We thank also Dr. Charles Doubleday for providing the software employed in our single photon counting analyses.

Registry No. Δ-Ru(phen)$_3^{2+}$, 19368-51-5; Λ-Ru(phen)$_3^{2+}$, 24162-09-2; guanine, 73-40-5; cytosine, 71-30-7.

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Communications to the Editor

Paramagnetic Cobalt(III) Complexes of Polyanionic Chelating Ligands

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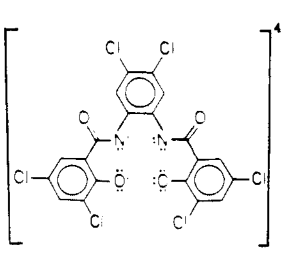
Our approach to the problem of finding new oxidizing agents[3] is to investigate the coordination chemistry of novel polyanionic chelating (PAC) ligands (e.g., **1**, **2**).[4-7] Ligand **1** forms stable octahedral cobalt(III) and cobalt(IV) complexes,[7] but here we show that **2**, which has a greater donor ability, stabilizes Co(III) in the rare square-planar geometry. Well-resolved, paramagnetically shifted, solution ^{1}H NMR spectra have been measured for these intermediate and high-spin cobalt(III) complexes.[8]



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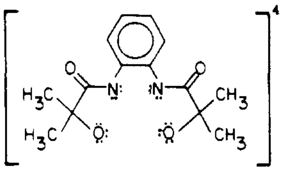

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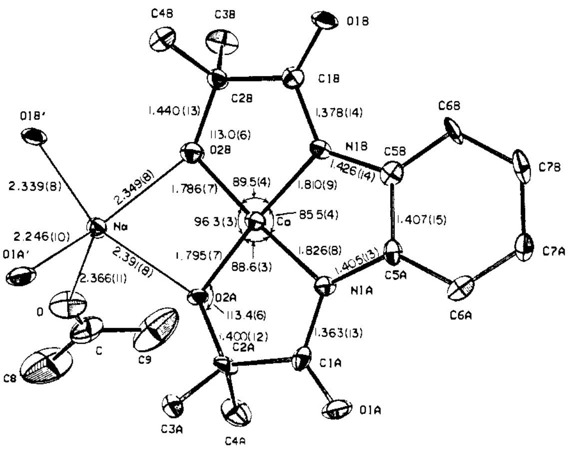

Figure 1. Molecular structure of Na[(η^4-**2**)]·acetone.

Reaction of $Co(O_2CCH_3)_2$ with 1 equiv of H_4-**2** and excess NaOH in ethanol under air gives a deep green solution containing [Co(η^4-**2**)]$^-$, which can be isolated (ca. 70%) as the Na$^+$ salt **3**

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Table I, ^1H NMR, Magnetic and UV/vis Data for Paramagnetic Cobalt(III) Complexes

solvent	salt	color	λ, nm	μ_{eff}[a]	chemical shift[b] Me	H_a	H_b
CH_2Cl_2	PPh_4	brown	558, 426	3.1	9.71	1.97	-17.12[c]
CH_2Cl_2	PPh_4	brown			8.08	-2.15	*d*
THF	PPh_4	brown	558, 426	3.3	8.57	1.04	-17.56
THF	Na^+	green	610, 444	*e*	6.98	-3.01	-22.64
MeOH	PPh_4	green	594, 436	3.9			
D_2O	Na^{+}[f]	green	606, 432	5.1	8.31[g]	-0.20	-26.25

[a] Average of two independent determinations using the Evans method at 303 K; estimated uncertainty ±0.1 μ_B. [b] Chemical shift in ppm relative to internal Me_4Si reference at 298 K; measured at 400 MHz on JEOL JNM-GX 400; aromatic ring coupling not well resolved. [c] Two water molecules of crystallization appear at δ 6.68; exchange with D_2O is rapid. [d] The hydrogen atom H_b is replaced by Cl in this complex. [e] Low solubility in THF precluded an accurate determination of μ_{eff}. [f] Identical behavior observed for Na[Co(η^4-**2**]·$2H_2O$ and for salt prepared in situ by addition of $NaClO_4$ to PPh_4[Co(η^4-**2**]·$2H_2O$. [g] Internal reference HOD at δ 4.65.

by recrystallization from acetone, or as the $[PPh_4]^+$ salt **4** by addition of $[PPh_4]Cl$ and crystallization from water.[9] The X-ray structure of **3**·acetone is shown in Figure 1.[10] The square-planar complex has short Co–O (1.791 (8) Å) and Co–N (1.818 (9) Å) bonds compared to octahedral Co(III) or square-planar Co(II) complexes of related ligands.[11,12] Bond distances and angles within the ligand framework are normal for complexes of this type.[4-7,12] There are no other short intermolecular contacts. The formal potential for the cobalt(III/II) couple of **4** in dichloromethane is -1.48 V vs. F_c^+/F_c (or -1.00 V vs. SCE).[13] Square-planar Co(III) is rare but has been structurally characterized for thiolate[14] and substituted biuret[15] complexes, where the ligand complements are also strongly donating.

Solid-state magnetic studies (6–300 K) showed that **3** and **4** obey the Curie–Weiss law over the temperature range 50–300 K (μ_{eff} of 3.26 μ_B for **3** and 3.41 μ_B for **4** at 300 K). Limiting values of μ_{eff} of 1.41 μ_B for **3** and 1.52 μ_B for **4** were observed at 6 K indicating zero-field splitting.[15b,16a] These data are consistent with spin triplet (S = 1) ground states.[8,15b,16] The solution magnetic behavior is solvent-dependent (see Table I). In aprotic solvents such as CH_2Cl_2 or THF, brown solutions of **4**·$2H_2O$ exhibit magnetic moments of 3.1–3.3 μ_B,[17] suggesting that the complex remains a spin triplet and probably four coordinate in these solutions as found for the Na^+ salt in the solid state, although it is not possible to rule out coordination of water of crystallization (or THF) in these aprotic media. In these solvents, no change is observed in the visible spectra upon addition of pyridine. In contrast, deep green solutions of **3** in water exhibit a magnetic moment of 5.1 μ_B, consistent with a high-spin quintet (S = 2) state. This species readily coordinates two molecules of pyridine, as demonstrated by equilibrium measurements,[18] to afford the yellow, diamagnetic, six-coordinate adduct.[19] In alcohols an intermediate magnetic moment (3.9 μ_B in methanol) and an intermediate affinity for pyridine are observed.[18] Ion pairing apparently plays a role in determining the electronic structure of the complexes. Brown solutions of **4** in THF or acetone acquire the deep green color associated with the quintet state upon addition of excess $NaClO_4$. Coordination of solvent or water might accompany the intermediate to high spin state change. Unfortunately, **4** is insoluble in water.

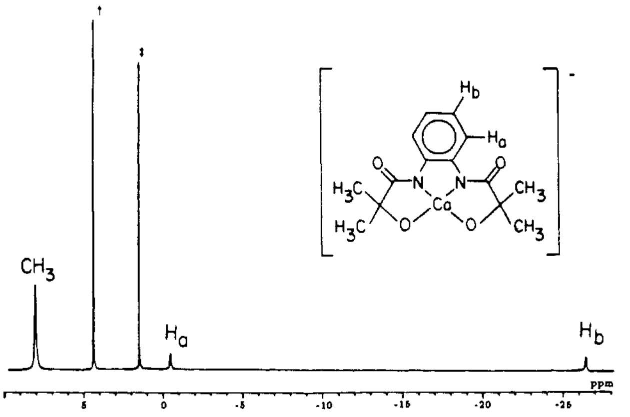

Figure 2, ^1H NMR spectrum of Na[(η^4-**2**)]·acetone measured in D_2O (μ_{eff} = 5.1 μ_B); †HOD, ‡$(CH_3)_2CO$.

It is important to note that reasonably sharp ($\nu_{1/2} \approx$10–20 Hz) ^1H NMR spectra can be observed for all these paramagnetic complexes (see Figure 2 and Table I). We are further examining the reactivity of these rare compounds. It is also noteworthy that the behavior described here for these square-planar cobalt(III) complexes is similar to that observed for isoelectronic square-planar ferrous porphyrin and related systems.[20]

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(9) Anal. Calcd for $[PPh_4]$[Co(η^4-**2**)]·$2H_2O$, $C_{38}H_{40}N_2CoO_6P$: C, 64.23; H, 5.67; N, 3.94. Found: C, 64.14; H, 5.45; N, 3.96. Anal. Calcd for Na[Co(η^4-**2**)]·$(CH_3)_2CO$ as highly crystalline material from which structural crystals were selected, $C_{17}H_{22}N_2CoNaO_5$: C, 49.05; H, 5.33; N, 6.73. Found: C, 48.46; H, 5.18; N, 6.62.

(10) Crystal data: space group $P2_12_12_1$, a = 10.358 (2) Å, b = 10.802 (3) Å, c = 17.794 (2) Å, V = 1991 (1) Å^3, Z = 4. Data were collected on a Nonius CAD4 diffractometer with graphite monochromator and Mo Kα (λ = 0.7107 Å) radiation to 2θ = 46°. Least-squares refinement led to goodness-of-fit S = 1.06 (1.14 for enantiomer), R_f = 0.125 (2390 reflections, $I >$ 0) R_F = 0.055 ($I > 3\sigma$ (I)). Full details given in supplementary material.

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(13) A cobalt(II)–ligand radical cation formulation for **4** is thus most unlikely as such a species would be expected to be a relatively strong oxidizing agent.

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(18) Equilibrium data determined by UV/vis for $[Co(\eta^4\text{-}\mathbf{2})]^- + 2py \rightleftharpoons [Co(\eta^4\text{-}\mathbf{2})(py)_2]^-$: (in H_2O) $k_{eq} = 2.6 \times 10^6$ mol^{-2} L^2 at 298 K, ΔH = -18.3 ± 0.4 kcal mol^{-1}, ΔS = -54.2 ± 1.3 cal K^{-1} mol^{-1}; (in ethanol) k_{eq} = 37 mol^{-2} L^2 at 298 K, ΔH = -23.3 ± 0.4 kcal mol^{-1}, ΔS = -48.8 ± 0.9 cal K^{-1} mol^{-1}.

(19) Attempts to crystallize this complex even in the presence of excess pyridine resulted in the isolation of the four-coordinate complex. ^1H NMR: ($[Co(\eta^4\text{-}2)(py\text{-}d^5)_2]^-$ in D_2O) δ 0.42 (s, Me, 12 H), 7.09 (dd, Ar H, 2 H), 8.52 (dd, Ar H, 2 H).

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Registry No. 3, 100909-69-1; 4, 100909-71-5; $[Co(\eta^4\text{-}2)(py)_2]^-$, 100909-72-6.

Supplementary Material Available: Complete details of data collection and refinement, listing of bond distances and angles, fractional atomic coordinates and Gaussian amplitudes, and structure factor amplitudes (18 pages). Ordering information is given on any current masthead page.

The First Total Syntheses of $\Delta^{9(12)}$-Capnellene-8β,10α-diol and $\Delta^{9(12)}$-Capnellene-3β,8β,10α-triol

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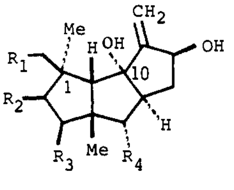
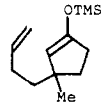
Capnellane is the generic name applied to the group of tricyclic sesquiterpene alcohols **1–6**and hydrocarbons, isolated from the soft coral *Capnella imbricata*.[1,2] These substances appear to act as chemical defense agents in the coral reef biomass to ward off algal and microbial growth and to prevent larval settlement.[3] A fascinating structural feature uniquely associated with compounds **1–6** is the presence of the unusual C-ring bisallylic alcohol unit, bringing about the severe steric repulsion between the hydroxyl group at C-10 and the 1α-methyl group. We describe herein the first total syntheses of (±)-**1** and (±)-**3** via the common intermediate **8**.



1, $R_1=R_2=R_3=R_4=H$
2, $R_1=R_3=R_4=H$, $R_2=OH$
3, $R_1=R_2=R_4=H$, $R_3=OH$
4, $R_1=R_2=R_3=H$, $R_4=OH$
5, $R_1=R_3=H$, $R_2=R_4=OH$
6, $R_2=R_4=H$, $R_1=R_3=OH$

Conjugate addition to 3-methyl-2-cyclopenten-1-one using 3-butenylmagnesium bromide (2.2 molar equiv) and cuprous iodide (1.1 molar equiv) followed by quenching with chlorotrimethylsilane and triethylamine afforded **7** in 78% yield (bp 85–85.5 °C (5.5 mmHg)). Treatment of **7** with 1.0 equiv of $Pd(OAc)_2$ and 1.0 equiv of NaOAc in CH_3CN at room temperature[4] gave a mixture of **8**, **9**, and **10** in a ratio of 3.6:0.4:1 (89%).[5] The *exo*-methylene **9** was quantitatively converted to **8** on exposure to DBU in benzene at room temperature. On the other hand, treatment of the mixture (**8–10**) with DBU in refluxing benzene for ca. 10 h provided **10** exclusively in 94% yield. Conversion of **8** to the key intermediate **14**, needed for the synthesis of **1**, was easily achieved by reaction with Me_2CuLi (96%). Similarly, **10** was also transformed into the key intermediate **15**, required for the synthesis of **3**, by the sequence: (1) reduction of **10** with $NaBH_4$ followed by silylation with *tert*-butyldimethylsilyl trifluoromethanesulfonate[6] to give **11** (84%);[7] (2) allylic oxidation of **11** with chromic anhydride and 3,5-dimethylpyrazole in CH_2Cl_2[8] and subsequent treatment with Me_2CuLi to form **12** in 60% yield (72% based on recovery of the enone); (3) reduction of **12** with Li in NH_3[9] followed by protection as the MPM (*p*-methoxybenzyl)[10] derivative to give **13** in ca. 60% yield; (4) exposure of **13** to $Bu_4N^+F^-$ and subsequent oxidation with PCC and 4A molecular sieves to provide **15** in 93% yield.



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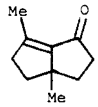

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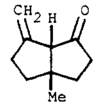

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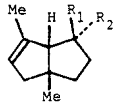
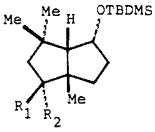
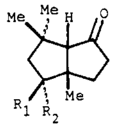


10, $R_1=R_2=O$
11, $R_1=H$, $R_2=OTBDMS$



12, $R_1=R_2=O$
13, $R_1=OMPM$, $R_2=H$



14, $R_1=R_2=H$
15, $R_1=OMPM$, $R_2=H$

With the two key synthetic intermediates in hand, the next subgoal of the synthetic effort was the efficient construction of the ABC ring systems **20** and **21**. In the first place the construction of **20** was examined. Reaction of the lithium enolate derived from **14** (LDA in THF) with ethyl 4-iodo-3-methoxycrotonate[11] gave **16** in 75% yield (83% based on recovery of **14**) as an isomeric mixture, which was converted to **18** in quantitative yield on exposure to 30% aqueous perchloric acid. The β-keto ester **18** was then subjected to the aldol cyclization. Unfortunately, all attemepts to obtain the tricyclic intermediate **20** employing a wide variety of different acidic and basic reagents met with failure. For example, under the conditions such as sodium ethoxide in ethanol at 25 °C only a trace amount of **20** was formed (<1%), **18** being recovered nearly exclusively. These results suggested that irreversible elimination reaction of the aldol derived from **18** could produce **20** in an acceptable yield. We speculated that β-silyloxy ketones may undergo irreversible elimination to conjugated ketones in the presence of trimethylsilyl trifluoromethanesulfonate, giving hexamethyldisiloxane which was expected not to undergo Michael addition to conjugated ketones. This expectation was fully realized and led to success. Thus, treatment of **18** with 3 molar equiv of trimethylsilyl trifluoromethanesulfonate and 2 molar equiv of triethylamine in refluxing benzene for 10 h afforded **20** in 42% yield together with recovery of **18** in ca. 32%. Since recovered **18** was again used for the cyclization reaction,[12] this novel conditions provided **20** in greater than 50% yield after two cycles.

[†] Sagami Chemical Research Center.

[‡] Teikyo University.

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(9) A ratio of the exo- to endo alcohol was ca. 3:2. The endo alcohol was quantitatively oxidized to **12** for recycling.

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(12) It was necessary to treat recovered **18** with sodium ethoxide in EtOH before the aldol cyclization.

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