0.065 for 238 parameters. The highest peak in the final difference Fourier map had a value of 1.55 e Å^{-3}, 1.58 Å from Pt(2). All calculations were performed with the Siemens SHELXTL PLUS (MicroVAX II) and PARST[16] programs. Atomic scattering factors and anomalous dispersion terms used were those supplied in the Siemens structure determination package.

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Supplementary Material Available: Figures showing an ORTEP diagram of **2c** and selected NMR spectra (Figures S1–S3) and full tables listing crystallographic and data collection parameters (Table SI), atomic coordinates (Table SII), bond lengths and angles (Tables SIII and SIV), anisotropic thermal parameters (Table SV), and hydrogen atom coordinates (Table SVI) (12 pages); a listing of calculated and observed structure factors (Table SVII) (17 pages). Ordering information is given on any current masthead page.

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A Tridecanuclear Ruthenium(II)–Polypyridine Supramolecular Species: Synthesis, Absorption and Luminescence Properties, and Electrochemical Oxidation

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Continuing our studies on the synthesis and characterization of metal complexes of high nuclearity,[2] we have prepared the luminescent and redox-active tridecanuclear Ru(II)–polypyridine supramolecular species $\mathrm{Ru\{(\mu\text{-}2,3\text{-}dpp)Ru(bpy)(\mu\text{-}2,3\text{-}dpp)Ru[(\mu\text{-}2,3\text{-}dpp)Ru(bpy)_2]_2\}_3}^{26+}$ (**1**) schematically represented in Figure 1 (2,3-dpp = 2,3-bis(2-pyridyl)pyrazine; bpy = 2,2′-bipyridine).[3]

The novel tridecanuclear species **1** has been obtained by using the "complexes as ligand and complexes as metals" synthetic strategy illustrated in Figure 2, i.e., by reacting a $\mathrm{Ru[(\mu\text{-}2,3\text{-}dpp)Ru(bpy)(2,3\text{-}dpp)]_3}^{8+}$ core (**2**),[2d] which contains three free chelating sites that can play the role of ligands, with three $\mathrm{Ru[(\mu\text{-}2,3\text{-}dpp)Ru(bpy)_2]_2Cl_2}^{4+}$ units (**3**),[2f] which contain easily replaceable Cl$^-$ ligands and can thus play the role of metals. The building blocks **2** and **3** are both well-characterized species that can be obtained as PF$_6^-$ salts.[2d,f,g,i,j]

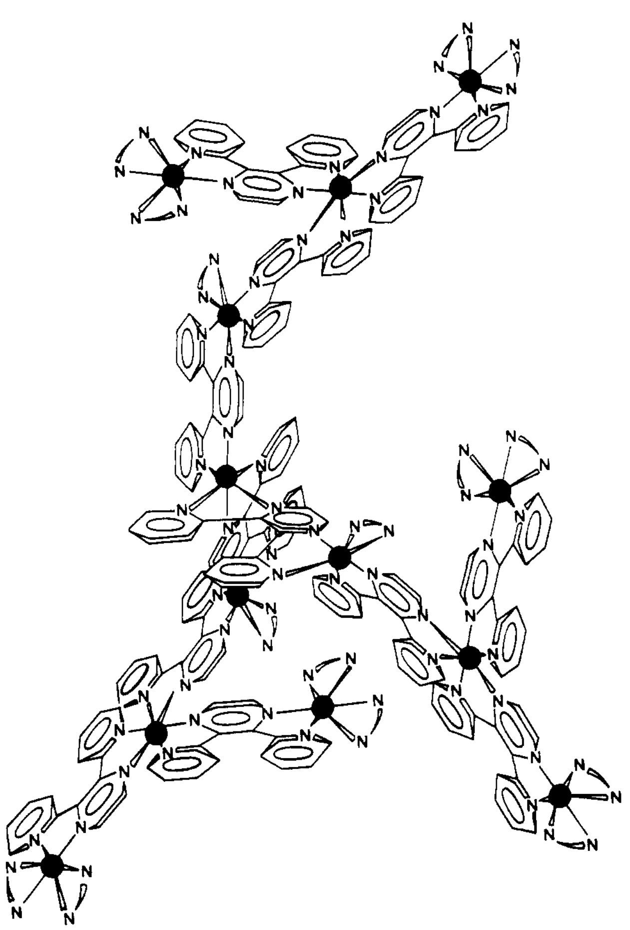

Figure 1. Schematic representation of the tridecanuclear complex **1**. N-N stands for bpy.

Experimental Section

The reaction illustrated in Figure 2 has been carried out as follows. To a solution of $\mathrm{\{Ru[(\mu\text{-}2,3\text{-}dpp)Ru(bpy)_2]_2Cl_2\}(PF_6)_4}$ (0.036 g, 0.018 mmol) in 2:1 (v/v) methanol–water (3 mL) was added solid AgNO$_3$ (0.006 g, 0.004 mmol). After 3 h of stirring at room temperature, $\mathrm{\{Ru[(\mu\text{-}2,3\text{-}dpp)Ru(bpy)(2,3\text{-}dpp)]_3\}(PF_6)_8}$ (0.020 g, 0.006 mmol) and ethylene glycol (3 mL) were added and the mixture was refluxed for 7 days. After removal of AgCl by repeated centrifugation, an excess of solid NH$_4$PF$_6$ was added to the mother liquor, and the violet solid thus obtained was filtered out. The product was purified by size-exclusion chromatography on Sephadex G-50 (acetonitrile eluant). From the eluant, the product was recovered as a violet powder by addition of ethanol and partial evaporation in vacuo. Yield after purification: 0.043 g (70%). Anal. Calcd. for $\mathrm{C_{318}H_{240}F_{156}N_{78}P_{26}Ru_{13}\cdot 10H_2O}$: C, 36.06; H, 2.51; N, 10.48. Found: C, 36.01; H, 2.48; N, 10.03. The infrared spectrum shows the characteristic bands of bridged 2,3-dpp,[2e] whereas the bands of unbridged 2,3-dpp[2e] are not present, indicating that only a 1:3 combination of **2** and **3** (Figure 2) has occurred. It should also be noted that the building blocks **2** and **3** display absorption, luminescence, and electrochemical properties clearly distinct from those of **1**, and this would also be the case for hypothetical side products containing Cl$^-$ ligands as **3** and/or unsaturated chelating sites as **2**.[2e,j]

The equipment used for spectroscopic, photophysical, and electrochemical measurements has been previously described.[2]

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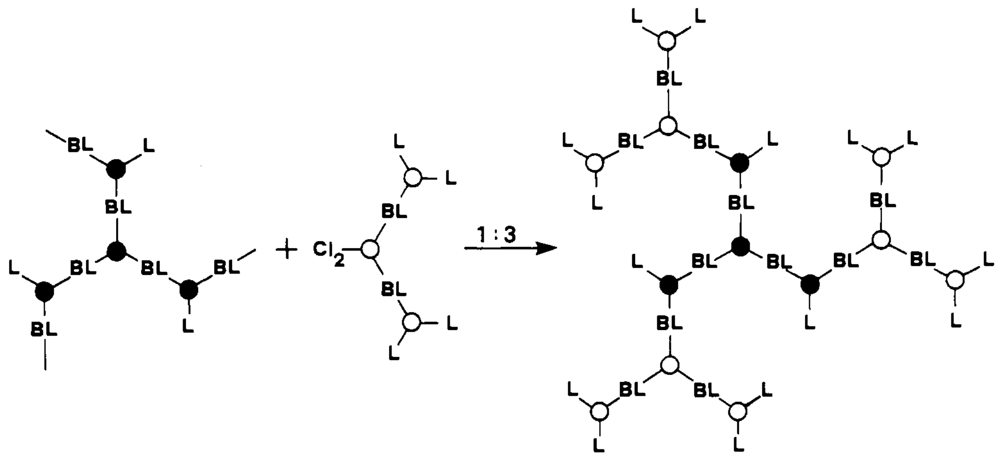

Figure 2. Schematic representation of the "complexes as metals and complexes as ligands" synthetic strategy to obtain **1**.

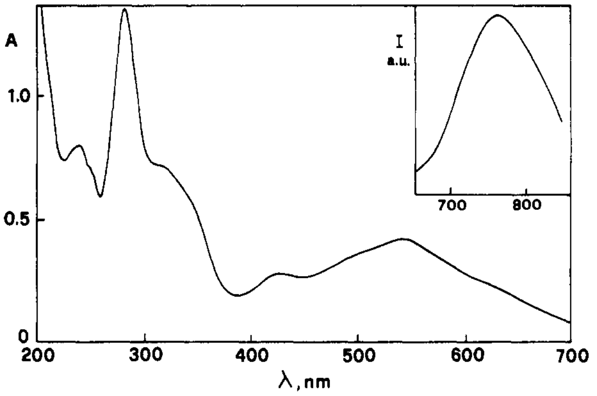

Figure 3. Absorption and (inset) uncorrected luminescence spectra of **1** in acetonitrile solution at room temperature.

Results and Discussion

The novel tridecanuclear species **1** exhibits a three-dimensional branching structure of the type shown by otherwise completely different compounds based on organic components described by Newkome et al.,[5] Tomalia et al.,[6] and Fréchet et al.[7] Quite interesting properties of **1** are the extremely high intensity absorption bands (Figure 3) in the UV (λ_{max} = 283 nm, ϵ_{max} = 429 000 M^{-1} cm^{-1}) and visible (λ_{max} = 544 nm, ϵ_{max} = 133 000 M^{-1} cm^{-1}) spectral regions and its electrochemical and luminescent behavior.

Neglecting differences that could arise from the presence of geometrical and optical isomers,[2h,j] in **1** there are four distinct types of metal ions (Figures 1 and 2): (i) the one located at the center, which is coordinated to three 2,3-dpp bridging ligands; (ii) the three equivalent metal ions coordinated to two bridging ligands and one bpy ligand; (iii) the three equivalent metal ions near the periphery coordinated to three bridging ligands; (iv) the six equivalent peripheral metal ions coordinated to a bridging ligand and two bpy ligands. It can be noticed that the metal ions of type i and iii are different from a topological point of view, but their first and second coordination spheres are identical. Therefore, they are expected to exhibit identical chemical properties.

The oxidation potential of the metal ion in Ru(II)–polypyridine complexes is known to depend on the coordinated ligands.[8] Since bpy is a better electron donor than μ-2,3-dpp,[2,4] one can expect that the oxidation potential will increase slightly on passing from the six peripheral Ru(μ-2,3-dpp)(bpy)$_2^{2+}$ units to the three Ru(μ-2,3-dpp)$_2$(bpy)$^{2+}$ units and three-plus-one Ru(μ-2,3-dpp)$_3^{2+}$ units. Previous electrochemical investigations carried out on oligonuclear complexes of the same family[2,4] have also shown that the interaction between different metal centers is negligible except for metals coordinated to the same bridging ligand when both electrostatic and through ligand interactions are significant. On the basis of the above arguments, the oxidation pattern of **1** is expected to consist of a "six-electron" wave corresponding to the independent one-electron oxidation of the Ru^{2+} ions of the six peripheral Ru(μ-2,3-dpp)(bpy)$_2^{2+}$ units, followed at slightly higher potential by a "three-electron" wave corresponding to the independent one-electron oxidation of the Ru^{2+} ions of the three Ru(μ-2,3-dpp)$_2$(bpy)$^{2+}$ units.[9] Oxidation of the remaining four Ru^{2+} ions (types i and iii above) should be displaced to much higher potential because of their interaction, through the bridging ligands, with three already oxidized metal ions. The experimental results are consistent with the above expectations. A broad, reversible oxidation wave is observed, with the following characteristics in differential pulse voltammetry: maximum at +1.50 V (vs SCE); half-width of 170 mV; area corresponding to 9 ± 1 electrons.[11] The waves of the six Ru(μ-2,3-dpp)(bpy)$_2^{2+}$ and three Ru(μ-2,3-dpp)$_2$(bpy)$^{2+}$ units are, as expected, so close as to overlap. No other oxidation wave is present in the potential window (<+1.90 V) examined.

Ru(II)–polypyridine complexes are known to exhibit luminescence from the lowest triplet metal-to-ligand charge-transfer (^{3}MLCT) excited state.[8] Since μ-2,3-dpp is a better electron acceptor (i.e., its LUMO lies at lower energy) and a worse electron donor than bpy, it follows that (a) the ligand involved in the lowest energy excited state is μ-2,3-dpp, not only in the Ru(μ-2,3-dpp)$_3^{2+}$ units but also in the mixed-ligand Ru(μ-2,3-dpp)$_2$(bpy)$^{2+}$ and Ru(μ-2,3-dpp)(bpy)$_2^{2+}$ ones and (b) the energy of the lowest excited state increases slightly in the order Ru(μ-2,3-dpp)(bpy)$_2^{2+}$ < Ru(μ-2,3-dpp)$_2$(bpy)$^{2+}$ < Ru(μ-2,3-dpp)$_3^{2+}$. The luminescence exhibited by **1** (77 K, λ_{max}^{cor} = 722 nm, τ = 1.46 μs; 300 K, λ_{max}^{cor} = 800 nm, τ = 62 ns; see also inset of Figure 3) is, as expected, typical of peripheral Ru(μ-2,3-dpp)(bpy)$_2^{2+}$ units.[2,4,12] A corrected excitation spectrum at room temperature has shown that the

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(9) The oxidation potential of the six peripheral Ru(μ-2,3-dpp)(bpy)$_2^{2+}$ units is known to be +1.43 V (*vs* SCE) from the behavior of the same units in the analogous tetra-,[2c,e,f,i,4c] hepta-,[2d] and decanuclear[2h,j] species. The oxidation potential of the Ru(μ-2,3-dpp)$_2$(bpy)$^{2+}$ units is not known, but it must be (i) slightly more positive than that of the previous units (+1.43 V), because μ-2,3-dpp is a worse electron donor than bpy, and (ii) considerably less positive than +1.78 V, which is the value of the oxidation potential of the Ru(μ-2,3-dpp)$_2$(bpy)$^{2+}$ unit when the two bridging ligands are linked to two Os^{3+} species.[10]

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(11) The number of electrons was established by comparison, through the internal standard wave of ferrocene oxidation, with the area of the six-electron oxidation wave of the analogous decanuclear complex.[2j]

(12) Luminescence from the (less numerous) Ru(μ-2,3-dpp)$_2$(bpy)$^{2+}$ units cannot be excluded because the bands are broad and the energy difference between the luminescent levels of the two types of units is small.

luminescent levels are reached with unitary efficiency (within an experimental error of ±20%) regardless of the excitation wavelength (in particular, regarding excitation in the ligand-centered bpy band at 282 nm and 2,3-dpp band at 350 nm).

Superamolecular species like **1** are very interesting because they contain many chromophoric and redox centers and exhibit luminescence from relatively long-lived excited states. Because of these properties, they are good candidates to play the roles of photosensitizers,[13–15] luminescent probes,[16,17] and multielectron-transfer catalysts.[18] By using different metals, bridging ligands, or terminal ligands, it is also possible to control the direction of electronic energy migration in supramolecular structures of this type (antenna devices).[2c,f,i,j,15]

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Complexes of Zinc Finger Peptides with Ni^{2+} and Fe^{2+}

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In 1983, it was discovered that the eukaryotic DNA binding protein transcription factor IIIA (TFIIIA) contained bound zinc ions as isolated and that these ions were required for site-specific DNA binding activity.[1] Treatment of the purified protein with EDTA abolished specific DNA binding activity. The activity could be restored by treatment of 15 μM Zn^{2+} but not by the same concentration of Fe^{2+}, Ni^{2+}, Co^{2+}, or Mn^{2+}. Analysis[2,3] of the deduced amino acid sequence of TFIIIA[4] revealed the presence of nine sequences that approximate the form (Phe,Tyr)-X-Cys-$X_{2,4}$-Cys-X_3-Phe-X_5-Leu-X_2-His-$X_{3,4}$-His-X_{2-6} where X represents relatively variable amino acids. It was proposed that each of these sequences binds a zinc ion through the invariant cysteinate and histidine residues to form a structural domain termed a "zinc finger". A variety of evidence has confirmed this hypothesis for TFIIIA.[5] In addition, a large number of other proteins have been found to contain similar sequences, and extant data are consistent with the proposition that these also form metal-based domains.[5,6]

While several studies have been reported concerning the structural and zinc binding properties of single zinc finger peptides,[7–16] the ability of these peptides to act as ligands for other metal ions has not been extensively described. We report that a prototypical zinc finger peptide, CP-1,[17] can indeed bind Ni^{2+} and Fe^{2+} in addition to Co^{2+} to produce complexes whose spectroscopic properties are consistent with distorted tetrahedral coordination by the cysteinate and histidine residues. In addition, studies of the affinity of this peptide for various metal ions reveal that the specificity of a single domain peptide in binding zinc over other metal ions is sufficient to explain the unique ability of zinc to restore the DNA binding activity of TFIIIA under the conditions reported.

Experimental Section

The peptides CP-1 and CP-1(H24C) were prepared and purified as described previously. All experiments were performed in an oxygen-free 98% nitrogen/2% hydrogen atmosphere. Optical spectra were recorded on a Perkin-Elmer Lambda 9 spectrophotometer. Nuclear magnetic resonance spectra were recorded on a Varian XL-400 spectrometer.

Results and Discussion

For these studies, the zinc finger peptide CP-1 which has the sequence ProTyrLys*Cys*ProGlu*Cys*GlyLysSerPheSerGlnLysSerAspLeuValLys*His*GlnArgThr*His*ThrGly was used.[17] This is a consensus peptide based on a large data base of zinc finger sequences. For comparison, the sequence variant CP-1(H24C)-ProTyrLys*Cys*ProGlu*Cys*GlyLysSerPheSerGlnLysSerAspLeuValLys*His*GlnArgThr*Cys*ThrGly was also studied. Previous studies have shown that CP-1 and CP-1(H24C) bind Co^{2+} and Zn^{2+}. The ability of these peptides to bind Cu^{2+}, Ni^{2+}, Fe^{2+}, and Mn^{2+} was investigated spectrophotometrically. Cu^{2+} was found to oxidize the peptides presumably to disulfide-linked species. The absorption spectra of the Ni^{2+} and Fe^{2+} complexes are shown in Figure 1. The spectra of the Ni^{2+} complexes are similar to those observed for distorted Ni^{2+} complexes such as Ni^{2+}-substituted aspartate transcarbamylase[18] and rubredoxin.[19] The ligand field transitions are blue shifted consistent with Ni(imidazole)$_{2,1}$(thiolate)$_{2,3}$ rather than Ni(thiolate)$_4$ chromophores, given the positions of thiolate and imidazole in the spectrochemical series.[20,21] In addition, the bands are somewhat more clearly resolved into components, consistent with the reduction of symmetry of the ligand fields compared with Ni(thiolate)$_4$. Additional ligand field bands were observed in the near infrared with maxima (nm) and extinction coefficients (M^{-1} cm^{-1}, in parentheses) as follows: CP-1(Ni2+), 1600 (15), 1725 (25); CP-1(H24C), 1550 (5), 1700 (10). The spectra of the Fe^{2+} complexes consist predominantly of a set of charge-transfer transitions in the ultraviolet. Examination of the spectra extending into the near-infrared region revealed the presence of bands at 1400–1800 nm with extinction coefficients near 80 M^{-1} cm^{-1}. Again, these features are similar

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