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HOMOLEPTIC COMPLEXES OF 2,2′-BIPYRIDINE

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I. Introduction

A. Introduction

At the time of writing, the ligand 2,2′-bipyridine (bpy, Fig. 1) is celebrating its centenary. The compound was first described by Fritz Blau in 1888 as the product obtained from the oxidative coupling of 2-pyridinecarboxylic acids or the oxidation and decarboxylation of 1,10-phenanthroline (phen, Fig. 2) (*73, 74*). The unique metal-binding properties of bpy were recognized from the outset, and in his very first paper, Blau describes the development of an intense red color in the presence of iron. Within 10 years, Blau had successfully demonstrated the coloration to be due to an iron complex ion of formulation $[Fe(bpy)_3]^{2+}$ and had shown that related complexes could be obtained with a wide range of other metal ions (*75*). The coordination chemistry of bpy was quiescent until improved synthetic methods were developed, and a spate of papers in the 1920s and 1930s can be traced to the discovery of direct methods for the oxidative dimerization of pyridine. The third renaissance in bpy chemistry can be traced to the commercial availability of the ligand in the 1950s, when large amounts were required for the preparation of Diquat insecticides (Fig. 3).

The availability of bpy coincided with the development of better physical and theoretical methods for the investigation of metal complexes, and the $[M(bpy)_3]^{n+}$ compounds provided an invaluable series of structurally related compounds with a wide range of metals. The current interest in complexes of bpy is associated with the extremely interesting electrochemical, photophysical, and photoelectrochemical properties that they exhibit.

This review is concerned with the class of complexes that contain

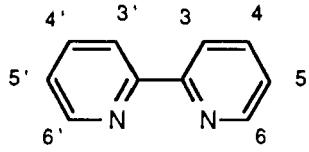


FIG. 1.

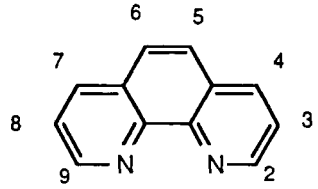


FIG. 2.

only bpy ligands bonded to the metal. This restriction is partly to limit the coverage to a reasonable size and partly because these are the classes of complex that have attracted most recent interest. In practice, the bulk of the review deals with complexes of stoichiometry $[M(bpy)_2]^{n+}$ and $[M(bpy)_3]^{n+}$. The origins of this review are in the author's own laboratory, where we frequently want to compare the properties of related homoleptic complexes of bpy and related ligands. The recent explosive expansion in the literature relating to the properties of bpy complexes renders such a treatment timely. The interest in the use of bpy complexes as photosensitizers, photocatalysts, redox catalysts, colorimetric reagents, catalysts, neurotoxins, or potentiometric indicators continues unabated. The coverage is intended to be comprehensive though numerous omissions must have occurred in such a large field. For these and any errors of interpretation we offer our apologies. The emphasis is on the complexes of metals other than the iron group triad, which has been adequately discussed elsewhere.

No attempt has been made to include coverage of the structurally related 1,10-phenanthroline ligand nor of substituted derivatives of bpy; once again, this was in an attempt to keep the literature to manageable proportions. We hope that this review will be of use to those working in the area of metal-bpy complexes. Complexes of the related ligand 2,2′:6′,2″-terpyridine have been reviewed previously (*158*).

A number of previous reviews have dealt with complexes of bpy and related ligands in various degrees of depth (*89, 372, 552, 621, 753*). Numerous other publications have dealt with aspects of the chemistry of bpy complexes; specific mention should be made of those dealing

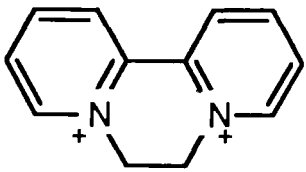


FIG. 3.

with the analytical (*108, 420, 727, 795*), photochemical, (*443*) and thermochemical (*611*). As a general rule, the reader is directed to ref. 621 for publications dated before 1960. Specialist reviews in the areas of solar energy photoconversion and water splitting are also relevant (*150, 186, 317, 334, 458, 987*).

II. Synthesis, Structure, and Properties of Bipy Complexes

A. Synthetic Considerations

The details for the preparations of complexes with specific metals are found at the appropriate point in the text. This section discusses the general strategies for the syntheses.

2,2'-Bipyridine is a strong field ligand that forms relatively stable complexes, with the inherent M—N bond strength enhanced by the chelate effect. These factors favor the formation of 4-coordinate bis and 6-coordinate tris complexes. The tris complexes of the first row transition metals in "normal" oxidation states (+2 or +3) are best prepared by the reaction of a suitable metal salt with an excess of bpy in water, methanol, or other organic solvent. The solid complexes can be obtained by crystallization or by the precipitation of the perchlorate, hexafluorophosphate, tetrafluoroborate, or other salts. Because bpy is a strong field ligand, the lower oxidation states tend to be favored, and reduction of M(III) complexes can occur in these preparations. The M(III) complexes are usually readily obtained by the chemical, aerobic, or electrochemical oxidation of the M(II) species.

In general, the tris complexes of the second and third row transition metals are not so readily obtained. This is in part due to the use of halides or complex halides as starting materials for M(II) and M(III) complexes of these elements and in part to the high activation barriers to substitution at these centers. The products of the reactions are commonly $[M(bpy)_2X_2]^{n+}$; the use of halide abstracting agents or halide free reaction media is frequently required for the synthesis of $[M(bpy)_3]^{n+}$. The great affinity of the metals for halide must be borne in mind when conducting reactions of $[M(bpy)_3]^{n+}$ in halide-rich medium. Similar considerations apply to the preparation of square planar bis complexes of d^8 ions; these have a tendency to form $[M(bpy)X_2]^{n+}$ in the presence of halide.

Complexes with main group metal ions, and with lanthanides and actinides, can be prepared in water or organic solvents. The complexes are probably (but see later) less robust than those of the transition

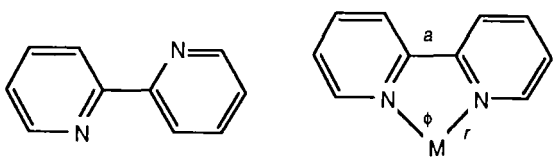
metals. Complexes with main group nonmetals are best prepared in nonaqueous conditions.

Numerous examples of homoleptic complexes in high or low formal oxidation states are known. In general, the high oxidation state complexes are best prepared by chemical or electrochemical oxidation of the "normal" oxidation state compounds, followed by further reaction *in situ* or precipitation with a suitable "inert" anion. In this respect, perchlorate is ideal as both oxidant and precipitant, but the complexes obtained are frequently violently explosive. Similarly, the low oxidation state complexes are best obtained by chemical or electrochemical reduction of available compounds (or "normal" oxidation salts in the presence of an excess of bpy). Commonly used reductants have included dissolving metals (zinc, sodium, lithium, magnesium) and the complexes Li(bpy) and Li_2(bpy). Isolated examples are known of the synthesis of low oxidation state complexes by reaction of M(0) complexes with bpy or by metal vapor synthesis.

B. Structure

Most of the complexes that are discussed in this review are of the stoichiometry $[M(bpy)_2]^{n+}$ or $[M(bpy)_3]^{n+}$, and it is these complexes we now consider. The free ligand 2,2′-bipyridine adopts a near-planar *transoid* arrangement in the solid state (Fig. 4a) though in solution there is the possibility of free rotation about the interannular C—C bond. In contrast, the chelating bonding mode requires a *cisoid* arrangement about the interannular bond (Fig. 4b). In general, a near-planar configuration of the two pyridyl rings is adopted. The C—C distance *a* is relatively independent of metal ion, whereas the M—L bond distance *r* and the N—M—N angle ϕ depends on the metal ion used.

The bis complexes are known for a range of metals and oxidation states though they tend to be more common for lower formal oxidation states. The precise geometry depends on the electronic configuration of



(a) (b)

FIG. 4.

the metal ion; in the absence of crystal field stabilization energy (CFSE) or other electronic constraints, the geometries are based upon a tetrahedral arrangement of the donor atoms about the metal (Fig. 5a) though the precise distortion depends on the metal. The alternative

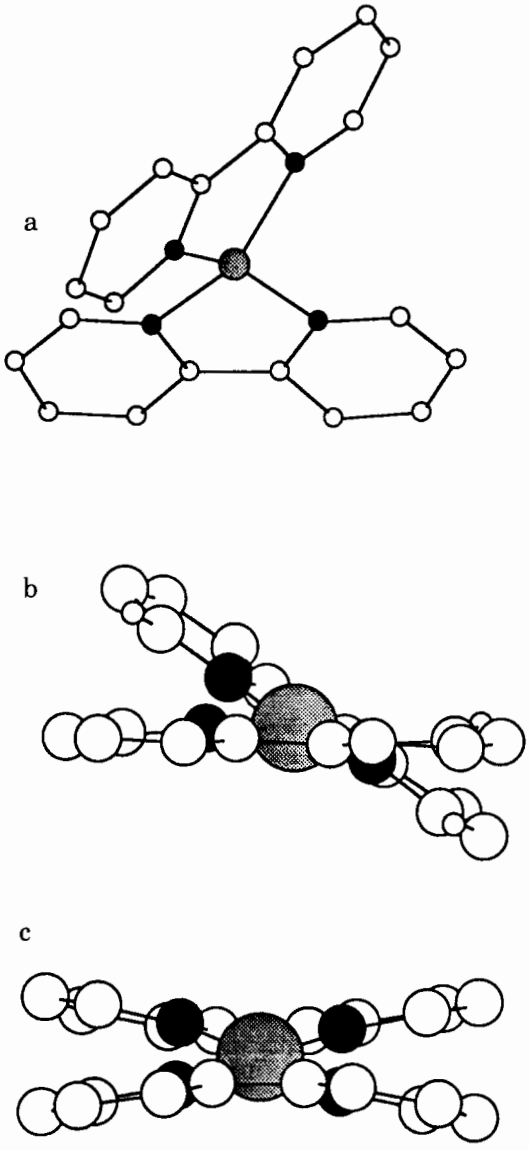


FIG. 5.

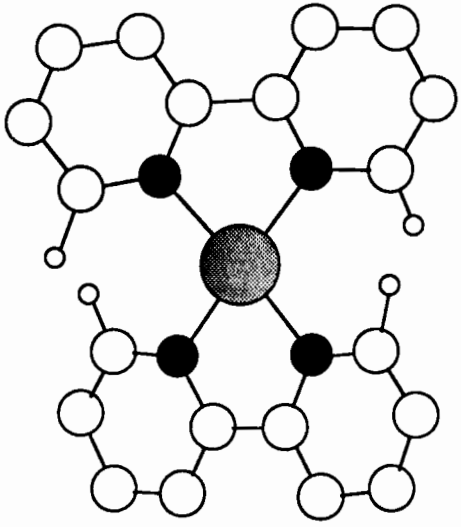


FIG. 6.

square-planar geometry is most commonly found associated with a d^8 configuration, but steric interactions result in a distortion towards a tetrahedral geometry (Fig. 5b) or a distortion of the ligand (Fig. 5c). This distortion is associated with the steric interactions between H_6 and $H_{6'}$ (Fig. 6) (*321*).

The tris complexes are based on an octahedral arrangement of the N_6 donor set about the metal (Fig.7), resulting in an overall D_3 symme-

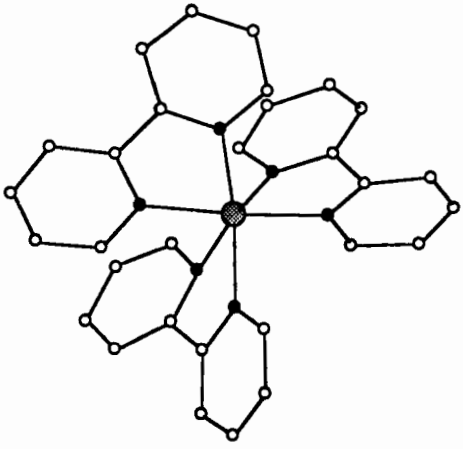


FIG. 7.

try. The change from O_h to D_3 symmetry has a number of interesting consequences, the most important of which is the resultant chirality. An $[M(bpy)_3]^{n+}$ ion is not superimposable on its mirror image, so it is, in principle, possible to isolate the two enantiomers of the compound. This has been achieved for many of the nonlabile tris (2,2′-bipyridine) complexes. Even in the case of labile metal ions, which racemize too rapidly for chemical resolution, the chirality may be expressed. The Pfeiffer effect, in which the optical activity is enhanced by the addition of a racemic labile metal complex to neutral, anionic, or cationic chiral environment compounds, was first detected in bpy and phen complexes. It is also of note that the adoption of the *cisoid* configuration results in close interatomic contacts between H_3 and $H_{3'}$ of the same ligand.

C. Properties

1. Oxidation States

The bpy ligand is remarkable for the wide range of formal oxidation states with which it is associated. Even in a single metal, it is possible to find bpy ligands associated with formal oxidation states ranging from -2 to $+7$. The principal interaction between the metal and the ligand is the σ-bonding resulting from an interaction of a vacant orbital on the metal and the lone pairs on the nitrogen atoms. Associated with this, is the presence of filled π and vacant π^* orbitals on the pyridine rings; the precise energies of the π and π^* orbitals depend on the metal ion, and the matching of energies enables bpy to act as a π-donor to high oxidation state complexes and a π-acceptor in low oxidation state complexes. The isolation of numerous low oxidation state complexes has led to discussion of the precise description of the bonding in the compounds. There is considerable evidence that the orbitals in which the excess of electrons are located possess predominantly ligand character. In other words, a complex $[M(bpy)_3]^0$ may be better represented as $[M^{III}(bpy^{-\cdot})_3]$. Electrochemical and spectroscopic studies, combined with calculations at various levels of sophistication, are in general agreement with this description (*372*).

2. Spectroscopic

The structures of bpy complexes have been probed by a remarkable variety of spectroscopic techniques, partly due to the availability of a wide range of isostructural complexes in which the electronic configu-

ration of the central metal ion varies. The complexes were widely used in the development of ligand field analyses, and their electronic spectra are particularly well studied. There are anomalies in the apparent ligand field strength of bpy on passing from one metal to another. In many cases the complexes are intensely colored, and the *d-d* transitions are swamped by charge transfer absorptions. The vibrational spectra have also been investigated in depth, and full analyses of the electronic and vibrational spectra of complexes are usually possible and indicate appropriate structural assignments.

The NMR spectra of both diamagnetic and paramagnetic complexes have also been widely investigated. The paramagnetic compounds have been of particular application in description of spin polarization in M—L systems. In general, the NMR spectra of the paramagnetic complexes are broadened and shifted. In contrast, the NMR spectra of the diamagnetic compounds are usually well resolved. The tris complexes are of particular interest, and the most noticeable feature is that the ^1H NMR signal from H_3 is often the lowest field resonance. This is interpreted in terms of Van der Waals deshielding of H_3, associated with the close H_3,$H_{3'}$ contacts, and shielding of H_6 by the metal.

Both the bis and tris complexes possess interesting photophysical properties and exhibit luminescent, phosphorescent, or fluorescent properties. These observations, combined with useful redox properties of both ground and excited states, have resulted in the investigation of such complexes as photocatalysts, photosensitizers, or photorelays for solar energy photoconversion systems. Indeed, such is their utility that it is rare to find a homogeneous or heterogeneous system that does not contain a bpy complex. Accordingly, numerous studies of the precise nature of the emitting states have been made; varying degrees of agreement have been reached.

3. Solution Properties

In this section general trends in the reactivity of specific complexes are discussed under the appropriate elements. The complexes of metals in low formal oxidation states (≥ 1) are very air sensitive and are readily oxidized to higher oxidation state compounds. Similarly, low oxidation state complexes may undergo oxidative addition reactions, with a change in oxidation state of +2.

The square-planar complexes $[M(bpy)_2]^{2+}$ are coordinatively unsaturated and undergo facile reactions with nucleophiles to yield $[M(bpy)_2X]^{n+}$, which may react further with loss of bpy.

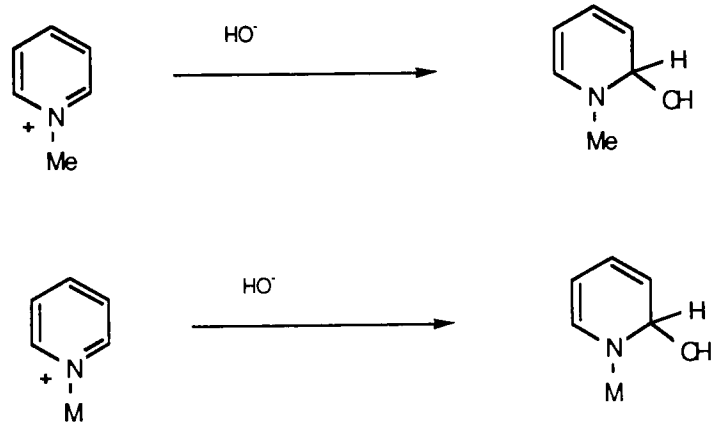


FIG. 8. Covalent hydration model.

The loss of bpy from octahedral tris complexes occurs readily only with labile metal ions; in general, forcing conditions are required. The Pfeiffer effects associated with $[M(bpy)_3]^{n+}$ complexes have also been discussed earlier. It is also clear that there are a number of unusual properties associated with $[M(bpy)_3]^{n+}$ ions, which may be categorized as intimate cation:nucleophile interactions. These have been interpreted in terms of nucleophilic attack at coordinated pyridine ligands, arguing an analogy between a metal-coordinated pyridine and a pyridinium ion (Fig. 8) though these proposals have not been accepted universally. At present the status of this "covalent hydration" model must be regarded as dubious; equally convincing explanations for the anomalous properties of $[M(bpy)_3]^{n+}$ complexes can be obtained by postulating tight ion-pairing or specific cation:nucleophile interactions (*157, 296, 299, 679, 814*). The formation and decomposition of $[M(bpy)_3]^{n+}$ complexes exhibits a marked pH dependence. This has been interpreted as being evidence for covalent hydration of the ligand but also fits for a simple Eigen–Wilkins type of mechanism involving monodentate bpy intermediates.

III. Homoleptic Complexes of 2,2'-Bipyridine

A. Group IA

The majority of reports dealing with Group IA complexes of 2,2'-bipyridine are concerned with the species $(M(bpy)_n$. The lithium complexes $Li_2(bpy)$ and Li(bpy) have been used (primarily by Herzog and

coworkers) as reagents for the preparation of a variety of low oxidation state transition metal complexes of 2,2′-bipyridine. The sodium complexes Na(bpy) and Na_2(bpy) are also known and behave similarly. The reaction of Na_2(bpy) with metallic tin gives Na(bpy) and Na_4Sn_9 (*209*).

The structures of these compounds have been the subject of some interest over the years, and a number of investigations have been reported. The complexes are extremely air sensitive and are prepared by the direct reaction of 2,2′-bipyridine with the alkali metal in an inert solvent. The compounds are paramagnetic [Li_2(bpy), μ_{eff} 1.04–1.58 μ_B over the temperature range 82–382.7 K; Li(bpy)·2dioxan, μ_{eff} 1.59–1.64 μ_B over the temperature range 81.4–293.2 K] (*364, 975*). The solutions are electron paramagnetic resonance (EPR) active, and the spectra are best interpreted in terms of close ion pairs containing the 2,2′-bipyridine radical anion $\{M^+ \cdots (bpy^{-\cdot})\}$ (*226, 449, 450*).

The interaction of alkali metal ions with 2,2′-bipyridine has also been investigated. A complex of stoichiometry $[Li(bpy)_2]^+$ has been established by ^7Li and ^{13}C NMR spectroscopy in $MeNO_2$ solution, though only very weak interactions were observed in Tetrahydrofuran (THF), dimethylformamide (DMF), MeOH, or propylene carbonate solution (*799*). Although a solid complex of this stoichiometry has not been isolated, the species Li(SCN)·bpy·H_2O (of unknown structure) has been reported (*943*). A similar ^{23}Na NMR study has established the formation of sodium complexes of 2,2′-bipyridine in $MeNO_2$ but not in better donor solvents (*799*). Although ^{39}K NMR studies indicate very weak interactions between K^+ and 2,2′-bipyridine in all solvents investigated (*799*), the solid complex [K(bpy)][BPh_4] is formed by the reaction of K[BPh_4] with 2,2′-bipyridine in Me_2CO (*319*).

B. Group IIA

There have been remarkably few studies of the homoleptic alkaline earth complexes of 2,2′-bipyridine. The complex $Be(bpy)_2$ is formed by the direct reaction of 2,2′-bipyridine with beryllium (*975*). The complex is paramagnetic (μ_{eff} 1.80–2.23 μ_B over the temperature range 79.9–383.1 K), and it is likely that the compound is of the type $\{Be^{2+} \cdots (bpy^{-\cdot})_2\}$ (*975*). The reaction of $Be(bpy)_2$ with lithium metal in THF, followed by treatment with dioxane, yields a diamagnetic material formulated $Li_2[Be(bpy)_2]$ (*356*).

The complex $Mg(bpy)_2$·3THF can be prepared by the direct reaction of magnesium with 2,2′-bipyridine in THF and is presumably of the

type $\{Mg^{2+} \cdots (bpy^{-\cdot})_2\}$ (*356*). The reaction of $\{Mg^{2+} \cdots (bpy^{-\cdot})_2\}$ with excess 2,2′-bipyridine in benzene yields $Mg(bpy)_3$, whereas reduction with sodium in THF/dioxane leads to $Na_4[Mg(bpy)_3]$ (μ_{eff} 0.95 μ_B) (*356*).

The paramagnetic complexes $Ca(bpy)_4$ (μ_{eff} 1.74–2.21 μ_B over the temperature range 81.5–293.7 K), $Sr(bpy)_4$, and $Ba(bpy)_4$ (μ_{eff} 2.33 μ_B) are prepared from the reaction of the metals with 2,2′-bipyridine in liquid ammonia at −78°C (*351, 352, 975*).

C. Lanthanum and the Lanthanides

A number of compounds of the stoichiometry $[M(bpy)_4]$ have been reported. The complex $[La(bpy)_4]$ (μ_{eff} 1.8 BM) is prepared by the reaction of $LaBr_3$ with $Li_2(bpy)$ in the presence of bpy (*257, 360*). Similar routes have been used for the synthesis of the extremely air-sensitive compounds $[Ce(bpy)_4]$ (μ_{eff} 2.85 μ_B) (*370*). $[Nd(bpy)_4]$ (μ_{eff} 3.62 μ_B) (*370*), and $[Tb(bpy)_4]$ (*715*). The reaction of bpy with the blue solutions of ytterbium or europium in liquid ammonia are reported to yield $[M(bpy)_3]$ or $[M(bpy)_4]$ species, depending on the precise conditions (*246, 715*). In solution, $[Yb(bpy)_4]$ exhibits a normal magnetic moment (μ_{eff} 2.79 BM) whereas $[Eu(bpy)_4]$ exhibits a novel form of temperature dependency that is attributed to an antiferromagnetic interaction between the metal center and the ligands (*246*).

Attempts to prepare $[Yb(bpy)_4]$ by the interaction of $YbBr_3$ with $Li_2(bpy)$ in the presence of excess bpy led to the formation of $Li[Yb(bpy)_4]\cdot 3THF$ (μ_{eff} 4.85 μ_B) (*370*). The complex $Na_5[Ce(bpy)_4]\cdot 5diox\cdot 2THF$ can be prepared by the reduction of $[Ce(bpy)_4]$ with sodium (*356*).

The complexes $[M(bpy)_3$ (M = Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, or Yb) have been spectroscopically identified as the products of the reaction of MCl_3 with $Li_2(bpy)$ and excess bpy in THF (*715*).

The compounds $[M(bpy)_3][SeCN]_3$ (M = Ce, Pr, Nd, Tb, Dy, Ho, or Er) have been prepared by the reaction of $M(SeCN)_3$ with bpy (*307, 849*). Conductivity studies of solutions of the compounds, together with infrared and crystallographic studies of the solids, suggested that a 6-coordinate formulation was appropriate. The ground-state magnetic properties and photophysical properties of the triplet excited states of $[M(bipy)_n]^{3+}$ complexes (M = Pr, Nd, Tb, Dy, or Er; n = 3 or 4) have been reported (*872*). The complexes are prepared by the reaction of the appropriae metal perchlorates with bpy in ethanol.

D. Transition Metals

1. Scandium and Yttrium

The black complex $[Sc(bpy)_3]$ is obtained from the reaction of $[Sc(THF)_3Cl_3]$ with $Li_2(bpy)$ in THF (*353*); the complex is paramagnetic with a markedly temperature-dependent magnetic moment (μ_{eff} 1.57–2.16 μ_B over the temperature range 80.8–353 K) (*353, 975*). A similar reaction of YCl_3 with bpy and $Li_2(bpy)$ in THF results in the formation of $[Y(bpy)_3]\cdot 3THF$ (*358*) or $Li[Y(bpy)_4]\cdot 3THF$ (*359*). The black, paramagnetic (μ_{eff} 1.84 μ_B) complex $[Y(bpy)_3]$ is formed on warming $Li[Y(bpy)_4]\cdot 3THF$ or $[Y(bpy)_3]\cdot THF$ or by the reaction of $[Y(THF)_3Cl_3]$ with $Na_2(bpy)$ (*358, 359*). On treatment with an excess of bpy, $[Y(bpy)_3]$ is converted to $[Y(bpy)_4]$ (*359*). Whereas sodium in THF converts it to $Na_3[Y(bpy)_3]\cdot 6THF$ (μ_{eff} 0.74 μ_B] (*355*).

The complex $[Sc(bpy)_3][SCN]_3$ is readily prepared by the reaction of an ethereal solution of $Sc(SCN)_3$ with a large excess of bpy (*165, 166*). The compound behaves as a 1:3 electrolyte in water and exhibits a single ionic thiocyanate stretching mode in the solid state. Loss of bpy occurs on treatment with ethanol to yield $[Sc(bpy)_2(SCN)_2][SCN]$.

2. Titanium, Zirconium, and Hafnium

The violet complex $[Ti(bpy)_3]$ is readily prepared by the reduction of THF solutions of $TiCl_4$ with bpy and $Li_2(bpy)$ (*371, 769*), or lithium (*621*). The compound is also obtained by the reduction of $TiCl_3$ in THF by sodium amalgam in the presence of an excess of bpy (*750*) or as a sublimate on heating $Li[Ti(bpy)_3]$ (*715*). The compound is near diamagnetic (μ_{eff} 0.55 μ_B) (*371, 372, 621*). The reduction of MCl_4 (M = Zr or Hf) by sodium amalgam (*750*) or by $Li_2(bpy)$ (*374*) in the presence of excess bpy leads to the related $[M(bpy)_3]$ complexes. $[Zr(bpy)_3]$ is also close to diamagnetic (μ_{eff} 0.31 μ_B) (*372, 374, 375*). There has been considerable interest in the nature of $[Ti(bpy)_3]$, which has been formulated as $[Ti^{III}(bpy^{-\cdot})_3]$ or $[Ti^0(bpy)_3]$ by various authors (*263, 326, 372, 454, 481, 503, 504, 621, 706, 715, 769*). In particular, there has been disagreement over the interpretation of the electronic (*326, 454, 481, 715*) and vibrational (*504, 769*) spectra of the compound. The complex does not give an EPR spectrum, but the upfield shift of the ring protons in the ^1H NMR spectrum is interpreted in terms of a singlet ground state (*226, 263*).

A structural analysis of $[Ti(bpy)_3]$ has shown that the complex possesses a distorted octahedral geometry (Ti—N, 2.09 ± 0.03 Å;

$\angle$N—M—N, 72.81 ± 1.5°) (*16, 17*). The M—N bond lengths appear to be more compatible with a Ti(0) rather than a Ti(III) formulation. Although [Ti(bpy)$_3$] does not react with pyridine, phosphines, or aromatic hydrocarbons (*361*), it has been shown to be of use as a reducing agent. Thus, reaction with benzophenone results in the formation of a Ti(IV) complex of $\{(Ph_2CO)_2\}^{2-}$ (*263*) whereas an unusual dimeric anion was obtained with $(NC)_2C{=}C(CN)_2$ (*205*). The chemical vapor decomposition of [Ti(bpy)$_3$] at low pressures (10^{-4}–10^{-6}) and moderate temperatures (200–500°C) has been investigated; thin films of titanium containing varying amounts of carbon, hydrogen, and nitrogen can be laid down on various substrates (*455, 639–642*).

Lower formal oxidation states are known, and Li[Ti(bpy)$_3$] is a second product from the reaction of lithium of Li_2(bpy) with $TiCl_4$ and bpy (*371, 621, 715*). The compound is blue-violet (*326, 503, 715*) and paramagnetic (μ_{eff} 1.7 μ_B, g_{av} 2.0074 ± 0.0002) (*371, 500, 503, 621*). A preliminary structural study of the [Ti(bpy)$_3$]$^-$ anion indicated a Jahn–Teller distorted octahedral geometry about the metal, with M—N_{av} $\approx$ 2.6 Å and $\angle$N—M—N $\approx$ 65° (*16, 17*). Oxidation of Li[Ti(bpy)$_3$] by PhNC gives [Ti(bpy)$_3$], but no further reaction appears to occur (*361*).

Electrochemical studies indicate that [Ti(bpy)$_3$] undergoes at least two reversible reductions to the Ti(-I) and Ti(-II) formal oxidation states (*773, 775*). The Ti(-II) state is accessible by a reversible chemical reduction of [Ti(bpy)$_3$] with lithium (*376*). The green complexes Li_2[Ti(bpy)$_3$]·*n*THF are diamagnetic (*375, 503*). The reduction of [Ti(bpy)$_3$] by sodium proceeds further, and a formally Ti(-III) complex, Na_3[Ti(bpy)$_3$]·7THF (μ_{eff} 1.75 μ_B) can be isolated (*356, 503*). The related complexes Li[Zr(bpy)$_3$] (μ_{eff} 1.7 μ_B) and Li_2[Zr(bpy)$_3$] (μ_{eff} 1.10 μ_B) are prepared by the reduction of [Zr(bpy)$_3$] with Li_2(bpy) (*374, 375*).

There is no evidence for the formation of $[M(bpy)_3]^{n+}$ (M = Ti; *n* = 1, 2, or 3) (*371*).

3. *Vanadium, Niobium, and Tantalum*

The complex [V(bpy)$_3$] was first reported as a violet, extremely airsesntivie compound obtained by the reduction of green aqueous ethanolic solutions of [V(bpy)$_3$]I_2 by magnesium or zinc (*621*). The complex is also obtained by the reduction of $[V(bpy)_3]^{2+}$ by Li[AlH_4] (*365*), sodium (*750*) or zinc amalgam (*188*), or the reaction of [$V(CO)_4$(dppe)], [$V(CO)_2$(dppe)$_2$] or [$V(CO)_3\{(Ph_2PCH_2)_3CCH_3\}$] with bpy (*59*). The compound is paramagnetic (μ_{eff} 1.78–1.82 BM over the temperature range 81.0–382.5 K) (*188, 502, 621, 975*) and EPR active (g_{av} 1.9831,

$|A^V|$ 83.5 ± 1.0 g) (*188, 226, 500, 502*). The niobium analog $[Nb(bpy)_3]$ (μ_{eff} 1.67–1.69 μ_B over the temperature range 81.0–383.4 K) can be prepared as a violet crystalline solid by the reaction of $NbCl_5$ with bpy and $Li_2(bpy)$ (*369, 944, 975*) or sodium amalgam (*750*). The reaction of $TaCl_5$ with bpy in the presence of sodium amalgam produces the tantalum compound $[Ta(bpy)_3]$ (μ_{eff} 1.77 μ_B) (*750*).

The bonding in $[V(bpy)_3]$ has been the subject of some conjecture, with various authors championing formulation $[V^{III}(bpy^{-\cdot})_3]$ and others $[V^0(bpy)_3]$ (*77*). The infrared (*504*) and electronic spectra (*275, 326, 454, 480, 769*) have been used to support these varying structures. The crystal structure of $[V(bpy)_3]$ has been reported; the metal is in a distorted octahedral environment, with (V—N, 2.10 ± 0.03 Å; ∠N—M—N, 73.6 ± 1.5°) (*16, 17*). The spectroscopic and structural data indicate that there is a considerable degree of interaction between the metals and the bpy ligands, which probably renders discussion of formal oxidation states in the compound futile.

The complex $[V(bpy)_3]$ is reported not to react with PhNC, py, Et_3P, Ph_3P, PhH, mesitylene, 1,2-bis(dimethylarsino)benzene, or 1,2-bis (diethylphosphino)-ethene (*361*).

The formally V(-I) (d^6) oxidation state is readily accessible, and $[V(bpy)_3]^-$ is prepared by the reduction of $[V(bpy)_3]$ or $[V(bpy)_3]^{2+}$ with $Li[AlH_4]$ (*621*) or by the reaction of VCl_3 with bpy and $Li_2(bpy)$ (*715*). The salt $Li[V(bpy)_3]\cdot 4THF$ is diamagnetic (*621*) and reacts with PhNC to yield $[V(bpy)_3]$ (*361*). Thermolysis of $Li[V(bpy)_3]\cdot 4THF$ yields $[V(bpy)_3]$ (*715*). A preliminary structural study of the lithium salt suggests that the vanadium is in a roughly octahedral environment with $M—N_{av} \approx 2.4$ Å and $\angle N—M—N \approx 65°$ (*16, 17*). The diamagnetic niobium analog $Li[Nb(bpy)_3]\cdot 3\cdot 5THF$ is prepared by the reduction of $NbCl_5$ by $Li_2(bpy)$ in the presence of excess bpy (*373*). The compound $Na_3[V(bpy)_3]\cdot 7THF$ (μ_{eff} 2.76 μ_B) has also been reported (*355*).

Electrochemical studies of solutions containing $[V(bpy)_3]^{n+}$ suggest that the formal oxidation states -I, 0, I, II, and III are all accessible (*66, 275, 502, 553, 619, 773–775, 819*). Violet-red solutions containing $[V(bpy)_3]^+$ are obtained by the redistribution reaction between $[V(bpy)_3]$ and $[V(bpy)_3]^{2+}$, and the salt $[V(bpy)_3]I\cdot py$ (μ_{eff} 2.8 ± 0.04 μ_B) can be isolated by the addition of iodide and pyridine (*454, 480, 502, 621*). Solutions containing $[V(bpy)_3]^+$ are EPR silent, and it has been suggested that the solid species contain equimolar amounts of $[V(bpy)_3]$ and $[V(bpy)_3]^{2+}$ (*502*). Solution studies have established the disproportionation of $[V(bpy)_3]I\cdot py$ to $[V(bpy)_3]$ and $[V(bpy)_3]^{2+}$ on redissolution (*621*), and it is presumably this equilibrium that pre-

vents the preparation of $[V(bpy)_3]^+$ by chemical reduction of $[V(bpy)_3]^{2+}$.

Salts of the green $[V(bpy)_3]^{2+}$ ion (μ_{eff} 3.81 μ_B) are well characterized and readily prepared by the direct reaction of aqueous vanadium(II) solutions with bpy (*66, 224, 275, 454, 480, 502, 621, 769*). Both violet and green isomers of $[V(bpy)_3][ClO_4]_2$ have been described (*621*). The equilibria in V(II)-bpy systems have been investigated; $\lg K_1 = 4.91$, $\lg K_2 = 4.67$; $\lg K_3 = 3.85$ (*164*). The dissociation of bpy from $[V(bpy)_3]^{2+}$ is avid dependent, and a mechanism involving competing pathways involving ligand loss from both protonated and neutral monodentate bpy intermediates has been proposed (*718*). The ion is readily oxidized to the labile vanadium(III) analog, but the rate of electron transfer is slower than that observed with $[Cr(bpy)_3]^{2+}$; the ion $[V(bpy)_3]^{2+}$ has been shown to reduce $[Ru(bpy)_3]^{3+}$ (*66*). Salts of $[V(bpy)_3]^{2+}$ have been evaluated as photographic developers (*741*). The electronic spectra are of some interest and display a charge transfer (CT) band at 1.92 eV (*66, 275, 454, 480, 502*). The self-exchange reaction of $[V(bpy)_3]^{2+/3+}$ in H_2O/*tert*-butanol mixtures has been investigated (*553*).

In view of available data, it is surprising that the detailed investigations of the photochemical and photophysical properties of $[V(bpy)_3]^{n+}$ systems were not made until 1986, though the fluorescence spectrum of $[V(bpy)_3]^{2+}$ had been reported in 1963 (*784, 872*). The lowest excited state of $[V(bpy)_3]^{2+}$ is probably $^2E/^2T_1$ or ^4MLCT in character and has a lifetime of 500 psec in EtOH at room temperature (*819*). The V(III) complexes are labile, and photolysis of $[V(bpy)_3]^{2+}$ in water in the presence of an electron acceptor yields $[(bpy)_2V(\mu\text{-}OH)_2V(bpy)_2]^{4+}$ (*819*). Despite the reported lability of vanadium(III) bpy complexes, the salts $[V(bpy)_3][SeCN]_3$ and $[V(bpy)_2[SeCN]_3$ have been described (*269, 916*).

4. Chromium, Molybdenum, and Tungsten

Electrochemical studies of solvents have indicated the existence of a wide range of $[M(bpy)_3]^{n+}$ (M = Mo or Cr) complexes ($n+ = 3, 2, 1, 0, -1$), (*217, 218, 402, 405, 621, 769, 772–775, 791, 856, 940*). The violet-red, diamagnetic, air-sensitive complex $[Cr(bpy)_3]$ can be formed by the reduction of $[Cr(bpy)_3]^{2+}$ or $[Cr(bpy)_3]^+$ by sodium (*621, 715*) or $Li[AlH_4]$ (*365*), the reaction of $Cr_2(OAc)_4$ with bpy in the presence of sodium amalgam (*750*), the reaction of bpy with $K_6[Cr(CN)_6]$, $[Cr(CO)_6]$, or *cis*-$[Cr(DPPM)_2(CO)_2]$ in liquid ammonia (*58, 61–63*). The crystal structure of $[Cr(bpy)_3]$ has been determined; the metal is in

an octahedral environment, with (Cr—N, 2.08 ± 0.03 Å; ∠N—M—N, 74.7 ± 1.5°) (*16, 17*). Attempts to prepare $[M(bpy)_3]$ (M = Mo or W) by reaction of $[M(CO)_6]$ with bpy lead to $[M(bpy)_2(CO)_2]$ (*63*), but the complexes can be prepared from bpy and $[Mo(C_6H_6)_2]$ (*217, 218*) or $[MCl_4(bpy)]$ (M = Mo or W) (*750*). The reduction of $[Mo(bpy)_3]Cl_3$ or $[WCl_6]$/bpy by $Li_2(bpy)$ has also been shown to yield $[M(bpy)_3]$ (M = Mo or W) (*363, 368*). The reaction of $[M(bpy)_3]$ (M = Cr, Mo, or W) with PhNC leads to the complex $[Cr(PhNC)_6]$; this reaction can be reversed in molten bpy (*361*). The low formal oxidation state complexes $Li[Cr(bpy)_3]\cdot 4THF$, $Na_3[Cr(bpy)_3]\cdot 7THF$, $Na_2[Cr(bpy)_3]\cdot 7THF$, and $Ca_3[Cr(bpy)_3]\cdot NH_3$ have also been reported (*357*).

Deep blue salts of $[Cr(bpy)_3]^+$ can be prepared by magnesium or sodium (*62, 769*) reduction of $[Cr(bpy)_3]^{2+}$. They can also be prepared by the reproportionation reaction between $[Cr(bpy)_3]$ and $[Cr(bpy)_3]^{2+}$ (*621*). In aqueous conditions, the $[Cr(bpy)_3]^{n+}$ system is complicated by a range of disproportionation and photolytic reactions (*33, 287, 621, 856, 857, 916*). The photochemical reaction of $[Cr(biphenyl)_2]Cl$ with bpy in MeOH leads to $[Cr(biphenyl)_2]$ and $[Cr(bpy)_3]^{2+}$ (*346*). Electron transfer reactions between $[Cr(bpy)_3]$ and $[Cr(bpy)_3]^+$ have been studied in dimethylformamide DMF (*770*). The vibrational and electronic spectra of $[Cr(bpy)_3]^{n+}$ complexes have been widely studied and variously interpreted in terms of localized or delocalized bonding schemes (*275, 326, 372, 406, 454, 481, 501, 504, 621, 769, 891*). The magnetic and EPR properties of the complexes have also been investigated (*226, 500, 621, 975*). An early study of $[Cr(bpy)_3]^+$ complexes suggests that significant electron density resides at the edges of the bpy rings (*947*).

Numerous salts of $[Cr(bpy)_3]^{2+}$ have been prepared by reaction of chromium(II) solutions with excess bpy in inert conditions; the complexes are paramagnetic and readily oxidized to $[Cr(bpy)_3]^{3+}$ (*33, 567, 621, 905, 909, 918*). Studies of the NMR spectra of $[Cr(bpy)_3]^{2+}$ salts have given some information on their electronic structure (*527, 528*).

Salts of $[Cr(bpy)_3]^{3+}$ are best prepared by chemical or electrochemical oxidation of chromium(II) systems (*857*). Care must be taken to minimize redox reactions that give rise to $[Cr(bpy)_2L_2]^{3+}$ (*52, 75, 219, 245, 589, 621, 721, 856, 857, 872, 917, 921*). Salts of $[Cr(bpy)_3]^{2+}$ have been evaluated as photographic developers (*741*) and used for the reduction of the binuclear $\{Fe^{III}, Fe^{III}\}$ center in hemerythrin (*26, 27*). A mass spectroscopic study of $[Cr(bpy)_3]^{2+}$ salts has been reported (*133*).

The principal interest in salts of $[Cr(bpy)_3]^{3+}$ lies in their photochemical properties; in particular the redox interactions between ground- and excited-state $[Cr(bpy)_3]^{3+}$ with ground- and excited-state $[Ru(bpy)_3]^{2+}$ are of great significance to photochemical systems (*37, 42,*

81, 99, 172, 231, 254, 258, 272, 405, 761, 817, 431). Related to these are the numerous studies of the electronic, photochemical, and redox properties of $[Cr(bpy)_3]^{3+}$ (*4, 20, 24, 28, 33, 35, 36, 40, 41, 53, 69, 80, 82, 116, 146, 148, 179, 227, 229, 230–232, 252, 255, 275, 283, 289, 321, 340, 349, 415, 427, 431, 432, 444, 446, 460–462, 472, 475, 487, 490, 505, 506, 510, 548, 568, 577, 578, 607, 621, 661, 684, 691, 694, 696, 697, 751, 760, 772, 786, 788, 798, 810, 813, 817, 822, 864, 865, 875, 888, 889, 897, 921, 922, 932, 933, 939, 942, 971*). These extremely important applications are reviewed elsewhere (*182, 350, 387, 428, 430, 471, 529, 814, 969*). The photochemical conversion of norbornadiene to quadricyclene is catalyzed by $[Cr(bpy)_3]^{3+}$ (*445*), as are a number of other photochemical reactions (*779*).

The $[Cr(bpy)_3]^{3+}$ cation is light sensitive, and photosolvation reactions are well characterized; there is rather convincing evidence for the involvement of a $\{Cr(bpy)_3(H_2O)\}^{3+}$ or $\{Cr(bpy)_3(OH)\}^{2+}$ intermediate in photoaquation processes (*427, 429, 433, 474, 577, 578, 657, 661, 869*). In DMF an autocatalytic photosolvation involving a chromium(II) intermediate is proposed (*732, 733*). Pulse radiolysis of $[Cr(bpy)_3]^{3+}$ has been investigated under a variety of conditions (*388, 934*).

As expected, the $[Cr(bpy)_3]^{3+}$ ion is chiral (*249, 302, 591*). The racemization of $[Cr(bpy)_3]^{3+}$ has been studied and the activation volume shown to favor an intramolecular twist mechanism (*533*). A partial photoresolution (giving 1–2% ee) by irradiation with circularly polarized light was reported in 1972 (*461*). The $[Cr(bpy)_3]^{3+}$ cation resembles $[Co(bpy)_3]^{3+}$ and exhibits strong ion-pairing interactions (*76, 633*).

Salts of $[Mo(bpy)_3]^{3+}$ with halides have been reported, but it was claimed that the perchlorates could not be obtained by metathesis, and the compounds merit further study (*368, 621*). Purple $[Mo(bpy)_3][BF_4]_2$ has been prepared by the reaction of bpy with *cis*-$[Mo(CO)_2(bpy)_2(MeCN)][BF_4]_2$ (*156*).

5. *Manganese, Technetium, and Rhenium*

The paramagnetic complex $[Mn(bpy)_3]\cdot 2THF$ (μ_{eff} 3.81–4.31 μ_B over the temperature range 81.2–413.4 K) (*329, 366, 367, 416, 975*) is prepared by the reaction of $[Mn(bpy)_3]Br_2$ with $Li_2(bpy)$ (*366*). The rhenium analog, $[Re(bpy)_3]$ (μ_{eff} 4.1 BM), is obtained from the reaction sodium amalgam with $ReCl_4(THF)_2$ in the presence of bpy, though it is noted that the corresponding reaction of $ReCl_5$ does not yield this product (*750*). The further reduction of $[Mn(bpy)_3]$ by lithium yields the formally Mn(-I) complex, $Li[Mn(bpy)_3]\cdot 4THF$, which can also be ob-

tained by the reaction of $[Mn(bpy)_3]Br_2$ with excess $Li_2(bpy)$ or of $[Mn(bpy)_3]$ with Li(bpy) [but not with $Li_2(bpy)$] (*367*). The complex $Na_3[Mn(bpy)_3]\cdot 8THF$ (μ_{eff} 6.01 BM) is reported to be the product of the reaction of vigorous reduction of $[Mn(bpy)_3]$ (*355*). Analysis of the infrared and electronic spectra of $[Mn(bpy)_3]$ and $Li[Mn(bpy)_3]\cdot n THF$ suggests that the compounds occupy an intermediate position between limiting ionic $[M^{III}(bpy^{-\cdot})_3]$ and covalent $[M^0(bpy)_3]$ systems (*326, 454, 769*). Whereas $[Mn(bpy)_3]\cdot 2THF$ exhibits Curie–Weiss behavior over the temperature range 20–160 K, the compound $Li[Mn(bpy)_3]\cdot 4THF$ does not (*416*).

The electrochemical behavior of $[Mn(bpy)_3]^{n+}$ systems has been investigated, and it is evident that a number of oxidation states are accessible. Reduction of $[Mn(bpy)_3]^{2+}$ to the $[Mn(bpy)_3]^+$, $[Mn(bpy)_3]$, and $[Mn(bpy)_3]^-$ states is well established (*105, 403, 646, 790, 896, 941*). Salts of $[Mn(bpy)_3]^+$ have not yet been isolated, and it seems likely that the formally Mn(I) complex exhibits a similar disproportionation reaction to that noted for $[Cr(bpy)_3]^+$ (*105*). Attempted chemical reduction of $[Mn(bpy)_3]^{2+}$ by $Li[AlH_4]$ led to the formation of $[Al(bpy)_3]$ (*365*).

Salts of $[Mn(bpy)_3]^{2+}$ are prepared by the direct reaction of an excess of bpy with an appropriate manganese(II) salt (*588, 621*). The complexes are high-spin paramagnetic (μ_{eff} 5.98 BM) (*149, 621, 721*) and exhibit characteristic EPR spectra (G = 2.01) (*646, 882*). The stability constants of manganese(II) bpy complexes have been determined (*621*). A number of studies of the infrared and electronic spectra of $[Mn(bpy)_3]^{2+}$ salts have been reported (*149, 308, 319, 454, 621, 721*). The rôle of manganese in the photosynthetic process has excited some interest, and $[Mn(bpy)_3]^{2+}$ has been shown to quench chlorophyll *a* singlet and triplet excited states but does not give rise to radical ion species (*94, 333*). No parent ion was observed in the laser ionization mass spectrum of $[Mn(bpy)_3]Br_2$ (*34*); the interaction of $[Mn(bpy)_3]^{2+}$ salts with Ar^+ has also been investigated (*924*). The thermal decomposition of $[Mn(bpy)_3]Br_2$ has also been investigated (*207*); the corresponding picrate salts are explosive (*712*). A Mössbauer study of low concentrations of $[Fe(bpy)_3][ClO_4]_2$ doped into the isomorphic $[Mn(bpy)_3][ClO_4]_2$ has been reported (*78*). The optical properties of $[Mn(bpy)_3]^{2+}$ have been examined (*621*) and Pfeiffer effects demonstrated with *l*-malic acid (*478*).

Salts of $[Mn(bpy)_3]^{2+}$ have proved to be of some interest as catalysts for the periodate oxidation of toluidine (*911*), the dioxygen oxidation of *iso*-propylbenzene to cumene hydroperoxide (*881*), the oxidation of tetrahydronaphthalene to tetralone (*756*), and aldol condensations (*419*), and as photocatalysts for the dimerization of phenyl vinyl ether (*638*).

The role of $[Mn(bpy)_3]^{2+}$ as an oxidation catalyst suggests the possible involvement of Mn(III) complexes, but no successful synthesis of $[Mn(bpy)_3]^{3+}$ salts has yet been reported. Electrochemical studies indicate that $[Mn(bpy)_3]^{2+}$ salts undergo an irreversible oxidation at moderately positive potentials (*403, 646*). In the presence of water, the product of the oxidation is the dimeric complex ion $[(bpy)_2MnO_2Mn(bpy)_2]^{3+}$ (*646*), which is also obtained from persulphate oxidation of $[Mn(bpy)_3]^{2+}$ (*687*).

Complexes of apparent 1 : 2 stoichiometry, $[Mn(bpy)_2][SbCl_6]_2$, have been reported (*565, 566*).

The violet complex $[Re(bpy)_3]Cl_3$ has been isolated in low yield from the reaction of $[Hbpy]_2[ReCl_5]$ with an excess of bpy; reaction with water results in ligand loss and the formation of $[ReO(bpy)Cl_3]$ (*132*).

6. Iron, Ruthenium, and Osmium

This triad of elements have played a crucial role in the development of the chemistry of 2,2'-bipyridine. The characteristic red color of $[Fe(bpy)_3]^{2+}$ was first observed by Blau in his pioneering studies on 2,2'-bipyridine (*73–75*), and iron complexes of bpy have continued to be of interest in the past century. The complexes of iron, ruthenium, and osmium probably account for about a third of all literature references to 2,2'-bipyridine complexes. This in part represents the facile synthesis of the complexes, their high stability, and extensive redox chemistry. The recent interest in the use of these compounds as photocatalysts has led to an explosive interest in the literature. Recent reviews have concerned themselves generally or partially with the chemistry of iron (*342, 552, 688, 814*) and ruthenium (*800, 803–806, 814*) complexes of 2,2'-bipyridine, so these complexes are not discussed further here. In particular, the reader is referred to excellent recent reviews of the photochemical applications of these compounds (*41, 43, 44, 176, 194, 443, 624, 625, 877, 954*).

Salts of $[Os(bpy)_3]^{2+}$ are best prepared by the reaction of bpy with $[Os(bpy)_2Cl_2]$ (*102*) or by prolonged reaction with other osmium complexes (*56, 318, 621*). The crystal structure of $[Os(bpy)_3][PF_6]_2$ reveals identical molecular properties to the corresponding ruthenium complex (*159*). Studies of the ^{13}C and ^{1}H NMR spectra of $[Os(bpy)_3]^{2+}$ salts have probed the metal-ligand interaction (*161, 204, 699*). The complex ion is nonlabile and is readily resolved as its antimonyl tartrate (*83, 590, 612, 621, 906*). The ^{189}Os Mössbaur spectra of $[Os(bpy)_3]^{2+}$ salts have been measured (*948*).

Like the iron and ruthenium analogs, $[Os(bpy)_3]^{2+}$ salts have been widely investigated as potential photocatalysts and electron transfer

agents (*18, 67, 77, 85, 99, 100, 101, 104, 119, 123, 124, 137, 151, 168, 172, 173, 175, 177, 197–201, 220, 247, 250–252, 254, 256, 257, 261, 274, 285, 289, 290, 295, 315, 391, 395, 438, 440, 444, 481, 491–496, 509, 517, 526, 549, 563, 571, 572, 574, 591, 592, 603, 606, 614, 645, 671, 678, 681, 698, 700, 713, 717, 759, 771–773, 775, 796, 808, 815, 816, 867, 876, 932, 938, 939*). The precise electronic nature of the excited and the emitting states of $[Os(bpy)_3]^{2+}$ has been debated as much as those for $[Os(bpy)_3]^{3+}$; similar controversy has raged over the localized or delocalized nature of the photochemically important excited states. Related to these studies are a number of investigations of $[Os(bpy)_3]^{2+}$ salts immobilized in various matrices (*25, 221, 423, 439, 555, 658, 764, 966*) or in micellar media (*97, 701, 702, 798*).

In aqueous solution, electrochemical studies indicate the ready availability of $[Os(bpy)_3]^{3+}$, $[Os(bpy)_3]^{2+}$, $[Os(bpy)_3]^{+}$, and $[Os(bpy)_3]$ though the description of the low-valence compounds is probably more appropriate in terms of ligand-centered radicals (*645*). Chemical oxidation of $[Os(bpy)_3]^{2+}$ salts leads to $[Os(bpy)_3]^{3+}$ (*131, 211, 621*). Among the numerous studies of electron transfer processes with $[Os(bpy)_3]^{n+}$, the use of NMR line-broadening methods to determine the self-exchange reactions of $[Os(bpy)_3]^{2+/3+}$ are of note (*134, 210, 211*), as are interactions of $[Os(bpy)_3]^{2+}$ with cytochrome *c* (*144*), metmyoglobin (*621*), and stellacyanin (*238*). The pulse radiolysis of $[Os(bpy)_3]^{n+}$ solutions has been reported (*681*). The interaction of $[Os(bpy)_3]^{3+}$ with water results in the evolution of O_2, and the mechanism of this reaction has concerned many workers (*151, 534, 570, 678, 683, 808, 815, 818*). Rival mechanisms have championed nucleophilic attack at the metal or at the ligand to give a pseudo-base; the situation is not yet conclusively resolved. The formation of outer sphere complexes with $[Os(bpy)_3]^{2+}$ and (in particular) $[Os(bpy)_3]^{3+}$ has been shown (*88, 296, 744, 814*); the role of this tight interaction in the reduction of water is not yet clear. However, the existence of unique interactions with hydroxide (but not H^+) (*797*) is clearly important and may be important in the specific deuteration reactions exhibited by $[Os(bpy)_3]^{2+}$ (*964*). In liquid sulphur dioxide solution, $[Os(bpy)_3]^{4+}$ is accessible (*285*). Like the other tris (2,2-bipyridine) complexes, $[Os(bpy)_3]^{2+}$ salts are toxic and inhibit the action of acetylcholinesterase (*497*).

7. *Cobalt, Rhodium, and Iridium*

The nature of the species formed by the chemical or electrochemical reduction of $[Co(bpy)_3]^{n+}$ has been the subject of some discussion. Chemical reduction with sodium amalgam, $Na[BH_4]$ (but not

$Li[AlH_4]$, or electrosynthesis leads to $[Co(bpy)_3]^+$ (μ_{eff} 2.71 BM) or dark blue $[Co(bpy)_3][ClO_4]$ (μ_{eff} 2.53 BM) (*6, 69, 103, 171, 262, 326, 347, 453, 454, 602, 621, 721, 769, 772, 785, 802*). The electronic spectra and stabilities of the two complexes differ (*802*), and electrochemical studies clearly indicate that ligand loss from $[Co(bpy)_3]^+$ is facile (*900*). In some of the earlier spectroscopic studies, it is not clear which of these two species was actually studied. The salt $[Co(bpy)_3]Cl \cdot H_2O$ has been structurally characterized and possesses nearly identical bond parameters to the corresponding cobalt(II) complex (Co—N_{av}, 2.11 Å (*884*). The report that the reaction of $[Co(bpy)_3]^{3+}$ with $[BH_4]^-$ over prolonged periods leads to ligand decomposition and the formation of NH_3 deserves further study (*139*). The d^6 cation $[Co(bpy)_2]^+$ may well prove to be a solvento species $[Co(bpy)_2S_2]^+$, but it has been shown to be active as a catalyst for the hydrodimerization of $MeCOCH{=}CH_2$ to $MeCO(CH_2)_4COMe$ (*459*) and is claimed to act as a catalyst precursor for acrylonitrile electroreduction (*643*). Reactions of $[Co(bpy)_2]^+$ with N_2O, alkyl halides, and nitrosyls have been reported (*46, 47, 941, 972*). The reaction of $[Co(bpy)_3]^+$ with bicarbonate yields predominantly CO, which is scavenged by excess $[Co(bpy)_3]^+$ (*465*). This is of interest in view of the observation that the direct reaction of cobalt with CO in the presence of bpy leads to $[Co(bpy)_3][Co(CO)_4]_2$ (*57*). It is claimed that in propylene carbonate, MeCN, or DMF solution, a further 2-electron reduction of $[Co(bpy)_n]^+$ to $[Co(bpy)_n]^-$ occurs (*208, 320, 789, 900*). The zero-valent complex $[Co(bpy)_2]$ can be prepared by MVS methods (*320*). The complex is EPR and NMR silent (μ_{eff} 2.02 μ_B), and readily oxidized to cobalt(I) species. In the presence of excess bpy, the MVS reaction yields $[Co(bpy)_3]$ (*320*), which can also be obtained from the reaction of $Li_2(bpy)$ with $CoCl_2$ (*362, 975*). Solutions containing $[Co(bpy)_3]^+$ (generated *in situ* from $[Co(bpy)_3]^{2+}$ and $[BH_4]^-$) have found some use as deoxygenation reagents (*392, 393*) and reductants (*730, 767*).

Numerous studies of the redox behavior of $[Co(bpy)_3]^{n+}$ ions in a range of solvents have been reported (*15, 33, 105, 110, 111, 140, 208, 347, 484, 531, 532, 582, 602, 621, 644, 668, 734–737, 739, 752, 766, 768, 772, 777, 793, 802, 900, 941, 959, 972*). It is clear that various 1 : 2 and 1 : 3 complexes in oxidation states ranging from −1 to +3 are accessible.

The ion $[Rh(bpy)_2]^+$ is obtained by 2-electron chemical or electrochemical reduction of $[Rh(bpy)_3]^{3+}$ and undergoes a further reduction to $[Rh(bpy)_2]$ (*114, 115, 588, 621*). The zero-valent complex $[Rh(bpy)_2]$ is EPR active. The oxidation of $[Rh(bpy)_2]^+$ by a variety of oxidants has been studied (*46, 114, 145, 647, 942*); in MeCN, dioxygen leads to

$[Rh_2L_4(MeCN)_2]$ whereas a mixture of Cl_2 and O_2 gives a red dimer that is thought to be peroxo-bridged (*114*). The ion undergoes a number of pH-dependent interactions with water (*145*). Salts of $[Rh(bpy)_2]^+$ have been investigated as catalysts for hydrogenation (*628, 989*) and the water-gas shift reaction (*579*). Pulse radiolysis of $[Rh(bpy)_3]^{3+}$ is thought to lead to transient $[Rh(bpy)_3]^{2+}$, which undergoes slow conversion to $[Rh(bpy)_2]^+$ (*651*). The reduction of *cis*-$[Rh(bpy)_2Cl_2]^+$ by hydrogen has been shown to be autocatalytic (*630*). Salts of $[Rh(bpy)_2]^+$ are reported to be formed in the oxidative thermolysis of $[Rh(CO)(bpy)(PPh_3)_2][ClO_4]$ (*519*). Electrochemical studies indicate that $[Rh(bpy)_3]^{3+/2+}$ and $[Rh(bpy)_2]^{2+/+/0/-}$ are all accessible in MeCN, with $[Rh(bpy)_3]^{2+}$ undergoing facile ligand loss (*466*).

The direct reaction of bpy with cobalt(II) salts in aqueous or polar nonaqueous solvents results in the formation of $[Co(bpy)_3]^{2+}$ salts. The d^7 $[Co(bpy)_3]^{2+}$ ion is labile and easily oxidized to $[Co(bpy)_3]^{3+}$, and the preparation of the cobalt(II) complexes is best performed under an inert atmosphere (*75, 245, 399, 418, 581, 621, 755*), though the ion does not directly bind O_2 (*855*). Salts of the paramagnetic $[Co(bpy)_3]^{2+}$ ion have been studied by a variety of spectroscopic and physical techniques (*13, 69, 149, 202, 327, 330, 404, 418, 581, 621, 649, 706, 711, 769, 785, 920*). The complexes are EPR active and show an apparent high-low spin transition in zeolite matrices (*64, 637*); the shifted ^1H NMR spectra have been used as probes for the degree and mechanism of spin transfer to the ligand (*262, 394, 399, 967*). Although salts of this labile ion were claimed not to be resolvable, Pfeiffer activity has been reported (*13, 298*), and the CD spectrum has been shown to be consistent with a D configuration for the *l* enantiomer (*249, 592*), and recently structural analyses of the complexes Λ-$[Co(bpy)_3]_2Cl_2$(*d*-tartrate), $Na\{\Delta$-$[Co(bpy)_3]\}Cl\square$(*d*-tartrate), and Δ-$[Co(bpy)_3]$(*d*-tartrate) have been described (*886, 887*). A number of thermal or solution reactions interconverting $[Co(bpy)_3]X_2$ and $[Co(bpy)_2X_2]$ have been described (*207, 539, 551, 649, 965*). A crystal structural analysis of the salt $[Co(bpy)_3]Cl_2 \cdot 2H_2O \cdot EtOH$ has been reported (*884*).

The cobalt(III) complexes are best prepared by the chemical or electrochemical oxidation of $[Co(bpy)_3]^{2+}$ salts (*204, 621, 765*) and are fully characterized by a range of spectroscopic techniques (*149, 179, 184, 225, 327, 442, 607, 621, 875*), including X-ray photoelectron spectroscopy (*330*), X-ray absorption spectroscopy (*703*), and ^{59}Co (*621*), ^{13}C (*929*) ^{15}N (*280*), and ^1H (*204, 463*) NMR spectroscopy (*621*). Salts of $[Co(bpy)_3]^{3+}$ are particularly amenable to study by Mössbauer spectroscopy and related techniques and enable the study of the chemical effects of the decay (and exchange) of ^{57}Co (*5, 6, 152, 322, 467–469, 520,*

594–596, 662–666, 669, 778, 782, 823, 866). Numerous heterobimetallic (*8–10, 12, 92, 498, 580, 758, 787, 825–828, 830, 832, 834–846, 848, 858, 930*) and other salts (*184, 185, 294, 535, 710*) of $[Co(bpy)_3]^{3+}$ have been reported. Numerous studies have indicated considerable outer-sphere interactions between $[Co(bpy)_3]^{3+}$ (and also $[Co(bpy)_3]^{2+}$) and various polar molecules or ions (*88, 412–414, 421, 528, 593, 631–634, 670, 746–749, 979*); these observations are clearly related to those made in the many chromatographic studies reported by various groups (*50, 122, 163, 241, 316, 536, 537, 707, 708, 742, 847, 984*).

Crystal structural analyses of Δ-$[Co(bpy)_3][Fe(CN)_6]\cdot 8H_2O$ (*690*) and $[Co(bpy)_3][Fe(CN)_6\cdot 8H_2O$ (*978*) have been reported. This complex is of some interest because it exhibits an intervalence band assigned to $\{Co^{III}, Fe^{III}\} \rightarrow \{Co^{IV}, Fe^{II}\}$ (*293*). The corresponding $[Fe(CN)_6]^{4-}$ salts are also known (*980*). The cobalt(III) salts can be resolved, and the $(-)_D$-enantiomer shown to possess the Δ configuration (*302, 304, 489, 591, 690*).

The pulse radiolysis of $[Co(bpy)_3]^{3+}$ salts has been investigated by several groups and a number of results claimed (*51, 189, 386, 388, 824, 934, 950*). There is relatively convincing evidence for the initial product being a transient high-spin cobalt(II) species.

A dizzying array of coordination compounds (*68, 127, 167, 187, 223, 279, 328, 545, 610, 616, 668, 729, 751, 879, 888, 889, 907*), organic compounds (*153–155, 817, 915, 916, 925*), and proteins or enzymes (*128, 138, 147, 521, 807*) have been shown to undergo redox reactions with $[Co(bpy)_3]^{3+}$. Motives for these investigations vary from studies of electron transfer kinetics to the preparation of organic compounds. Particular attention has been paid to the self-exchange couple $[Co(bpy)_3]^{3+}/[Co(bpy)_3]^{2+}$ and the pseudo self-exchange couple $[Co(bpy)_3]^{3+}/[Co(terpy)_2]^{2+}$ (*90, 98, 225, 242, 243, 281, 406, 743, 878, 959, 960, 981*). Some applications of $[Co(bpy)_3]^{3+}$ that are relevant to the area of solar energy photoconversion have been noted (*49, 68, 110, 111, 140, 223, 231, 385, 404, 508, 511, 556, 695, 705, 817, 915*). Theoretical investigations of the $[Co(bpy)_3]^{3+}$ ion have been made at extended Hückel [65] and CNDO/2 levels (*757*).

Salts of $[Co(bpy)_3]^{2+}$ and $[Co(bpy)_3]^{3+}$ possess curariform activity (*904*), are toxic to *E. coli* (*276*), and show photochemical interactions with DNA (*48*). These properties are thought to be related to the highly charged species present in solution, which is in accord with the observed coagulation of hydrosols by the salts (*125, 126, 598, 599, 601, 880*). The use of the salts as oxidation (*206, 586*) and reduction (*821*) catalysts has been studied.

Salts of $[Rh(bpy)_3]^{3+}$ can be prepared by the reaction of $RhCl_3\cdot 3H_2O$

with an excess of bpy under mildly reducing conditions; presumably the reaction is autocatalysis by rhodium(II) or rhodium(I) intermediates. In the absence of reducing agents, the major products are $[Rh(bpy)_2Cl_2]^+$ salts. When pure, $[Rh(bpy)_3]^{3+}$ salts are very pale though they frequently contain highly colored impurities (*176, 179, 204, 336, 588, 621*). The major interest in $[Rh(bpy)_3]^{3+}$ lies in its photochemical and photophysical properties and its use as a relay in photoconversion cells (*95, 120, 170, 176, 180, 181, 192, 193, 195, 228, 235–237, 323, 324, 382, 387, 400, 401, 456, 470, 499, 540, 546, 547, 648, 674, 792, 820, 897, 902, 951, 952, 957, 958, 990–993*). These are frequently based on $[Ru(bpy)_3]^{2+}/(HOCH_2CH_2)_3N/[Rh(bpy)_3]^{3+}/Pt$ systems and have been excellently reviewed elsewhere (*434*).

A crystal structural analysis of the salt $[Rh(bpy)_3]Cl_3 \cdot 4H_2O$ has been reported (*401*). The natural abundance ^{15}N NMR spectrum of $[Rh(bpy)_3]^{3+}$ has been recorded (*84*). Flash photolysis or pulse radiolysis of $[Rh(bpy)_3]^{3+}$ produces a transient formally rhodium(II) species, which is best formulated $[Rh^{III}(bpy)_2(bpy^{-\cdot})]^{2+}$ (*169, 652, 801*). The ion has been resolved as its (+-tris-{L-cysteinesulfinato(2)*S,N*}cobaltate(III) salt (*214*) and circular dichroism (CD) spectra described (*591, 592*). The toxicity of $[Rh(bpy)_3]^{3+}$ salts has been noted and interpreted in terms of specific interactions (*264, 297, 953*). The reduction of CO_2 to formate in aqueous solution is catalyzed by $[Rh(bpy)_3]^{3+}$ (*278*).

In contrast to the moderately well characterized $[Rh(bpy)_3]^{3+}$, the literature regarding $[Ir(bpy)_3]^{3+}$ is confused and confusing, and early reports should be regarded with care. Initial reports of the preparation of $[Ir(bpy)_3]^{3+}$ in a chloride-rich medium almost certainly gave $[Ir(bpy)_2Cl_2]^+$ (*143, 179, 203, 301, 588, 621*). Later preparations in chloride-free media led to materials of apparently correct formulation that possessed anomalous properties (*265, 448*). A number of groups favored differing explanations for the origins of the anomalous properties; among these, the presence of a monodentate bpy (*447, 448, 957*) and the presence of a covalently hydrated bpy (*157, 300*) seemed to be most consistent with the observations. The first crystal structural analysis of the anomalous compound revealed the presence of a previously unique bonding mode for bpy, in which the two pyridyl rings are *transoid*, with one of them cyclometalated, and so presenting a *C,N*-donor set (Fig. 9a) (*970*). Independent crystallographic studies of $[Ir(N,N\text{-bpy})_2(C,N\text{-bpyH})]^{3+}$ and $[Ir(N,N\text{-bpy})_2(C,N\text{-bpy})]^{2+}$ (Fig. 9b) have confirmed this result (*344, 680*), as have detailed NMR studies (*448*). Although this result is of some interest, the spate of studies on these compounds are due to the fact that the photochemical and photophysical properties of the cyclometalated and nonmetalated com-

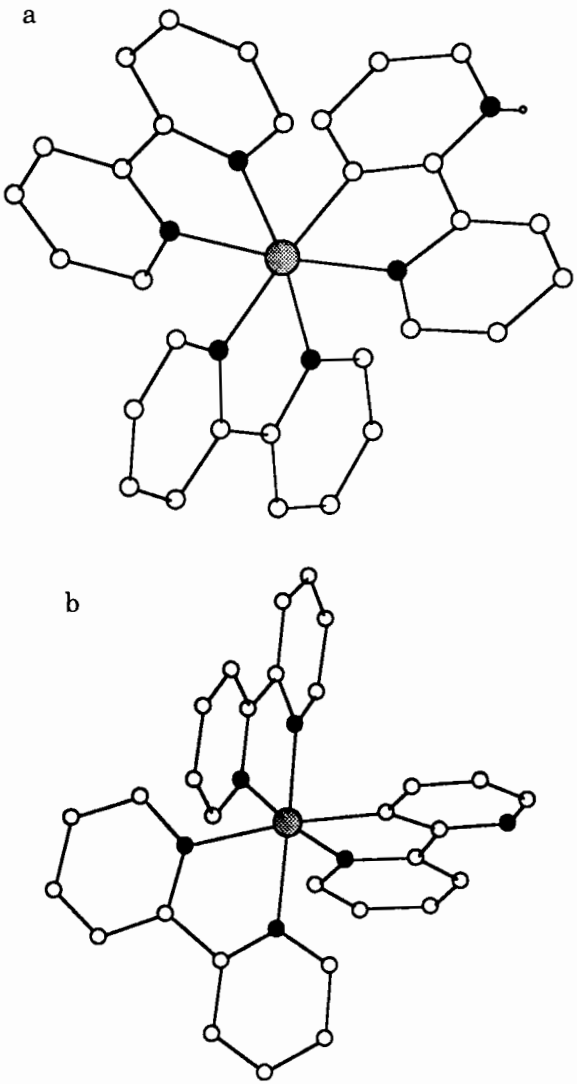


FIG. 9.

plexes are of great interest (*162, 259, 265, 382, 387, 447, 448, 689, 775, 861, 862, 955–957, 976*). These developments and a discussion of the various structural proposals for the anomalous complexes are detailed elsewhere (*809*).

The authentic, yellow $[Ir(bpy)_3]^{3+}$ cation can best be prepared by the reaction of $[Ir(bpy)_2(CF_3SO_3)_2]^+$ with bpy (*874*). A crystal structural

analysis of $[Ir(bpy)_3][ClO_4]_3 \cdot 2H_2O$ has been reported, and the complex shown to possess six equivalent Ir—N distances, in contrast to the cyclometalated compound (*344, 680*).

8. Nickel, Palladium, and Platinum

The violet complex $[Ni(bpy)_2]$ is readily prepared by the reaction of bpy with $[Ni(cp)_2]$ (*59*) or $[Ni(cod)_2]$ (*425*), or by metal vapor synthesis (*348*). The 18-electron complex is presumably tetrahedral. Mixtures of nickel(0) compounds with bpy react with dihaloalkanes to yield $[NiL_3]Br_2$ and nickelacyclopentanes (*890, 919*). Numerous studies of the electrochemical behavior of $[Ni(bpy)_3]^{2+}$ in a range of solvents have been made (*347, 418, 621, 734–737, 739, 766, 793, 794, 868, 899, 901, 994*). The behavior varies with solvent, but it is clear that the formal oxidation states −1, 0, +1, +2, and +3 are accessible in MeCN. The electrochemical behavior is complicated by a number of dissociative processes. The initial reduction product, $[Ni(bpy)_3]^+$, can be isolated as its air-sensitive perchlorate salt after controlled potential electrolysis. The further reduction of $[Ni(bpy)_3]^+$ yields $[Ni(bpy)_3]$, which undergoes loss of bpy to form $[Ni(bpy)_2]$. The reaction of $K_4[Ni(CN)_4]$ with excess bpy in liquid ammonia gives a product formulated as $[Ni(bpy)_3]$ (*60*). Further reduction of either of the nickel(0) complexes leads to $[Ni(bpy)_3]^-$ or $[Ni(bpy)_2]^-$, which are also in equilibrium by ligand loss or gain. Electrochemical studies of the isolated $[Ni(bpy)_2]$ complex clearly indicate the formation of $[Ni(bpy)_2]^-$; ESR studies of these nickel(−1) complexes suggest that the radical is ligand-centered (60, 348). Attempts to reduce $[Ni(bpy)_3]^{2+}$ salts by $Li[AlH_4]$ led to the formation of $[Al(bpy)_3]$ (*365*). This is in contrast to the reaction with aqueous $Na[BH_4]$, with which the initial product is $[Ni(bpy)_3][BH_4]_2$; in ethanol, deep-blue solutions containing $[Ni(bpy)_2]^+$ are obtained, from which $[PF_6]$ or $[BPh_4]$ salts can be isolated. The $[Ni(bpy)_2]^+$ ion disproportionates to metallic nickel and $[Ni(bpy)_3]^{2+}$ (*389*). It is not possible to prepare $[Ni(bpy)_3]^+$ salts by $Na[BH_4]$ reduction (*389*).

Salts of the pink complex ion $[Ni(bpy)_3]^{2+}$ are readily prepared by the reaction of an excess of bpy with suitable nickel(II) compounds and are paramagnetic (*75, 380, 418, 435, 558, 559, 585, 588, 621, 626–628,787, 873*). The d^8 ion is labile, and equilibrium mixtures of the 1 : 1, 2 : 1, and 3 : 1 complexes are formed with less bpy (*190, 390, 621*). Studies of the electronic and ligand field spectra of the $[Ni(bpy)_3]^{2+}$ ion abound (*91, 178, 179, 326, 327, 338, 441, 544, 621, 659, 693, 724–726, 759, 791, 876, 934, 979*). The vibrational spectra of the salts are unremarkable (*149, 407, 418, 621, 721*). A number of studies of the NMR

spectra of the paramagnetic $[Ni(bpy)_3]^{2+}$ ion have probed the mechanism for spin delocalization onto the ligands (*216, 268, 394, 963, 968*). The apparent molar volume of the $[Ni(bpy)_3]^{2+}$ ion is 380 $cm^3\ mol^{-1}$ (*86*). Related studies include photoelectron spectroscopy (*597, 985, 986*), X-ray absorption (*621*), and laser mass spectroscopic (*34*) investigations.

A range of bimetallic compounds incorporating $[Ni(bpy)_3]^{2+}$ counterions have been isolated, usually by the direct reaction of bpy with appropriate bimetallics (*7, 9–11, 92, 286, 297, 335, 337, 575, 781, 825–833, 836–838, 845, 859*). The reduction of $[Os_3(CO)_{12}]$ by Na in liquid ammonia generates $[Os(CO)_4]^{2-}$, which can be isolated as the $[Ni(bpy)_3]^{2+}$ salt. Salts with the pseudohalides $[PhC(NO_2)_2]$ (*29*), $[H_2NCOC(CN)NO]$ (*851*), $[H_2NCOC(CN)_2]$ (*850*), $[N(CN)_2]$ (*498*), and [SCN] (*783*) have been reported. The anion $[B_6H_7]^-$ was first isolated as the salt $[Ni(bpy)_3][B_6H_7]_2$ by the reaction of $[Ni(bpy)_3]^{2+}$ with $[B_6H_6]^{2-}$ (*525, 740, 936, 937*); the related complexes $[Ni(bpy)_3][B_nH_n]$ (n = 10 or 12) (*282*).

Structural studies of a number of $[Ni(bpy)_3]^{2+}$ salts have been reported (*260, 621, 724, 725, 886, 887, 945, 946*); the cation exhibits the expected approximate D_3 symmetry with Ni—N_{av} 2.09 Å. The $(+)_{589}$ cation has been shown to possess the Λ absolute configuration, from a structural analysis of $(+)_{589}$ tartrate salt (*946*). The diastereomeric $[Ni(bpy)_3]^{2+}\cdot(+)_{589}$ tartrate system is extremely complex, and the complexes $\{\Lambda\text{-}[Ni(bpy)_3]\}_2Cl_2\cdot(+)_{589}$ tartrate, and $\Delta\text{-}[Ni(bpy)_3]\cdot(+)_{589}$ tartrate have been prepared from racemic $[Ni(bpy)_3]^{2+}$ (*886, 887*).

The $[Ni(bpy)_3]^{2+}$ cation can be resolved as tartrate, antimonyl tartrate, or arsenyl tartrate salts (*621*). Once resolved, the salts racemize rapidly in aqueous solution (*182, 621*) and less rapidly in the solid state. The solution racemization shows an intriguing solvent (*621*) and pH dependence, and it has been proposed that nucleophilic attack at the ligand is a preliminary to racemization or ligand loss. Whatever the precise mechanism, $[Ni(bpy)_3]^{2+}$ salts are labile in aqueous solution and exchange with $^{63}Ni(aq)$ (*222, 621*). Ligand loss appears to be acid-promoted as expected for an Eigen–Wilkins mechanism, but the complex is remarkably stable, being resistant to attack by polysulphide or $[Hdmg]^-$ (*621*). The ion does not dissociate in concentrated sulphuric acid, an observation used to implicate covalent hydration (*303*). The solid-state racemization is anion dependent, and various mechanisms for the process have been proposed (*277, 903*). The Δ and Λ enantiomers can be resolved by paper electrophoresis of the arsenyl $(+)_{589}$-tartrate salts (*118*). The CD, magnetic circular dichromism (MCD), and optical rotatory dispersion (ORD) of the ions

have been reported (*79, 249, 331, 332, 478, 592*). Solutions containing *racemic* $[[Ni(bpy)_3]^{2+}$ salts are Pfeiffer active (*298, 477, 478, 608*), an observation in general accord with their lability and facile formation of outer-sphere complexes (*464, 621, 745, 869*).

Miscellaneous studies of the thermal decomposition of a range of $[Ni(bpy)_3]^{2+}$ salts have been reported (*213, 539, 557, 561, 649, 667*). The magnetic properties of the salts with tetracyanoquinodimethane (tcnq) have excited some interest (*709, 780*). The large $[Ni(bpy)_3]^{2+}$ ions have been shown to coagulate silver halide hydrosols (*126, 598, 600*) and possess curariform neurotoxicity (*497, 904*). Other applications of $[Ni(bpy)_3]^{2+}$ salts have been found as catalysts for aldol condensations (*425*), Grignard cross-couplings (*409*), and the H_2O_2 oxidation of indigocarmine (*910*), and in nitrate ion-selective electrodes (*408*).

Although 1 : 2 complexes with nickel are known in solution, in the solid state, the existence of 4-coordinate compounds in the solid state is less certain. Complexes of the stoichiometry $\{Ni(bpy)_2X_2\}$ often contain axially ligated X groups (*69, 107, 335, 551, 564, 566, 621*) or exist as coordination isomers (*335*). Claims for diamagnetic (*621*) or pseudotetrahedral (*787*) $[Ni(bpy)_2]^{2+}$ complexes remain to be substantiated.

In contrast to the dominance of the 3 : 1 stoichiometry for nickel(II), all bpy complexes with palladium(II) and platinum(II) are based on the square-planar geometry. No evidence has been presented for the preparation of $[Pd(bpy)_3]^{2+}$ or $[Pt(bpy)_3]^{2+}$ (*160*). The complex ions $[M(bpy)_2]^{2+}$ [M = Pd or Pt] can be prepared by the reaction of $[MCl_4]^{2-}$ or MCl_2 with an excess of bpy; the ions are very sensitive to halide ion, and readily revert to $[M(bpy)Cl_2]$. In general, $[M(bpy)_2]^{2+}$ ions are sensitive to nucleophilic attack and ligand displacement ($\lg K_2$ for M = Pd; 8.9) (*21, 666*), and it is necessary to precipitate them from the reaction mixture as a relatively insoluble salt (*109, 424, 554, 621, 704, 759, 845*). Dimorphic forms of a number of the salts are known, and $[M(bpy)_2][MX_4]$ complexes are of particular interest in that they may show anisotropic magnetic, optical, and electrical phenomena along the principal (stacking) axis (*70, 109, 554, 621*).

Structural studies of a number of $[M(bpy)_2]^{2+}$ salts have been reported, and it is clear that the steric interaction between the H_6 positions of the two ligands may result in a distortion from D_{4h} symmetry (*121, 141, 215, 234, 343, 345, 383, 576*). In $[Pd(bpy)_2][NO_3]_2 \cdot H_2O$ (Fig. 10), the ion is distorted toward a tetrahedral geometry by the tilting of one bpy with respect to the other and exhibits a weak interaction with the nitrate ion (*121, 141, 383*). Similarly, in $[Pd(bpy)_2]X_2$ (X = picrate), the planar PdN_4 unit shows a bow-shaped distortion resulting from the

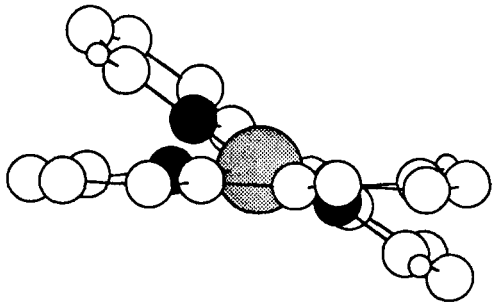


FIG. 10.

twisting of one bpy ring, maintaining a square-planar N_4-donor set and exhibits stacking interactions with the lattice picrate ions (Fig. 11) (*576*). In $[Pt(bpy)_2][(tcnq)_2]$, the same distortion is observed; the two TCNQ radicals have formed a covalent C—C bond (1.65 Å) in the dimeric anion (*215*). This is related to the observed diamagnetism of $[Pt(bpy)_2][(TCNQ)_2]$ and the facile conversion to a triplet state 0.25 eV higher (*215, 233*). A structural analysis of the diamagnetic complex $[Pt(bpy)_2][(tcnq)_3]$ (which also shows a transition to a paramagnetic state 0.15 eV above the ground state) has been reported (*234*).

Reversible changes in the electronic and NMR spectra of $[Pt(bpy)_2]^{2+}$ in the presence of OH^- or MeO^- led to the suggestion that nucleophilic attack on the ligand was occurring (*19, 71, 305*); in view of the formation of outer-sphere (*494*) and 5-coordinate complexes (*557*) by attack at the *metal*, these results are best reinterpreted in terms of nucleophilic attack at Pt (*244, 677*).

The redox and photochemical properties of $[M(bpy)_2]^{2+}$ salts have excited some interest (*212, 507, 518, 961, 977*), and $[Pt(bpy)_2][Pt(CN)_4]$ has found some application as a photosensitizer for the production of dihydrogen from water (*397, 398*). The use of $[Pd(bpy)_2]^{2+}$ salts as catalysts for MeCN hydration (*935*) and the oxidative coupling of phthalate esters has been investigated (*422*).

Salts of $[Ni(bpy)_3]^{3+}$ can be prepared by electrochemical oxidation of the corresponding nickel(II) complex (*93, 962*); the ion is a powerful

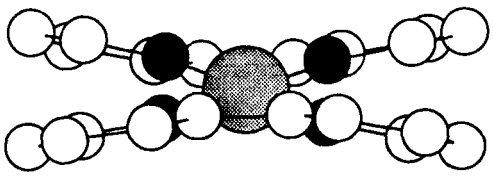


FIG. 11.

outer-sphere oxidizing agent, and its redox reactions with various organic and inorganic reductants have been investigated (*106, 298, 299, 569, 571, 573, 579, 962, 963*). Chemical oxidation of $[Ni(bpy)_3]F_2$ did not yield the nickel(III) complex (*380*). A structural analysis of $[Ni(bpy)_3][ClO_4]_3 \cdot 2MeCN \cdot \square CH_2Cl_2$ revealed an axial compression of the ion ($Ni—N_{ax}$, 1.9246 Å; $Ni—N_{eq}$, 2.022, 2.000 Å) (*885*).

Although the palladium(II) and platinum(II) complexes can be oxidized to various metal(IV) derivatives, $[M(bpy)_3]^{4+}$ complexes have not yet been isolated.

9. *Copper, Silver, and Gold*

No formally zero-valent complexes of the type $[M(bpy)_n]$ have been reported for this triad, which appears not to have been investigated by Herzog and his co-workers.

The complex ion $[Cu(bpy)_2]^+$ is formed in the irreversible electrochemical reduction of $[Cu(bpy)_3]^{2+}$ salts in a process involving the loss of a bpy ligand (*240, 426, 583, 621, 623, 655, 719, 764, 898*) or by the direct reaction of excess bpy with a suitable copper(I) salt. The electrochemical behavior of $[Cu(bpy)_3]^{2+}$ salts in a range of solvents has been investigated, as has the rate of self-exchange between $[Cu(bpy)_2]^+$ and $[Cu(bpy)_2X_2]^{2+}$ (X = various) ions (*538*). The redox potential for the process is solvent and counterion dependent. The reduction of the salts $[Cu(bpy)_2X_2]^{2+}$ (X = Cl, ClO_4, or H_2PO_2) to $[Cu(bpy)_2]^+$ derivatives has been investigated as an image-forming stage in a silver-free photographic process (*515*). A number of estimates of the stability constant of the $[Cu(bpy)_2]^+$ ion have been made, and a value for $\lg\beta_2$ in the region of 14.4 would seem to be appropriate (*240, 621, 716*). Several authors have reported the electronic (*69, 306, 483*) and vibrational (*722*) spectra of $[Cu(bpy)_2]^+$ salts; these data are fully in accord with the expected tetrahedral structure of the complex.

The reddish-brown complex ion $[Cu(bpy)_2]^+$ is air sensitive and undergoes an autoxidation reaction with dioxygen. Numerous studies of the oxidation of $[Cu(bpy)_2]^+$ by dioxygen or hydrogen peroxide have been described. It is proposed that the initial step involves the formation of the blue dioxygen adduct $[Cu(bpy)_2(O_2)]^+$ (*69, 310–313, 655, 673, 719, 881, 912*), which then reacts with a second equivalent of $[Cu(bpy)_2]^+$ to yield hydrogen peroxide and $[Cu(bpy)_2]^{2+}$. The addition of dioxygen is readily reversed by passing N_2 through a solution of the blue $[Cu(bpy)_2(O_2]^+$ complex (*655*). The effects of added ascorbate, acetate, malate, or ethylene diaminetetraacetic acid (edta) on the autoxidation reaction has been studied (*853, 912*). In the case of malate,

evidence was presented for the formation of $[Cu(bpy)(malate)]^{n+}$ complexes with the release of O_2^- (*853*). The mechanism indicated below is consistent with the data though the reaction is pH-dependent and processes involving $HO_2^{\cdot}$ or $HO^{\cdot}$ have been implicated (*306, 313, 314, 852*).

$$[Cu(bpy)_2]^+ + O_2 = [Cu(bpy)_2(O_2)]^+$$

$$[Cu(bpy)_2(O_2)]^+ + [Cu(bpy)_2]^+ + 2H^+ = 2[Cu(bpy)_2]^{2+} + H_2O_2$$

The oxidation of $[Cu(bpy)_2]^+$ by O_2 is reported to be second order in Cu(I) whereas the oxidation by H_2O_2 is reported to be first order with respect to Cu(I), in general accord with the above mechanism (*306, 309, 719, 988*). The dioxygen is most likely present as a superoxide O_2^- ion in the complex $[Cu(bpy)_2(O_2)]^+$ (*306*). The $[Cu(bpy)_2]^+/O_2$ system is an active catalytic oxidation systems (presumably by the intermediacy of $[Cu(bpy)_2(O_2)]^+$) for the oxidation of alcohols to aldehydes and ketones (*655*). The importance of predissociation of bpy ligands is not yet established though evidence has been presented for the equilibrium

$$[Cu(bpy)_2]^+ = [Cu(bpy)]^+ + bpy$$

in $MeNO_2$ or MeCN (*988*). The isolated product of the oxidation of $[Cu(bpy)_2][ClO_4]$ by dioxygen in $MeNO_2$ is $[Cu(bpy)_2(NO_3)][ClO_4]$, which has been structurally characterized (*284*).

The interaction of $[Cu(bpy)_2]^+$ with a range of other oxidants, including $[Cu(bpy)_3]^{3+}$ and $[Co([14]\text{-ane-}N_4)(O_2)(OH_2)]^{2+}$, has been studied; no evidence for an inner-sphere pathway in the reaction with $Co([14]\text{-ane-}N_4)(O_2)(OH_2)]^{2+}$ was found (*659, 983*).

The complex ion $[Cu(bpy)_2]^+$ has been shown to possess no affinity for binding carbon monoxide, in contrast to the related $[\{Cu(bpy)X\}_2]$ complexes (*482*).

The related diamagnetic silver(I) complex ion $[Ag(bpy)_2]^+$ is also known and is readily prepared by the direct reaction of silver(I) salts with excess bpy (*516, 588, 621*). The stability of $[Ag(bpy)_2][NO_3]$ has been investigated and a value for $\lg\beta_2$ of 7.6 reported (*106, 621, 871*). The vibrational spectra of $Ag(bpy)_2]^+$ complexes are consistent with a tetrahedral geometry about the metal (*620, 621*). The complex ion is unstable with respect to loss of bpy, and equilibria of the type

$$Ag(bpy)_2]^+ = [Ag(bpy)_2]^+ + bpy$$

have been established (*731*). A number of physical techniques have been applied to the study of $Ag(bpy)_2]^+$ salts, and X-ray photoelectron

spectra (*656*), laser ionization mass spectra (*34*), fast atom bombardment (FAB) mass spectra (*129*), FD mass spectra (*129*), and secondary ion mass (SIM) (*726*) spectra being reported. The light-sensitive salt $Ag(bpy)_2][NO_3]$ is reported to possess fungicidal properties (*524*).

Salts of $Ag(bpy)_2]^+$ are reported to be effective catalysts for the H_2O_2 decomposition or persulphates; $Ag(bpy)_2]^{2+}$ salts are implicated in the process (*621*).

The discussion of $[Cu(bpy)_2]^{2+}$ salts is fraught with peril. These ions are implicated in numerous speciation studies of the Cu(II)-bpy system. However, it seems likely that the majority of such ions in solution possess relatively strongly coordinated solvent molecules in the axial sites and so do not fall in the category of homoleptic complexes. The distinction between a Jahn–Teller distorted octahedral complex with weakly bonded axial ligands and a homoleptic square-planar complex is fine. However, even such weakly coordinating ligands as perchlorate and tetrafluoroborate are bonded to the metal in $[Cu(bpy)_2(ClO_4)][ClO_4]$ and $[Cu(bpy)_2(BF_4)_2]$ (*266, 514*), and only $[Cu(bpy)_2][PF_6]_2$ appears to be a true 4-coordinate $[Cu(bpy)_2]^{2+}$ salt (*267*). The crystal structural analysis of $[Cu(bpy)_2][PF_6]_2$ reveals the metal to be in a compressed tetragonal N_4 environment (Fig. 12), a conclusion fully in accord with the EPR data for the complex (*267*). The situation is complicated by the following equilibrium:

$$[Cu(bpy)_3]^{2+} = [Cu(bpy)_2]^{2+} + bpy$$

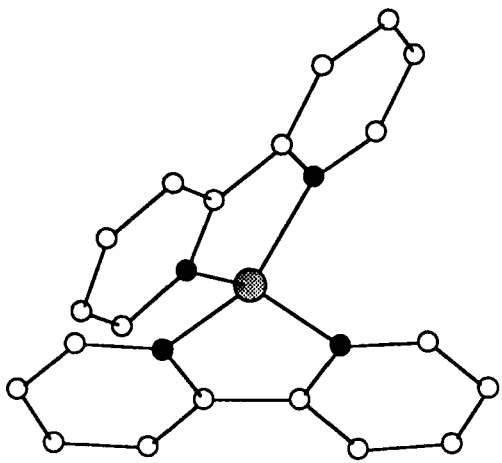


FIG. 12.

which is well established (*426, 621*). Numerous studies of the electrochemical behavior of $[Cu(bpy)_3]^{2+}$ have established $[Cu(bpy)_2]^{+}$ as the reduction product (*426, 538, 585, 621, 625, 764, 898*). In addition to the electrochemical reduction, $[Cu(bpy)_2]^{2+}$ species have been shown to undergo photoreduction to $[Cu(bpy)_2]^{+}$ (*483, 514, 672*). Although further discussion of the properties of reported $[Cu(bpy)_2]^{2+}$ complexes is not given here, it must be remembered that many of the reported reactions of $[Cu(bpy)_3]^{2+}$ complexes may be due to $[Cu(bpy)_2]^{2+}$ formed by the equilibration just indicated. A typical example of such a process is the use of $[Cu(bpy)_2][ClO_4]_2$ catalysts for the dioxygen oxidation of DMF to CO_2 and Me_2NH (*206*).

The position of $[Ag(bpy)_2]^{2+}$ salts is slightly clearer. Although crystal structural determinations indicate weak interactions between $[Ag(bpy)_2]^{2+}$ cations and anions or solvent molecules, the cations can be regarded as homoleptic $[Ag(bpy)_2]^{2+}$ complexes (*1, 617, 728*). Reddish-brown salts of $[Ag(bpy)_2]^{2+}$ are readily obtained by electrochemical, persulphate, or ozone oxidation of either $[Ag(bpy)_2]^{+}$ or silver(I) salts in the presence of excess bpy (*543, 621, 629, 641, 926, 967*). The salt $[Ag(bpy)_2][SO_3F]_2$ is prepared by the reaction of bpy with $Ag(SO_3F)_2$, obtained from the direct reaction of silver with $S_2O_6F_2$ (*541, 542*).

The crystal structural analysis of $[Ag(bpy)_2][NO_3]_2 \cdot H_2O$ reveals a chain structure in which weakly bonded bridging nitrate ions link $[Ag(bpy)_2]^{2+}$ units; the two bpy ligands on each silver are nonequivalent, and the $Ag—O_{nitrate}$ distances are 2.78 and 2.82 Å (*30*).

In solution, the following equilibria have been established in a range of solvents (*39, 377, 894, 900*).

$$Ag(bpy)_2]^{2+} = [Ag(bpy)]^{2+} + bpy$$

$$Ag(bpy)_2]^{2+} + H^{+} = [Ag(bpy)]^{2+} + [Hbpy]^{+}$$

Numerous electrochemical studies have established the facile oxidation of $[Ag(bpy)_2]^{+}$ to $[Ag(bpy)_2]^{2+}$ (*315, 621, 892–894, 926*). It has been suggested that the electroactive species in the preparation of $[Ag(bpy)_2]^{2+}$ from $[Ag(bpy)_2]^{+}$ is $[Ag(bpy)]^{+}$ (*926*). An oscillating reaction involving a bromate/malonate reaction in sulphuric acid in the presence of the $[Ag(bpy)_2]^{2+}/[Ag(bpy)_2]^{+}$ couple has been reported; the system is heterogeneous as a result of the insolubility of $[Ag(bpy)_2]^{+}$ salts (*517*).

Salts of $[Ag(bpy)_2]^{2+}$ are paramagnetic (*541, 542, 618, 621, 908*) and EPR active (*113, 239, 325, 378, 476, 541, 542, 908*). The reported magnetic moments vary from 2.12 to 1.80 μ_B, with the lower values proba-

bly being the more reliable. Many of the reported EPR spectra have been recorded in solvents that promote bpy loss according to the preceding equation, and ambiguities in their interpretation may arise from this source (*239*). The vibrational (*588*) and electronic (*39, 45, 130, 377, 542*) spectra of $[Ag(bpy)_2]^{2+}$ salts are in general accord with structures exhibiting weak interactions with solvent molecules, but it is evident that some of the reported spectra refer to partially solvated or anated species. Salts of $[Ag(bpy)_2]^{2+}$ have also been investigated by SIM (*726*), FAB mass (*129*), FD mass (*129*), and X-ray photoelectron (*656*) spectroscopy.

Silver(II) compounds are powerful oxidizing agents, and the stability of $[Ag(bpy)_2]^{2+}$ salts has rendered them particularly useful as sources of silver(II) (*621*). Salts of $[Ag(bpy)_2]^{2+}$ have been shown to oxidize H_2O_2 (*378, 729*), $RN(CH_2CO_2H)_2$ (*31*), or propan-2-ol (*379*), and also to be useful for the oxidative acetoxylation of arenes in the presence of acetate (*685, 686*). The oxidative decarboxylation of organic acids is promoted by $[Ag(bpy)_2]^{2+}$, and the mechanism (shown later) is proposed (*22*). Aqueous solutions containing $[Ag(bpy)_2]^{2+}$ salts are not indefinitely stable, and in addition to the bpy loss reactions, a number of other processes have been suggested. A photoreduction of $[Ag(bpy)_2]^{2+}$ to $[Ag(bpy)_2]^{+}$ and Ag metal has been established (*117*). This process is accelerated by alcohols and, presumably, other reducing agents (*38*). A number of less well-established equilibria have been proposed (*39, 410, 411*), including

$$[Ag(bpy)_2]^{2+} + H_2O = [Ag(bpy)_2]^{+} + H^{+} + HO^{\cdot}$$

and the disproportionation

$$2[Ag(bpy)_2]^{2+} = [Ag(bpy)_2]^{+} + [Ag(bpy)_2]^{3+}$$

The complex ion $[Cu(bpy)_3]^{2+}$ has been well established as a solution species in the Cu(II)-bpy systems (*196, 240, 621, 714, 721, 722*). A number of studies of the EPR spectra $[Cu(bpy)_3]^{2+}$ in solution and solid phases have been reported (*339, 480, 587, 650, 675, 676, 843, 854, 949*); some of the earlier solution EPR spectra have been shown to be due to mixtures of $[Cu(bpy)_3]^{2+}$ and $[Cu(bpy)_2]^{2+}$ species and must be treated with caution (*949*).

The EPR spectra are consistent with a distorted octahedral environment about the metal, a conclusion that has been fully confirmed by crystal structural analyses of $[Cu(bpy)_3]^{2+}$ salts (Fig. 13). (*23, 724, 725*). The electronic (*179, 339, 480, 481, 530, 621, 711, 724, 725, 764,*

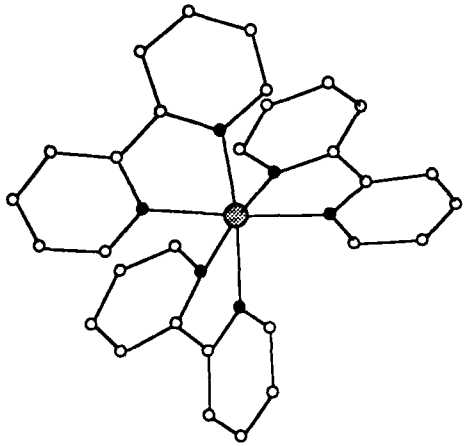


FIG. 13.

854, 928) and vibrational (*418, 619, 621, 720–722, 769, 873*) spectra of $[Cu(bpy)_3]^{2+}$ salts are also best interpreted in terms of this distorted structure. The bulk of evidence is in favor of a dynamic Jahn–Teller effect operating in $[Cu(bpy)_3]^{2+}$ salts. The X-ray photoelectron spectrum of $[Cu(bpy)_3][SO_4]\cdot 7H_2O$ has been reported (*273*).

Solid complexes containing $[Cu(bpy)_3]^{2+}$ are readily prepared from appropriate copper(II) salts and excess bpy (*23, 207, 418, 621, 724, 725, 873*), and include $[Cu(bpy)_3]X_2$ (X = $C(CN)_3$ (*848*), $PhC(NO_2)_2$, $N(CN)_2$, $ArPS_2$, and $[Cu(bpy)_3]X$ (X = $OsCl_6$, $OsBr_6$, or OsI_6 (*859, 860*). The complexes are paramagnetic (*627*) and redox active, as discussed. The salt $[Cu(bpy)_3][TCNQ]_2$ is a highly conducting mixed oxidation state species (*417, 780*).

Salts of $[Cu(bpy)_3]^{2+}$ have been used as catalysts for aldol condensations (*419*) or the *tert*-butyl hydroperoxide oxidation of cyclic alkenes (*54, 55*). The oxidation of water by $[Ru(bpy)_3]^{3+}$ in the presence of $[Cu(bpy)_3]^{2+}$ catalysts has been investigated in alkaline and slightly acidic solutions; the active catalysts were probably $[Cu(bpy)_2]^{2+}$ complexes (*223*). The autoxidation of EtCHO in $MeCO_2H$ is catalyzed by $[Cu(bpy)_3]^{2+}$ salts (*776*). The complex $[Cu(bpy)_3]Cl_2$ is reported to add HCl reversibly; the product is not characterized, but could be $[Cu(bpy)_2(bpyH)Cl]^{2+}$ (*873*). The binding of $[Cu(bpy)_3]^{2+}$ to hectorite (*913*) and Dowex 50W (*923*) has been described.

The ion $[Cu(bpy)_3]^{2+}$ is labile, and attempts to resolve the Δ and Λ forms with bromocamphorsulphonate were unsuccessful (*621*).

Reports of $[Ag(bpy)_3]^{2+}$ must be regarded with suspicion (*621, 892*).

10. Zinc, Cadmium, and Mercury

Low oxidation state complexes of this triad are extremely uncommon: however, a spectroscopic study of the complex ion $[Hg_2(bpy)_2]^{2+}$ has been reported (*895*). The ill-defined compound $[Zn(bpy)_2 \cdot 2NH_3]$ is reported to be the product of the reaction of $Li_2(bpy)$ with $[Zn(bpy)_3]I_2$ in liquid ammonia (*354*).

The interaction of 2,2′-bipyridine with zinc salts was first described by Blau (*75*), and speciation studies have established the formation of $[Zn(bpy)_3]^{2+}$ in a range of solvents (*531, 621, 721, 928*), and a range of salts of this diamagnetic ion have been prepared (*91, 183, 291, 354, 418, 588, 621, 721*). The d^{10} configuration renders $[Zn(bpy)_3]^{2+}$ salts particularly good models for investigating the metal-ligand interactions in $[M(bpy)_3]^{n+}$ complexes, and numerous studies of the electronic (*91, 142, 176, 192, 621, 928*), ^1H NMR, (*436*), photoacoustic (*142*), and vibrational spectra have been reported (*142, 291, 407, 418, 721*).

Detailed studies of the crystal morphology of a range of salts have been described (*621*). The intense interest in the photochemical and photophysical properties of $[M(bpy)_3]^{n+}$ complexes (see earlier discussion) led to a number of studies of the iron(II) (*78, 197, 251, 252*), ruthenium(II) (*32, 248, 251–253, 384, 513, 711*), and osmium(II) complexes (*197, 251, 252*) in $[Zn(bpy)_3]^{2+}$ hosts. The coadsorption of $[Zn(bpy)_3]^{2+}$ and $[Ru(bpy)_3]^{2+}$ in interlayers in colloidal smectite hosts has also been reported (*292*). Although many photochemical studies have been made in $[Zn(bpy)_3]^{2+}$ hosts, salts of this ion are fluorescent (*91, 192, 381, 692*).

Although the $[Zn(bpy)_3]^{2+}$ ion is labile, a number of optical studies have been made (*91*). The phenomenon in which the optical activity of a labile tris (chelate) complex is enhanced in the presence of a chiral environment compound is known as the Pfeiffer effect (*298, 449*) and was first observed in complexes of 1,10-phenanthroline and 2,2′-bipyridine. A number of examples involving $[Zn(bpy)_3]^{2+}$ salts are known (*298, 478, 636*).

The large $[Zn(bpy)_3]^{2+}$ ion has been used to isolate a range of unusual anions; and salts with $[Mn(CO)_5]^-$ (*609*), $[Hg(SCN)_4]^{2-}$ (*836, 845*), $[RSO_2]^-$ (*550*), and $[PhC(NO_2)_2]^-$ (*29*) have been described. The thermal decomposition of $[Zn(bpy)_3]Br_2$ has been investigated (*207*). Electrochemical studies of $[Zn(bpy)_3]^{2+}$ salts give an indication of the potential for ligand-based reduction in $[M(bpy)_3]^{2+}$ species; adsorption at mercury electrodes is well characterized (*15, 793, 794*). Salts of $[Zn(bpy)_3]^{2+}$ (like those of other $[M(bpy)_3]^{2+}$ ions) are known to exhibit curariform activity and be potent inhibitors of acetylcholinesterase

(*497*). The use of $[Zn(bpy)_3]^{2+}$ salts as catalysts for aldol condensations (*419*) and the autoxidation of EtCHO in AcOH (*776*) has been investigated.

In general, salts of $[Cd(bpy)_3]^{2+}$ closely resemble those of the zinc analogs (*2, 15, 75, 106, 437, 562, 584, 621*). A ^{113}Cd NMR study of $[Cd(bpy)_3]^{2+}$ has been made (δ 243 vs. 0.1 M $Cd(ClO_4)_2$) (*654*). There is good evidence for a $[Cd(bpy)_2]^{2+}$ complex in the Cd^{2+}-bpy system (*2*), and it is suggested that this ion is near tetrahedral (*112, 437*). The complex $[Hg(bpy)_3][CF_3SO_3]_2$ is formed from the reaction of $Hg(CF_3SO_3)_2$ with bpy and represents the sole example of a $[Hg(bpy)_3]^{2+}$ salt (*191*).

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