though the equatorial chlorine atoms (Cl(1)-Tc-C(1) = 84.71 (4)°) but not the equatorial phosphines (P-Tc-C(1) = 89.65°), are significantly displaced toward the axial carbonyl ligand. The equatorial Tc-Cl(1) bond lengths at 2.322 (2) Å are shorter than expected for technetium(III) complexes of this type.[16] Furthermore, the technetium-phosphorus bonds are quite long (2.525 (2) Å) when compared with those of other triphenylphosphine complexes of technetium(III) (2.44-2.52 Å).[16,17]

The technetium carbonyl complex has an intense infrared band at 2054 cm^{-1} assigned as ν(CO). The high frequency of ν(CO) in this complex is suggestive of terminal carbonyl ligation with minimal π-back-bonding.[18] This hypothesis is supported by the unusually long technetium-carbon bond length of 1.985 (9) Å, though the carbon-oxygen distance of 1.12 (1) Å is within experimental error of the norm.[16] Although *mer*-$TcCl_3(PMe_2Ph)_3$ reacts with CO to form[19] the diamagnetic C_{3v} seven-coordinate complex $TcCl_3(PMe_2Ph)_3(CO)$, there is no evidence for the formation of a seven-coordinate dicarbonyl complex in the acetonitrile substitution reaction described above. This difference in reactivity may be attributed to the greater σ-basicity of PMe_2Ph compared to PPh_3, leading to a higher affinity for π-acids (e.g. CO) in complexes of the former.

The reaction of $TcCl_3(PPh_3)_2(MeCN)$ with nitric oxide is similar to the reaction with CO, except that there is now a change in the formal oxidation state of the technetium, yielding trichloronitrosylbis(triphenylphosphine)technetium(II). The assignment of the unpaired electron onto the metal is verified by the presence of a ten-line pattern in the electron spin resonance (ESR) spectrum of the isolated complex dissolved in CH_2Cl_2 attributed to hyperfine coupling to a ^{99}Tc nucleus, which has nuclear spin $I = {}^9/_2$. No phosphorus superhyperfine coupling, however, was noted in this solution spectrum. The NO stretching mode in the infrared spectrum at 1805 cm^{-1} is consistent with a terminal linear NO^+ linkage.[20] This complex is entirely analogous to the complex $TcCl_3(PMe_2Ph)_2(NO)$, previously prepared[21] from the tris(phosphine)-trichloride starting material. Although the solution and low-temperature glass ESR spectra for these two analogues appear identical, a slight lowering of ν(NO) is noted for the PMe_2Ph derivative, which may be understood similarly to the carbonyl phosphines discussed above.

Conclusions

The complex $TcCl_3(PPh_3)_2(MeCN)$ is readily prepared in high yields. By analogy to its rhenium cogener, it promises to be a useful starting material for low-valent technetium chemistry. It reacts with the small potentially π-accepting molecules CO and NO, but they do not participate appreciably in back-bonding.

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Supplementary Material Available: For $TcCl_3(PPh_3)_2(CO)$, listings of anisotropic thermal parameters and torsion angles and, for $TcCl_3(PPh_3)_2(NO)$, a frozen-glass EPR spectrum (5 pages); for $TcCl_3(PPh_3)_2(CO)$, a table of calculated and observed structure factors (15 pages). Ordering information is given on any current masthead page.

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Efficient Syntheses of Dioxorhenium(V) Complexes

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An improved general synthesis for $[ReO_2L_4]X$ complexes is presented. The amine complex $[ReO_2(en)_2]I$ can be made in 90% yield from $[ReO_2(PPh_3)_2I]$ and ethylenediamine in less than 15 min at room temperature. Refluxing methanol suspensions of $[ReO_2(PPh_3)_2I]$ with 12-50 equiv of electron-rich pyridines results in the formation of $[ReO_2L_4]I$ compounds in 50-85% yield.

Introduction

The complex $[ReO_2(py)_4]^+$ has been shown to exhibit a long-lived luminescence in aprotic solvents ($\tau = 10\ \mu s$) that is efficiently quenched by hydroxylic compounds[1] and by electron transfer to viologen acceptors.[2] The luminescence lifetime of this compound is highly dependent on the environment. It has been found that this complex is an effective probe of the hydrophobicity of binding regions in micelles[3] and the intracrystalline environments of complex-layered oxides.[4] For these reasons, we became interested in the tunability of ReO_2^+ species in terms of both ground-state and excited-state properties. Unfortunately, the existing synthetic methods[5] are not suitable for use with hydrophobic or high melting pyridines. We therefore sought an alternative route for smooth insertion of the ReO_2^+ unit into a variety of ligand environments.

The Re(IV) compound K_2ReCl_6 is the common starting material for the syntheses of coordination complexes containing the ReO_2^+ unit. For example, the compound $[ReO_2(py)_4]Cl$ is obtained (in 34-60% yield) by bubbling O_2 through an aqueous pyridine solution of K_2ReCl_6.[5a]

$$2K_2ReCl_6 + 14py + {}^1/_2O_2 + 7H_2O \rightarrow 2[ReO_2(py)_4]Cl\cdot 2H_2O + 6(pyH)Cl + 4KCl$$

In a similar fashion, the ethylenediamine complex $[ReO_2(en)_2]Cl$ is synthesized by reacting K_2ReCl_6 in aerated 90% ethylenediamine for a 12-h period.[5c,6]

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$$4K_2ReCl_6 + 8C_2H_4(NH_2)_2 + O_2 + 6H_2O \rightarrow 4[ReO_2(en)_2]Cl + 8KCl + 12HCl$$

These reactions are slow (likely because the Re(IV) center must be oxidized by dioxygen to Re(V)) and are not readily adapted for the syntheses of complexes containing high-melting, hydrophobic, or water-sensitive ligands.

In 1969, Freni[7] reported the synthesis and reactivity of the complex $[ReO_2(PPh_3)_2I]$. When this material is heated in neat pyridine for 15 min, $[ReO_2(py)_4]I$ of high purity precipitates in 91% yield.[7] It was also reported that when $[ReO_2(PPh_3)_2I]$ was treated with benzene solutions of pyridine, an inseparable mixture of products resulted. We have found, however, that when $[ReO_2(PPh_3)_2I]$ is treated with a moderate excess of electron-rich pyridines, the corresponding tetrapyridine compounds $[ReO_2L_4]I$ can be isolated in 50–85% yields. We report here the characterization of a series of $[ReO_2L_4]^+$ complexes prepared by using this new method.

Experimental Section

Materials. All materials used were either of reagent grade or the best grade commercially available and used as received. The ligand 4-methoxypyridine was prepared from the commercially available *N*-oxide by using the method of Ochiai.[8]

Physical Measurements. ^1H NMR spectra were recorded at 90 MHz on a Varian EM-390 spectrometer or at 89.93 MHz on a JEOL FX90-Q spectrometer. ^1H chemical shifts are reported in ppm (δ) with the solvent ($CHCl_3$, δ 7.24; $CHDCl_2$, δ 5.32; CD_3SOCD_2H, δ 2.49; CD_2HCN, δ 1.93; CD_3COCHD_2, δ 2.04) used as an internal standard. Infrared spectra were recorded on a Beckman IR 4240 spectrometer. Electronic absorption spectra were obtained by using a Shimadzu UV-260 recording spectrometer. Elemental analyses were obtained at the Caltech analytical facility.

Syntheses. The purple-violet compound $[ReO_2(PPh_3)_2I]$ (**1**) was prepared by hydrolysis of $[ReO(OEt)(PPh_3)_2I_2]$ using the literature method[9] except that $KReO_4$ was used in place of $HReO_4$. All manipulations were carried out in air. Four representative preparations are presented below.

$[ReO_2(4\text{-pyrrpy})_4]I$ and $[ReO_2(4\text{-pyrrpy})_4]PF_6$ (4-pyrrpy = 4-Pyrrolidinopyridine). **1** (1.00 g, 1.15 mmol) and 4-pyrrolidinopyridine (2.20 g, 14.8 mmol) were combined with 10 mL of methanol. Immediately upon mixing, a deep orange-brown solution was obtained. After the mixture was stirred for about 10 min, an orange-brown solid began precipitating. The reaction slurry was then refluxed for about 1 h, cooled to room temperature and placed in a refrigerator to stand overnight. The precipitate was collected on a medium-porosity frit and washed successively with 3 × 15 mL of toluene, 3 × 15 mL of diethyl ether and 2 × 15 mL of pentane. The product was then aspirated to dryness. Yield: 920 mg, 85%.

The iodide salt was metathesized to the hexafluorophosphate salt by dissolving the iodide salt in a minimum volume of 2:1 (v/v) methanol–acetone and then adding 2–3 equiv of NH_4PF_6. The material obtained was then washed successively with 2 × 7 mL of cold (4 °C) methanol, 2 × 15 mL of toluene, 2 × 15 mL of diethyl ether and 2 × 15 mL of pentane. The product was aspirated to dryness. Yields were typically 50% or greater.

Anal. Calcd for $C_{36}H_{48}N_8O_2RePF_6$: C, 45.23; H, 5.06; N, 11.72. Found: C, 45.02; H, 5.24; N, 11.59. UV–vis λ_{max} (CH_3OH solution): 490, 335 nm.

$[ReO_2(4\text{-MeOpy})_4]I$ and $[ReO_2(4\text{-MeOpy})_4]PF_6$ (4-MeOpy = 4-Methoxypyridine). **1** (420 mg, 0.48 mmol) was combined with 4-methoxypyridine (890 mg, 8.2 mmol) in 5 mL of methanol. A dark orange solution formed initially, and this became lighter orange after being stirred for 5 min. This solution was refluxed for 2 h and then allowed to cool to room temperature. Toluene (10 mL) was added and the solution volume reduced by rotary evaporation until an orange product precipitated. The solution volume was about 5 mL at this point. The mixture was placed in a refrigerator to stand overnight. The solid was collected, washed successively with 3 × 15 mL of toluene, 3 × 15 mL of diethyl ether, and 3 × 15 mL of pentane. The material was aspirated to dryness. Yield: 290 mg, 75%.

Conversion to the hexafluorophosphate salt was accomplished as follows. $[ReO_2(4\text{-MeOpy})_4]I$ (160 mg, 0.2 mol) was dissolved in 20 mL of 50% aqueous methanol and filtered by gravity. The filter paper was rinsed with 2 mL of distilled water. NH_4PF_6 (190 mg, 1.2 mmol) was added to the filtrate, and a bright yellow solid immediately precipitated. The mixture was left to stand for 10 min to ensure complete precipitation. The solid was collected on a medium-porosity frit, washed successively with 2 × 15 mL of distilled water, 3 × 15 mL of toluene, 3 × 15 mL of diethyl ether, and 3 × 15 mL of pentane, and aspirated to dryness. Recrystallization from acetone–hexane mixtures gave yellow-orange needles. These were dried at room temperature in vacuo ($<10^{-3}$ Torr) overnight. Yield: 115 mg, 70%. Anal. Calcd for $C_{24}H_{28}N_4O_6RePF_6$: C, 36.05; H, 3.53; N, 7.01. Found: C, 35.66; H, 3.44; N, 6.96. UV–vis λ_{max} (CH_3OH solution): 452, 334 nm.

$[ReO_2(4\text{-Phpy})_4]I$ and $[ReO_2(4\text{-Phpy})_4]PF_6$ (4-Phpy = 4-Phenylpyridine). **1** (200 mg, 0.23 mmol) and 4-phenylpyridine (710 mg, 4.57 mmol) were placed in a 25-mL round-bottom flask. About 5 mL of methanol was added, and the pale orange suspension was stirred. As the solution was heated to reflux, a deep orange color developed and an orange solid began to precipitate. The mixture was refluxed for 15 min, cooled to room temperature, and then placed in a refrigerator for 4 h. The orange solid was collected on a medium frit, washed successively with 3 × 10 mL of diethyl ether, 1 × 10 mL of toluene, and 2 × 10 mL of pentane, and aspirated to dryness. Yield: 120 mg, 54%. Anal. Calcd for $C_{44}H_{36}N_4O_2ReI$: C, 54.71; H, 3.76; H, 5.80. Found: C, 54.96; H, 4.15; N, 5.05. UV–vis λ_{max} (CH_3OH solution): 371 nm.

Metathesis to the hexafluorophosphate salt was accomplished as follows. The iodide salt (105 mg) was dissolved in 100 mL of methanol and filtered by gravity. To this yellow-orange solution was added 100 mg of NH_4PF_6, and the solution was vigorously stirred. Within 2 min, a precipitate began forming. The mixture was placed in a refrigerator for 4 h to encourage further precipitation. The product was then collected, washed successively with 2 × 15 mL of cold (4 °C) methanol, 2 × 15 mL of toluene, 2 × 15 mL of diethyl ether, and 2 × 15 mL of pentane, and then aspirated to dryness. Yield: 55 mg, 51%.

$[ReO_2(en)_2]I$. **1** (210 mg, 0.24 mmol) and 10 mL of methanol were combined in a 50-mL round-bottom flask. To this purple slurry were added 340 mg (5.7 mmol, 24 equiv) of ethylenediamine and another 10 mL of methanol. In less than 2 min, all of the purple solid disappeared and $[ReO_2(en)_2]I$ began to settle out. The pale yellow slurry was stirred for 15 min to ensure complete reaction. The precipitate was collected on a coarse frit, washed with 3 × 15 mL of diethyl ether, and aspirated to dryness. Yield: 90 mg, 80%. The identity of the product was confirmed by measuring UV–visible[5c,11] and infrared spectra.[10]

Results and Discussion

We have found that the complex $[ReO_2(PPh_3)_2I]$ is an ideal starting material for the synthesis of pyridine and amine complexes of the ReO_2^+ unit. By the use of our new method, $[ReO_2(en)_2]I$ can be prepared in 90% yield from $[ReO_2(PPh_3)_2I]$ in less than 15 min at room temperature.

$$[ReO_2(PPh_3)_2I] + 2en \xrightarrow{\text{excess en}} [ReO_2(en)_2]I\downarrow + 2PPh_3$$

The pyridine complexes are efficiently made by refluxing $ReO_2(PPh_3)_2I$ and 12–40 equiv of substituted pyridine in methanol for 0.25–12 h. Complexes of the form $[ReO_2L_4]^+$ can be made from the following pyridines by using this method: L = 4-(dimethylamino)pyridine (dmap), 4-pyrrolidinopyridine (4-pyrrpy), 4-methoxypyridine (4-MeOpy), 3,5-lutidine (3,5-lut), 4-picoline (4-pic), and 4-phenylpyridine (4-Phpy). In general, the more basic pyridines require fewer equivalents and shorter reflux periods.

$$[ReO_2(PPh_3)_2I] + 4L \xrightarrow{\text{excess L}} [ReO_2L_4]I + 2PPh_3$$

Metatheses to hexafluorophosphate salts were accomplished by using NH_4PF_6 in methanol, aqueous methanol, or methanol–acetone mixtures. ^1H NMR spectra indicate that salts containing noncoordinating anions such as ClO_4^- and PF_6^- tend to be more resistant to pyridine loss. The instability of halide salts has been noted previously.[5a,12]

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Table I. ^1H NMR Shift Data for $[ReO_2L_4]^+$ Complexes.

complex	solvent	peak positions
$[ReO_2(4\text{-pyrrpy})_4]PF_6$	CD_2Cl_2	δ 8.31 d, 2 H, J = 7 Hz
		δ 6.34 d, 2 H, J = 7 Hz
		δ 3.36 m, 4 H
		δ 2.00 m, 4 H
$[ReO_2(dmap)_4]PF_6$	DMSO-d_6	δ 8.24 d, 2 H, J = 7 Hz
		δ 6.68 d, 2 H, J = 7 Hz
		δ 3.30 s, 6 H
$[ReO_2(4\text{-MeOpy})_4]PF_6$	$CDCl_3$	δ 8.74 d, 2 H, J = 7 Hz
		δ 6.99 d, 2 H, J = 7 Hz
		δ 3.94 s, 3 H
$[ReO_2(3,5\text{-lut})_4]PF_6$	CD_3CN	δ 8.70 s, 2 H
		δ 7.37 s, 1 H
		δ 2.20 s, 6 H
$[ReO_2(4\text{-pic})_4]PF_6$	acetone-d_6	δ 8.95 d, 2 H, J = 7 Hz
		δ 7.42 d, 2 H, J = 7 Hz
		δ 2.58 s, 3 H
$[ReO_2(4\text{-Phpy})_4]I$	$CDCl_3$	δ 9.21 d, 2 H, J = 7 Hz
		δ 7.59 m, 7 H
$[ReO_2(py)_4]PF_6$	CD_3CN	δ 9.03 d, 2 H, J = 7 Hz
		δ 7.77 m, 1 H
		δ 7.47 m, 2 H

^1H NMR data are set out in Table I. ^1H NMR spectroscopy has been found to be particularly useful in the characterization of $[ReO_2L_4]^+$ complexes; resonances due to the protons at the 2- and 6-positions of pyridines are sensitive to the coordination environment of the nitrogen. Incomplete product formation and decomposition reactions are indicated by the appearance of multiple signals in the region δ 8.2-9.2 of the proton NMR spectrum.

The complexes $[ReO_2L_4]^+$ show two prominent bands in their UV-visible absorption spectra. Band I, which has been assigned to a $^1A_{1g}[(b_{2g})^2] \rightarrow {}^1E_g[(b_{2g})^1(e_g)^1]$ LF transition, is found in the region 410-490 nm ($\epsilon \approx 1600$) and shifts to lower energy as the pyridine basicity increases. The absorption maximum of band II is located between 330 and 370 nm ($\epsilon \approx 25\,000$) and undergoes a slight blue shift as the pyridine basicity increases. This band has been attributed both to LMCT (oxo to Re)[4] and to MLCT (Re to π^*-pyridine)[13] transitions.

In summary, the complex $[ReO_2(PPh_3)_2I]$ rather than K_2ReCl_6 is the material of choice for high-yield syntheses of $[ReO_2L_4]X$ complexes. Reactions proceed quickly and give higher yields of product than former methods did. The new method also has the potential to accommodate a wide variety of ligands and reaction conditions.

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Synthesis, Protonation Sequence, and NMR Studies of Polyazamacrocyclic Methylenephosphonates

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Macroscopic and microscopic protonation of a series of cyclic polyamino polyphosphonic acids (NOTP, DOTRP, and DOTP) was studied by using potentiometry and multinuclear magnetic resonance spectroscopy. The macroscopic protonation constants of these ligands were compared with those of the cyclic amines and corresponding acetate derivatives. Chemical shifts for the various protonated species derived from the ^{31}P and ^1H resonances are interpreted in terms of preferred conformational features due to intramolecular hydrogen bonding between protonated nitrogens and nonprotonated phosphonates and changes in phosphonate electronic structures with pH. Protonation sequences were obtained from the proton data by using published procedures. The data suggest that two nitrogens are protonated first in each compound followed by protonation of the phosphonate oxygens. In the triaza ligands, the third and fourth protonations occur at the phosphonate oxygens, and subsequent protons distribute between the remaining nitrogen and oxygens. This protonation scheme is quite similar to that previously observed for the analogous macrocycles containing acetate pendant groups.

Introduction

Considerable interest has emerged regarding the properties of polyamino polyphosphonates as chelating agents for metal ions[1] and the comparison between these and the corresponding polyamino polycarboxylates. Various open-chain amino polyphosphonates, including iminobis(methylenephosphonic acid), nitrilotris(methylenephosphonic acid), ethylenediaminetetrakis(methylenephosphonic acid), and diethylenetriaminepentakis(methylenephosphonic acid), and various metal ion complexes of these ligands have been studied by potentiometry and NMR.[2-26] In the present study, protonation of three polyazamacrocyclic poly(methylenephosphonate) ligands, 1,4,7-triazacyclononane-*N*,*N*′,*N*″-tris(methylenephosphonic acid) (NOTP), 1,5,9-triazacyclododecane-*N*,*N*′,*N*″-tris(methylenephosphonic acid) (DOTRP) and 1,4,7,10-tetraazacyclododecane-*N*,*N*′,*N*″,*N*‴-tetrakis(methylenephosphonic acid) (DOTP) (see structures in Figures 1-3), has been investigated by means of potentiometry and ^{31}P, ^1H, and ^{13}C NMR spectroscopy. The results are compared with earlier

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