itiated by a change of two electrons, forming B_{12a} (the cobalt(III) analogue of B_{12s}), which has been shown[27] to undergo rapid comproportionation (eq 4). Previous examinations of B_{12s} reactions[7,28] appear to favor 2e initiation, whereas studies of halate reductions by metal center reductants that can partake in either 1- or 2e changes demonstrate the feasibility of a series of single-electron transfers.[23b,29] Resolution of this matter awaits further study.

$$Co^{I}{-}H + IO_3^- \rightarrow [H{-}Co^{I}{-}O{-}IO_2] \rightarrow Co^{III}{-}OH + IO_2^- \qquad (3)$$

$$Co^{III}{-}OH + Co^{I}{-}H \rightarrow 2Co^{II}{-}OH_2 \quad \text{(rapid)} \qquad (4)$$

Registry No. BrO_3^-, 15541-45-4; IO_3^-, 15454-31-6; ClO_2^-, 14998-27-7; $H_2PO_4^-$, 14066-20-7; H_3BO_3, 10043-35-3; HCO_3^-, 71-52-3; B_{12s}, 18534-66-2.

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(27) Ryan, D. A.; Espenson, J. H.; Meyerstein, D.; Mulac, W. A. *Inorg. Chem.* **1978**, *17*, 3725.

(28) Banks, R. G. S.; Henderson, R. J.; Pratt, J. M. *J. Chem. Soc. A* **1968**, 2886.

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A High-Valent Ruthenium(VI) Dioxo Cation of 1,4,8,11-Tetramethyl-1,4,8,11-tetraazacyclotetradecane

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A high-valent *trans*-$[Ru(TMC)O_2]^{2+}$ (TMC = 1,4,8,11-tetramethyl-1,4,8,11-tetraazacyclotetradecane) cation has been produced from *trans*-$[Ru(TMC)Cl_2]^+$ through H_2O_2 oxidation. This novel diamagnetic ruthenium(VI) dioxo species has been subjected to detailed spectroscopic and electrochemical characterization. The UV–vis absorption spectra of *trans*-$[Ru(TMC)O_2]^{2+}$ and the previously reported *trans*-$[Ru(NH_3)_4O_2]^{2+}$ exhibit a weak vibronic structured absorption band at ~390 nm. Cyclic voltammetric and controlled-potential coulometric experiments showed that the reduction of *trans*-$[Ru(TMC)O_2]^{2+}$ to *trans*-$[Ru(TMC)O(H_2O)]^{2+}$ proceeded by a single-step two-electron-transfer process.

Introduction

The chemistry of high-valent ruthenium(IV) oxo amine complexes has received much attention in recent years.[1] However, there have been relatively few reports on high-valent ruthenium(VI) oxo species[2] despite the fact that *trans*-dioxoosmium(VI) species have been known for more than 1 decade. We have recently found that H_2O_2 oxidation of an aqueous solution of *trans*-$[Ru(TMC)Cl_2]Cl$ (TMC = 1,4,8,11-tetramethyl-1,4,8,11-tetraazacyclotetradecane) led to the formation of a yellow solid that we previously formulated as $[Ru(TMC)O(H_2O)](ClO_4)_2$.[3] Here, we would like to report the preparation, together with the spectroscopic and electrochemical characterization, of this high-valent ruthenyl ion. Our work clearly indicates that the previously reported $[Ru(TMC)O(H_2O)]^{2+}$ is best formulated *trans*-$[Ru^{VI}(TMC)O_2]^{2+}$.

Experimental Section

$K_2[RuCl_5H_2O]$ (Johnson and Matthey) and 1,4,8,11-tetramethyl-1,4,8,11-tetraazacyclotetradecane (TMC) (Strem) were used as supplied. All chemicals and solvents used were of analytical grade.

***trans*-$[Ru(TMC)O_2](Y)_2$ (Y = ClO_4, PF_6).** *trans*-$[Ru(TMC)Cl_2]Cl$[4] (0.3 g) and silver toluene-*p*-sulfonate (0.54 g) in deionized water (25 mL) were heated on a steam bath for 1/2 h. The resulting yellow solution (sometimes appeared as brownish yellow) was filtered off to remove the insoluble silver(I) chloride. H_2O_2 (3 mL; 30%) was slowly added to the filtrate that was then heated on a steam bath for about 5 min. The course of the reaction was followed by monitoring the UV–vis spectral changes of the solution. When the vibronic structured absorption band at ~390 nm had fully developed in 5–10 min, a saturated aqueous solution of $NaPF_6$ (5 mL) was added. Upon cooling, a yellow microcrystalline solid of *trans*-$[Ru(TMC)O_2]PF_6$ slowly deposited. This was then filtered off, washed with a diethyl ether/acetone mixture (10:1) and dried under vacuum at room temperature. Analytically pure samples of *trans*-$[Ru(TMC)O_2](PF_6)_2$ could be obtained by dissolving the crude yellow solid in acetone, filtering if necessary, followed by the subsequent addition of diethyl ether. The perchlorate salt was similarly prepared with $NaClO_4$ instead of $NaPF_6$. Yields were usually greater than 60%. Anal. Calcd for *trans*-$[Ru(TMC)O_2](ClO_4)_2$: C, 28.57; H, 5.44; N, 9.52; Cl, 12.0. Found: C, 28.60; H, 5.40; N, 9.60; Cl, 12.10. IR (Nujol mull): ν(Ru=O) 855 cm^{-1}. μ_{eff} ~0 μ_B at 25 °C. Λ_M for *trans*-$[Ru(TMC)O_2](PF_6)_2$ in H_2O 256 Ω^{-1} cm^2 mol^{-1} at 20 °C. UV–vis spectral data [λ_{max}, nm (ϵ)] in H_2O: ~445 (sh) (50), 388 (560), ~305 (sh) (960), 256 (1.03×10^4), 225 (1.28×10^4).

***trans*-$[Ru(NH_3)_4O_2]Cl_2$.** This complex was prepared by the method of Griffith and his co-workers.[2] Hydrated ruthenium hydroxide was prepared by the hydrolysis of $RuCl_3 \cdot xH_2O$ in 0.1 M NaOH. The complex has been found to be unstable upon exposure to moisture. IR (Nujol mull): ν(Ru=O) 845 cm^{-1}. UV–vis spectral data [λ_{max}, nm (ϵ_{max})] in 1 M $HClO_4$: ~450 sh (~40), 390 (~800), 300 (~800), 255 (br) (~10^4), 210 (~5×10^3).

Physical Measurements. Elemental analyses of the newly prepared complexes were performed by the Australian National Laboratory. IR spectra were measured in Nujol mulls on a Perkin-Elmer 577 spectrophotometer (4000–200 cm^{-1}). Electronic absorption spectra of freshly prepared solutions were measured with a Beckman Acta CIII spectrophotometer. Magnetic susceptibilities of solid samples were measured by the Guoy method using mercury tetrakis(thiocyanato)cobaltate(II) as the calibrant. ^1H NMR spectra were measured in D_2O or acetonitrile with a JEOL FX90Q Fourier transform NMR spectrometer. Tetramethylsilane was used as the internal standard.

Cyclic voltammograms were obtained with Princeton Applied Research (PAR) instruments: Model 175 (Universal Programmer); Model 173 (potentiostat-galvanostat). Pyrolytic graphite and glassy-carbon electrodes were used as appropriate as the working electrode. The reaction cell was a standard PAR Model K64 three-electrode jacketed polarographic cell that was maintained at ±0.1 °C by thermostated water. Cyclic voltammograms were recorded with a Houston Model 2000 X-Y recorder at slow scan rates (<1 V s^{-1}) and with a Tektronix Model 5103N storage oscilloscope at high scan rates (1 V s^{-1}). Controlled-potential coulometric electrolysis was carried out with a PAR Model 9610 coulometric cell system that was supplied with a PAR Model 377 synchronous stirring motor. A platinum-wire-gauze electrode was

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(1) See, for example: Samuels, G. J.; Meyer, T. J. *J. Am. Chem. Soc.* **1981**, *103*, 307–312.

(2) Griffith, W. P.; Pawson, D. *J. Chem. Soc., Dalton Trans.* **1973**, 1315–1320.

(3) Che, C. M.; Tang, T. W.; Poon, C. K. *J. Chem. Soc., Chem. Commun.* **1984**, 641–642.

(4) Tang, T. W. Ph.D. Thesis, University of Hong Kong, 1982.

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used as the working electrode during controlled-potential electrolysis experiments. All reaction solutions were deaerated before the CV scans and during the constant-potential electrolysis process.

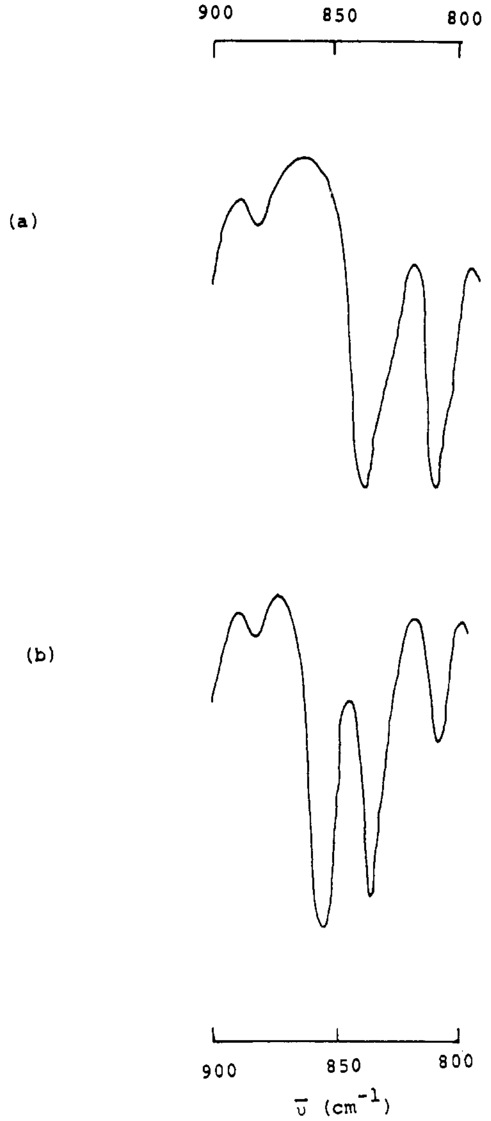

Figure 1. IR absorption spectra (Nujol mull) of (a) *trans*-$[Ru(TMC)Cl_2]ClO_4$ and (b) *trans*-$[Ru(TMC)O_2](ClO_4)_2$ in the 800–900-cm^{-1} region.

Results and Discussion

We have previously communicated that the oxidation of *trans*-$[Ru(TMC)Cl_2]Cl$ by H_2O_2 (30%) in boiling water produced a high-valent ruthenyl complex.[3] Recent kinetic studies showed that the major species present in a boiling aqueous soluton of *trans*-$[Ru(TMC)Cl_2]^+$ are probably a mixture of $[Ru(TMC)Cl(H_2O)]^{2+}$ and $[Ru(TMC)(H_2O)_2]^{3+}$[5]. A clean method for the preparation of this ruthenyl species is the direct oxidation of $[Ru(TMC)(H_2O)_2]^{3+}$ by H_2O_2. Other oxidants such as Ce(IV) have also been found to give the same results. This procedure cannot be applied for the preparation of the tetraammineruthenyl analogue. Reactions of *trans*- or *cis*-$[Ru(NH_3)_4(H_2O)_2]^{3+}$ with H_2O_2 or Ce(IV) in H_2O yielded a reddish brown solution of ill-defined products. This TMC ruthenyl species has been found to be stable in dark and moisture-free conditions but slowly deteriorated to a green substance on exposure to UV light, the nature of which has not yet been identified. Conductivity measurements showed that it is a 2:1 electrolyte. This species has been found to be diamagnetic both in solid state and in D_2O and acetonitrile-d_6 solutions.[6] The observed diamagnetism, together with the electrochemical behavior of this species, which will be described later in the text, clearly supports the formulation of a ruthenium(VI) dioxo species, *trans*-$[Ru(TMC)O_2]^{2+}$. The previously assumed formulation of $[Ru^{IV}(TMC)O(H_2O)]^{2+}$ would have resulted in a paramagnetic ground state $[(d_{xy})^2(d_{xz})^1(d_{yz})^1]$. This *trans*-$[Ru(TMC)O_2]^{2+}$ species is probably isomerically pure as only one band was developed upon chromatography through a Sephadex C-25 column with HCl (0.1 M) as the eluent. Reduction of *trans*-$[Ru(TMC)O_2]^{2+}$ in HCl (1 M) yielded a species that has exactly the same UV–vis absorption spectrum as the starting *trans*-$[Ru(TMC)Cl_2]^+$.[7] This finding, together with the presence of only one ν(Ru=O) stretch in the IR spectrum of *trans*-$[Ru(TMC)O_2](ClO_4)_2$ (Figure 1), indicates a trans configruation for this ruthenium(VI) dioxo complex. The observed ν(Ru=O) is also in agreement with that for the few *trans*-dioxoruthenium(VI) complexes reported by Griffith and co-workers.[2]

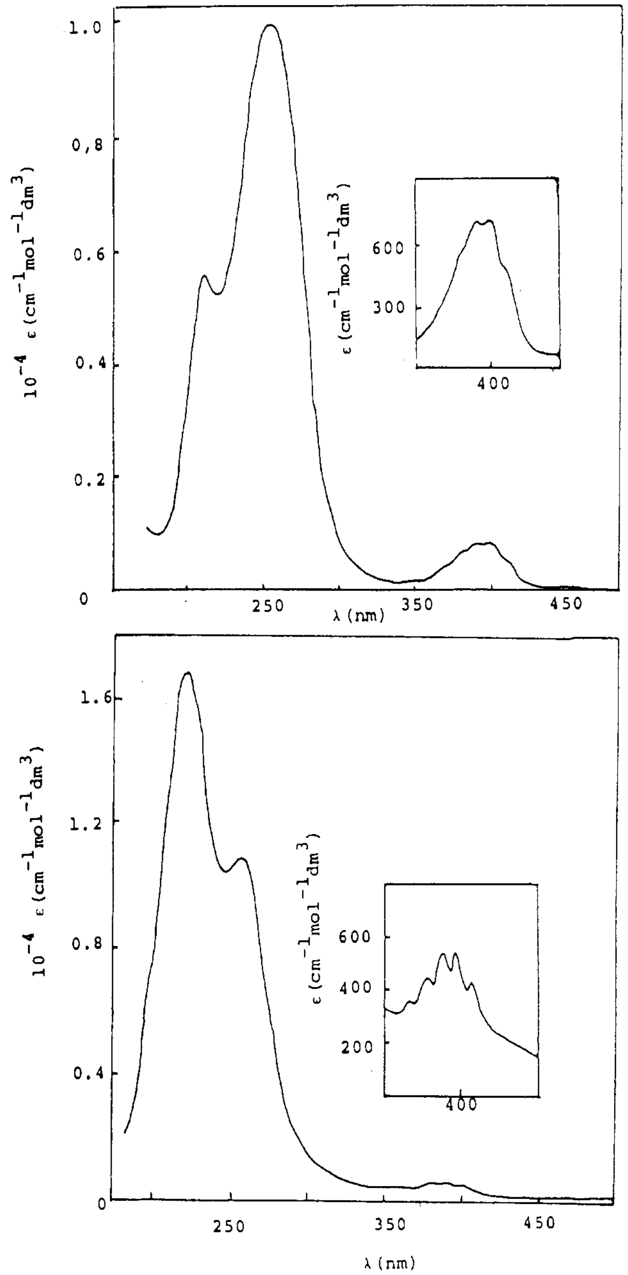

Figure 2. UV–Vis absorption spectra of (top) *trans*-$[Ru(NH_3)_4O_2]^{2+}$ and (bottom) *trans*-$[Ru(TMC)O_2]^{2+}$ in $HClO_4$ (0.1 M) at 25 °C.

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(5) Kwong, S. S., unpublished work.

(6) Well-resolved ^1H NMR spectra of *trans*-$[Ru(TMC)O_2]^{2+}$ have been obtained in D_2O and acetonitrile-d_6 solutions. However, the spectra were too complex to draw any useful structural information.

(7) Che, C. M., unpublished results.

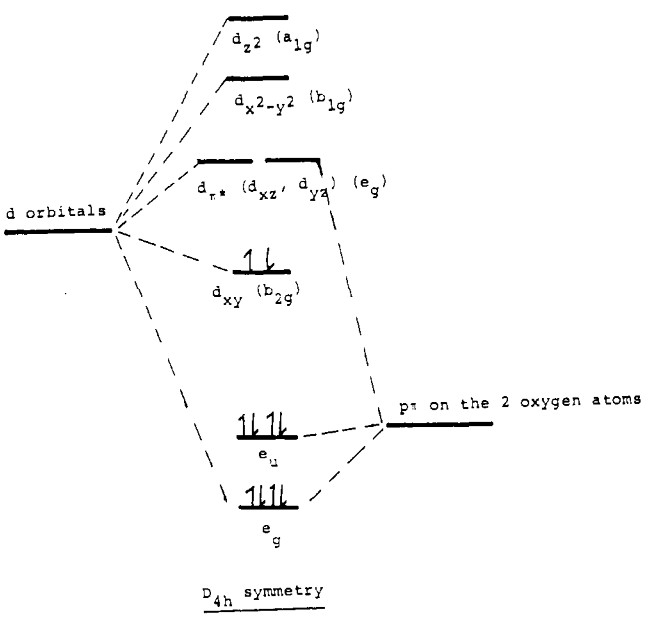

Figure 3. Simplied molecular orbital diagram of *trans*-$[ML_4O_2]^{n+}$ (L = unidentate amine ligand) with a d^2 electronic configuration (assuming a D_{4h} symmetry).

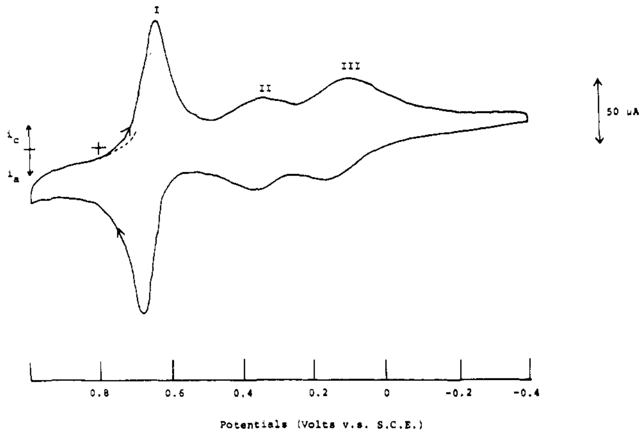

Figure 4. Cyclic voltammogramm of *trans*-$[Ru(TMC)O_2]^{2+}$ (~1 mM) in 0.1 M $HClO_4$ using a graphite electrode at a scan rate of 50 mV s^{-1}.

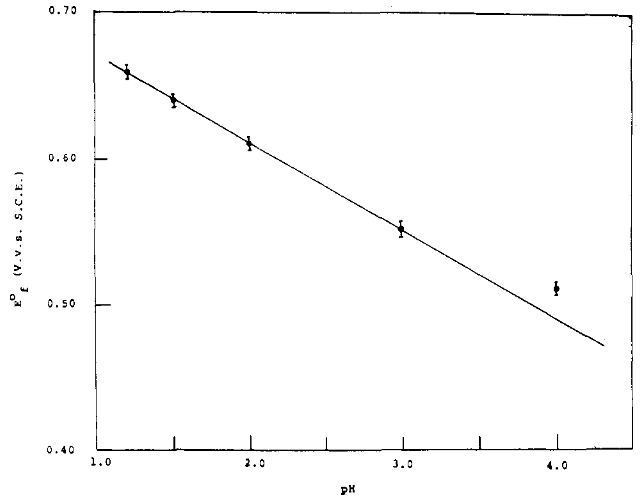

Figure 5. Linear plot of $E_f°$ vs. pH, according to the equation $E_f° = E°_{298} - 0.059pH$, for the *trans*-$[Ru(TMC)O_2]^{2+}$/*trans*-$[Ru(TMC)O(H_2O)]^{2+}$ couple in acidic aqueous solutions.

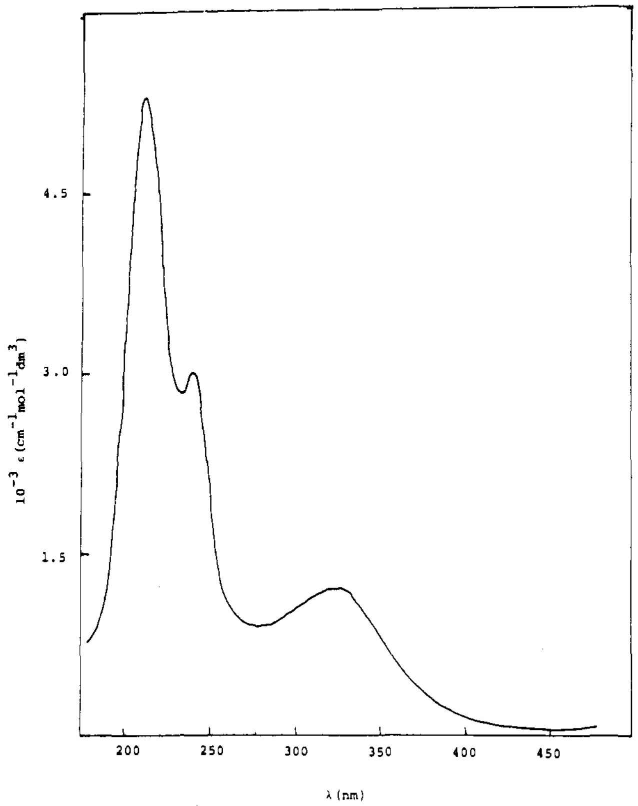

Figure 6. UV-Vis absorption spectrum of *trans*-$[Ru(TMC)(H_2O)_2]^{3+}$ in $HClO_4$ (0.1 M), freshly generated by controlled-potential reduction of *trans*-$[Ru(TMC)O_2]^{2+}$ at 0.25 V vs. SCE.

The UV–vis absorption spectra of *trans*-$[Ru(TMC)O_2]^{2+}$ and *trans*-$[Ru(NH_3)_4O_2]^{2+}$ in $HClO_4$ (0.1 M) are shown in Figure 2. Figure 3 is a simplified version of the molecular orbital diagram of *trans*-dioxo transition-metal complexes with a d^2 electronic configuration after the work of Gray and co-workers.[8] Simple ligand field analysis would predict only one fully allowed ligand-to-metal $p_\pi(O)$ $(e_u) \rightarrow d_\pi^*$ (e_g) transition ($^1A_{1g} \rightarrow {}^1A_{2u}$ transition in D_{4h} symmetry), and in the present case, this is assigned to the very intense band at 255 nm. The band at ca. 210–220 nm has been found in most ruthenium amino complexes,[7] the nature of which has not yet been identified. Perhaps the most interesting feature is the presence of a weak vibronic structured absorption band centered at ~390 nm. We previously assigned this band as due to the spin-forbidden $p_\pi(O) \rightarrow d_\pi^*$ transition. However, this band may be better considered to arise from the $(d_{xy})^2 \rightarrow (d_{xy})^1 (d_\pi^*)^1$ $(^1A_{1g} \rightarrow {}^1E_g)$ transition, which is vibronically coupled to the A_{2u} vibrational stretching mode [ν(Ru–O)]. The estimated vibrational spacing, 550–650 cm^{-1}, is attributable to the ν(Ru–O) stretch in the excited state. The much reduced ν(Ru–O) stretch in the excited-state as compared with the ground-state value (~840–860 cm^{-1}) indicates that the e_g orbitals possess tremendous $\pi^*(RuO_2)$ character. Work is in progress to study the electronic spectroscopy of this ruthenium(VI) dioxo system.

The cyclic voltammogram of *trans*-$[Ru(TMC)O_2]^{2+}$ in $HClO_4$ (0.1 M) at pH 1.2 is shown in Figure 4. Three quasi-reversible couples, I–III (ΔE_p ~ 30–100 mV), at 0.66, 0.36, and 0.15 V vs. SCE respectively, were observed at slow scan rates (50 mV s^{-1}). Previously the 0.66-V couple was tentatively assigned to a Ru^{IV}/Ru^{III} couple by reference to other known Ru^{IV}/Ru^{III} couples appearing at approximately the same potential.[3] However, the relative peak area of couple I is nearly twice that of the other two. Controlled-potential reductions of *trans*-$[Ru(TMC)O_2]^{2+}$ in $HClO_4$ (0.1 M) at 0.58 V vs. SCE.[9] showed that $n = 1.90$ where

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(8) Winker, J. R.; Gray, H. B. *J. Am. Chem. Soc.* **1983**, *105*, 1373–1374.

(9) Experiments were performed in $HClO_4$ (0.1 M; *trans*-$[Ru(TMC)O_2](PF_6)_2$, 10^{-4} mol) using a platinum-wire-gauze working electrode.

n is the electrochemical stoichiometry. This result, together with the 30–40-mV peak-to-peak separation (ΔE_p) of this couple, strongly suggests that it is a reversible single-step two-electron-transfer process, as represented by eq 1. This is further supported

$$trans\text{-}[\mathrm{Ru(TMC)O_2}]^{2+} + 2\mathrm{H}^+ + 2e^- \rightleftharpoons trans\text{-}[\mathrm{Ru^{IV}(TMC)O(H_2O)}]^{2+} \quad (1)$$

by the linear plot of $E_f°$ vs. pH with a slope of 60 mV over the pH range 1–3 (Figure 5) as required by the equation $E°_{298} = E_f° + 0.059\mathrm{pH}$.

In acid solutions couples II and III could be assigned to reactions 2 and 3, respectively.

$$trans\text{-}[\mathrm{Ru^{IV}(TMC)(O)(H_2O)}]^{2+} + 2\mathrm{H}^+ + e^- \rightleftharpoons trans\text{-}[\mathrm{Ru(TMC)(H_2O)_2}]^{3+} \quad (2)$$

$$trans\text{-}[\mathrm{Ru^{III}(TMC)(H_2O)_2}]^{3+} + e^- \rightleftharpoons trans\text{-}[\mathrm{Ru(TMC)(H_2O)_2}]^{2+} \quad (3)$$

Controlled-potential coulometry of *trans*-$[\mathrm{Ru^{IV}(TMC)O(H_2O)}]^{2+}$ in $\mathrm{HClO_4}$ (0.1 M) at 0.25 V vs. SCE established n = 1 for reaction 2.[9] All three couples were shifted cathodically, with the two one-electron waves broadening substantially on increasing the pH of the medium. We have also found that Ru(VI) dioxo complexes are particularly unstable at high pH. The $E_f°$ value for the *trans*-$[\mathrm{Ru(TMC)(H_2O)_2}]^{3+/2+}$ couple is ~300 mV more anodic than that for the corresponding *trans*-$[\mathrm{Ru(NH_3)_4(H_2O)_2}]^{3+/2+}$ couple.[10] It is quite likely that this is due to the effect of the TMC ligand as the same difference in $E_f°$ values has also been found for *trans*-$[\mathrm{Ru(TMC)Cl_2}]^+$ and *trans*-$[\mathrm{Ru(NH_3)_4Cl_2}]^+$.[7] Whereas *trans*-$[\mathrm{Ru(NH_3)_4O_2}]^{2+}$ undergoes complicated chemical reactions upon reduction,[11] the redox interconversion between *trans*-$[\mathrm{Ru(TMC)O_2}]^{2+}$ and *trans*-$[\mathrm{Ru(TMC)(H_2O)_2}]^{2+}$ is entirely reversible in strong acids. The cyclic voltammogram of *trans*-$[\mathrm{Ru(TMC)O_2}]^{2+}$ remains relatively unchanged upon repetitive scanning. The inherent stability of *trans*-$[\mathrm{Ru^{IV}(TMC)O(H_2O)}]^{2+}$ as compared with the analogous $[\mathrm{Ru^{IV}(NH_3)_4O(H_2O)}]^{2+}$ (if it does exist) is probably attributed to the macrocyclic as well as the tertiary amine nature of the TMC ligand. Both *trans*-$[\mathrm{Ru^{IV}(TMC)O(H_2O)}]^{2+}$ and *trans*-$[\mathrm{Ru(TMC)(H_2O)_2}]^{3+}$ could be generated by controlled-potential coulometry. The UV–vis absorption spectrum of the latter cation is shown in Figure 6. Recent kinetic work showed that the chloride anation of *trans*-$[\mathrm{Ru(TMC)(H_2O)_2}]^{3+}$, freshly generated electrochemically from *trans*-$[\mathrm{Ru(TMC)O_2}]^{2+}$, is just the reverse of the chloride aquation of *trans*-$[\mathrm{Ru(TMC)Cl_2}]^+$.[7] This indicates that both *trans*-$[\mathrm{Ru(TMC)O_2}]^{2+}$ and *trans*-$[\mathrm{Ru(TMC)Cl_2}]^+$ may have the same geometrical configuration and ligand conformation.

Conclusion

We have shown here unambiguously the existence of a stable trans Ru(VI) dioxo tetraamine complex. The stability of *trans*-$[\mathrm{Ru(TMC)O_2}]^{2+}$ and *trans*-$[\mathrm{Ru(TMC)O(H_2O)}]^{2+}$ indicates the possibility of developing long-lived oxidative catalysts with high-valent ruthenium macrocyclic amine oxo species.

Acknowledgment. We thank the Committee on Research and Conference Grants, University of Hong Kong, for support.

Registry No. *trans*-$[\mathrm{Ru(TMC)O_2}](\mathrm{ClO_4})_2$, 95978-18-0; *trans*-$[\mathrm{Ru(TMC)O_2}](\mathrm{PF_6})_2$, 95978-19-1; *trans*-$[\mathrm{Ru(TMC)Cl_2}]\mathrm{Cl}$, 92141-42-9; *trans*-$[\mathrm{Ru(NH_3)_4O_2}]\mathrm{Cl_2}$, 38882-90-5; *trans*-$[\mathrm{Ru(TMC)O(OH_2)}]^{2+}$, 92141-47-4; *trans*-$[\mathrm{Ru(TMC)(H_2O)_2}]^{3+}$, 95978-20-4; *trans*-$[\mathrm{Ru(TMC)(H_2O)_2}]^{2+}$, 95978-21-5.

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(10) Poon, C. K.; Kwong, S. S.; Che, C. M.; Kan, Y. P. *J. Chem. Soc., Dalton Trans.* **1982**, 1457–1463.

(11) Preliminary experiments showed that the reduction of *trans*-$[\mathrm{Ru(NH_3)_4O_2}]^{2+}$ or the $\mathrm{H_2O_2}$ oxidation of *trans*-$[\mathrm{Ru(NH_3)_4(H_2O)_2}]^{3+}$ yielded some ill-defined μ-oxo ruthenium amine species: Che, C.M., unpublished results.

Contribution No. 3539 from the Central Research & Development Department,
Experimental Station, E. I. du Pont de Nemours and Company, Wilmington, Delaware 19898

Shape Selectivity in Olefin Hydrogenation Using Rhodium-Containing Zeolites

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Received August 8, 1984

Competitive hydrogenation of two cyclic olefins using rhodium zeolite catalysts shows a selectivity based on the molecular dimensions of these substrates. A maximum selectivity of 47 (based on the ratio of hydrogenation rates) is achieved with the medium-pore zeolite ZSM-11 for the preferential hydrogenation of cyclopentene in competition with 4-methylcyclohexene. This selectivity appears to be a composite of the intrinsic size selectivity of the zeolite framework as modified by the presence of intracrystalline water. Rh–X shows selectivities ranging from 1 to 30 as the water content rises while Rh–ZSM-11 displays a maximum in selectivity at an intermediate water content. Selective poisoning of metal sites on the external zeolite surface with bulky phosphines is essential in order to obtain selectivity.

Introduction

Zeolites are microporous crystalline solids that have been used for ion exchange, as detergents, as drying agents and in the catalytic cracking of petroleum.[1] Recently, chemists have begun to explore the ability of zeolites to impose shape selectivity upon transition-metal-catalyzed reactions since such selectivity may far exceed that possible in homogeneous systems.[2] In their pioneering study, Huang and Schwartz[3] reported that treatment of Linde 13X zeolite with tris(π-allyl)rhodium results in a catalyst with improved selectivity toward hydrogenation of small olefins. However, this selectivity was deduced via a comparison of rates for hydrogenation of individual olefins, while to demonstrate true reaction selectivity, one would prefer to compete the two olefins in a single reaction medium. Schwartz also showed that treatment of this catalyst with $\mathrm{P(Bu)_3}$, which can only react with surface Rh sites, has little effect on rate, while $\mathrm{P(Me)_3}$, which can enter the zeolite, stops catalytic activity.

We have now prepared other rhodium-containing zeolites for comparison with Schwartz's catalyst. In the competitive hydrogenation of cyclopentene and 3- or 4-methylcyclohexene, we observe selectivity increases over that measured for Schwartz's catalyst of an order of magnitude, from 4 to 47. In this paper, we describe the preparation of zeolite catalysts made either by

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(1) Schwochow, F.; Puppe, L. *Angew Chem., Int. Ed. Engl.* **1974**, *14*, 620.

(2) Dessau, R. M. *J. Catal.* **1982**, *77*, 304. Chen, N. Y.; Weisz, P. B. *Chem. Eng. Prog., Symp. Ser.* **1967**, *63* (No. 73), 86.

(3) (a) Huang, T.-N.; Schwartz, J. *J. Am. Chem. Soc.* **1982**, *104*, 5245. (b) Huang, T.-N.; Schwartz, J.; Kitajima, N. *J. Mol. Catal.* **1984**, *22*, 389-93.

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