Contribution from the Department of Chemistry,
University of Southern California, Los Angeles, California 90007

Improved Synthesis of *nido*-Dicarbaoctaborane (10)

Thomas J. Reilly and Anton B. Burg*

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We wish to report a more efficient synthesis of the interesting nido carborane $C_2B_6H_{10}$. Although the records indicate our work to have been the first actual isolation of this unstable compound, it is clear that others accomplished the same reaction independently, after earlier mass spectral evidence had suggested its existence.[1] Also, we are glad to acknowledge their essential interpretations, without which the structural character of our product would have been more difficult to recognize.

Our work began with the 25° vapor-phase reaction of $C_2B_3H_5$ with twice as much B_2H_6, during 2 days in a sealed tube at 2 atm pressure. The $C_2B_3H_5$ was 85% consumed, with 6% conversion to $C_2B_6H_{10}$, accompanied by an equimolar amount of hydrogen. The mass spectrum directly indicated the formula $C_2B_6H_{10}$, with H-abstraction showing nido character. The ^{11}B nmr spectrum was identical with that which was obtained later on the same instrument, for a sample furnished by Gotcher, *et al.*[1] Our apparently pure sample had an equilibrium vapor pressure of 24 mm at 0° and decomposed at a rate comparable to that of tetraborane(10).

A better synthesis was accomplished by means of a 750-ml concentric-cylindrical hot-cold reactor having a diameter of 90 mm and an interwall distance of 11 mm. The internal cold finger was held at $-78°$ and the outer wall was heated to 165°. A 4-hr operation with 2.15 mmol of $C_2B_3H_5$ and 4.37 mmol of B_2H_6 led to 78% consumption of the $C_2B_3H_5$ and a 0.61-mmol yield (36.5%) of $C_2B_6H_{10}$. The accompanying less volatile mixture of other carboranes (0.40 mmol) was not resolved but showed a mass spectrum having the correct isotopic patterns for $C_2B_7H_9$ and $C_2B_8H_{10}$.

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Registry No. *nido*-$C_2B_6H_{10}$, 50442-30-3; $C_2B_3H_5$, 20693-66-7; B_2H_6, 19287-45-7.

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Contribution from the Department of Chemistry,
University of North Carolina, Chapel Hill, North Carolina 27514

Unusual Activation Parameters in the Oxidation of $Fe(H_2O)_6^{2+}$ by Polypyridine Complexes of Iron(III). Evidence for Multiple Paths for Outer-Sphere Electron Transfer

Jeffrey L. Cramer and Thomas J. Meyer*

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We recently reported the kinetics of oxidation of $Fe(H_2O)_6^{2+}$ by a series of polypyridine complexes of ruthenium(III)[1]

$$Fe(H_2O)_6^{2+} + Ru(III) \rightarrow Fe(H_2O)_6^{3+} + Ru(II) \quad (1)$$

(Ru(III) is $Ru(terpy)_2^{3+}$, $Ru(phen)_3^{3+}$, $Ru(bipy)_3^{3+}$, or $Ru(bipy)_2(py)_2^{3+}$ [2]). The activation parameters for the reactions are remarkable in that the $\Delta H^{\ddagger}$ values are small or even slightly negative ($\Delta H^{\ddagger} = -2.80 \pm 0.50$ to $\Delta H^{\ddagger} = 0.30 \pm 0.05$ kcal/mol). It was suggested by us that the activation parameters are indicative of an activation process which differs in detail from the Marcus-Hush model for outer-sphere electron transfer.[3]

The rates of oxidation of $Fe(H_2O)_6^{2+}$ by a series of iron(III)-polypyridine complexes have been studied by Ford-Smith and Sutin[4] and Sutin and Gordon,[5] *e.g.*

$$Fe(H_2O)_6^{2+} + Fe(phen)_3^{3+} \rightarrow Fe(H_2O)_6^{3+} + Fe(phen)_3^{2+} \quad (2)$$

Because of the similarity in activation parameters for reactions 1 and 2 it was also suggested by us that the Fe(III)- and Ru(III)-polypyridine reactions occur by the same "non-Marcus" path.

We report here the kinetics of oxidation of $Fe(H_2O)_6^{2+}$ by $Fe(terpy)_2^{3+}$ and $Fe(bipy)_3^{3+}$. The activation parameters for the two reactions provide further support for a similarity in mechanism for the Fe(III)- and Ru(III)-polypyridine reactions.

Experimental Section

Materials. Water used for the kinetic experiments was doubly distilled from alkaline permanganate. Perchloric acid (Reagent grade from MCB), ferrous sulfate (Reagent ACS from Allied Chemical), sodium perchlorate (G. F. Smith), 2,2′,2″-terpyridine (G. F. Smith), 2,2′-bipyridine (Aldrich Chemical Co.), lead dioxide (Alfa Inorganics), and tetra-*n*-butylammonium chloride (Eastman) were used without further purification. Ceric ammonium nitrate (G. F. Smith) was vacuum-dried at 84° for 4 hr. Ferrous perchlorate (G. F. Smith) was recrystallized from perchloric acid. Other solvents used were reagent grade.

Preparations. The complexes $Fe(bipy)_3(ClO_4)_2$ and $Fe(terpy)_2(ClO_4)_2$ were prepared using published procedures.[4,6] Conversions to the more water-soluble chloride salts were carried out by dissolving the perchlorates in acetone and adding a saturated solution of tetra-*n*-butylammonium chloride. The chloride salts, which precipitated, were collected on a frit and washed with acetone. The iron polypyridine content of the salts was determined spectrophotometrically using known extinction coefficients.[7] The salts are extensively hydrated, as has been found, for example, for $Os(phen)_3Cl_2 \cdot 8H_2O$[8] and can best be formulated as $Fe(bipy)_3Cl_2 \cdot nH_2O$ and $Fe(terpy)_2Cl_2 \cdot nH_2O$ ($n \approx 10$).

Solutions. Solutions of the iron(III) complexes $Fe(terpy)_2^{3+}$ and $Fe(bipy)_3^{3+}$ were prepared by oxidizing stock solutions of the appropriate iron(II) complex by ceric ammonium nitrate. The iron(III) solutions were 0.45 *M* in $HClO_4$. The use of lead dioxide as the oxidant gave erratic results. Stock solutions of $Fe(H_2O)_6^{2+}$ were prepared in 0.1 *M* $HClO_4$ and the concentration of iron(II) was deter-

(1) J. N. Braddock and T. J. Meyer, *J. Amer. Chem. Soc.*, **95**, 3158 (1973).

(2) Abbreviations used here include terpy for 2,2′,2″-terpyridine, phen for 1,10-phenanthroline, bipy for 2,2′-bipyridine, and py for pyridine.

(3) For pertinent reviews see (a) R. A. Marcus, *Annu. Rev. Phys. Chem.*, **15**, 155 (1966); (b) J. E. Earley, *Progr. Inorg. Chem.*, **13**, 243 (1970); (c) R. G. Linck, *MTP (Med. Tech. Publ. Co.) Int. Rev. Sci.: Inorg. Chem., Ser. One*, 9, 303 (1971); (d) W. L. Reynolds and R. W. Lumry, "Mechanisms of Electron Transfer," Ronald Press, New York, N. Y., 1966; (e) N. Sutin, *Chem. Brit.*, **8**, 148 (1972).

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mined spectrophotometrically as the $\mathrm{Fe(phen)_3^{2+}}$ complex at 510 nm.[6]

Measurements. Ultraviolet-visible spectra were obtained using Cary Models 14 and 17, Guilford Model 240, and Unicam SP800B spectrophotometers. The kinetics experiments were carried out under nitrogen using a stopped-flow spectrometer which has been described previously.[1,9] The photomultiplier voltage output from the spectrometer was amplified to *ca.* 20 V, passed through an analog to digital converter, and then analyzed directly by a Raytheon 706 computer using the program WIGWAM 03.[10] The kinetic runs were carried out under pseudo-first-order conditions with $\mathrm{Fe(H_2O)_6^{2+}}$ in excess.

Results

The observed second-order rate constants for the oxidation of $\mathrm{Fe(H_2O)_6^{2+}}$ by both $\mathrm{Fe(terpy)_2^{3+}}$ and $\mathrm{Fe(bipy)_3^{3+}}$ in 0.28 *M* $\mathrm{HClO_4}$ are given in Table I. The net reaction in both cases is

$$\mathrm{Fe(H_2O)_6^{2+} + Fe(III) \rightarrow Fe(H_2O)_6^{3+} + Fe(II)}$$

Kinetic runs were carried out over a range of $\mathrm{Fe(H_2O)_6^{2+}}$ concentrations ($\sim$(3-10) × 10^{-4} *M*) in order to verify the first-order dependence on iron(II). The initial iron(III) concentrations were varied in the range (1.4-3.5) × 10^{-5} *M*. As indicated by the error limits given in Table I, which represent the range in observed rate constants, reproducibility between different experiments was good. For purposes of comparison the data of Ford-Smith and Sutin at 25.0° in 0.50 *M* $\mathrm{HClO_4}$[4] is also given. The remarkable agreement between the values reported here and the earlier data may be partly fortuitous since the media used are slightly different (0.28 and 0.50 *M* $\mathrm{HClO_4}$).

The activation parameters for the two reactions studied here and for some closely related reactions are given in Table II. The error limits for the activation parameters reported here were calculated using standard methods of error analysis.[11] The calculations were made using an estimated uncertainty in temperature and the error limits for the rate constants given in Table I. It is interesting to note that within experimental error the $\Delta H^{\ddagger}$ values for the reactions involving $\mathrm{Fe(terpy)_2^{3+}}$ and $\mathrm{Fe(bipy)_3^{3+}}$ are zero or slightly negative and that the pattern in activation parameters—small or even negative $\Delta H^{\ddagger}$ values, large negative $\Delta S^{\ddagger}$ values—is similar for both the iron(III) and ruthenium(III) complexes.

Table I. Rate Constants for the Oxidation of $\mathrm{Fe(H_2O)_6^{2+}}$ by $\mathrm{Fe(terpy)_2^{3+}}$ and $\mathrm{Fe(bipy)_3^{3+}}$ in 0.28 *M* $\mathrm{HClO_4}$

Oxidant	T,[a] °C	$10^{-4}k$,[b] M^{-1} sec^{-1}	No. of runs[c]
$\mathrm{Fe(terpy)_2^{3+}}$	5.0	8.45 ± 0.18	5
	15.0	8.31 ± 0.23	4
	25.0	8.42 ± 0.05	2
	25.0	8.5[d]	
$\mathrm{Fe(bipy)_3^{3+}}$	5.0	2.76 ± 0.16	5
	15.0	2.61 ± 0.16	4
	25.0	2.69 ± 0.08	3
	25.0	2.7[d]	

[a] ±0.2°. [b] The deviation given is the *range* of rate constants; averaged values are given for *k*. [c] Each "run" was actually the average of four or five stopped-flow experiments using the same stock solutions. [d] Reference 5; in 0.5 *M* $\mathrm{HClO_4}$.

Table II. Activation Parameters for the Oxidation of $\mathrm{Fe(H_2O)_6^{2+}}$ by Iron(III)- and Ruthenium(III)-Polypyridine Complexes

Oxidant	$\Delta G^{\ddagger}$(25°), kcal/mol	$\Delta H^{\ddagger}$, kcal/mol	$\Delta S^{\ddagger}$(25°), eu
$\mathrm{Fe(terpy)_2^{3+}}$ [a]	10.7 ± 0.1	−0.6 ± 0.6	−38 ± 2
$\mathrm{Fe(bipy)_3^{3+}}$ [a]	11.4 ± 0.1	−0.8 ± 0.5	−41 ± 2
$\mathrm{Fe(phen)_3^{3+}}$ [b]	11.3	0.2 ± 0.2	−37
$\mathrm{Ru(terpy)_2^{3+}}$ [c]	9.40 ± 1.9	−2.80 ± 0.50	−41 ± 4
$\mathrm{Ru(phen)_3^{3+}}$ [c]	9.40 ± 1.9	−1.35 ± 0.33	−36 ± 4
$\mathrm{Ru(bipy)_3^{3+}}$ [c]	9.53 ± 0.9	−0.30 ± 0.05	−33 ± 3
$\mathrm{Ru(bipy)_2(py)_2^{3+}}$ [c]	9.53 ± 0.9	+0.30 ± 0.05	−31 ± 3

[a] This work; 0.28 *M* $\mathrm{HClO_4}$. [b] Reference 5; 0.5 *M* $\mathrm{HClO_4}$. [c] Reference 1; 1.0 *M* $\mathrm{HClO_4}$.

Discussion

Similarity in Mechanism for the Fe(III) and Ru(III) Reactions.

The reactions studied here and in the earlier work[1]

$$\mathrm{Fe(H_2O)_6^{2+} + M(III) \rightarrow Fe(H_2O)_6^{3+} + M(II)}$$

(M = iron- or ruthenium-polypyridine complex) are necessarily outer sphere. They are outer sphere at least in the sense that a mechanism involving a ligand bridge between *normal* coordination positions of both reacting ions can be ruled out. Both the iron(III) and ruthenium(III) complexes are substitution inert on the time scale for electron transfer.

The negative $\Delta H^{\ddagger}$ values for the oxidation of $\mathrm{Fe(H_2O)_6^{2+}}$ by ruthenium(III)-polypyridine complexes have led to the suggestion that the reactions occur by a "non-Marcus" path which includes at least one distinctive feature not considered in the Marcus model for outer-sphere electron transfer.[1] The similarity in activation parameters for the Fe(III) and Ru(III) reactions suggests that they occur by the same path.

For the oxidation of $\mathrm{Fe(H_2O)_6^{2+}}$ by a series of substituted 1,10-phenanthroline complexes of Fe(III) Ford-Smith and Sutin found a linear relationship between free energies of activation and overall free energy changes.[4] The ruthenium(III)-polypyridine reactions fit the same linear relationship which also implies a common mechanism, but there is some uncertainty because of the different media used.

Multiple Paths for Outer-Sphere Electron Transfer. Agreement between observed and calculated rates for many outer-sphere electron-transfer reactions has been obtained using the Marcus free energy correlation[3]

$$\Delta G_{12}^{\ddagger} = \tfrac{1}{2}[\Delta G_{11}^{\ddagger} + \Delta G_{22}^{\ddagger} + \Delta G_{12}^{\circ} - (RT/2)\ln f] \qquad (3)$$

$$\ln f = (\ln K_{12})^2/[4\ln(k_{11}k_{22}/Z^2)]$$

where ΔG_{12}°, $\Delta G_{12}^{\ddagger}$, and K_{12} refer to the overall reactions. $\Delta G_{11}^{\ddagger}$, $\Delta G_{22}^{\ddagger}$, k_{11}, and k_{22} are the free energies of activation and rate constants for the $\mathrm{Fe(H_2O)_6^{3+}}$-$\mathrm{Fe(H_2O)_6^{2+}}$ and M(III)-M(II) polypyridine self-exchange reactions. Z is the collision frequency between two neutral molecules in solution. The agreement extends to reactions between the $\mathrm{Fe(H_2O)_6^{3+,2+}}$ and Ru(III)-Ru(II) couples, namely, to the oxidations of $\mathrm{Ru(NH_3)_6^{2+}}$ and $\mathrm{Ru(en)_3^{2+}}$ by $\mathrm{Fe(H_2O)_6^{3+}}$.[12] For such reactions the model used by Marcus in deriving eq 3 appears to account satisfactorily for the mechanistic details of the activation process. However, the agreement does not extend to the oxidation of $\mathrm{Fe(H_2O)_6^{2+}}$ by the iron(III)- and ruthenium(III)-polypyridine complexes. Using eq 3, calculated values for the M(III)-M(II) polypyridine self-exchange rates are too low by $\sim10^5$.[1,8,13]

The oxidation of $\mathrm{Fe(H_2O)_6^{2+}}$ by the Fe(III) and Ru(III) polypyridine complexes appears to occur by a distinct path.

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A feature which characterizes the path is the negative or small positive $\Delta H^{\ddagger}$ values.

An implication of this work is that it may be possible to obtain experimental data which reveal details of the activation process not considered in the Marcus–Hush model for outer-sphere electron transfer. In fact, it may be possible by experimental means to identify a series of paths for outer-sphere electron transfer which differ in detail from the "normal" Marcus–Hush path. In addition to the path discussed here, Lavallee, *et al.*, have suggested that the oxidation of Np(III) by $Ru(NH_3)_6{}^{3+}$ and $Ru(NH_3)_5H_2O^{3+}$ may occur by an "unorthodox" transition state characterized by large negative $\Delta S^{\ddagger}$ values in which one of the oxygens bound to neptunium attacks an octahedral face of the ruthenium complexes.[14] Outer-sphere electron-transfer reactions involving Co(III)–Co(II) complexes may also occur by a distinct path; Rillema and Endicott have recently inferred that certain key factors which make major contributions to such reactions are not considered in current theoretical models of outer-sphere electron transfer.[15]

Mechanistic Details of the Non-Marcus Path. The full mechanistic details of the non-Marcus path discussed here are not clear. However, the oxidation of $Fe(H_2O)_6{}^{2+}$ by iron(III)-polypyridine complexes has been studied in detail by Sutin and coworkers. From their work certain facts are known which allow inferences to be drawn.

1. The pattern of activation parameters—negative or small positive $\Delta H^{\ddagger}$ values, large negative $\Delta S^{\ddagger}$ values—is paralleled by the thermodynamic parameters for the overall reactions: for $Fe(H_2O)_6{}^{2+} + Ru(bipy)_3{}^{3+} \rightarrow Fe(H_2O)_6{}^{3+} + Ru(bipy)_3{}^{2+}$, $\Delta H_{12}{}^{\circ} = -27$ kcal/mol and $\Delta S_{12}{}^{\circ} = -43$ eu;[16,17] and for $Fe(H_2O)_6{}^{2+} + Fe(bipy)_3{}^{3+} \rightarrow Fe(H_2O)_6{}^{3+} + Fe(bipy)_3{}^{2+}$, $\Delta H_{12}{}^{\circ} = -23$ kcal/mol and $\Delta S_{12}{}^{\circ} = -47$ eu.[16,17]

2. Ford-Smith and Sutin found no evidence for specific steric effects in the reactions of the 5,6- and 3,4,7,8-methyl-substituted iron–phenanthroline complexes with $Fe(H_2O)_6{}^{2+}$. The absence of specific steric effects led Ford-Smith and Sutin to suggest that in the activated complex for electron transfer, $Fe(H_2O)_6{}^{2+}$ is located in the space between phenanthroline groups rather than at the periphery of a phenanthroline group.[4]

3. In other solution reactions where negative $\Delta H^{\ddagger}$ (or E_a) values have been observed, mechanisms have been invoked in which enthalpically favorable preequilibria exist.[18-21] For the reactions studied here, an equilibrium involving ion-pair formation between the reacting ions must exist. Electron transfer presumably follows ion-pair formation, *e.g.*

$$Fe(H_2O)_6{}^{2+} + Fe(phen)_3{}^{3+} \rightleftharpoons Fe(H_2O)_6{}^{2+},Fe(phen)_3{}^{3+} \quad (K) \qquad (4)$$

$$Fe(H_2O)_6{}^{2+},Fe(phen)_3{}^{3+} \rightarrow Fe(H_2O)_6{}^{3+} + Fe(phen)_3{}^{2+} \quad (k) \qquad (5)$$

For a reaction involving unlike-charged ions, direct evidence has been obtained that ion-pair formation precedes electron transfer.[22] Hemmes has calculated that ion pairing between large cations can be thermodynamically favorable.[23] From attempts to observe ion pairing between $Co(H_2O)_6{}^{2+}$ and $Ru(bipy)_3{}^{3+}$ spectrophotometrically, it was concluded that $K < 0.5$.[1]

Ion pairing preceding electron transfer could make a negative contribution to the observed enthalpy of activation.[24] In media of high ionic strength, electrostatic effects are small and expected to make a minor contribution.[25] However, nonelectrostatic effects may be important. Such effects might include charge fluctuation forces[26] or the effect of solvent structure which has been suggested to enhance ion pairing when both ions are large.[27]

The existence of important nonelectrostatic effects could also explain the poor agreement obtained using eq 3. Marcus has suggested that this result may be due to a failure of nonelectrostatic terms to cancel in the work done in bringing the reactants together.[28]

4. The reactions between $Fe(H_2O)_6{}^{2+}$ and the iron(III)-polypyridine complexes show strong specific anion effects.[4,29] Especially striking is the path for catalysis by SCN^- in 1.0 *M* $HClO_4$ at 25.0° in which $FeNCS^{2+}$ is formed quantitatively.[26] The specific anion effects for anions like SCN^- or $N_3{}^-$ may arise as a consequence of preequilibria involving complex ion formation, *e.g.*, $FeNCS^+$; however, with other anions, most notably $ClO_4{}^-$, energetically favorable ion pairing with the polypyridine complex may be an important part of the overall electron-transfer reaction.

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Registry No. $Fe(H_2O)_6{}^{2+}$, 15365-81-8; $Fe(terpy)_2{}^{3+}$, 47779-99-7; $Fe(bipy)_3{}^{3+}$, 13479-48-6.

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Contribution from the Department of Chemistry,
University of Guelph, Guelph, Ontario, N1G 2W1, Canada

Organoantimony Compounds. IV. Preparation, Characterization, and Vibrational Spectra of Trimethyl- and Triphenylantimony Diazides and Diisocyanates[1,2]

R. G. Goel* and D. R. Ridley

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In continuation of our investigation into the preparation, structure, and chemical properties of triorganoantimony(V) derivatives, we initiated a study[3] on triorganoantimony(V)

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