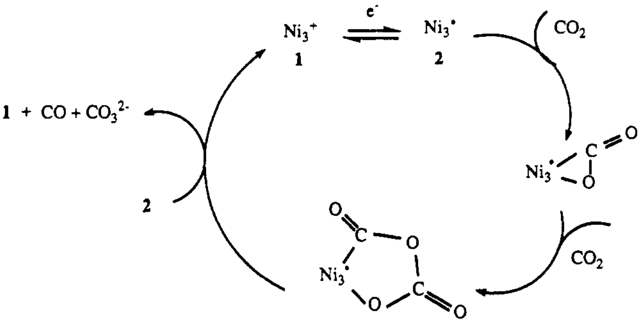
Scheme III



electrode, the heterogeneous electron transfer rate constant, k_e, for the reduction of 1 was found to be 0.023 cm s^{-1}. The homogeneous rate constant, k_{CO_2}, for the reaction between 2 and CO_2 was studied by cyclic voltammetry. When the scan rate was varied, the value of the steady-state limiting current was found (i.e. the region where catalysis, not diffusion, determines the current). The value for k_{CO_2} so determined was 0.011 M^{-1} s^{-1}.[34]

Further experiments were performed to obtain the turnover number of the catalyst. Using a dilute sample of 1 (2 μM in CH_3CN with TBAP as supporting electrolyte), seven turnovers were seen within 3 h using a Pt-gauze electrode. In addition, a steady-state current was reached in the current vs time plot, indicative of a catalytic reaction.[34]

The reduction of CO_2 by the electrocatalyst 1 occurs at a potential of −1.09 V vs Ag/AgCl, which is over 1 V less than the value of $E°(CO_2/CO_2^{\bullet-}) = -2.16$ V vs Ag/AgCl reported by Saveant and Amatore.[23] This result is clearly inconsistent with direct "outer sphere" reduction of CO_2 to $CO_2^{\bullet-}$. It is, however, consistent with the formation of an adduct of CO_2 with the reduced form of the trinickel cluster 2. A second CO_2 molecule may then be inserted into the adduct in a "head-to-tail" fashion. Subsequent electron transfer from a second equivalent of 2, followed by asymmetric disproportionation of the head-to-tail dimer of CO_2, results in CO and CO_3^{2-}. The proposed mechanism is outlined in Scheme III.

THF solutions of 2 react with carbon dioxide in the presence of $NaPF_6$ to re-form complex 1. The reduction of CO_2 to CO was verified by gas chromatography. The solvent was removed under vacuum, and the resulting solid was washed with CH_2Cl_2 to obtain a purple solution of pure complex 1 and a white solid. Infrared data for the white solid showed a strong band at 1450 cm^{-1} and a weaker band at 1632 cm^{-1}, consistent with the presence of carbonate. Indeed, the carbonate was precipitated with barium triflate. Formate was not observed in scrupulously dried solvents. However, when CO_2 entrained in D_2O vapor is passed through a THF solution of 2, DCO_2^- becomes the primary CO_2 product by IR spectroscopy. Under these conditions, only traces of CO are observed by GC. The results suggest that cluster-bound $CO_2^{\bullet-}$ may be intercepted by protonation to ultimately produce formate. Isotope labeling studies with $^{13}CO_2$ followed by IR analysis confirmed that CO and CO_3^{2-} were derived only from CO_2. Our results are consistent with an "inner-sphere" reduction of CO_2 by 2 to produce cluster-bound $CO_2^{\bullet-}$. The $CO_2^{\bullet-}$ undergoes head-to-tail condensation with a second CO_2 molecule and reduction by 2 to afford the reductive disproportionation products CO and CO_3^{2-}. Oxalate was not observed under any conditions employed in these studies.

Our continued studies are focusing on electrocatalytic systems related to 1 to effect carbon–carbon bond-forming reactions between $CO_2^{\bullet-}$ groups.

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Reaction of $[Fe\{C(=O)N^iPr_2\}(CO)_4]^-$ with Trifluoroacetic Anhydride: Molecular Structure of $[Fe(CF_3)\{\eta^2\text{-}C(=O)N^iPr_2\}(CO)_2(PPh_3)]$

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Summary: The reactions of the (carbamoyl)tetracarbonylferrate $[Fe\{C(=O)N^iPr_2\}(CO)_4]^-$ with $(CF_3CO)_2O$ and PPh_3 provide the structurally characterized trifluoromethyl-carbamoyl complex $[Fe(CF_3)\{\eta^2\text{-}C(=O)N^iPr_2\}(CO)_2(PPh_3)]$. A similar reaction with Ph_3SnCl and PPh_3 provides the stannyl derivative $[Fe(SnPh_3)\{\eta^2\text{-}C(=O)N^iPr_2\}(CO)_2(PPh_3)]$.

We have recently reported that the metal carbonyls $[M(CO)_n]$ ($n = 6$, M = Cr, Mo, W; $n = 5$, M = Fe) react successively with lithium diisopropylamide, trifluoroacetic anhydride, and triphenylphosphine to provide the aminomethylidyne complexes $[M(\equiv CN^iPr_2)(O_2CCF_3)(CO)_{n-3}(PPh_3)]$.[1] The formulation of these compounds as alkylidyne complexes was based on spectroscopic and elemental microanalytical data, and we have confirmed this for the chromium complex by its reaction with potassium hydrotris(pyrazol-1-yl)borate to provide $[Cr(\equiv CN^iPr_2)(CO)_2\{\kappa^3\text{-}HB(pz)_3\}]$ (pz = pyrazol-1-yl), which we have structurally characterized.[2] While subsequent reactivity studies on the group 6 derivatives have provided results

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(1) Anderson, S.; Hill, A. F. *J. Organomet. Chem.* **1990**, *394*, C24.
(2) Hogarth, G. Unpublished results.

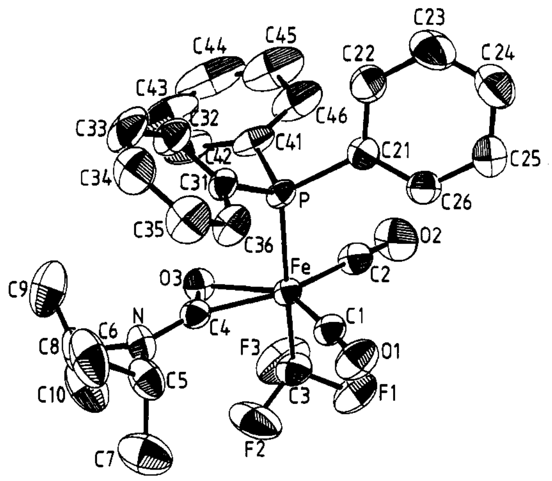

Figure 1. Molecular geometry of $[\mathrm{Fe(CF_3)}\{\eta^2\text{-}\mathrm{C({=}O)N^iPr_2}\}\mathrm{(CO)_2(PPh_3)}]$. Average bond distances (Å) and angles (deg): Fe–P, 2.290 (1); Fe–C(1), 1.744 (3); Fe–C(2), 1.803 (3); Fe–C(3), 1.986 (3); Fe–C(4), 1.875 (2); Fe–O(3), 2.030 (2); C(4)–O(3), 1.251 (3); Fe–C(4)–O(3), 78.2 (1); O(3)–C(4)–N, 127.9 (2).

typical of this class of compounds, the reactivity of "$[\mathrm{Fe({\equiv}CN^iPr_2)(O_2CCF_3)(CO)_2(PPh_3)}]$" appeared anomalous. Thus, for example, the reaction of this compound with $\mathrm{HBF_4}$ provided the *tricarbonyl*-carbamoyl complex $[\mathrm{Fe}\{\eta^2\text{-}\mathrm{C({=}O)N^iPr_2}\}\mathrm{(CO)_3(PPh_3)}]^+$ in high yield. We were therefore eager to unequivocally confirm the alkylidyne formulation for this complex. A single-crystal X-ray diffraction analysis of the compound shows, however, that for iron the reaction takes a completely different course to provide instead the trifluoromethyl compound $[\mathrm{Fe(CF_3)}\{\eta^2\text{-}\mathrm{C({=}O)N^iPr_2}\}\mathrm{(CO)_2(PPh_3)}]$. The results of the structure determination are summarized in Figure 1. Both the alkylidyne and carbamoyl formulations are consistent with the initially reported spectroscopic and elemental microanalytical data.[3,4] Subsequent consideration of ^{19}F NMR and solid-state IR data, however, also reveals features consistent with the trifluoromethyl–carbamoyl structure,[3] most notably the observation of coupling to phosphorus-31 (29.3 Hz). The solid-state IR spectrum is complicated in the region typical of M–$\mathrm{CF_3}$. However, the peaks tentatively assigned to ν(CF) appear at 1050 vs and 990 vs cm^{-1} and are to lower energy of those observed for $[\mathrm{Cr({\equiv}CN^iPr_2)(O_2CCF_3)(CO)_3(PPh_3)}]$ at 1124 and 1112 cm^{-1}.

The outcome of the reaction is, in retrospect, easily understood (Scheme I), when the ambident nature of the $[\mathrm{Fe}\{\eta^1\text{-}\mathrm{C({=}O)N^iPr_2}\}\mathrm{(CO)_4}]^-$ nucleophile is appreciated. Electrophilic attack by the trifluoroacetylating agent may occur either at the carbamoyl oxygen (as in the case of the group 6 complexes) or at the iron center. In the latter case the trifluoroacetyl complex $[\mathrm{Fe}\{\eta^1\text{-}\mathrm{C({=}O)N^iPr_2}\}\{\eta^1\text{-}\mathrm{C({=}O)CF_3}\}\mathrm{(CO)_4}]$ would be formed initially, and while subsequent loss of carbonyl ligands, migration of the $\mathrm{CF_3}$ group to iron, and carbonyl substitution by triphenylphosphine are all quite reasonable processes, the very mild conditions (0–10 °C) under which these steps occur are worthy of note. The decarbonylation of trifluoroacetyl ligands to provide trifluoromethyl ligands is normally irreversible and is central to the synthesis of metal–$\mathrm{CF_3}$ complexes.[5] However, these reactions typically require photolytic or thermolytic initiation; e.g., the complex $[\mathrm{Fe}\{\eta^1\text{-}\mathrm{C({=}O)CF_3}\}\mathrm{(CO)_2}(\eta\text{-}\mathrm{C_5H_5})]$ requires irradiation for 16 h to effect low (6%) conversion to $[\mathrm{Fe(CF_3)(CO)_2}(\eta\text{-}\mathrm{C_5H_5})]$.[6]

The formation of $[\mathrm{Fe}\{\eta^2\text{-}\mathrm{C({=}O)N^iPr_2}\}\mathrm{(CO)_3(PPh_3)}]\mathrm{BF_4}$[7] from $[\mathrm{Fe(CF_3)}\{\eta^2\text{-}\mathrm{C({=}O)N^iPr_2}\}\mathrm{(CO)_2(PPh_3)}]$ and aqueous $\mathrm{HBF_4}$ provides a further example of the electrophilic conversion of a trifluoromethyl ligand into a (hydrolytically sensitive) difluorocarbene (i.e., $[\mathrm{Fe}\{\eta^2\text{-}\mathrm{C({=}O)N^iPr_2}\}\mathrm{({=}CF_2)(CO)_2(PPh_3)}]^+$), a reaction class which has been so important in the renaissance of (perfluoroalkyl)metal chemistry.[8,9]

The reactions of the acyl anions $[\mathrm{Fe\{C({=}O)R\}(CO)_4}]^-$ toward alkylating agents have been extensively studied, and it has been shown by Semmelhack and co-workers that hard electrophiles attack the acyl oxygen while soft electrophiles attack the metal center.[10] Fischer's report that $[\mathrm{Fe}\{\eta^1\text{-}\mathrm{C({=}O)N^iPr_2}\}\mathrm{(CO)_4}]^-$ with the harder electrophile $[\mathrm{Et_3O}]^+\mathrm{BF_4}^-$ provides the carbene complex $[\mathrm{Fe\{{=}C(N^iPr_2)OEt\}(CO)_4}]$,[11] and our own observations that

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[3] Data for $[\mathrm{Fe(CF_3)}\{\eta^2\text{-}\mathrm{C({=}O)N^iPr_2}\}\mathrm{(CO)_2(PPh_3)}]$: IR ($\mathrm{CH_2Cl_2}$) 2021 vs, 1951 vs [$\nu$(CO)]; FAB-MS[4] m/z (543 [M − CO]$^+$), 515 ([M − 2CO]), 465 ([M − 2CO − $\mathrm{CF_3}$]$^+$), 337 ([$\mathrm{FePPh_3}$]$^+$); ^{1}H NMR ($\mathrm{CDCl_3}$, 25 °C) δ 0.53, 1.04, 1.16, 1.29 [d × 4, 12 H, $\mathrm{CHC}H_3$, J(HH) 6.7 Hz], 3.28, 4.79 [h × 2, 2 H, $\mathrm{C}H\mathrm{CH_3}$, J(HH) 6.7 Hz], 7.18–7.41 [m, 15 H, $\mathrm{C_6}H_5$]; $^{13}\mathrm{C}\{^1\mathrm{H}\}$ NMR ($\mathrm{CDCl_3}$, 25 °C) δ 218.3, 211.6, 197.1 [d (br) × 3, Fe(CO)$_2$ and C(O)$\mathrm{N^iPr_2}$, J(PC) not resolved], 133.6, 130.3, 128.5 [PC_6H$_5$], 54.7, 48.5 [s × 2, CH(CH$_3$)$_2$], 21.2, 20.5, 20.2, 19.9 [s × 4, CH(CH$_3$)$_2$], eight-line resonance due to $\mathrm{CF_3}$ not detected; ^{31}P NMR ($\mathrm{CDCl_3}$, 25 °C) δ 49.6 [q, J(PF) 27.2 Hz], ^{19}F NMR ($\mathrm{CDCl_3}$, 25 °C) δ −7.15 ppm [d, ^3J(PF) 29.3 Hz]. For a discussion of ^{19}F NMR data for trifluoromethyl complexes see ref 8b. Anal. Calcd for $\mathrm{C_{28}H_{29}F_3FeO_3PN}$: C, 58.9; H, 5.1; N, 2.5. Found: C, 58.8; H, 5.2; N, 2.4. Crystallography: yellow prism; diffracted intensities (ω–2θ scans) at 302 K in the range 2° < 2θ < 25°, on a CAD4 four-circle diffractometer. Of 8451 unique reflections, 6248 having $I > 3\sigma(I)$ were used in the structure solution and refinement. Corrections were applied for Lorentz, polarization, and X-ray absorption effects, the last by an empirical method based on azimuthal scan data. M_r = 526.38, crystal class monoclinic, a = 11.520 (6) Å, b = 28.659 (6) Å, c = 17.157 (2) Å, β = 99.79 (2)°, V = 5581.8 (30) Å^3, Z = 8, D_c = 1.25 g cm^{-3}, F(000) = 2480, space group $P2_1/c$ (No. 14), Mo Kα X-radiation (λ = 0.71069 Å, Zr filter), μ(Mo Kα) = 6.53 cm^{-1}. The structure was solved, and all non-hydrogen atoms located, by direct methods and difference Fourier maps which indicated that there were two virtually identical molecules in the asymmetric unit. Phenyl hydrogen atoms were included in calculated positions (C–H = 0.95 Å) with fixed isotropic thermal parameters. All non-hydrogen atoms were refined anisotropically (blocked-matrix least squares; 670 parameters), the refinement converging at R = 0.038 (R_w = 0.042) with a weighting scheme of the form $w^{-1} \propto [\sigma^2(F) + 0.000853|F|^2]$. Residual electron-density maxima were between −0.33 and +0.39 e Å^{-3}. Atomic positions, bond lengths and angles, thermal parameters, and full procedural details have been deposited as supplementary material. These data and structure factor values are available from G.R.C.

[4] The absence of a molecular ion and presence of an [M − 2CO]$^+$ ion in the FAB mass spectrum was not considered unusual for the hypothetical carbyne complex or iron carbonyl compounds in general.

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[6] King, R. B.; Bisnette, M. B. *J. Organomet. Chem.* **1964**, *2*, 15. See also ref 9 for an improved procedure, which nevertheless requires prolonged photolysis.

[7] Data for $[\mathrm{Fe}\{\eta^2\text{-}\mathrm{C({=}O)N^iPr_2}\}\mathrm{(CO)_3(PPh_3)}]\mathrm{BF_4}$: IR ($\mathrm{CH_2Cl_2}$) 2102 vs, 2058 vs, 2023 [ν(CO)], 1650 m [acyl]; FAB-MS m/z 530 ([M]$^+$), 502 ([M − CO]), 474 ([M − 2CO]), 446 ([M − 3CO]), 337 ([$\mathrm{FePPh_3}$]$^+$); ^{1}H NMR ($\mathrm{CDCl_3}$, 25 °C) δ 0.86, 1.24, 1.35, 1.44 [s (br) × 4, 12 H, CHMe_2], 3.64, 4.68 [s (br) × 2, 2 H, CHMe$_2$], 7.30, 7.53 [m (br) × 2, $\mathrm{PPh_3}$]; $^{13}\mathrm{C}\{^1\mathrm{H}\}$ NMR ($\mathrm{CDCl_3}$, 25 °C) δ 206.6 [d, Fe(CO), J(PC) 31.4 Hz], 202.2 [d, Fe(CO), J(PC) 26.5 Hz], 197.1 [d, Fe(CO), J(PC) 51.0 Hz], 185.9 [d, OCN^{i}Pr$_2$, J(PC) 18.8 Hz], 133.1 [d, $\mathrm{C^{3,5}(C_6H_5)}$, J(PC) 8.4 Hz], 132.6 [s, $\mathrm{C^4(C_6H_5)}$], 129.6 [d, $\mathrm{C^{2,6}(C_6H_5)}$, 9.8 Hz], 127.5 [d, $\mathrm{C^1(C_6H_5)}$, J(PC) 48.2 Hz], 56.7, 50.3 [s × 2, NCHMe$_2$], 21.8, 19.7, 19.4, 19.1 [s × 4, NCH(CH$_3$)$_2$]; ^{31}P NMR ($\mathrm{CDCl_3}$, 25 °C) δ 22.4 [s]. Anal. Calcd for $\mathrm{C_{28}H_{29}BF_4FeNO_4P}$: C, 54.5; H, 4.7; N, 2.3. Found: C, 54.1; H, 4.7; N, 2.2.

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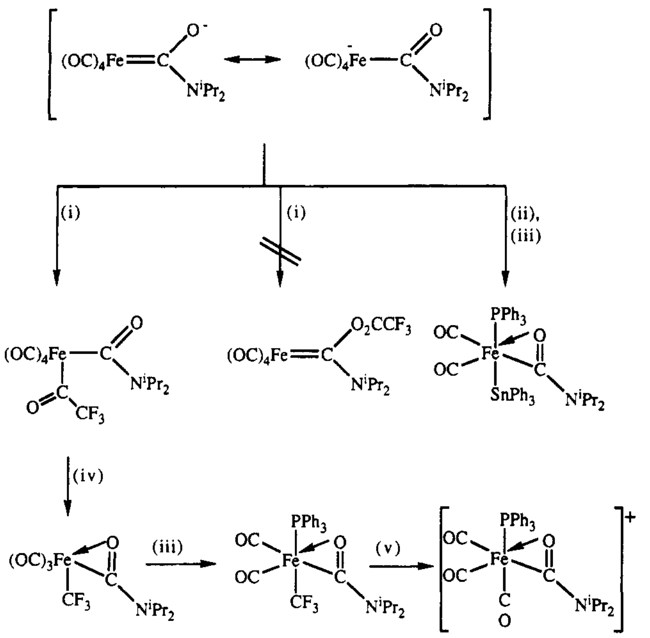
Scheme I[a]



[a] Legend: (i) $(CF_3CO)_2O$; (ii) Ph_3SnCl; (iii) PPh_3; (iv) –CO; (v) HBF_4/H_2O, –3HF.

treating the carbamoylate with the soft electrophile Ph_3SnCl followed by triphenylphosphine provides the stannyl complex $[Fe(SnPh_3)\{\eta^2\text{-}C(=O)N^iPr_2\}(CO)_2(PPh_3)]$ (Sn trans to P)[12] further support this. The divergence in reactivity between the carbamoylates $[Cr\{C(=O)N^iPr_2\}(CO)_5]^-$ and $[Fe\{C(=O)N^iPr_2\}(CO)_4]^-$ is presumably due in part to the increased accessibility of the 5-coordinate iron center toward electrophiles relative to the 6-coordinate chromium complex.

We are currently investigating the versatility of the conveniently accessible (carbamoyl)carbonylferrate $[Fe\{\eta^1\text{-}C(=O)N^iPr_2\}(CO)_4]^-$ in reactions with other electrophiles which provide an expedient entry into the carbamoyl chemistry of iron(II).

Supplementary Material Available: Tables of crystal data and data collection and refinement details, atomic positional and thermal parameters, and bond lengths and angles for $[Fe(CF_3)\{\eta^2\text{-}C(=O)N^iPr_2\}(CO)_2(PPh_3)]$ (8 pages). Ordering information is given on any current masthead page.

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[12] Data for $[Fe(SnPh_3)\{\eta^2\text{-}C(=O)N^iPr_2\}(CO)_2(PPh_3)]$: IR ($CH_2Cl_2$) 1979 s, 1908 vs [$\nu$(CO)], 1585 m (acyl); FAB-MS *m/z* 851 ($[M]^+$), 795 ($[M - 2CO]^+$), 720 ($[M - 2CO - C_6H_5]^+$), 668 ($[Fe(PPh_3)(SnPh_3)]$), 563 ($[M - PPh_3 - CO]^+$), 535 ($[M - PPh_3 - 2CO]^+$), 351 ($[SnPh_3]^+$), 318 ($[FePPh_3]^+$); $^{13}C\{^1H\}$ NMR ($CDCl_3$, 25 °C) δ 218.0 [d (br), Fe(CO)], 196.7 [d, OCN^iPr_2, 12.9 Hz], 145.7 [d, $C^1(SnC_6H_5)$, *J*(PC) 5.1 Hz, *J*(SnC) ca. 258 Hz, $J(^{119}SnC/^{117}SnC)$ not resolved], 137.2 [$C^{2,6}(SnC_6H_5)$, *J*(SnC) ca. 32.9 Hz], 133.8 [d, $C^1(PC_6H_5)$, *J*(PC) 35.0 Hz], 133.4 [d, $C^{2,6}(PC_6H_5)$, *J*(PC) 10.5], 129.7, 127.1 [s × 2, $C^4(PC_6H_5)$ and $C^4(SnC_6H_5)$], 128.2 [d, $C^{3,5}(PC_6H_5)$, *J*(PC) 9.1], 127.4 [$C^{3,5}(SnC_6H_5)$, *J*(SnC) ca. 37.5 Hz], 54.4, 47.5 [s × 2, $NCHMe_2$], 20.7, 20.1 [br, $NCH(CH_3)_2$]; ^{31}P NMR ($CDCl_3$, 25 °C) δ 53.8 [$J\ (P^{11x,y}Sn)$ 637, 608 Hz]. Anal. Calcd for $C_{45}H_{44}FeNO_3PSn\cdot CH_2Cl_2$: C, 59.0; H, 4.9; N, 1.7. Found: C, 59.8; H, 5.0; N, 1.4.

Water-Soluble Organometallic Compounds. 2. Catalytic Hydrogenation of Aldehydes and Olefins by New Water-Soluble 1,3,5-Triaza-7-phosphaadamantane Complexes of Ruthenium and Rhodium

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Summary: Water-soluble phosphine complexes of ruthenium(II) and rhodium(I) have been prepared by the reduction of $RuCl_3$ and $RhCl_3$ in ethanol in the presence of the air-stable phosphine 1,3,5-triaza-7-phosphaadamantane (**1**). The complex $RuCl_2(PTA)_4$ (**2a**) and its protonated analog $RuCl_2(PTA)_4\cdot 2HCl$ (**2b**) have been characterized by X-ray crystallography. Similarly, the *cis*-bis(phosphine) complex $RhCl(PTA)_2\cdot 2HCl$, derived from the protonation of $RhCl(PTA)_3$ by HCl, has been characterized by X-ray crystallography. The water-soluble ruthenium phosphine derivative **2a** is catalytically quite active for the conversion of unsaturated aldehydes to unsaturated alcohols using a biphasic aqueous/organic medium with formate as the source of hydrogen. By way of contrast, under similar conditions $RhCl(PTA)_3$ is a very active catalyst for olefin hydrogenation.

Much attention has been focused recently on reactions catalyzed by organometallic reagents in aqueous or biphasic aqueous–organic media.[1] Despite the impressive progress in the synthesis of organometallic complexes

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