metal organometallic complexes.

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Registry No. $[(CO)_4V(\mu\text{-}P(CH_3)_2)]_2$, 68365-83-3; $[(CO)_4Cr(\mu\text{-}P(CH_3)_2)]_2$, 19599-74-7; $[(CO)_4Mn(\mu\text{-}P(CH_3)_2)]_2$, 19599-80-5; $[(CO)_4Cr(\mu\text{-}P(C_2H_5)_2)]_2$, 95527-25-6; $[(CO)_4Mo(\mu\text{-}P(C_2H_5)_2)]_2$, 34482-49-0; $[(CO)_4W(\mu\text{-}P(C_2H_5)_2)]_2$, 42830-84-2; $[(CO)_4Mn(\mu\text{-}P(C_2H_5)_2)]_2$, 110551-58-1.

Carbon-13 Monoxide Enrichment of Metal Carbonyl Compounds Employing KH and $NaBH_4$ as Exchange Promoters

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Carbon-13 monoxide enrichment of the metal carbonyls $Fe(CO)_5$, $M_3(CO)_{12}$ (M = Fe, Ru, Os), $M_2(CO)_{10}$ (M = Mn, Re), and $M(CO)_6$ (M = Cr, Mo, W) at pressures up to 1 atm of ^{13}CO and 25 °C has been examined by using KH and $NaBH_4$ as exchange promoters. With the exception of $Ru_3(CO)_{12}$, it was found that $NaBH_4$ is more effective or at least equal to KH as an exchange promoter and appears to be more useful due to its ease of handling and greater solubility in the solvent tetrahydrofuran.

Introduction

In conjunction with earlier work on the catalysis of the water-gas shift reaction by $[HRu_3(CO)_{11}]^-$[1] we observed that the reaction of KH with triruthenium dodecacarbonyl, $Ru_3(CO)_{12}$, occurred according to the following equilibrium[2] (reaction 1). When a large excess of CO gas was present

$$KH + Ru_3(CO)_{12} \rightleftharpoons K[HRu_3(CO)_{11}] + CO \qquad (1)$$

in the system, the forward reaction of reaction 1 was inhibited. When that gas was ^{13}CO, statistical exchange of ^{13}CO–^{12}CO occurred between gas and condensed phases but did not occur when KH was absent.[1a,3] The anion $[HRu_3(CO)_{11}]^-$ readily exchanges bound CO with free ^{13}CO.[1a,5,6] Carbon monoxide exchange is also promoted by $NaBH_4$.[3] More recently,[4] MeO^- was shown to promote ^{13}CO exchange with $Ru_3(CO)_{12}$.

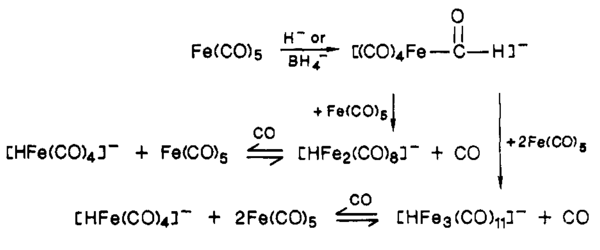
Since utilization of the carbon-13 nuclide as a probe for the characterization and study of organometallic compounds is widespread,[7] a convenient general procedure for enriching metal carbonyls is desirable. Although a variety of methods for ^{13}CO enrichment are known,[4,8] the present report provides a general route that involves mild conditions for enrichment of a variety of carbonyl compounds.

Table I. Carbon-13 Monoxide Enrichment of Metal Carbonyls under 1 atm[a] of ^{13}CO and 0.02 mol % of $NaBH_4$[a] as an Exchange Promoter at 25 °C

expt	metal carbonyl	expt enrichment, %	statistical enrichment, %	time, h
1	$Fe(CO)_5$	69	70	24
2	$Ru_3(CO)_{12}$[b]	54	59	46
3	$Ru_3(CO)_{12}$	16	52	60
4	$Os_3(CO)_{12}$	60	66	24
5	$Fe_3(CO)_{12}$[c]	9	10	8
6	$Mn_2(CO)_{10}$	61	69	24
8	$Re_2(CO)_{10}$	1.1	62	48
9	$Cr(CO)_6$	52	59	24
10	$Mo(CO)_6$	38	50	48
11	$W(CO)_6$	33	55	48

[a] Except where noted. [b] KH used as the exchange promoter. [c] 120 Torr of ^{13}CO.

Scheme I



Results and Discussion

Carbon-13 monoxide enrichment of a number of metal carbonyls at pressures up to 1 atm of ^{13}CO and 25 °C has been examined by using KH and $NaBH_4$ as exchange promoters. With the exception of $Ru_3(CO)_{12}$, it was found that $NaBH_4$ is more effective or at least equal to KH as an exchange promoter and appears to be more useful due to its ease of handling and greater solubility in the solvent THF. Table I provides a summary of the most favorable results obtained. Detailed consideration of the systems studied is provided below.

$Fe(CO)_5$. No carbon monoxide exchange between ^{13}CO and $Fe(CO)_5$ was observed over a period of 24 h at 25 °C and 1 atm of ^{13}CO pressure. This is consistent with earlier results.[9] However, when a small amount of KH or $NaBH_4$

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[2] We have shown that the equilibrium is completely reversible, that is, KH and $Ru_3(CO)_{12}$ can be isolated from a THF solution of $K[HRu_3(CO)_{11}]$ that has an atmosphere of CO gas above it (see Experimental Section).

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(molar ratio of promoter to metal cluster = 0.02/1) was added to the system, $Fe(CO)_5$ was rapidly enriched with ^{13}CO at 25 °C (experiment 1, Table I). Pathways by which H^- and BH_4^- are able to induce ^{13}CO exchange are shown below (Scheme I).

Each step in the above scheme is a known reaction.[10-12] Initial reaction of $Fe(CO)_5$ with H^- and also BH_4^- gives the formyl anion[10] $[(CO)_4FeCHO]^-$, which reacts with additional $Fe(CO)_5$ in a 1:1 ratio to give $[HFe_2(CO)_8]^-$ and in a 1:2 ratio to give $[HFe_3(CO)_{11}]^-$. Both $K[HFe(CO)_4]$[3] and $Na[HFe(CO)_4]$[13] readily exchange their carbon monoxide with free ^{13}CO. Thus, exchange between bound carbon monoxide and free ^{13}CO occurs in $[HFe(CO)_4]^-$, which is in equilibrium with $Fe(CO)_5$.

$Ru_3(CO)_{12}$, $Os_3(CO)_{12}$, and $Fe_3(CO)_{12}$. Reactions of KH with the trinuclear dodecacarbonyls of the iron triad occur according to eq 2. Relative rates of reaction at 25 °C were found to be $Fe_3(CO)_{12}/Ru_3(CO)_{12}/Os_3(CO)_{12}$ = 24/15/1. Earlier studies have shown that the reaction between $M_3(CO)_{12}$ and $NaBH_4$ in THF also produces $[HM_3(CO)_{11}]^-$.[14-16] We found that while molar ratios of promoter (KH or $NaBH_4$) to cluster as small as 0.02/1 were capable of inducing ^{13}CO enrichment in $Fe_3(CO)_{12}$, $Ru_3(CO)_{12}$, and $Os_3(CO)_{12}$, no apparent exchange occurred under similar conditions of temperature and time when the promoter was absent from the system. Exchange between ^{13}CO and metal-bound carbon monoxide has been shown to occur for the anions $[HRu_3(CO)_{11}]^-$ and $[HOs_3(CO)_{11}]^-$[6] and is probably true for the anion $[HFe_3(CO)_{11}]^-$, as well.

$$KH + M_3(CO)_{12} \xrightleftharpoons{THF} K[HM_3(CO)_{11}] + CO \qquad (2)$$

$$M = Fe, Ru, Os$$

When KH was used to promote ^{13}CO–^{12}CO exchange in $Ru_3(CO)_{12}$ (experiment 2), near statistical exchange occurred within 48 h at 25 °C, in accord with reaction 1. However, when $NaBH_4$ was used as an exchange promoter (experiment 3), after 60 h at 25 °C only 16% enrichment was observed while statistical exchange would have produced 52% enrichment. This is the only case we observed in which KH was more effective than $NaBH_4$ as an exchange promoter.

In the case of $Os_3(CO)_{12}$, detectable enrichment using KH as an exchange promoter did not occur at 25 °C. At 60 °C, however, significant although not statistical exchange occurred within 48 h. On the other hand, use of $NaBH_4$ as an exchange promoter (experiment 4) resulted in near statistical exchange *within 24 h at 25 °C.*

Under the conditions used to enrich $Ru_3(CO)_{12}$ and $Os_3(CO)_{12}$, $Fe_3(CO)_{12}$ is transformed to $Fe(CO)_5$ (reaction 3). The reaction appears to be catalyzed by H^- and also BH_4^-. In the absence of these anions, under otherwise identical conditions, there was less than 1% fragmentation of $Fe_3(CO)_{12}$ to $Fe(CO)_5$. Fragmentation of $Fe_3(CO)_{12}$ to $Fe(CO)_5$ in the presence of H^- or BH_4^- is probably due to the formation of the intermediate $[HFe_3(CO)_{11}]^-$ ion which has been shown to fragment readily in the presence of CO.[12] Statistically enriched $Fe(CO)_5$, from $Fe_3(CO)_{12}$, was obtained under ^{13}CO at 1 atm of pressure in the presence of either KH or $NaBH_4$. On the other hand, statistically enriched $Fe_3(CO)_{12}$ was obtained in the presence H^- or BH_4^- at a reduced ^{13}CO pressure[17] (120 Torr, (experiment 5)) in about 8 h, although some degradation to enriched $Fe(CO)_5$ also occurred. Sodium borohydride appears to be slightly more effective than KH in its ability to promote enrichment of $Fe_3(CO)_{12}$.

$$Fe_3(CO)_{12} + 3CO \xrightarrow[H^- \text{ or } BH_4^-]{THF} 3Fe(CO)_5 \qquad (3)$$

$Mn_2(CO)_{10}$ and $Re_2(CO)_{10}$. Reaction between $Mn_2(CO)_{10}$ and KH has been observed[18] (eq 4). An analogous reaction occurs between $NaBH_4$ and $Mn_2(CO)_{10}$.[14b] Both KH and $NaBH_4$ function as exchange promoters in the ^{13}CO enrichment of $Mn_2(CO)_{10}$. Results with $NaBH_4$ are reported as experiment 6, in Table I. Since detectable enrichment does not occur in the absence of exchange promoters under otherwise identical conditions, enrichment of $Mn_2(CO)_{10}$ might occur through the interaction of $K[Mn(CO)_5]$ with $Mn_2(CO)_{10}$.

$$Mn_2(CO)_{10} + 2KH \xrightarrow[25\ °C]{THF} 2K[Mn(CO)_5] + H_2 \qquad (4)$$

At 25 °C, $Re_2(CO)_{10}$ does not react significantly with either KH or $NaBH_4$ under the conditions used to enrich $Mn_2(CO)_{10}$ (experiment 8). Thus, enriched $Re_2(CO)_{10}$ could not be obtained by using KH or $NaBH_4$ as exchange promoters.

$Cr(CO)_6$, $Mo(CO)_6$, and $W(CO)_6$. Sodium borohydride proved to be superior to KH as an exchange promoter for ^{13}CO enrichment of $Cr(CO)_6$, $Mo(CO)_6$, and $W(CO)_6$ (experiments 9–11). No enrichment was observed in the absence of promoters. Potassium hydride was found to be adequate as an exchange promoter for ^{13}CO enrichment of $Cr(CO)_6$, $Mo(CO)_6$, and $W(CO)_6$ but required higher temperature (60 °C) for statistical enrichment in an equivalent period of time. Hydride donors react with $M(CO)_6$ (M = Cr, Mo, W) to form hydrido carbonyl anions that have been shown[19] to exchange with ^{13}CO. It is likely that such anions are intermediates in the exchange process in the presence of an exchange promoter.

Experimental Section

Materials. $Ru_3(CO)_{12}$, $Os_3(CO)_{12}$, $Fe_3(CO)_{12}$, $Mn_2(CO)_{10}$, $Re_2(CO)_{10}$, $Cr(CO)_6$, $Mo(CO)_6$, and $W(CO)_6$ were used as obtained from Strem Chemical Co. $Fe(CO)_5$ (Alpha-Ventron) was purified prior to use by trap to trap distillation on the vacuum line. KH (Aldrich) in a 50% mineral oil suspension was washed repeatedly with hexane until a free-flowing, oil-free powder resulted. The KH was stored under N_2 in a sealed vessel in a nitrogen-filled drybox. ^{13}CO (95% enriched) was obtained from the Monsanto

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[17] Pressures of ^{13}CO higher than 120 Torr produced much lower yields of enriched $Fe_3(CO)_{12}$ and higher yields of enriched $Fe(CO)_5$. At pressures above 300 Torr, there was quantitative production of enriched $Fe(CO)_5$ (90% yield). At pressures below 120 Torr of ^{13}CO the enrichment of $Fe_3(CO)_{12}$ was less than 10%.

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Chemical Co. Mound Laboratory. Tetrahydrofuran (THF) was distilled from and stored over sodium benzophenone ketyl. $NaBH_4$ (MCB) was used as received and stored in a dessicator. $K[HRu_3(CO)_{11}]$[2b] was prepared by using a previously described procedure.

Apparatus. All manipulations were carried out on a standard high-vacuum line, using standard Schlenk methods, or in a glovebox under an atmosphere of dry, pure N_2. Analysis of all gas mixtures were carried out on an AEI MS-10 mass spectrometer. Infrared spectra were recorded on a Perkin-Elmer 457 spectrophotometer or a Mattson Instruments FTIR spectrophotometer using either matched 0.1-mm KBr cells (liquid) or a gaseous KBr cell with a 10-cm path length. Carbon-13 NMR spectra[20] were obtained on Bruker-300 (75.43 MHz), Bruker-250 (62.90 MHz), and Bruker-80 (20.11 MHz) NMR spectrometers operating in the FT mode.

Enrichments Promoted by $NaBH_4$. Enrichment of $Fe(CO)_5$. In air, a 1.0-mg quantity of $NaBH_4$ was placed in a 140-mL reaction vessel containing a Teflon-covered magnetic stirring bar. THF, 5.0 mL, was distilled into the reaction vessel at −78 °C, followed by distillation of 0.38 mmol of $Fe(CO)_5$ at −196 °C. Approximetely 1 atm (5.93 mmol) of ^{13}CO was expanded over the reaction solution at −78 °C. The reaction vessel was then allowed to warm to ambient temperature and was covered with aluminum foil to exclude light, and the solution was stirred for 20 h. Very brief visual inspection of the reaction solution from time to time indicated that its initially yellow color was maintained throughout the course of the reaction. The gas in the reaction vessel was trapped and measured with the use of a Toepler pump. Mass spectral analysis of the $^{13}CO-^{12}CO$ gas mixture revealed that statistical exchange between $Fe(CO)_5$ and free ^{13}CO had occurred. The $Fe(CO)_5$ was recovered in greater than 90% yield by trap to trap distillation (−15, −45, −78 °C). The infrared spectrum of recovered $Fe(CO)_5$ also indicated that enrichment had occurred: ν(CO) 2033 (m), 2015 (s), 1983 (m), 1978 (m), 1973 (m), 1969 (m) cm^{-1}. In the absence of $NaBH_4$ no measurable (mass and IR spectra) $^{13}CO-^{12}CO$ exchange occurs under identical conditions of time and temperature.

Enrichment and Isolation of $Ru_3(CO)_{12}$, $Os_3(CO)_{12}$, and Group VI (6[22]) and Group VII (7[22]) Carbonyls. In air, $NaBH_4$ (1-2 mg) was placed in a 140-mL reaction vessel containing a Teflon-covered magnetic stirring bar and the solid metal carbonyl. Between 0.1 and 1.0 mmol of metal carbonyl was employed. THF was distilled into the reaction vessel at −78 °C, and the solution was maintained at this temperature while 1 atm (~6 mmol) of ^{13}CO (95%) was expanded over the solution. The solution was allowed to warm to 25 °C in the absence of light and was stirred continuously. Mass spectral analyses of the gases over the solution were performed from time to time to determine the extent of isotopic exchange. The extent of enrichment is given in Table I. After gas sampling had indicated statistical or significant $^{13}CO-^{12}CO$ exchange had occurred, the carbon monoxide was removed from the vessel, measured, and analyzed. $Ru_3(CO)_{12}$, $Os_3(CO)_{12}$, $Cr(CO)_6$, $Mo(CO)_6$, and $W(CO)_6$ are only slightly soluble in THF so that separation from the THF-soluble hydrido metal carbonyl anions formed was accomplished by filtration, with only small loss of the neutral metal carbonyl. Yields of isolated metal carbonyls: $Ru_3(CO)_{12}$ (85%); $Os_3(CO)_{12}$ (88%); $Cr(CO)_6$ (90%); $Mo(CO)_6$ (75%); $W(CO)_6$ (86%). Isolation of $Mn_2(CO)_{10}$ and $Re_2(CO)_{10}$ was accomplished by reducing the volume of THF to 1 mL and cooling to −78 °C. Crystals of $Mn_2(CO)_{10}$ and $Re_2(CO)_{10}$ formed and were collected by rapid filtration. Yield: $Mn_2(CO)_{10}$ (84%); $Re_2(CO)_{10}$ (79%).

Infrared spectra, carbon monoxide mass spectral analyses, and ^{13}C NMR spectra were employed to determine both degree of ^{13}CO incorporation and the purity of the isolated metal carbonyl. Blank reactions (no hydride promoter) were carried out under identical conditions (Table I) and did not produce measurable ^{13}CO exchange.

Enrichment of $Fe_3(CO)_{12}$. In air, $NaBH_4$ (1–2 mg) was placed in a 250-mL reaction flask containing a Teflon-covered magnetic stirring bar and 600 mg of $Fe_3(CO)_{12}$ and equipped with a vacuum adapter. THF was distilled into the flask at −78 °C and 120 Torr of ^{13}CO was expanded over the solution.[17] The solution was allowed to warm to 25 °C in the absence of light and was stirred for 8 h. At this time, mass spectral analysis of the carbon monoxide revealed that near statistical $^{13}CO-^{12}CO$ exchange had occurred. Removal of the solvent and a subsequent IR spectrum showed formation of enriched $Fe(CO)_5$. Isolation of the $Fe(CO)_5$ was carried out as described above; a yield of 40% was obtained. Once the $Fe(CO)_5$ was removed from the reaction mixture, the isolation procedure for $Fe_3(CO)_{12}$ was the same as for $Ru_3(CO)_{12}$ and $Os_3(CO)_{12}$, as described above. The yield of $Fe_3(CO)_{12}$ was found to be 50%. IR and ^{13}C NMR spectra were used to determine the degree of ^{13}CO incorporation and the purity of the product. A blank reaction under identical conditions (no initiator) showed less than a 1% loss of $Fe_3(CO)_{12}$ (forming $Fe(CO)_5$) and no detectable enrichment of $Fe_3(CO)_{12}$.

Enrichment Promoted by KH. The procedure was the same as that for $NaBH_4$ except that KH was handled inside of a N_2-filled glovebox.

Isolation of KH and $Ru_3(CO)_{12}$ from the Reaction of $K[HRu_3(CO)_{11}]$ and CO. In the drybox 510 mg (0.783 mmol) of $K[HRu_3(CO)_{11}]$ was weighed into a 100-mL bulb equipped with a Teflon-coated stir bar and a vacuum line adapter. The bulb was attached to the line and evacuated, and approximately 30 mL of dried THF was condensed into the bulb at −78 °C. About 1 atm of CO (passed through a −196 °C trap) was expanded into the bulb. The bulb was then warmed to room temperature, and the contents were stirred vigorously. After several days small white particles began to wash up on the side of the bulb and orange crystals were observed in solution. After about 3 weeks the CO was removed from the solution and the bulb was placed in the glovebox, with care being taken to not disturb the white particles on the side. The THF solution was pipetted off, and the orange precipitate was washed with fresh THF. The white solid (3–4 mg) was scraped from the bulb and placed in a 0.3-mm X-ray capillary that was sealed with grease and subsequently flame sealed. An IR spectrum of the brown THF solution and the subsequent orange THF wash was taken. The IR spectrum of the first THF solution showed $K[HRu_3(CO)_{11}]$ with a slight $Ru_3(CO)_{12}$ impurity. The second THF wash solution showed only $Ru_3(CO)_{12}$. The existence of KH was demonstrated from its X-ray powder diffraction.

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Registry No. $Fe(CO)_5$, 13463-40-6; $Fe_3(CO)_{12}$, 17685-52-8; $Ru_3(CO)_{12}$, 15243-33-1; $Os_3(CO)_{12}$, 15696-40-9; $Mn_2CO)_{10}$, 10170-69-1; $Re_2(CO)_{10}$, 14285-68-8; $Cr(CO)_6$, 13007-92-6; $Mo(CO)_6$, 13939-06-5; $W(CO)_6$, 14040-11-0; KH, 7693-26-7; $NaBH_4$, 16940-66-2; ^{13}CO, 1641-69-6; $K[HRu_3(CO)_{11}]$, 80662-65-3; ^{13}C, 14762-74-4.

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[22] In this paper the periodic group notation in parentheses is in accord with recent actions by IUPAC and ACS nomenclature committees. A and B notation is eliminated because of wide confusion. Groups IA and IIA become groups 1 and 2. The d-transition elements comprise groups 3 through 12, and the p-block elements comprise groups 13-18. (Note that the former Roman number designation is preserved in the last digit of the new numbering: e.g., III → 3 and 13.)