

properties of a series of systematically substituted macrocyclic compounds.

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**Registry No.** 1, 56276-51-8; 2, 70320-24-0; 3, 70320-25-1; Ni<sup>II</sup>-1, 51223-51-9; Ni<sup>II</sup>-2, 70354-80-2; Ni<sup>II</sup>-3, 70320-77-3; Ni<sup>II</sup>(CH<sub>3</sub>C-N)<sub>6</sub>(BF<sub>4</sub>)<sub>2</sub>, 15170-11-3; *p*-phenylazobenzoyl chloride, 104-24-5; azobenzene, 103-33-3.

**Supplementary Material Available:** An extended version of Table I containing additional polarographic and voltammetric data (2 pages). Ordering information is given on any current masthead page.

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- The macrocyclic ligands **1**, **2**, and **3** deprotonate in the presence of transition-metal ions. Thus Ni<sup>II</sup>-**1**, **-2**, and **-3** contain the dianionic forms of **1**, **2**, and **3**.
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## Complexes of the Platinum Metals. 16.<sup>1</sup> New Carboxylato Derivatives of Ruthenium(III) and Osmium(III)

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Since their discovery over ten years ago, the ruthenium(II) triphenylphosphine complexes RuX<sub>2</sub>(PPh<sub>3</sub>)<sub>n</sub> (X = Cl or Br; n = 3 or 4)<sup>2</sup> and, to a lesser degree, their osmium analogues<sup>3</sup> have attracted much attention. Their structures,<sup>4,5</sup> dissociative behavior,<sup>5,6</sup> chemical reactivity,<sup>7</sup> and catalytic activity<sup>8</sup> have all been intensively studied, and their aerobic oxidation in organic media has been well documented.<sup>6,9</sup> However, the nature of the ruthenium and osmium species present in the dark colored solutions after oxidation has not been properly elucidated. Moreover, aerobic oxidation of the complexes MX<sub>2</sub>(PPh<sub>3</sub>)<sub>3</sub> in organic media has attracted little attention as a possible route to ruthenium(III) and osmium(III) derivatives.

We now find that under aerobic conditions the complexes MX<sub>2</sub>(PPh<sub>3</sub>)<sub>3</sub> react with carboxylic acids, RCO<sub>2</sub>H (R = alkyl

or aryl), in benzene to afford red-brown solutions from which air-stable carboxylato derivatives MX<sub>2</sub>(O<sub>2</sub>CR)(PPh<sub>3</sub>)<sub>2</sub> rapidly crystallize (R = aryl) or may be precipitated with petroleum ether (R = alkyl). In the absence of air, unchanged ruthenium(II) and osmium(II) complexes are recovered in good yield.

## Experimental Section

The ruthenium and osmium halide complexes MX<sub>2</sub>(PPh<sub>3</sub>)<sub>n</sub> (M = Ru, X = Cl or Br; M = Os, X = Br; n = 3 or 4) were prepared by standard literature procedures<sup>2,3</sup> with platinum metal salts obtained from Johnson Matthey and Co., Ltd. Benzene, petroleum ether (bp 40-60 °C), and carboxylic acids were used as purchased. All reactions were performed in open conical flasks over a steam bath situated in an efficient fumehood. Infrared spectra were taken on a Perkin-Elmer 621 grating spectrometer by using KBr disks, and molecular weights were determined with a Perkin-Elmer Hitachi osmometer. Analyses were performed by the microanalytical laboratories at University College, London, and the School of Pharmacy, London. Melting points were taken in sealed tubes under nitrogen with a Buchi melting point apparatus.

**Dichloro(benzoato)bis(triphenylphosphine)ruthenium(III)** was prepared by the following two methods.

**Method a.** Dichlorotris(triphenylphosphine)ruthenium (0.48 g, 0.5 mM) and benzoic acid (0.12 g, 1.0 mM) were warmed together in benzene (15 mL) at ca. 70 °C for 15 min. The resultant red-brown solution was then filtered, cooled to room temperature, and set aside to crystallize. After ca. 24-48 h the precipitated solid was filtered off, washed with petroleum ether (bp 40-60 °C), and dried in vacuo to yield the required product as red-brown platelets (0.389 g, 95%).

**Method b.** Dichlorotris(triphenylphosphine)ruthenium (0.48 g, 0.5 mM) and benzoyl peroxide (0.125 g, 0.5 mM) were warmed together in benzene (15 mL) at ca. 70 °C for 15 min. The resultant red-brown solution was worked up as described above to yield the required product as red-brown platelets (0.372 g, 90%). The following were prepared by method a with the appropriate carboxylic acid; where necessary, crystallization of the required product from the benzene solution was induced by addition of petroleum ether (bp 40-60 °C): **dichloro(acetato)bis(triphenylphosphine)ruthenium(III)** as flesh pink needles (41%), **dichloro(propionato)bis(triphenylphosphine)ruthenium(III)** as flesh pink microcrystals (62%), **dichloro(chloroacetato)bis(triphenylphosphine)ruthenium(III)** as cream needles (57%), **dichloro(p-chlorobenzoato)bis(triphenylphosphine)ruthenium(III)** as flesh pink platelets (38%), **dichloro(o-bromobenzoato)bis(triphenylphosphine)ruthenium(III)** as red-brown platelets (55%), **dichloro(p-bromobenzoato)bis(triphenylphosphine)ruthenium(III)** as orange-brown platelets (60%), **dichloro(p-methoxybenzoato)bis(triphenylphosphine)ruthenium(III)** as orange-brown platelets (84%), **dichloro(o-toluato)bis(triphenylphosphine)ruthenium(III)** as red-brown platelets (56%), **dichloro(p-toluato)bis(triphenylphosphine)ruthenium(III)** as orange-brown microcrystals (52%), **dichloro(p-nitrobenzoato)bis(triphenylphosphine)ruthenium(III)** as red-brown platelets (63%).

The following were prepared by method a with dibromotris(triphenylphosphine)ruthenium and the appropriate carboxylic acid: **dibromo(acetato)bis(triphenylphosphine)ruthenium(III)** as lustrous brown platelets (62%), **dibromo(benzoato)bis(triphenylphosphine)ruthenium(III)** as dark brown microcrystals (58%), **dibromo(p-bromobenzoato)bis(triphenylphosphine)ruthenium(III)** as brown rhombic crystals (40%), **dibromo(p-toluato)bis(triphenylphosphine)ruthenium(III)** as yellow-brown rhombic crystals (44%).

**Dibromo(benzoato)bis(triphenylphosphine)ruthenium(III)**, as dark brown microcrystals (65%), was prepared by method b with dibromotris(triphenylphosphine)ruthenium.

The following were prepared by method a with dibromotris(triphenylphosphine)osmium: **dibromo(benzoato)bis(triphenylphosphine)osmium(III)** as bright yellow microcrystals (31%), **dibromo(p-bromobenzoato)bis(triphenylphosphine)osmium(III)** as bright yellow microcrystals (40%), **dibromo(p-toluato)bis(triphenylphosphine)osmium(III)** as bright yellow microcrystals (40%).

## Results and Discussion

The ruthenium and osmium complexes MX<sub>2</sub>(PPh<sub>3</sub>)<sub>3</sub> dissolve in warm benzene under aerobic conditions and in the presence of carboxylic acids to afford dark red-brown (M = Ru) or yellow-brown (M = Os) solutions from which products of

**Table I.** Melting Point, Analytical, and Molecular Weight Data

complex	R	mp, °C	% C <sup>a</sup>	% H <sup>a</sup>	% P <sup>a</sup>	% halogen <sup>a</sup>	mol wt <sup>a</sup>
RuCl <sub>2</sub> (O <sub>2</sub> CR)(PPh <sub>3</sub> ) <sub>2</sub>	Me	222–225	61.05 (60.41)	4.45 (4.4)			720 (756)
	C <sub>2</sub> H <sub>5</sub>	218–220	61.34 (60.87)	4.58 (4.58)			766 (769)
	CH <sub>2</sub> Cl	202–203	57.69 (57.77)	4.06 (4.08)			789 (789)
	Ph <sup>b</sup>	250–252	64.15 (63.16)	4.40 (4.31)	7.3 (7.58)	8.65 (8.67)	
	Ph <sup>c</sup>	245–246	63.0 (63.16)	4.35 (4.31)			
	<i>p</i> -C <sub>6</sub> H <sub>4</sub> Cl	222–224	60.65 (60.61)	4.31 (4.02)	6.96 (7.27)	12.8 (12.48)	843 (852)
	<i>o</i> -C <sub>6</sub> H <sub>4</sub> Br	195–196	58.07 (57.6)	3.85 (3.82)			
	<i>p</i> -C <sub>6</sub> H <sub>4</sub> Br	230–231	57.63 (57.6)	3.84 (3.82)			
	<i>p</i> -C <sub>6</sub> H <sub>4</sub> OMe	231–232	62.63 (62.34)	4.61 (4.40)	7.15 (7.31)	8.75 (8.36)	833 (848)
	<i>o</i> -C <sub>6</sub> H <sub>4</sub> Me	217–219	63.93 (63.5)	4.52 (4.48)		8.37 (8.55)	
	<i>p</i> -C <sub>6</sub> H <sub>4</sub> Me	239–240	62.47 (63.5)	4.69 (4.48)	8.1 (7.45)	8.2 (8.55)	
<i>p</i> -C <sub>6</sub> H <sub>4</sub> NO <sub>2</sub> <sup>d,e</sup>	205–206	62.3 (62.5)	4.18 (4.28)			949 (863)	
RuBr <sub>2</sub> (O <sub>2</sub> CR)(PPh <sub>3</sub> ) <sub>2</sub>	Me	221–222	54.09 (54.05)	3.92 (3.94)			
	Ph	225–227	57.14 (56.97)	3.87 (3.89)	7.14 (6.83)	18.0 (17.63)	
	Ph <sup>c</sup>	226–228	57.22 (56.97)	3.92 (3.89)			
	<i>p</i> -C <sub>6</sub> H <sub>4</sub> Br	223–224	52.11 (52.41)	3.49 (3.48)			
	<i>p</i> -C <sub>6</sub> H <sub>4</sub> Me	229–231	57.43 (57.41)	4.13 (4.05)			
OsBr <sub>2</sub> (O <sub>2</sub> CR)(PPh <sub>3</sub> ) <sub>2</sub>	Ph	245–247	51.82 (51.87)	3.51 (3.54)			
	<i>p</i> -C <sub>6</sub> H <sub>4</sub> Br	240–241	46.82 (48.06)	3.05 (3.19)			
	<i>p</i> -C <sub>6</sub> H <sub>4</sub> Me	255–256	52.51 (52.34)	3.73 (3.69)			

<sup>a</sup> Calculated figures in parentheses. <sup>b</sup> % oxygen 3.83 (3.91). <sup>c</sup> Sample prepared from benzoyl peroxide. <sup>d</sup> Calculated figures include benzene of solvation (1 mol). <sup>e</sup> % nitrogen 1.51 (1.49).

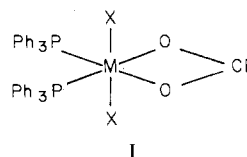
**Table II.** Infrared Data<sup>a</sup>

complex	R	carboxylate (RCO <sub>2</sub> ) vibrations, cm <sup>-1</sup>			ν(Ru-Cl), cm <sup>-1</sup>		
RuCl <sub>2</sub> (O <sub>2</sub> CR)(PPh <sub>3</sub> ) <sub>2</sub>	Me			1480 sh	362 s		
	C <sub>2</sub> H <sub>5</sub>			1480 sh			
	Ph	1607 s		1507 s	1450 s	352 s	
	<i>p</i> -C <sub>6</sub> H <sub>4</sub> Cl	1601 s		1505 s	1440 sh	338 s	
	<i>o</i> -C <sub>6</sub> H <sub>4</sub> Br	1590 s	1570 m		1430 sh	340 s	
	<i>p</i> -C <sub>6</sub> H <sub>4</sub> Br	1590 s		1500 m	1430 sh	355 s	
	<i>p</i> -C <sub>6</sub> H <sub>4</sub> OMe	1605 s			1470 m	1450 s	338 s
	<i>o</i> -C <sub>6</sub> H <sub>4</sub> Me	1602 m	1580 m		1420 sh	340 s	
	<i>p</i> -C <sub>6</sub> H <sub>4</sub> Me	1600 s		1520 s	1470 m	1430 sh	335 s
	<i>p</i> -C <sub>6</sub> H <sub>4</sub> NO <sub>2</sub> <sup>b</sup>	1605 m		1500 s	1430 sh	335 s	
	RuBr <sub>2</sub> (O <sub>2</sub> CR)(PPh <sub>3</sub> ) <sub>2</sub>	Me			1480 sh		
Ph		1605 s		1505 s	1440 sh		
<i>p</i> -C <sub>6</sub> H <sub>4</sub> Me		1610 m		1520 s	1430 sh		
<i>p</i> -C <sub>6</sub> H <sub>4</sub> NO <sub>2</sub> <sup>b</sup>		1610 vw		1505 m	1440 sh		
OsBr <sub>2</sub> (O <sub>2</sub> CR)(PPh <sub>3</sub> ) <sub>2</sub>	Ph	1610 m		1510 m	1450 s		

<sup>a</sup> s = strong, m = medium, w = weak, sh = shoulder. <sup>b</sup> Also very strong ν(NO<sub>2</sub>) bands at ca. 1530 and 1345 cm<sup>-1</sup>.

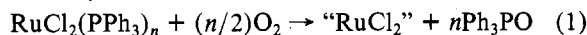
stoichiometry MX<sub>2</sub>(O<sub>2</sub>CR)(PPh<sub>3</sub>)<sub>2</sub> rapidly crystallize (R = aryl) or are precipitated by petroleum ether (R = alkyl). The benzoato complexes MX<sub>2</sub>(O<sub>2</sub>CPh)(PPh<sub>3</sub>)<sub>2</sub> can also be prepared by treatment of the precursors, MX<sub>2</sub>(PPh<sub>3</sub>)<sub>3</sub>, with benzoyl peroxide under anaerobic or aerobic conditions. The new ruthenium complexes (Table I) vary in color from red-brown to flesh pink depending upon particle size; their osmium analogues are all bright yellow. All are air-stable, paramagnetic solids (μ<sub>eff</sub> = 1.9 μ<sub>B</sub>); the arylcarboxylato derivatives in particular are very sparingly soluble in all common organic solvents. Molecular weight determinations (chloroform, 303 K) for the more soluble products establish that these at least are monomeric in solution. The paramagnetism and low solubility of the new complexes preclude <sup>31</sup>P NMR studies. However, the infrared spectra are more informative; bands attributable to the carboxylate ligand (ca. 1620–1420 cm<sup>-1</sup>) are clearly visible (Table II). For the arylcarboxylato complexes, absorptions in the region 1620–1420 cm<sup>-1</sup> fall into three frequency ranges: ca. 1620–1590, 1535–1500, and 1450–1420 cm<sup>-1</sup>. These we attribute to aromatic ring stretching, asymmetric carboxylate stretching [ν(OCO)<sub>asym</sub>], and symmetric carboxylate stretching [ν(OCO)<sub>sym</sub>] vibrations, respectively.<sup>10,11</sup> The absorptions at 1535–1500 cm<sup>-1</sup> probably also include a contribution from an aromatic ring stretching mode.<sup>10</sup> These conclusions are supported by the spectra of the corresponding alkylcarboxylato complexes which display only one band or shoulder (ca. 1480 cm<sup>-1</sup>) attributable to the

carboxylate ligand. Assignment of the bands at ca. 1535–1500 and 1450–1420 cm<sup>-1</sup> to ν(OCO)<sub>asym</sub> and ν(OCO)<sub>sym</sub>, respectively, is fully consistent with the presence of chelate arylcarboxylate ligands.<sup>12</sup> The ruthenium chloro complexes all show a single, strong, sharp absorption at ca. 365–335 cm<sup>-1</sup> which is absent from the spectra of the corresponding bromo complexes and can be attributed to the asymmetric vibration of a trans Cl–Ru–Cl moiety.<sup>13</sup> Ruthenium- and osmium-bromide stretching vibrations (<~200 cm<sup>-1</sup>) lie beyond the range of our spectrometers and were therefore not examined. However, it seems probable that the bromo complexes adopt the same stereochemistry as their chloro analogues. The presence of a chelate carboxylate ligand and a trans X–M–X moiety would establish stereochemistry I for the new complexes.

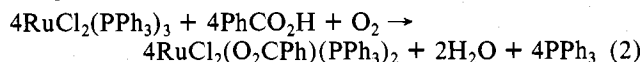


Although participation of dioxygen in the formation of the carboxylato complexes MX<sub>2</sub>(O<sub>2</sub>CR)(PPh<sub>3</sub>)<sub>2</sub> is clearly established by the repeated failure of syntheses attempted under anaerobic conditions, the exact mechanism of the reaction remains unclear. Solutions of the ruthenium complexes

$\text{RuX}_2(\text{PPh}_3)_n$  ( $n = 3$  or  $4$ ) in particular are highly air-sensitive and undergo rapid oxidation to form dark green or brown products containing triphenylphosphine oxide and/or ruthenium(III) species.<sup>2,6,9</sup> Previous workers<sup>6,9</sup> have established that uptake of dioxygen by the  $\text{RuCl}_2(\text{PPh}_3)_n$  complexes in benzene solution at ambient temperature corresponds to the stoichiometry



and is accompanied by a color change from orange to green but does not lead to formation of characterizable intermediates or isolable products. In particular, this system affords no positive evidence for the formation of dioxygen adducts.<sup>14</sup> We have found that the stoichiometry of dioxygen uptake by  $\text{RuCl}_2(\text{PPh}_3)_3$  at 45 °C is the same as that previously measured at ambient temperature and have observed that subsequent addition of benzoic acid to the oxygenated solution does not afford the complex  $\text{RuCl}_2(\text{O}_2\text{CPh})(\text{PPh}_3)_2$ . We have also found that uptake of dioxygen during the reaction of  $\text{RuCl}_2(\text{PPh}_3)_3$  with benzoic acid to form  $\text{RuCl}_2(\text{O}_2\text{CPh})(\text{PPh}_3)_2$  amounts to ca. 0.25–0.30 mol  $\text{O}_2$ /mol of ruthenium complex. The lower limit is in accord with the reaction



The “extra” oxygen, which is slowly taken up over a period of ca. 90 min, is probably consumed in the oxidation of a small amount of the liberated triphenylphosphine to phosphine oxide. However, <sup>31</sup>P NMR measurements on the mother liquors after removal of the ruthenium complex establish that whereas substantial quantities of triphenylphosphine are present very little triphenylphosphine oxide is formed. This observation in turn implies that the reaction of carboxylic acids with the ruthenium complexes occurs sufficiently rapidly to prevent significant  $\text{RuCl}_2(\text{PPh}_3)_n$ -catalyzed oxidation of triphenylphosphine and that the carboxylato complexes  $[\text{RuCl}_2(\text{O}_2\text{CR})(\text{PPh}_3)_2]$  do not act as catalysts for the same reaction. The formation of  $\text{RuCl}_2(\text{O}_2\text{CPh})(\text{PPh}_3)_2$  from  $\text{RuCl}_2(\text{PPh}_3)_3$  and benzoyl peroxide does not require the presence of dioxygen and probably proceeds by an oxidative-addition mechanism possibly involving the known<sup>5</sup> binuclear halide-bridged species  $[\text{RuX}_2(\text{PPh}_3)_2]_2$ .

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**Registry No.**  $\text{RuCl}_2(\text{O}_2\text{CPh})(\text{PPh}_3)_2$ , 70046-96-7;  $\text{RuCl}_2(\text{O}_2\text{CMe})(\text{PPh}_3)_2$ , 70046-97-8;  $\text{RuCl}_2(\text{O}_2\text{CC}_2\text{H}_5)(\text{PPh}_3)_2$ , 70046-98-9;  $\text{RuCl}_2(\text{O}_2\text{CCH}_2\text{Cl})(\text{PPh}_3)_2$ , 70084-48-9;  $\text{RuCl}_2(\text{O}_2\text{C}-p\text{-C}_6\text{H}_4\text{Cl})(\text{PPh}_3)_2$ , 70046-99-0;  $\text{RuCl}_2(\text{O}_2\text{C}-o\text{-C}_6\text{H}_4\text{Br})(\text{PPh}_3)_2$ , 70047-00-6;  $\text{RuCl}_2(\text{O}_2\text{C}-p\text{-C}_6\text{H}_4\text{Br})(\text{PPh}_3)_2$ , 70047-01-7;  $\text{RuCl}_2(\text{O}_2\text{C}-p\text{-C}_6\text{H}_4\text{OMe})(\text{PPh}_3)_2$ , 70047-02-8;  $\text{RuCl}_2(\text{O}_2\text{C}-o\text{-C}_6\text{H}_4\text{Me})(\text{PPh}_3)_2$ , 70047-03-9;  $\text{RuCl}_2(\text{O}_2\text{C}-p\text{-C}_6\text{H}_4\text{Me})(\text{PPh}_3)_2$ , 70047-04-0;  $\text{RuCl}_2(\text{O}_2\text{C}-p\text{-C}_6\text{H}_4\text{NO}_2)(\text{PPh}_3)_2$ , 70047-05-1;  $\text{RuBr}_2(\text{O}_2\text{CMe})(\text{PPh}_3)_2$ , 70047-06-2;  $\text{RuBr}_2(\text{O}_2\text{CPh})(\text{PPh}_3)_2$ , 70047-07-3;  $\text{RuBr}_2(\text{O}_2\text{C}-p\text{-C}_6\text{H}_4\text{Br})(\text{PPh}_3)_2$ , 70047-08-4;  $\text{RuBr}_2(\text{O}_2\text{C}-p\text{-C}_6\text{H}_4\text{Me})(\text{PPh}_3)_2$ , 70047-09-5;  $\text{OsBr}_2(\text{O}_2\text{CPh})(\text{PPh}_3)_2$ , 70047-10-8;  $\text{OsBr}_2(\text{O}_2\text{C}-p\text{-C}_6\text{H}_4\text{Br})(\text{PPh}_3)_2$ , 70047-11-9;  $\text{OsBr}_2(\text{O}_2\text{C}-p\text{-C}_6\text{H}_4\text{Me})(\text{PPh}_3)_2$ , 70047-12-0;  $\text{RuBr}_2(\text{O}_2\text{C}-p\text{-C}_6\text{H}_4\text{NO}_2)(\text{PPh}_3)_2$ , 70047-13-1;  $\text{RuCl}_2(\text{PPh}_3)_3$ , 15529-49-4;  $\text{RuBr}_2(\text{PPh}_3)_3$ , 15709-75-8;  $\text{OsBr}_2(\text{PPh}_3)_3$ , 36543-21-2.

## References and Notes

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## Bis(chlorozinc)tetracarbonyliron: Preparation, Characterization, and Metathetical Reactions with Organotin Chlorides

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Derivatives of  $\text{H}_2\text{Fe}(\text{CO})_4$  are commonly made from neutral iron carbonyls<sup>1</sup> or from  $\text{Fe}(\text{CO})_4^{2-}$ .<sup>2,3</sup> Vigorous conditions required for reaction of the former with organotin halides frequently give unpredictable products.<sup>1</sup> Little is known about the metathetical reactions of zinc-to-transition-metal bonds or their use in synthetic intermediates. Previous work in our laboratory showed that Zn–Co and Zn–Mn bonds were cleaved efficiently by triorganotin chlorides.<sup>4</sup> Herein we describe the synthesis of  $(\text{ClZn})_2\text{Fe}(\text{CO})_4\text{C}_6\text{H}_{14}\text{O}_3$  and its reactivity with organotin halides.<sup>5</sup> Chlorozinc and bromozinc derivatives of some molybdenum complexes have been prepared and structurally characterized by Oliver and co-workers.<sup>6–9</sup>

## Experimental Section

All reactions were carried out in modified (two-necked) Schlenk tubes (MST) under an atmosphere of purified argon unless otherwise specified.<sup>10</sup> All melting points were determined in sealed capillary tubes under argon and are corrected. Infrared spectra were obtained with a Perkin-Elmer Model 337 spectrometer. NMR spectra were taken on a Varian A-60A spectrometer calibrated against external  $\text{Me}_4\text{Si}$ . Elemental analyses were performed by the Pascher Microanalytisches Laboratorium, Bonn, West Germany. Analyses of CO were done with the apparatus described by Orchin and Wender.<sup>11,12</sup> Tin was determined gravimetrically.<sup>13</sup> *cis*-Bis(chloromercury)-tetracarbonyliron was prepared by the method of Hock and Stuhlman<sup>14</sup> and dried over  $\text{P}_2\text{O}_{10}$  at 56 °C under vacuum. Diglyme was doubly distilled from sodium.

**Preparation of Bis(chlorozinc)tetracarbonyliron Bis(2-methoxyethyl) Etherate.**  $(\text{ClHg})_2\text{Fe}(\text{CO})_4$  (1.28 g, 2.0 mmol), powdered zinc (2.61 g, 40 mg-atoms), and diglyme (40 mL) were stirred together for 18 h during which a yellow precipitate formed. When the mixture was heated to a gentle reflux for 5 min, the precipitate dissolved. After cooling, the mixture was filtered and the solvent was removed by trap-to-trap distillation at 40 °C. The nearly white residue was recrystallized from a mixture of diglyme and toluene yielding 0.56 g (69%) of colorless crystals, mp 162–164 °C dec. Anal. Calcd for  $\text{C}_{10}\text{H}_{14}\text{Cl}_2\text{FeO}_3\text{Zn}_2$ : C, 23.84; H, 2.80; Cl, 14.08; CO, 22.24. Found: C, 24.29; H, 3.39; Cl, 14.30; CO, 22.5. In one preparation, the zinc