member of the class $RC_2R'Fe_3(CO)_9$, which may be regarded as an alkyne-substituted $Fe_3(CO)_{12}$,[42] thereby fills the missing structural link in observing the detailed stereochemical changes in trinuclear iron carbonyl–acetylene complexes on successive chemical replacement of carbonyl groups by alkynes. The sequence illustrated in Figure 7a–d demonstrates how the linkage of the alkynes can be achieved within the framework of the trinuclear organorion complexes. The given configuration of $Fe_3(CO)_{12}$ (Figure 7a) represents the probable structure[43] obtained from the structure of the trinuclear anion $HFe_3(CO)_{11}^-$[26] by the substitution of a bridging carbonyl group in the regular coordination site occupied by a bridging hydrogen atom. The proposed structure, regarded as being formed by the insertion of a *cis*-$Fe(CO)_4$ group in place of one of the three bridging carbonyl positions of $Fe_2(CO)_9$, is in accord with the X-ray, reinterpreted Mössbauer, and *solid-state* infrared studies of $Fe_3(CO)_{12}$ and *no doubt* is correct.[43]

Figure 7 also shows the structural relation between $Fe_3(CO)_{12}$ (a) and $C_6H_5C_2C_6H_5Fe_3(CO)_9$ (b) to involve the substitution of the diphenylacetylene for the two bridging carbonyls and one of the carbonyls of the *cis*-$Fe(CO)_4$ fragment. Since one alkyne (with four π electrons available for bonding) has displaced three carbonyl groups (with six bonding σ-type electrons) to give $RC_2R'Fe_3(CO)_9$ complexes, an electron-deficient compound results with the electronic configuration involving two three-center bonds. The further addition of the second alkyne with the loss of only one carbonyl group to give the nonelectron-deficient violet isomer of $(C_6H_5C_2C_6H_5)_2Fe_3(CO)_8$ (c) can be visualized in terms of the displacement of a terminal carbonyl from one of the equivalent iron atoms of $C_6H_5C_2C_6H_5Fe_3(CO)_9$ (b) with the more or less simultaneous attachment of the olefinic group to the other formerly equivalent iron along with the formation of new bonds from the addition of the second alkyne as an olefinic coordinating group. The isomerization of the violet compound to give the ferracyclopentadiene group of the black $(C_6H_5C_2C_6H_5)_2Fe_3(CO)_8$ (d) involves the breaking of an iron–iron bond and formation of the butadiene bond and iron carbonyl (bridge) bonds.[27] Although the reaction mechanisms involving the bond-breaking and -making sequence with formation of the new complexes are not known, the above pictorial representation of structural change does illustrate the complexity of metal–alkyne reactions involving molecular reorganization of the carbonyls due to the stereochemical requirements of the alkyne groups in their distinctly different types of interaction with the three iron atoms.

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(42) Although the diphenyl derivative was isolated from the alkyne reaction with $Fe_2(CO)_9$, the methylphenyl derivative was obtained from $Fe_3(CO)_{12}$.

(43) L. F. Dahl and J. F. Blount, *Inorg. Chem.*, **4**, 1373 (1965).

Lewis Basicity of Metals. II. The Interaction of Group VI and VII Transition Metal Cyclopentadienyl Derivatives with BF_3, BCl_3, and B_2H_6

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Abstract: The reaction of $(C_5H_5)_2MoH_2$, $(C_5H_5)_2WH_2$, and $(C_5H_5)_2ReH$ with Lewis acids leads to the following adducts: $(C_5H_5)_2WH_2 \cdot BF_3$, $(C_5H_5)_2WH_2 \cdot BCl_3$, $(C_5H_5)_2MoH_2 \cdot BF_3$, $(C_5H_5)_2ReH \cdot BF_3$, and $(C_5H_5)_2ReH \cdot BCl_3$. However, borane addition compounds do not form. Infrared spectra of the adducts indicate the presence of tetracoordinated boron and the most reasonable structures involve metal–BX_3 donor–acceptor bond formation. Attempts to add BF_3 to $(C_5H_5)_2Mo(CO)_3^-$ and $(C_5H_5)_2W(CO)_3^-$ resulted in their neutral dimers as the principal products.

In a recent communication, an example of the apparent basicity of a metal toward BF_3 was reported.[1] This report on $(C_5H_5)_2WH_2 \cdot BF_3$ was soon followed by Parshall's observation that some manganese and rhenium complexes are basic toward BH_3.[2] Very recently Brunner, Wailes, and Kaesz reported the stable adducts $(C_5H_5)_2WH_2 \cdot AlMe_3$ and $(C_5H_5)_2ReH \cdot AlMe_3$.[3] We are now aware of a system which appears analogous to those above—$Co_2(CO)_8 \cdot AlBr_3$—which was reported by Chini and Ercoli in 1958.[4]

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(1) D. F. Shriver, *J. Am. Chem. Soc.*, **85**, 3509 (1963).

(2) G. W. Parshall, *ibid.*, **86**, 361 (1964).

(3) H. Brunner, P. C. Wailes, and H. D. Kaesz, Abstracts, Proceedings of the Second International Symposium on Organometallic Chemistry, Madison, Wis., 1965, p. 40.

(4) P. Chini and R. Ercoli, *Gazz. chim. ital.*, **88**, 1170 (1958).

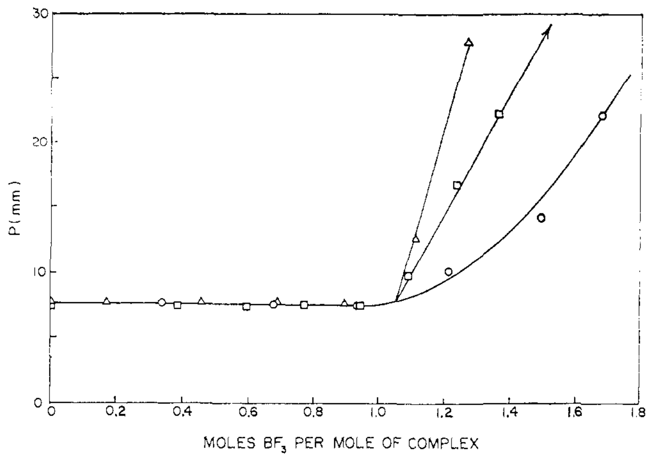

Figure 1. Tensimetric titration of biscyclopentadienylmetal hydrides with BF_3: ○, $(C_5H_5)_2MoH_2 + BF_3$; □, $(C_5H_5)_2WH_2 + BF_3$; △, $(C_5H_{52})ReH + BF_3$. Toluene was the solvent and the temperature was 0°. Curvature in the region of the break is not unique to these systems; it frequently occurs when a solid phase is present. Presumably, this is due to adsorption.

In this paper we describe attempts to prepare Lewis salts of cyclopentadienyl metal derivatives which are known to protonate on the metal.[5]

Experimental Section

Except for methylcyclopentadienylmanganese tricarbonyl, all of the compounds encountered in this work are air sensitive. Accordingly, the metal complexes were prepared and manipulated under a blanket of nitrogen[6] and the Lewis acids were generally transferred, stored, and measured in a preparative high-vacuum system. Samples for infrared spectra and X-ray powder patterns were handled in a drybox.

All infrared spectra reported here were obtained on a Beckman IR-9. Visible spectra were obtained on a Cary Model 14 spectrophotometer and n.m.r. spectra were run on a Varian A-60.

Solvents were dried by distillation from calcium hydride and they were freed from oxygen by either a nitrogen purge or by outgassing under vacuum. Dimethoxyethane was purified by distillation from lithium aluminum hydride in a nitrogen atmosphere.

The Lewis acids were purified by trap-to-trap distillation; vapor pressures agreed with literature reports: BF_3, 301.4 mm. at $-111.6°$ (lit.[7] 301 mm.); BCl_3, 476.0 mm. at 0° (lit.[8] 476 mm.); B_2H_6, 224.9 mm. at $-111.6°$ (lit.[9] 225 mm.). In addition, infrared spectra of these gases agreed with those in the literature.

Cyclopentadienyl Hydrides. Biscyclopentadienyltungsten dihydride, the corresponding molybdenum compound, and biscyclopentadienylrhenium hydride were prepared by the methods of Wilkinson and co-workers.[10,11] $(C_5H_5)_2WH_2$ melted at 193 to 195° (lit.[12] 185 to 190°); the n.m.r. and infrared spectra agreed with those reported by Wilkinson. Also the X-ray powder pattern was indexed on the basis of a slightly larger C-centered unit cell than that reported for $(C_5H_5)_2MoH_2$.[13] The latter compound melted at 163 to 165° (lit.[12] 165 to 168°) and the infrared spectrum agreed with the literature. $(C_5H_5)_2ReH$ melted at 160 to 162° (lit.[14] 161–162°) and the infrared spectrum agreed with the literature.[11]

Cyclopentadienyl Carbonyls. Methylcyclopentadienylmanganese tricarbonyl was a gift of the Ethyl Corp.; it was listed as 99.8% pure and was used directly.

A solution of 10 g. of $NaMoC_5H_5(CO)_3$ in tetrahydrofuran was prepared according to King's recipe[15] and the solvent was removed under reduced pressure leaving a brown residue; 50 ml. of 1 *M* NaOH was added and the mixture was stirred for 2 hr. The resulting yellow solution was filtered into another solution containing 10 g. of $[(C_6H_5)_4As]Cl$ and a yellow precipitate resulted. This product was filtered under N_2, washed with water, and dried under high vacuum. An infrared spectrum of a Nujol mull showed CO bands at 1751 (s), 1776 (s), and 1892 (s), with additional bands at 1175 (m), 1000 (s), 885 (m), 760 (s), 750 (s), and 695 (s) cm.$^{-1}$. The CO frequencies are in the correct region for the anionic complex.

Tetrapropylammonium cyclopentadienyltungsten tricarbonyl was prepared in a manner analogous to the molybdenum compound. Its infrared spectrum in a Nujol mull showed CO bands at 1774 (s), 1782 (s, sh), and 1897 (s) with additional bands at 1264 (mw), 1177 (m), 1100 (m), 1054 (m), 977 (m), 780 (m), 724 (m), 615 (m), and 515 (m) cm.$^{-1}$.

Results

Cyclopentadienylmetal Hydride–BF_3 Adducts. Direct reaction of solid $(C_5H_5)_2WH_2$ with BF_3 gas for 3 days at 33.6° led to the absorption of 0.954 mole of boron trifluoride per mole of complex. A more rapid synthesis was achieved by adding BF_3 to a toluene solution of the complex, which resulted in immediate precipitation of the adduct. This reaction was followed by incremental addition of BF_3 at -22.9 and 0°, and a pressure break was evident at 1:1 stoichiometry (Figure 1). The dark yellow adduct displayed a broad infrared absorption in the 1000–1100-cm.$^{-1}$ region which is in the correct position for the asymmetric BF_n stretching frequency of four-coordinated boron.[16] The complete spectrum contained bands at 521 (mw), 577 (mw), 601 (m), 776 (m), 720 (s), 822 (ms), 906 (w), ~1004 (s), ~1053 (s), ~1097 (m) ~1286 (w), 1917 (m), 1922 (sh), and 3110 (mw), cm.$^{-1}$.

A similar tensimetric titration of BF_3 with $(C_5H_5)_2MoH_2$ in toluene indicated a 1:1 adduct (Figure 1). Removal of BF_3 and solvent under high vacuum followed by separation of these components by trap-to-trap distillation and pressure–volume–temperature measurement of the BF_3 showed that 1.03 moles of acid was absorbed per mole of complex. Purity of the BF_3 was established by an infrared spectrum which showed no spurious bands.

The toluene-insoluble, green adduct displayed characteristic coordinated B–F stretching vibrational transition in the 1000–1100-cm.$^{-1}$ region. The complete spectrum contains the following bands: 723 (ms), 765 (ms), 819 (ms), 845 (m), 1007 (s), 1057 (s), 1093 (s), 1102 (s), 1853 (m), and 3140 (m) cm.$^{-1}$.

A tensimetric titration (Figure 1) and measurement of BF_3 consumed (1.05:1) were used to establish the formation of $(C_5H_5)_2ReH \cdot BF_3$. This yellow adduct exhibited the characteristic BF_3 band around 1000–1100 cm.$^{-1}$; the complete spectrum included 526 (m), 607 (m), 725 (m), 819 (s), 1000 (m), 1007 (m), 1039 (s), 1053 (s), 1085 (s), 1282 (m), 2057 (w), and 3153 (m) cm.$^{-1}$.

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(8) H. C. Brown and R. R. Holmes, *ibid.*, **78**, 2173 (1956).

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(11) M. L. H. Green, L. Pratt, and G. Wilkinson, *ibid.*, 3916 (1958).

(12) E. O. Fischer and Y. Hristidu, *Z. Naturforsch.*, **15b**, 135 (1960).

(13) M. Gerloch and R. Mason, *J. Chem. Soc.*, 296 (1965).

(14) R. B. King, *Organometal. Syn.*, **1**, 80 (1965).

(15) R. B. King, *Inorg. Syn.*, **7**, 107 (1963).

(16) (a) A. R. Katritzky, *J. Chem. Soc.*, 2049 (1959); (b) G. L. Cote and H. W. Thompson, *Proc. Roy. Soc.* (London), **A210**, 217 (1951).

Cyclopentadienylmetal Hydride–BCl_3 Adducts. A solution of 0.345 mmole of $(C_5H_5)_2WH_2$ in toluene was allowed to react with 0.805 mmole of BCl_3 at 0°. A dark orange-brown precipitate formed immediately. The solvent and excess BCl_3 were removed overnight under high vacuum to leave a yellow solid, and weight gain of the solid indicated that 1.08 moles of BCl_3 was absorbed per mole of complex. In an attempt to determine excess BCl_3, pyridine was added to the toluene–BCl_3 mixture which had been removed from the sample (BCl_3 and toluene cannot be quantitatively separated by trap-to-trap distillation). However, the last traces of pyridine and/or toluene could not be removed from the complex, which resulted in gummy py–BCl_3. The weight of this complex indicated 0.92 BCl_3 absorbed per tungsten complex. An infrared band in the spectrum of the metal adduct at *ca.* 700 cm.$^{-1}$ probably corresponds to the asymmetric stretch found at 755 cm.$^{-1}$ for Me_3NBCl_3.[17] A very weak band at 550 cm.$^{-1}$ may be the BCl_3 symmetrical stretch.[17] The complete spectrum included: 430 (ms), 444 (ms), *ca.* 550 (vw), 620 (m), 668 (s), 678 (s), 696 (s), 704 (s), 724 (s), 811 (m), 816 (m), 831 (m), 846 (m), 906 (m), 1017 (m), 1967 (m), and 3112 (m) cm.$^{-1}$.

A similar experiment was performed with 0.218 mmole of $(C_5H_5)_2ReH$ and 0.461 mmole of BCl_3. The weight gain of the orange solid indicated 1.12 BCl_3 absorbed per $(C_5H_5)_2ReH$. The compound was very air sensitive and decomposed before infrared spectra could be obtained.

Cyclopentadienylmetal Hydrides with B_2H_6. No isolable adducts were observed when B_2H_6 was exposed to toluene solutions of $(C_5H_5)_2WH_2$ or $(C_5H_5)_2ReH$.[18] A sample of biscyclopentadienyltungsten dihydride (0.716 mmole) in toluene was exposed to about 0.5 mmole of diborane. After 5 hr. at 0° the pressure in the tensimeter was unchanged and 98% of the diborane was recovered unchanged.

In a similar experiment 0.106 mmole of B_2H_6 and 0.12 mmole of $(C_5H_5)_2ReH$ were left overnight at 0° and a slight pressure increase was noted. This increase was probably due to decomposition of the diborane to hydrogen and higher boron hydrides. The amount of noncondensables in the system was 0.073 mmole, and 0.070 mmole of condensable material was recovered. The infrared of the reaction residue did not show any bands in the terminal or bridging BH region and the $(C_5H_5)_2ReH$ bands appeared unchanged.

Cyclopentadienylmetal Carbonyls with BF_3. A solution of 1.35 mmoles of methylcyclopentadienylmanganese tricarbonyl in a few milliliters of toluene was exposed to an equivalent amount of BF_3. The solution changed from orange to red, but negligible BF_3 absorption was found at either 0 or −78°. Boron trifluoride was recovered quantatively from the mixture.

The observation that $MeC_5H_4Mn(CO)_3$ will not add BF_3 is not surprising, since this compound is protonated, if at all, in very strong acid.[5] However, the isoelectronic anions of group six are known to protonate with ease,[19] and on this basis, their reaction with boron trifluoride appeared promising.

$[(C_6H_5)_4As][C_5H_5Mo(CO)_3]$ (5 g.) was dissolved in 150 ml. of methylene chloride, and a BF_3–N_2 mixture was bubbled through to yield a dark red solution.

Pentane was added to this solution and a gray precipitate formed. The infrared spectrum of this material was nearly identical with that of $[(C_6H_5)_4As][BF_4]$ but the X-ray powder patterns were different. Qualitative tests indicated the presence of chloride, boron, and arsenic and the absence of molybdenum. It was tentatively postulated that this material could be $[(C_6H_5)_4As][BClF_3]$ for which there is some precedent[20] and which might be explained by the following reaction.

$$2[(C_6H_5)_4As][Mo(cp)(CO)_3] + 2BF_3 + 2CH_2Cl_2 = Mo_2(cp)_2(CO)_6 + 2[(C_6H_5)_4As][BClF_3] + C_2H_4Cl_2$$

However, an attempt to prepare $[(C_6H_5)_4As][BClF_3]$ by bubbling BF_3 through a methylene chloride solution of $[(C_6H_5)_4As][Cl]$ followed by precipitation with pentane led to a chloride-containing product which was very similar in infrared spectrum and powder pattern to $[(C_6H_5)_4As][BF_4]$. Unfortunately, the characteristic absorption[20] for BF_3Cl^- at 456 cm.$^{-1}$ is obscured by the $(C_6H_5)_4As^+$ cation. Since the gray precipitate did not display CO and C_5H_5 infrared absorptions, its complete characterization was not attempted. In subsequent discussion it is referred to as "the chlorofluoroborate."

The solvent from the original reaction was evaporated leaving a dark residue, from which yellow crystals were sublimed at 50°. These crystals were identified as the hydride $C_5H_5Mo(CO)_3H$ by their melting point which was 53 to 55° (lit.[19] 50–52°) and CO bands at 2031 and 1935 cm.$^{-1}$ (lit.[21] 2028 and 1934 cm.$^{-1}$). This was also confirmed by the 5:1 ratio of low-field to high-field n.m.r. signals. The residue was chromatographed on silica gel with methylene chloride as an eluent. The major constituent of this material was the red compound $[C_5H_5Mo(CO)_3]_2$ as evidenced by a visible maximum at 508 mμ, a proton signal at 5.48 p.p.m. *vs.* TMS, and CO stretching frequencies at 1958 and 1898 cm.$^{-1}$; these results agreed with those of an authentic sample purchased from Alfa Inorganics. The reaction was also carried out on a vacuum line and the same three products were isolated. Apparently, BF_3 has promoted reaction of the anion with the solvent. Formation of the dimer involves an oxidation which may occur in partially chlorinated solvents but at a much slower rate than observed in the presence of BF_3. Slightly more than 2 moles of BF_2 was consumed per mole of original anion; however, the fate of boron, other than that going into "chlorofluoroborate," is unknown. The only unidentified metal-containing product was a moderate amount of dark brown material which was retained on the chromatography column and which had the appearance of the nondescript oxidation products of $[C_5H_5Mo(CO)_3]_2$.

Since the stabilities of group VI cyclopentadienyl tricarbonyl hydrides increase in the order Cr < Mo

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(17) (a) R. C. Taylor, Advances in Chemistry Series, No. 42, American Chemical Society, Washington, D. C., 1964, p. 59; (b) R. L. Amster and R. C. Taylor, *Spectrochim. Acta*, **20**, 1487 (1964).

(18) Prior to our work on the diborane systems, Parshall (private communication) attempted the reaction of B_2H_6 with a benzene solution of $(C_5H_5)_2WH_2$ but an adduct was not formed.

(19) E. O. Fischer and W. Hafner, *Z. Naturforsch.*, **10b**, 140 (1955).

(20) T. C. Waddington and F. Klanberg, *J. Chem. Soc.*, 2339 (1960).

(21) H. G. Schuster-Woldan, private communication.

$\leq$ W,[22] and since ethers are generally poorer oxidizing agents than chlorinated hydrocarbons, we attempted the reaction of $[pr_4N][C_5H_5W(CO)_3]$ with BF_3 in dimethoxyethane. The reaction was carried out on a vacuum line under rigorously air-free conditions. The solid reaction mixture, which remained after volatiles were removed, contained $C_5H_5W(CO)_3H$ which displayed carbonyl bands assignable to the hydride at 2029 (s) and 1931 (s) cm.$^{-1}$. The other products were a pentane-insoluble material, presumably $[(n\text{-}C_3H_7)_4N][BF_4]$, and the red dimer $[(C_5H_5)_2W(CO)_3]_2$, which had infrared absorptions at 1959 (s), 1931 (s), and 1899 (sh) cm.$^{-1}$.

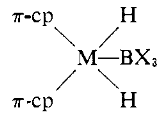
Discussion

Infrared absorptions due to metal-hydrogen vibrations are collected in Table I where it may be seen that adduct formation either leaves the frequency unchanged or causes an increase except in the case of $(C_5H_5)_2WH_2 \cdot BF_3$. In general, BF_3 has little effect, but a rather large frequency shift was observed upon formation of the BCl_3 adduct of $(C_5H_5)_2WH_2$. These shifts are smaller and in the opposite direction from those of hydrogen stretching frequencies of main group Lewis bases; *e.g.*, NH_3 stretching frequencies at 3414 and 3336 cm.$^{-1}$ shift to 3397 and 3312 cm.$^{-1}$ in $[Ni(NH_3)_6][ClO_4]_2$.[23] Protonated forms of these cyclopentadienylmetal hydrides have irregular M-H frequency shifts from the parent complex and in several cases frequency increases are observed.[24]

Table I. Comparison of M-H Stretching Frequencies

Compound	M-H frequency, cm.$^{-1}$
$Re(cp)_2H$	2040
$Re(cp)_2H \cdot BF_3$	2057
$W(cp)_2H_2$	1922
$W(cp)_2H_2 \cdot BF_3$	1917
$W(cp)_2H_2 \cdot BCl_3$	1967
$Mo(cp)_2H_2$	1853
$Mo(cp)_2H_2 \cdot BF_3$	1853

Adducts of the cyclopentadienylmetal hydrides were either insoluble in or reactive with common solvents, so molecular weight and conductivity experiments were not possible. Therefore, it is necessary to rely on less direct structural evidence. The most plausible structures for the adducts formed here involve simple donor-acceptor bond formation. All of the adducts have absorptions in the correct region for coordinated BF_3 or BCl_3 (or for any tetrahedrally bonded B-F or B-Cl group). The M-H frequencies remain upon coordination and do not shift greatly. This suggests that M-H-BX_3 linkages similar to those of $B_2H_7^-$ are not formed,[25] and it also indicates that M-H bonds are not broken. Vibrations characteristic of the cyclopentadienyl groups are not greatly affected by BX_3 addition. In particular, bands characteristic of pentagonal symmetric C_5H_5 ligands are present, whereas frequencies found only in diene or σ-bonded C_5H_5 are absent.[24] The simplest structure consistent with these data is I; however, ionic formulations such as II cannot be excluded but appear unlikely for steric reasons.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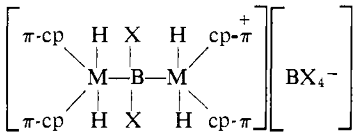
II

Despite the fact that $(C_5H_5)_2ReH$ has two unshared electron pairs, which should be available for donor-acceptor bond formation,[26] it takes up only 1 mole of Lewis acid. This is analogous to the observation that the compound adds only one proton.[11] However, the electrostatic argument which was invoked in the protonation study does not seem likely for the BF_3 system.

It is surprising that BF_3 adducts of the cyclopentadienylmetal hydrides form, while BH_3 adducts do not. By contrast, Lewis bases containing main group elements are sometimes known to coordinate with BH_3 but not BF_3; for example, H_3BCO is known but F_3BCO is not. According to Pearson's scheme of classification this implies that these cyclopentadienylmetal hydrides are "hard" bases.[27]

Acknowledgment. This research was supported by Grants GP-1977 and GP-3804 from the National Science Foundation.

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[24] H. P. Fritz, *Advan. Organometal. Chem.*, **1**, 239 (1964).

[25] (a) H. C. Brown and P. A. Tierney, *J. Am. Chem. Soc.*, **80**, 1552 (1958); (b) D. F. Gaines, *Inorg. Chem.*, **2**, 523 (1963).

[26] C. J. Ballhausen and J. P. Dahl, *Acta Chem. Scand.*, **15**, 1333 (1961).

[27] R. G. Pearson, *J. Am. Chem. Soc.*, **85**, 3533 (1963).