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REACTIONS OF COORDINATED MOLECULES

XXIII *. THE PREPARATION OF SEVERAL METALLA-β-DIKETONATE COMPLEXES OF BORON

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Summary

When the metalla-β-diketone molecules, [*cis*-$(OC)_4Re(CH_3CO)(RCO)$]H (where R is methyl, isopropyl or benzyl) and [$(C_5H_5)(OC)Fe(CH_3CO)(RCO)$]H (where R is methyl or isopropyl), are treated with anhydrous trigonal boron compounds of the type BX_2Y (where X is a halogen and Y is either halogen or phenyl) the corresponding (metalla-β-diketonato)B(X)(Y) complexes are formed. Twelve such complexes are reported. Also, when the mangana-acetylacetonate anion, [*cis*-$(OC)_4Mn(CH_3CO)_2$]Li, is treated with gaseous BF_3, the corresponding (mangana-acetylacetonato)BF_2 complex is formed. The preparation, characterization and chemical reactivity of these complexes are discussed.

Introduction

When the β-diketones are treated with the boron trihalides, the reaction with BF_3 affords the (β-diketonato)BF_2 complex while the other boron trihalides give only the corresponding *bis*-(β-diketonato) boronium salts [2]. However, we recently reported that the reaction of the rhena-acetylacetone molecule, [*cis*-$(OC)_4Re(CH_3CO)_2$]H, with all four boron trihalides afforded only the boron dihalide complexes, [*cis*-$(OC)_4Re(CH_3CO)_2$]BX_2 [3]. Presumably the metalla-acetylacetonate ligand is too weak a Lewis base to displace the remaining two halogen atoms to form the boronium complexes.

Subsequent to the above report, we realized from a comprehensive ^{13}C NMR study of the metalla-β-diketonate molecules that the acyl-carbon resonances of the above boron dihalide complexes appeared at ca. 16 ppm to lower field than the corresponding resonances of the metalla-β-diketone molecules [4]. This downfield shift might indicate that the acyl-carbon atoms in the boron com-

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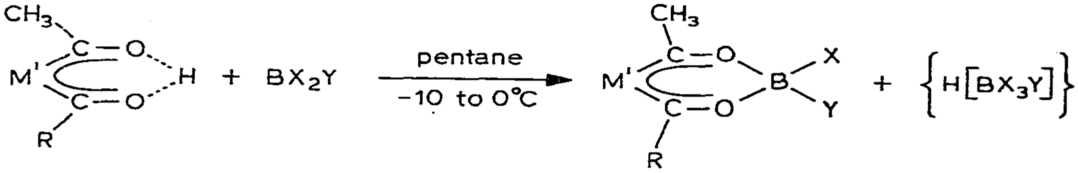
* For Part XXII see ref. 1.

plexes are more positively charged than the acyl-carbon atoms in the metalla-β-diketone molecules, and may imply that the (metalla-β-diketonato)boron dihalide complexes are more suitable reagents for anticipated condensation chemistry than are the metalla-β-diketones. We now wish to report an extended study of the preparation of these rhena-β-diketonato boron complexes. The ^{13}C NMR spectra of several of these compounds were reported earlier [4].

We realized that the greater thermal and air stability of the (rhena-β-diketonate) boron dihalide complexes in comparison to the corresponding rhena-β-diketones might enable the isolation of ferra- and mangana-β-diketonato boron complexes even when the initial metalla-β-diketones of these metals were known to be very air sensitive or were never isolated. We report here the successful preparation of these ferra and mangana complexes which may be useful as new synthetic reagents.

Results and discussion

The rhena- and ferra-β-diketonato boron complexes Ia—Va were prepared by treating a cold pentane solution of the metalla-β-diketone with two molar-equivalents of the appropriate trigonal boron compound as shown below:



(I: M′ = $(OC)_4Re$, R = CH_3; (Ia: X = Y = F
Ib: X = Y = Cl
Ic: X = Y = Br
Id: X = Y = I
Ie: X = Cl; Y = Ph;
II: M′ = $(OC)_4Re$, R = i-Pr; IIa: X = Y = F
IIb: X = Y = Cl
IIc: X = Y = Br
IId: X = Y = I
IIe: X = Cl; Y = Ph;
III: M′ = $(OC)_4Re$, R = $PhCH_2$; IIIa: X = Y = F
IIIb: X = Y = Cl
IIIc: X = Y = Br
IIId: X = Cl; Y = Ph;
IV: M′ = $(\eta^5\text{-}C_5H_5)(OC)Fe$, R = CH_3; IVa: X = Y = F
V: M′ = $(\eta^5\text{-}C_5H_5)(OC)Fe$, R = i-Pr) Va: X = Y = F)

The boron complexes crystallize from organic solvents as white to orange-colored solids, and are obtained in 20—65% yield.

The rhena-β-diketonato boron complexes are air stable for at least several days. The rhena-acetylacetonato boron dihalide complexes Ia—Id were reported previously [3]. Except for the thermally unstable diiodide complex, Id, the complexes Ia—Ie melt or decompose at temperatures from 27—76°C higher than the rhena-acetylacetone molecule I. A similar trend is observed for the rhena-isobutyrylacetonato boron complexes IIa—IIe which melt or decompose from 52—59°C higher than the rhena-enol complex II. The rhena-benzyrylacetonate complexes IIIa—IIIc melt or decompose at temperatures from 13—28°C higher than complex III. Complex IIId has an unusually broad decomposition range.

The thermal stability of these complexes appears to be dependent on the various substituents within the molecule. The boron diiodide complexes could be isolated when R is methyl or isopropyl, but not when R is benzyl. This instability may not arise completely from steric factors since the complexes Id and IId also decompose rapidly even in the solid state with the elimination of iodine. The $B(C_6H_5)(Cl)$ complexes, where R is isopropyl or benzyl, are less stable than when R is methyl. This trend in thermal stabilities might be caused by steric forces since the isopropyl complex IIe appears to be the least stable complex of this type.

The IR spectra of the rhena-β-diketonate boron complexes exhibit the expected four-band pattern in the terminal, carbonyl C—O stretching region [3]. These bands appear at ca. 30 cm^{-1} to higher frequency than the same vibrational bands of the rhena-β-diketone complexes [5]. Presumably, the B(X)(Y) unit is more electron withdrawing than is the enolic proton.

An interesting feature of these spectra is that the axial carbonyl B_1 bands are shifted ca. 15 cm^{-1} to lower energy relative to the position of the equatorial carbonyl-A_1 bands [6]. This frequency separation is only ca. 7 cm^{-1} for the rhena-β-diketone molecules. A similarly large shift has been observed in the spectrum of the bis(diacetyltetracarbonylrhenium)copper complex, and a subsequent X-ray structural determination revealed that the atoms defining the chelate rings of the rhena-acetylacetonate ligands are nonplanar [1]. Although these rather large frequency separations may be due to electronic effects, this analogy may imply that the chelate rings of these rhena-β-diketonato boron complexes are significantly nonplanar, also.

The ^1H NMR spectra show that the protons on the carbon atoms which are bonded to the acyl carbon atoms resonate at lower fields than do the corresponding protons of the metalla-β-diketone molecules. The chelate-ring methyl resonances of the boron complexes appear between (δ) 2.90 and 3.12 ppm which represents a downfield shift of ca. 0.25 ppm. The position of this methyl resonance is a diagnostic indication of the formation of these rhena-β-diketonato boron complexes. The resonances of the methine protons of the isopropyl groups, and the benzylic protons of the benzyl groups appear at 0.25—0.49 ppm to lower field than do the corresponding resonances of the rhena-β-diketones. These downfield shifts are consistent with the greater electron withdrawal by the B(X)(Y) groups. The anisochronism [7] observed for the methyl groups of the isopropyl substituent of IIe and for the benzylic protons of IIId is consistent with the C_1 symmetry of these complexes.

The ^{13}C NMR spectra of I, Ia, Ib, Ie, II, IIa, IIe and IIIa were reported previously [4]. These spectra confirm the idealized molecular symmetries of these complexes in the solution phase. For example, complexes I, Ia and Ib have C_{2v} symmetry while complexes Ie, II, IIa and IIIa have C_s symmetry, and complex IIe possesses only C_1 symmetry.

If the chelate rings within these rhena-β-diketonato boron complexes are significantly nonplanar forming either chair- or boat-shaped structures, then the complexes Ie, IIe and IIId should exhibit geometrical isomerism, and the methyl groups of the isopropyl substituents and the benzylic protons of each isomer should be nonequivalent. Since the recorded ^{1}H and ^{13}C NMR spectra are consistent with a planar rhena-β-diketonate ring, the expected anisochronism is either not resolved experimentally or it is time-averaged due to the rapid interconversion of two or more isomers. The only evidence supporting a nonplanar chelate ring structure for these rhena complexes is the tenuous IR evidence mentioned above. The consideration of a boat-shaped structure for these rhena compounds is based on the known structural features of the ferra-β-diketonate boron complexes.

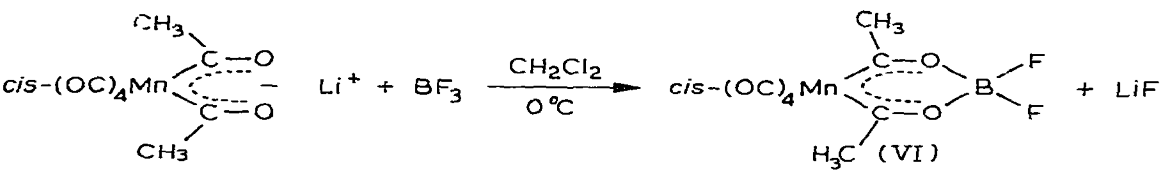
The ferra-β-diketonate boron difluoride complexes IVa and Va are significantly more stable to air and heat than are the parent complexes IV and V. The melting point of IVa is 60°C higher than that of IV, and while complex Va begins to decompose at 126°C, complex V exists as an oil at ambient temperature. Compounds IV and V decompose rapidly upon exposure to the atmosphere while the boron complexes IVa and Va are air stable for at least a week.

The IR spectra of solutions of IVa and Va show two carbonyl stretching vibrations. When a crystalline sample of each complex is dissolved in CS_2, two carbonyl bands of nearly equal intensity are observed. Upon removing the CS_2 from the sample solutions and completely redissolving the residue in CH_2Cl_2, the two band pattern is observed again, but the relative intensities have changed to ca. 1.5/1. The reduced intensity of the low energy band due to a solvent change strongly indicates the presence of equilibrating, geometrical isomers. The most reasonable molecular structures that would generate two geometrical isomers involve a chair- or boat-shaped ferra-β-diketonate chelate ring. Preliminary data from a single crystal X-ray structural determination of complex Va confirms a distinct boat-shaped molecular geometry [8].

The ^{1}H NMR spectrum of IVa and Va show singlet resonances for the methyl and cyclopentadienyl groups indicating a rapid interconversion of the two geometrical isomers on the NMR time scale. These resonances exhibit an average downfield shift of ca. 0.23 ppm relative to the corresponding resonances of IV and V due, presumably, to the greater electronegativity of the BF_2 groups. Surprisingly, the methyl resonances of the isopropyl substituent of Va appear as a single doublet. Two doublets are expected because these methyl groups are nonequivalent at all rotational positions of the isopropyl group. If the ferra-β-diketonate ligand ring were planar, then the methyl groups of the isopropyl substituent would experience the two very different environments of the C_5H_5 and the CO ligands. However, the X-ray structure reveals that the isopropyl substituent is located below the boat-shaped, ferra-ligand ring, and apparently, each methyl group experiences a very similar environment which is dominated primarily by the carbonyl ligand. Any anisochronism between these methyl groups must be less than 3 Hz which is the peak width at half height for the two resonances defining this doublet.

The mangana-acetylacetonato boron difluoride complex VI is prepared directly from the mangana-acetylacetonate anion [9] as shown below:

Compound VI is a yellow solid which melts at 120°C. It can be isolated in a 41% yield, and it appears to be air stable for at least a day. The IR and ^{1}H NMR



data of VI are very similar to that of the rhena analogues. If the metalla-β-diketonato boron complexes react similarly to the metalla-β-diketones, then compounds like VI represent the only route available for preparing mangana derivatives since the mangana-β-diketones can not be prepared using the normal procedures.

When a pentane solution of the rhena-acetylacetone molecule I is treated with two molar-equivalents of $BH_3 \cdot THF$, the BH_2 analogue to Ia is not formed. The off-white solid which is isolated from this reaction appears to be $H_3Re_3(CO)_{12}$ based on the IR and ^1H NMR spectra and the elemental analysis [10]. The fate of the acetyl ligands was not determined. The rhenium hydride cluster was isolated in a 48% yield, and although the expected BH_2 complex was not prepared, this reaction might be useful for the synthesis of other hydrido-polymetallic cluster compounds.

The boron dihalide moiety in these compounds is quite inert chemically. A 5/1 hexane/CH_2Cl_2 solution of the boron dichloride complex IIIb did not react with excess acetylacetone during four hours at ambient temperature. However, a trigonal boronium complex is formed, presumably, when Ib is treated with $AgBF_4$ in THF solution. Silver chloride precipitates from this reaction solution with the concomitant polymerization of the THF. The THF polymerization is probably initiated by the presence of a trigonal boronium cationic complex. The reaction of Ib with sodium methoxide or sodium thiophenolate afforded only recovered starting material. The inertness of these boron dihalide moieties to mildly nucleophilic and moderately basic reagents may indicate that these metalla-β-diketonate complexes of boron might be able to survive reaction conditions that would decompose or deprotonate the metalla-β-diketones.

Experimental

All reactions and other manipulations were performed under dry, prepurified nitrogen at 25°C, unless otherwise stated. Solvents were distilled from Na/K alloy or P_2O_5 (halogenated solvents) before use. Complexes I—V and Ia—Id were prepared by literature methods [3,4]. BF_3 (Research Organic/Inorganic Chemical Corp.), BCl_3 (Matheson Gas Products), BBr_3, BI_3, $BPhCl_2$ (Ventron Corp.), $BH_3 \cdot THF$ (Aldrich Chemical Co.) and all other reagents were used as purchased.

Infrared spectra were recorded on a Perkin-Elmer 727 spectrometer in 0.10 mm sodium chloride cavity cells using the solvent as a reference and polystyrene film as a calibration standard. ^1H NMR spectra were recorded on a Jeol MH-100 spectrometer using TMS as an internal reference and $CDCl_3$ as the solvent, unless otherwise stated. Microanalyses were performed by Galbraith Laboratories, Inc., Knoxville, Tennessee.

General Preparation of the [cis-$(OC)_4Re(CH_3CO)(RCO)]B(X)(Y)$ Complexes Ia—IIId

To a solution of 0.10—0.25 g of I, II or III in 30 ml of pentane at —10°C was added an amount in slight excess of two molar-equivalents of the appropriate boron reagent as a gas (BF_3 and BCl_3), liquid (BBr_3 and $BPhCl_2$) or solid (BI_3). The product precipitated as a white or yellow powder during the mixing of the reagents. The reaction mixture was stirred at —10°C for 20 min., and then was warmed to 25°C. After stirring for 20 min. at 25°C, the solvent was removed at reduced pressure. The products were extracted into and were crystallized from a minimum amount of ether or toluene. Specific data for each new compound are provided below.

[cis-$(OC)_4Re(CH_3CO)_2]B(Ph)Cl)$, (Ie). Light yellow solid (42%): dec. 106—110°C; IR(C_5H_{12}): ν(CO) 2120m, 2030s, 2020vs, 1980vs cm^{-1}; ^{1}H NMR: δ 3.09 (singlet, 6, CH_3); 7.44—7.77 (multiplet, 5, Ph) ppm. Anal. Found: C, 32.96; H, 2.30; B, 2.26; Cl, 6.89. $C_{14}H_{11}BClO_6Re$ calcd.: C, 33.12; H, 2.18; B, 2.13; Cl, 6.98.

[cis-$(OC)_4Re(CH_3CO)(i\text{-}PrCO)]BF_2$, (IIa). White solid (65%): m.p. 95—95.8°C; IR (C_6H_{12}): ν(CO) 2120m, 2030s, 1998vs, 1978vs; ν($C\overset{\cdots}{-}O$) 1481m cm^{-1}; ^{1}H NMR: δ 1.04 (doublet, J = 7 Hz, 6, Me_2HC); 2.90 (singlet, 3, CH_3); 3.53 (heptet, J = 7 Hz, 1, HC) ppm. Anal. Found: C, 26.10; H, 2.15; F, 8.11. $C_{10}H_{10}BF_2O_6Re$ calcd.: C, 26.04; H, 2.19; F, 8.24.

[cis-$(OC)_4Re(CH_3CO)(i\text{-}PrCO)]BCl_2$, (IIb). Light yellow solid (53%): dec. 109—120°C; IR (hexane): ν(CO) 2120m, 2025s, 2010vs, 1980vs cm^{-1}; ^{1}H NMR: δ 1.10 (doublet, J = 7 Hz, 6, Me_2HC); 3.06 (singlet, 3, CH_3); 3.71 (heptet, J = 7 Hz, 1, HC) ppm. Anal. Found: C, 24.50; H, 2.13; Cl, 14.55. $C_{10}H_{10}BCl_2O_6Re$ calcd.: C, 24.31; H, 2.04; Cl, 14.35.

[cis-$(OC)_4Re(CH_3CO)(i\text{-}PrCO)]BBr_2$, (IIc). Yellow solid (40%): dec. 102—104°C; IR (C_5H_{12}): ν(CO) 2110m, 2030s, 2025vs, 1985vs cm^{-1}; ^{1}H NMR: δ 1.10 doublet, J = 7 Hz, 6, Me_2HC); 3.08 (singlet, 3, CH_3); 3.73 (heptet, J = 7 Hz, 1, HC) ppm. Anal. Found: C, 21.05; H, 1.79. $C_{10}H_{10}BBr_2O_6Re$ calcd.: C, 20.60; H, 1.73.

[cis-$(OC)_4Re(CH_3CO)(i\text{-}PrCO)]BI_2$, (IId). Yellow solid which decomposes rapidly even in the solid state; IR(C_5H_{12}): ν(CO) 2120m, 2035s, 2025vs, 1985vs cm^{-1}; ^{1}H NMR: δ 1.10 (doublet, J = Hz, 6, Me_2HC); 3.12 (singlet, 3, CH_3); 3.76 (heptet, J = 7 Hz, 1, HC) ppm.

[cis-$(OC)_4Re(CH_3CO)(i\text{-}PrCO)]B(Ph)(Cl)$, (IIe). White solid (31%): m.p. 95—98°C; IR(C_6H_{14}): ν(CO) 2100m, 2025s, 2000vs, 1975vs cm^{-1}; ^{1}H NMR: δ 1.10, 1.14 (pair of doublets, J = 7 Hz, 6, Me_2HC); 2.96 (singlet, 3, CH_3); 3.59 (heptet, J = 7 Hz, 1, HC); 7.13, 7.47 (multiplets, 5, Ph) ppm. Anal. Found: C, 34.97; H, 1.98. $C_{16}H_{15}BClO_6Re$ calcd.: C, 35.87; H, 2.82.

[cis-$(OC)_4Re(CH_3CO)(PhCH_2CO)]BF_2$, (IIIa). Yellow solid (55%): m.p. 100—100.5°C; IR(C_6H_{12}): ν(CO) 2120m, 2025s, 2010vs, 1975vs cm^{-1}; ^{1}H NMR: δ 2.91 (singlet, 3, CH_3); 4.37 (singlet, 2, CH_2); 7.30 (multiplet, 5, Ph) ppm. Anal. Found: C, 33.17; H, 1.94; F, 7.64. $C_{14}H_{10}BF_2O_6Re$ calcd.: C, 33.02; H, 1.98; F, 7.46.

[cis-$(OC)_4Re(CH_3CO)(PhCH_2CO)]BCl_2$, (IIIb). Light yellow solid (47%): dec. 107—116°C; IR(C_5H_{12}): ν(CO) 2120m, 2030s, 2020s, 1985vs cm^{-1}; ^{1}H NMR: δ 3.00 (singlet, 3, CH_3); 4.44 (singlet, 2, CH_2); 7.34 (multiplet, 5, Ph) ppm.

Anal. Found: C, 30.86; H, 1.96; Cl, 12.71. $C_{14}H_{10}BCl_2O_6Re$ calcd.: C, 31.01; H, 1.86; Cl, 13.08.

[cis-(OC)$_4$Re(CH$_3$CO)(PhCH$_2$CO)]BBr$_2$, (IIIc). Yellow solid (25%): dec. 92—100°C; IR(C_6H_6): ν(CO) 2115m, 2030vs, 2020vs, 1985vs cm^{-1}; ^{1}H NMR: δ 2.96 (singlet, 3, CH_3); 4.47 (singlet, 2, CH_2); 7.31 (multiplet, 5, Ph) ppm. Anal. Found: C, 26.49; H, 1.73; Br, 24.08. $C_{14}H_{10}BBr_2O_6Re$ calcd.: C, 26.64; H, 1.60; Br, 25.33.

[cis-(OC)$_4$Re(CH$_3$CO)(PhCH$_2$CO)]B(Ph)(Cl), (IIId). Yellow solid (20%): dec. 75—95°C; IR(C_6H_{14}): ν(CO) 2120m, 2030s, 2015s, 1980vs cm^{-1}; ^{1}H NMR: δ 3.02 (singlet, 3, CH_3); 4.50, 4.52 (2 singlets, 2, CH_2); 7.46, 8.35 (multiplets, 10, 2 C_6H_5) ppm. Anal. Found: C, 42.32; H, 2.83. $C_{22}H_{15}BClO_6Re$ calcd.: C, 41.15; H, 2.59.

General preparation of the [(C$_5$H$_5$)(OC)Fe(CH$_3$CO)(RCO)]BF$_2$ complexes IVa and Va

To a solution of the appropriate ferra-β-diketone in 70 ml of pentane at 0°C was added BF_3 gas by bubbling the BF_3 into the solution for 2 min. The reaction solution was stirred at 0°C for 10 min, and at 25°C for 20 min. During this time a red or brown oil formed, and then the solvent was removed at reduced pressure. The products were extracted into and were crystallized from ether solution. Specific data are provided below.

[(C$_5$H$_5$)(OC)Fe(CH$_3$CO)$_2$]BF$_2$, (IVa). Orange solid (20%): m.p. 147—148°C; IR (CH_2Cl_2): ν(CO) 2010vs, 1975s; (in CS_2) 2020s, 1975s cm^{-1}; ^{1}H NMR: δ 2.88 (singlet, 6, 2 CH_3); 5.10 (singlet, 5, C_5H_5); (in CS_2) 2.87 (singlet, 6, 2 CH_3); 4.98 (singlet, 5, C_5H_5) ppm. Anal. Found: C, 42.43; H, 3.92. $C_{10}H_{11}BF_2FeO_3$ calcd.: C, 42.31; H, 3.91.

[(C$_5$H$_5$)(OC)Fe(CH$_3$CO)(i-PrCO)]BF$_2$, (Va). Yellow solid (58%): dec. 126—130°C; IR (CH_2Cl_2): ν(CO) 2020vs, 1975vs; (in CS_2) 2020s, 1965vs cm^{-1}; ^{1}H NMR: δ 1.04 (doublet, J = 7 Hz, 6, *Me*$_2$HC); 2.88 (singlet, 3, CH_3); 3.71 (heptet, J = 7 Hz, 1, HC); 5.00 (singlet, 5, C_5H_5) ppm. Anal. Found: C, 46.25; H, 4.85. $C_{12}H_{15}BF_2FeO_3$ calcd.: C, 46.21; H, 4.85.

Preparation of [cis-(OC)$_4$Mn(CH$_3$CO)$_2$]BF$_2$, (VI). Gaseous BF_3 was bubbled through a CH_2Cl_2 solution of [*cis*-(OC)$_4$Mn(CH$_3$CO)$_2$]Li at 0°C for three min. The reaction solution was stirred at 25°C for 20 min, and then the solvent and excess BF_3 were removed at reduced pressure. The product was extracted into and was crystallized from a toluene solution in 41% yield as a yellow solid: m.p. 120—121; IR (C_6H_{12}) ν(CO) 2100m, 2040s, 2010vs, 1990s cm^{-1}; ^{1}H NMR: δ 3.05 (singlet, CH_3) ppm. Anal. Found: C, 31.62; H, 2.12. $C_8H_6BF_2O_6Mn$ calcd.: C, 31.83; H, 2.00.

Reaction of I with BH$_3$ · THF. A solution of 0.27 g (7 mmol) of I in 60 ml of pentane was cooled to —7°C (ice/acetone), and 2 molar equivalents of $BH_3 \cdot$THF was added to the solution. The reaction solution was warmed to 25°C, and was stirred for 2 h. The solvent was removed at reduced pressure, and the products was extracted into and was crystallized from CH_2Cl_2 as an off-white solid (48%) identified as H_3Re_3 $(CO)_{12}$: dec. 195—200°C; IR(C_5H_{12}) ν(CO) 2099m, 2035s, 2010s, 1975s cm^{-1}; ^{1}H NMR: δ — 17.4 (singlet, H) ppm. Anal. Found: C, 16.03; H, 0.44. $C_{12}H_3O_{12}Re_3$ calcd.: C, 16.04; H, 0.34.

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References

1 P.G. Lenhert, C.M. Lukehart and L.T. Warfield, in press.
2 W. Dilthey, F. Eduardof and F.J. Schumacher, Ann., 344 (1906) 300.
3 C.M. Lukehart and L.T. Warfield, Inorg. Chem., 17 (1978) 201.
4 K.P. Darst and C.M. Lukehart, J. Organometal. Chem., 161 (1978) 1.
5 C.M. Lukehart and J.V. Zeile, J. Amer. Chem. Soc., 99 (1977) 4368.
6 C.M. Lukehart and G.P. Torrence, J. Chem. Soc., Dalton, (1978) 93.
7 W.B. Jennings, Chem. Rev., 85 (1975) 307.
8 P.G. Lenhert, C.M. Lukehart and L.T. Warfield, work in progress.
9 C.M. Lukehart, G.P. Torrence and J.V. Zeile, Inorg. Synth., 18 (1978) 56.
10 M.A. Andrews, S.W. Kirtley and H.D. Kaesz, Inorg. Synth., 17 (1977) 66.