The reaction between $Mo_2(OCMe_3)_6$ and 1-pentyne (1:10) in the presence of 8 equiv of quinuclidine in pentane (4 h) yields a pentane-soluble crude reaction mixture that contains $Mo(CH)(OCMe_3)_3(quin)$ and what can be identified as $Mo(CPr)(OCMe_3)_3(quin)$ by comparison of its ^1H NMR spectrum with that of an authetic sample[7b] in ~50% total yield by ^1H NMR (vs. CH_2Cl_2). The formation of oily oligomers has thus far prevented isolation of the product. In the absence of quinuclidine the reaction between $Mo_2(OCMe_3)_6$ and 1-pentyne yields oils (thought to be 1-pentyne oligomers) that contain $Mo(CPr)(OCMe_3)_3$ (δ-(*C*Pr) 286.6 in C_6D_6) in about 30% yield by ^1H NMR.[7b]

Addition of 1 equiv of acetylene itself to $Mo_2(OCMe_3)_6$ in the presence or absence of quinuclidine produces what appears to be $Mo_2(OCMe_3)_6(\mu\text{-}C_2H_2)$ by ^{13}C NMR. In particular, the acetylenic carbon atom signal is found at 127.1 ppm in dioxane-d_8 with J_{CH} = 198 Hz and the acetylene proton signal at 5.99 ppm in C_6D_6 (cf. $\mu\text{-}C_2H_2$ complexes of tungsten; ref 3c and references therein). The conversion of $Mo_2(OCMe_3)_6$ to $Mo_2(OCMe_3)_6(\mu\text{-}C_2H_2)$ in pentane is high, but attempted recrystallization of the crude red solid reaction product yields more and more $Mo_2(OCMe_3)_6$, apparently due to loss of acetylene from $Mo_2(OCMe_3)_6(\mu\text{-}C_2H_2)$. This reaction is far from definitive but provides some evidence that the first step in the cleavage of the $Mo\equiv Mo$ bond by an acetylene is addition of the acetylene to give a tetrahedral Mo_2C_2 core, as all evidence to date overwhelmingly suggests.[3]

The reactions outlined here do not appear to be as selective for scission of triple bonds as those involving $W_2(OCMe_3)_6$.[2] Apparently other reactions (acetylene dimerization, etc.) compete effectively with the scission reaction. Nevertheless, we have at least demonstrated that the principles, if not yet the synthetic utility, of $W\equiv W$ bond cleavage extend to the analogous $Mo\equiv Mo$ systems. We should note, however, that so far we have not observed scission of acetonitrile or benzonitrile by $Mo_2(OCMe_3)_6$, a reaction in the tungsten system that is qualitatively at least as fast, if not faster than, scission of acetylenes.

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Registry No. $Mo_2(OCMe_3)_6$, 60764-63-8; $Mo(CPh)(OCMe_3)_3$, 91760-26-8; $Mo(CPh)(OCMe_3)_3(quin)$, 91780-94-8; $Mo(CH)(OCMe_3)_3(quin)$, 91780-95-9; $Mo(CPh)(OCMe_3)_3(py)$, 91760-27-9; $Mo(CPr)(OCMe_3)_3(quin)$, 91780-96-0; $Mo_2(OCMe_3)_6(\mu\text{-}C_2H_2)$, 91760-28-0; $Mo(CCMe_3)(OCMe_3)_3$, 82209-30-1; $Mo(CPr)(OCMe_3)_3$, 91780-93-7; $PhC\equiv CH$, 536-74-3; quin, 100-76-5; py, 110-86-1; 1-pentyne, 627-19-0; acetylene, 74-86-2.

Selective Reduction of Acyl Chlorides to Aldehydes by Anionic 6B Transition-Metal Hydrides

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Summary: Acyl chlorides can be selectively and rapidly reduced, under mild conditions, by group 6B anionic hydrides, $HM(CO)_4L^-$ (M = Cr, W; L = CO, PR_3), giving the corresponding aldehydes and the metal chlorides $ClM(CO)_4L^-$. The reaction is nearly quantitative for both aliphatic and aromatic acyl chlorides using solvent systems such as dichloromethane, tetrahydrofuran, or acetonitrile. Only in the presence of acid will the aldehydes consume a second equivalent of hydride, subsequently being reduced to alcohols. The anionic hydrides selectively reduce acyl chlorides in the presence of other reducible groups such as alkyl bromides or nitro aromatics. In situ preexchange of hydrogen by deuterium ($HM(CO)_4L^-/CH_3OD \rightarrow DM(CO)_4L^-/CH_3OH$) allows for deuterium delivery, giving RCDO products, as indicated by ^2H NMR.

The monomeric group 6B transition-metal hydrides $HM(CO)_4L^-$, where M = Cr or W and L = CO or PR_3, reduce alkyl halides, with reactivities approaching that of the powerful main-group metal hydrides such as Brown's HBR_3^- reagents.[2] We have now learned that the more reactive acyl chlorides can be selectively reduced to the corresponding aldehydes without further reduction to alcohols—a complication which is unavoidable with Brown's HBR_3^- reagents.[3] This exceptional utility of group 6B hydrides for selective reduction of acyl chlorides has prompted this preliminary report.

Acyl chlorides react within the time of mixing with the group 6B hydrides $HM(CO)_4L^-$ at 25 °C to generate the corresponding aldehydes and the metal chlorides $ClM(CO)_4L^-$ as described in eq 1.[4,5] The organometallic

$$RC(=O)Cl + HM(CO)_4L^- \rightarrow RC(=O)H + ClM(CO)_4L^- \quad (1)$$

product $ClM(CO)_4L^-$ was identified by its IR spectrum, e.g., for $ClCr(CO)_5^-$, ν_{CO} (cm^{-1}) = 2058 (w), 1932 (s), and 1864 (m). The conversion from acyl chloride to aldehyde was monitored in every case by IR and ^1H NMR spectroscopies as well as by GC analysis. In most cases, the deuterium-labeled aldehyde (vide infra) was also identified by ^2H NMR. The benzaldehyde product of reduction of benzoyl chloride was positively identified by GC/MS analysis. Yields as checked by GC analysis always agreed with those checked by spectroscopic means. As shown in Table I, the reaction is nearly quantitative for both aliphatic and aromatic acyl chlorides using solvent systems such as dichloromethane, tetrahydrofuran, or acetonitrile. One reduction (benzoylchloride/$HCr(CO)_5^-$) was done at the synthetic level (0.5 g of $PPN^+HCr(CO)_5^-$, 0.050 g of C_6H_5COCl in 25 mL of THF). The THF solvent was removed by trap to trap distillation (liquid N_2 as trappant) and the benzaldehyde product separated from the oily orange residue by hexane extraction. Following filtration through a short Celite column the hexane was removed by trap to trap distillation. Benzaldehyde, free of any organometallic products, was thus isolated in 59% yield (nonoptimized) based on benzoyl chloride.

To take advantage of the facile hydrogen–deuterium exchange reaction of eq 2,[6] the acyl chlorides were reacted

$$HM(CO)_4L^- + CH_3OD \rightleftharpoons DM(CO)_4L^- + CH_3OH \quad (2)$$

with the hydrides (ca. 0.1 mmol) in THF/CH_3OD, (5 mL

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[1] On leave from the Department of Chemistry, The College of Wooster, Wooster, OH.

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Table I. Products of Acyl Chloride Reductions by Anionic 6B Metal Hydrides in THF Solvent (25 °C)[a]

entry	acid chloride	$HM(CO)_4L^-$	reductn product	yield,[b] %
1	CH_3COCl	$HCr(CO)_5^-$	CH_3CHO	90
2	$(CH_3)_2CHCOCl$	$HCr(CO)_5^-$	$(CH_3)_2CHCHO$	93
3	$CH_3(CH_2)_3COCl$	$HW(CO)_5^-$	$CH_3(CH_2)_3CHO$	95
4	$CH_3(CH_2)_3COCl$	$HCr(CO)_5^-$	$CH_3(CH_2)_3CHO$	95
5[c]	$CH_3(CH_2)_3COCl$	$HCr(CO)_5^-$	$CH_3(CH_2)_3CDO$	95
6[d]	$CH_3(CH_2)_3COCl$	$HCr(CO)_5^-$	$CH_3(CH_2)_3CH_2OH$	90
7	PhCOCl	$HW(CO)_5^-$	PhCHO	92
8	PhCOCl	$HW(CO)_4P(OMe)_3^-$	PhCHO	50
9	PhCOCl	$HCr(CO)_5^-$	PhCHO	95
10[e]	PhCOCl	$HCr(CO)_5^-$	PhCHO	81
11[f]	PhCOCl	$HCr(CO)_5^-$	PhCHO	70
12[c]	PhCOCl	$HCr(CO)_5^-$	PhCDO	95
13	PhCOCl $PhNO_2$	$HCr(CO)_5^-$	PhCHO	95
14	PhCOCl PhBr	$HCr(CO)_5^-$	PhCHO	90
15	$BrCH_2(CH_2)_2COCl$	$HCr(CO)_5^-$	$BrCH_2(CH_2)_2CHO$	95
16[g]	$BrCH_2(CH_2)_2COCl$	$HCr(CO)_5^-$	$CH_3(CH_2)_2CHO$	90
17	$C_6H_5CH_2COCl$	$HCr(CO)_5^-$	$C_6H_5CH_2CHO$	92

[a] 1:1 molar equivalents of hydride to acyl chloride, except where noted. [b] GC yields, determined by comparison with gravimetric standards of the pure products. [c] Reaction carried out in the presence of MeOD. [d] Two equivalents of $HCr(CO)_5^-$ and 1 equiv of HOAc. [e] CH_2Cl_2. [f] CH_3CN. [g] Two equivalents of $HCr(CO)_5^-$.

of THF/4 μL of CH_3OD). The metal deuterides thus prepared[6] delivered deuterium to the aldehydic carbon, giving RCDO products. The d-labeled aldehydes were identified with ^2H NMR, e.g., for PhCDO, δ 9.95. For the reduction of benzoyl chloride using $HCr(CO)_5^-/CH_3OD$, 1:1 in THF, GC/MS analysis indicated that 50% of the deuterium originally available as CH_3OD was delivered to the PhCDO product. The level of enrichment ultimately available by this in situ method of generating $DM(CO)_4L^-$ (and hence PhCDO) depends on the position of equilibrium in eq 2 and on kinetic isotope effects that may be associated with the reduction.

The reduction of acyl chlorides by the group 6B hydrides can be accomplished in the presence of other reducible groups. Mixtures of nitrobenzene and benzoyl chloride yield benzaldehyde as the sole reduction product in the presence of 1 equiv of $HCr(CO)_5^-$, entry 13, Table I. Benzaldehyde is also selectively produced in the reaction mixture of bromobenzene and benzoyl chloride, entry 14. Even highly reactive aliphatic bromides are tolerated at an equal molar ratio of hydride and substrate: γ-bromobutyraldehyde was generated from the corresponding acyl chloride upon treatment with $HCr(CO)_5^-$; the reduction of the bromo group requires a second equivalent of the hydride, entries 15 and 16.

Reduction of simple acyl chlorides with group 6B hydrides gives no contamination of the aldehyde product from further reduction to alcohols,[7] even in the presence of excess hydride reagent. On treating a THF/CH_3OD solution of *n*-valeryl chloride with 2 equiv of $HCr(CO)_5^-$, the ^2H NMR spectrum of the solution reveals stoichiometric reduction to the aldehyde (δ 9.95 (s)) and one unreacted equivalent of deuteride (δ −6.91 (s)). Integrated intensities of the resonances here indicated that, of the deuterium available as $DCr(CO)_5^-$, >95% of it was incorporated into the aldehyde product. This spectrum remains unchanged for hours, until the proper reagent was added to trap the excess deuteride. Addition of a second equivalent of acyl chloride consumes the remaining deuteride to give a second equivalent of aldehyde. However, when acetic acid-d_1 was added as the trapping reagent, the two singlets that were assigned to the deuteride and aldehyde CDO disappear with a concomitant formation of a signal at δ 3.40, assignable to deuterium of a methylene unit adjacent to the OH group of the alcohol product. GC analysis confirmed the formation of *n*-pentyl alcohol in about 90% yield based on the starting chloride. Thus $HCr(CO)_5^-$ is an effective reducing agent for aldehydes in acidic conditions (eq 3). This new finding broadens the applicability of group 6B hydrides as reducing agents, and the scope of this reaction is currently under investigation.[8]

$$\mathrm{RC({=}O)H + HCr(CO)_5^- \xrightarrow{HOAc} RCH_2OH + AcOCr(CO)_5} \tag{3}$$

Other methods for the selective reduction of acyl chlorides to aldehydes include use of the sterically hindered lithium tri-*tert*-butoxyaluminum hydride[9] and of sodium borohydride in dimethylformamide, either at low temperature[10a] or in the presence of pyridine,[10b] as well as cadmium or copper dichloride.[11] Similar selectivity can also be obtained with complex copper borohydrides[12] or complex copper cyanoborohydrides.[13] The transformation of acyl halides to aldehydes has also been achieved with tributyltin hydride catalyzed by zerovalent palladium complexes.[14] Transition-metal reductants such as $Fe(CO)_4^{2-}$,[15] $HFe(CO)_4^-$,[16] or $CpV(CO)_3H^-$,[17] have also been shown to be useful for such reactions. Some of these methods employ relatively mild conditions and offer good yields. The unique feature of the group 6B reagents, however, is their capacity for controlled reduction (i.e., acyl chloride to aldehyde and subsequent reduction of the aldehyde to alcohol only if acid is added) and their con-

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(7) This statement assumes the acyl chloride to be free of any contaminant acid. If acid is present, the aldehyde will be further reduced to alcohol.

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venience in providing deuterated aldehydes and alcohols through the in situ hydrogen–deuterium exchange of the hydride reagent prior to reduction. The explicit development of anionic transition-metal hydrides as useful synthetic reagents continues to be an attractive goal since, in principle at least, they can be regenerated from inexpensive starting materials: CO and base.

The mechanisms associated with the reduction of acyl chlorides by transition-metal hydrides is still a subject of debate. The reaction of $HFe(CO)_4^-$ and acyl chloride is claimed to involve initial displacement of chloride by $HFe(CO)_4^-$ to form an acyl hydride complex, followed by reductive elimination of aldehyde.[16] The reaction of $CpV(CO)_3H^-$ with acyl chloride has been studied quite extensively, but due to the complex nature of the reaction, the authors failed to reach a definite conclusion as to mechanism.[17] Even the seemingly well-established radical chain process for tributyltin hydride reductions has recently been challenged.[18] We have examined the reaction between $HCr(CO)_5^-$ and $C_6H_5CH_2COCl$ and found neither $C_6H_5CH_3$ nor $C_6H_5CH_2COOCH_2C_6H_5$ among the products (entry 17). Observation of either product can serve as an indication of a radical process.[19] We have also noted a correlation between the rates of acyl chloride reductions and hydridic activity (i.e., the apparent charge polarization in the $M^{\delta+}–H^{\delta-}$ bond as implied by ion-pairing studies[20] and hydride-transfer abilities[21]). The relative rate of reaction of $C_6H_5CH_2COCl$ with a range of different transition-metal hydrides was found to be $HW(CO)_4P(OMe)_3^- > HW(CO)_5^- > HCr(CO)_5^- > HFe(CO)_4^-$.[22] This order of reactivity also parallels the hydride-transfer ability of these hydrides toward the reduction of nonhindered alkyl halides.[2] We are inclined to expect that ionic (hydride-transfer) character is associated with the key step in the reductions reported here: however conjecture as to possible intermediates is premature.

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(22) This order established by competitive reaction of an equimolar mixture of the hydrides with a deficiency of acyl chloride.

Reaction of (DPPE)Pt(CH$_3$)(OCH$_3$) with Tetrafluoroethylene. Synthesis and Structural Characterization of (DPPE)Pt(CH$_3$)(CF$_2$CF$_2$OCH$_3$)[†]

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Summary: In order to study the feasibility of catalytic olefin hydration, we prepared (DPPE)Pt(CH_3)(OCH_3) (**1**) by metathesis with $NaOCH_3$ on the (DPPE)Pt(CH_3)Cl precursor. A mixed benzene/methanol solvent system typically resulted in isolated yields of 70%. Complex **1** crystallizes from THF/pentane in space group $P2_1/c$ with cell dimensions a = 11.547 (2) Å, b = 13.152 (6) Å, c = 16.658 (5) Å, and γ = 98.51 (2)° with Z = 4. While complex **1** does not react with unactivated olefins, it does generate small amounts of insertion adducts with acrylonitrile and methyl acrylate. Reaction of **1** with tetrafluoroethylene (TFE) generates a single product in quantitative yield that was identified by spectroscopic methods and by X-ray crystallography as (DPPE)Pt(CH_3)($CF_2CF_2OCH_3$) (**2**); the product expected of olefin insertion into the metal–oxygen bond. TFE adduct **2** also crystallizes in space group $P2_1/c$ with cell dimensions a = 11.764 (1) Å, b = 15.503 (2) Å, c = 16.069 (3) Å, and γ = 105.03 (1)° with Z = 4.

The sometimes severe conditions required for acid-catalyzed olefin hydration and the poor high-temperature thermodynamics of such a reaction make a facile transition-metal-catalyzed route attractive. Low-valent metals capable of activating O–H bonds in water[1] present such a possibility for hydration catalysts if they can coordinate olefins and facilitate olefin insertion into metal–oxygen bonds present.[2] So far, this reaction has been a mere speculation in the Wacker literature.[3] We report here the first example of a late transition-metal alkoxide reacting with an olefin to give the product expected of such an insertion reaction.

In order to study these possible steps, we prepared (DPPE)Pt(CH_3)(OCH_3), 1, by metathesis techniques[4] (eq 1). The complex features a chelating bisphosphine ligand

$$\mathrm{(DPPE)Pt(CH_3)Cl + NaOCH_3 \xrightarrow[25\ °C,\ 6\ h]{C_6H_6,\ CH_3OH} (DPPE)Pt(CH_3)(OCH_3)} \quad (1)$$

$$\mathbf{1}\ (75\%\ \text{yield})$$

[†] Contribution No. 3264.

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