bond of 1.[8] Finally, this reaction demonstrates that all steps in a hypothetical catalytic olefin hydration are possible. Investigations into the use of Pt(0) phosphine complexes as olefin hydration catalysts are in progress.

Acknowledgment. The technical competence of M. P. Stepro and the assistance of G. Kruppa and B. Dunn in the acquisition of NMR spectra have been appreciated.

Registry No. 1, 82405-05-8; 2, 91993-68-9; 3, 93503-83-4; C_2F_4, 18251-90-6; perfluorocyclopentene, 559-40-0; perdeuterated tetrahydrofuran, 1693-74-9.

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(8) Which is particularly interesting in light of calculations in: Bäckvall et al. *J. Am. Chem. Soc.* **1984**, *106*, 4369–4373 which suggest hydroxide ligands will always migrate more slowly to coordinated olefins (on four-coordinate Pd complexes) than alkyl ligands.

Hydride Transfer to Transition-Metal Carbonyls in the Gas Phase. The Heat of Formation of $(CO)_4FeCHO^-$

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Summary: The hydride affinity of iron pentacarbonyl, $D[(CO)_4FeCO\text{–}H^-]$, has been determined from binary hydride-transfer reactions in the gas phase to be 56.1 ± 4.0 kcal/mol. From this, the heat of formation of the iron-formyl anion, $\Delta H_f[(CO)_4FeCHO^-,g] = -194.4 \pm 4.3$ kcal/mol, and the associated metal–acyl bond energies, $D[(CO)_4Fe^-\text{–}CHO] = 43.9 \pm 9.1$ kcal/mol and $D[(CO)_4Fe\text{–}CHO^-] = 101.1 \pm 6.1$ kcal/mol, have been derived. The direct reaction of hydride ion with $Fe(CO)_5$ occurs at 40.0% of the collision-limited rate and produces roughly equal amounts of $HFe(CO)_4^-$ and $HFe(CO)_3^-$ as products. The relationship of these data to metal–formyl reactivity in solution is discussed.

Transition-metal formyls and hydrides have received considerable attention over the last decade as key intermediates in the metal-catalyzed reduction of CO and CO_2 by H_2.[1,2] Model formyl complexes have been generated in solutions of the corresponding metal carbonyl and borohydride reagents and have been reported to function as hydride reducing agents in the presence of simple ketones, aldehydes, and other metal carbonyls.[3] While the synthesis and reactivity of metal formyls has been extensively investigated,[4] there are only scant thermochemical data available to guide the rational design off more effective formyl-based catalysts. Recently, a wealth of new thermodynamic data for transition-metal compounds has emerged from studies of organometallic ions in the gas phase.[5] In the course of our own studies of the gas-phase chemistry of anionic metal acyls related to homogeneous catalysis,[6] we have recently observed the simple iron–formyl ion, $(CO)_4FeCHO^-$, as a product of binary hydride-transfer reactions. In this note we report a determination of the hydride affinity of $Fe(CO)_5$ and a derivation of $\Delta H_f[(CO)_4FeCHO^-,g]$.

Our experiments have been carried out at 300 ± 2 K in a flowing afterglow apparatus that has been described in detail previously.[6] Hydride donor anions such as $C_6H_5\bar{C}HCH_3$, HCO_2^-, and $CH_3\bar{C}HCOCH_3$ are produced from proton-transfer reactions between an appropriate neutral precursor ($C_6H_5CH_2CH_3$, HCO_2H, and $CH_3CH_2COCH_3$, respectively) and NH_2^-, the latter being generated by electron impact on NH_3. A fast flow of helium buffer gas[7] carries these ions the length of a 100 cm × 7 cm i.d. flow reactor where they interact with $Fe(CO)_5$ added to the system through a moveable injector. The steady-state ion composition in the flow tube is continuously monitored by a quadrupole mass spectrometer located behind a small sampling orifice.

Before proceeding on to the results for hydride transfers, it is important to first consider the direct reaction of $Fe(CO)_5$ with hydride ion itself. Stable signals of H^- may be produced in the flow reactor from adding excess H_2 to NH_2^- and the ensuing proton transfer. An exceedingly rapid reaction with $Fe(CO)_5$ is observed which yields two hydridoiron carbonyl ions as primary products in roughly equal abundance (eq 1).[8]

$$H^- + Fe(CO)_5 \longrightarrow \begin{cases} \xrightarrow{50\%} HFe(CO)_4^- + CO \\ \xrightarrow{50\%} HFe(CO)_3^- + 2CO \end{cases} \qquad (1)$$

The bimolecular rate coefficient for this reaction has been determined to be $(4.93 \pm 0.66) \times 10^{-9}$ cm^3 molecule^{-1} s^{-1} which indicates, when compared with the ion–molecule collision rate ($k_{\mathrm{Langevin}} = 1.24 \times 10^{-8}$ cm^3 molecule^{-1} s^{-1}),[9] that reaction is occurring on approximately every other encounter. Interestingly, we find that the $HFe(CO)_3^-$ ion undergoes secondary reactions with excess $Fe(CO)_5$ to produce the dinuclear hydrides $HFe_2(CO)_6^-$ and $HFe_2(CO)_7^-$ as well as a fast addition reaction with CO_2 to produce $HFe(CO)_3(CO_2)^-$.[10] In contrast, the 18-electron tetracarbonyl ion $HFe(CO)_4^-$ appears to be relatively inert toward either $Fe(CO)_5$ or CO_2. We have noted similar gas-phase reactivity patterns in other saturated (18 electron) vs. unsaturated (16-electron) $M(CO)_nL^-$ ions.[11]

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Table I. Hydride-Transfer Reactions with $Fe(CO)_5$ (300 K)

anion, XH^-	neutral, X	hydride affinity, HA(X), kcal mol^{-1}	k^{II}_{obsd} [a]	% H^- transfer [b]	other products (%)
$C_6H_5\bar{C}HCH_3$	$C_6H_5CH{=}CH_2$	50.4 ± 2.5	1.08 (0.72)	3.0 (0.03)	$(C_6H_5CHCH_3)Fe(CO)_5^-$ (77) $(C_6H_5CHCH_3)Fe(CO)_4^-$ (10) $HFe(CO)_4^-$ (10)
HCO_2^-	CO_2	51.7 ± 2.5	0.16 (0.08)	13.0 (0.01)	$(HCO_2)Fe(CO)_5^-$ (75) $HFe(CO)_4^-$ (12)
$(CH_3)_2\bar{C}CN$	$CH_2{=}C(CH_3)CN$	56.1 ± 2.5	1.16 (0.67)	5.0 (0.03)	$((CH_3)_2CCN)Fe(CO)_5^-$ (69) $((CH_3)_2CCN)Fe(CO)_4^-$ (23) $HFe(CO)_4^-$ (3)
$CH_3\bar{C}HCN$	$CH_2{=}CHCN$	57.6 ± 2.5	1.16 (0.61)	$\leqslant$0.5 (0.003)	$(CH_3CHCN)Fe(CO)_5^-$ (78) $(CH_3CHCN)Fe(CO)_4^-$ (20) $(CH_3CHCN)Fe(CO)_3^-$ (2)
$CH_3\bar{C}HCOCH_3$	$CH_2{=}CHCOCH_3$	59.3 ± 2.5	1.05 (0.61)	$\leqslant$0.1 (0.0006)	$(CH_3CHCOCH_3)Fe(CO)_5^-$ (100)

[a] Observed bimolecular rate coefficient for disappearance of reactant anion in units of 10^{-9} cm^{-3} $molecule^{-1}$ s^{-1}. Overall reaction efficiency = k(obsd)/k(Langevin) shown in parentheses.[9] [b] Efficiency for hydride-transfer channel shown in parentheses.

Binary hydride-transfer reactions are of particular interest since their occurrence may give rise to useful thermodynamic data pertaining to hydride reductions[12] (i.e., hydride affinities). The hydride affinity of a neutral compound, HA(X), is defined as the enthalpy of dissociation of the corresponding anion HX^- to free hydride and the neutral X (eq 2).[13] These data may be readily cal-

$$HX^- \xrightarrow{\Delta H} H^- + X \qquad (2)$$

$$HA(X) = \Delta H = \Delta H_f(H^-) + \Delta H_f(X) - \Delta H_f(HX^-)$$

culated from known heats of formation of neutrals and the corresponding anions, the latter being derived from gas-phase acidity measurements.[13] From known hydride affinities, values for other neutrals (i.e., $Fe(CO)_5$) may be determined experimentally by bracketing techniques, much the same way as *proton* affinities are bracketed.[13]

Table I presents a summary of results for $Fe(CO)_5$. Hydride transfer yielding the iron–formyl anion (m/z 197) is a minor, though observable reaction channel for the 1-phenylethyl anion, formate, and isobutyronitrile anions, but not for the propionitrile anion and 2-butanone enolate. Rate coefficients and complete primary product distributions were determined[6] for the five reactant anions shown in the table. These data are further supported by qualitative observations involving other anion/neutral pairs which span a 40 kcal/mol range in hydride affinity.[13] The reverse reaction, wherein $(CO)_4FeCHO^-$ donates hydride to a neutral acceptor is not observed under our conditions (eq 3), even when thermodynamically favorable. Thus, the

$$(CO)_4FeCHO^- + X \not\rightarrow Fe(CO)_5 + HX^- \qquad (3)$$

preformed iron formyl ion does not appear to react with $CH_2{=}CHCN$ (HA = 57.6 kcal/mol), $CH_2{=}CHCOCH_3$ (HA = 59.3 kcal/mol), or $CH_2{=}CF_2$ (HA = 63.1 kcal/mol) within the time scale of the flowing afterglow. We prefer a kinetic as opposed to a thermodynamic interpretation for these observations and suggest that the back-reaction (eq 3) is impeded by unfavorable charge localization requirements in $(CO)_4FeCHO^-$.

We therefore assign the hydride affinity of $Fe(CO)_5$ to be 56.1 ± 4.0 kcal/mol.[14] Combining this value with $\Delta H_f[Fe(CO)_5,g] = -173.0 \pm 1.5$ kcal/mol and $\Delta H_f[H^-,g] = 34.7 \pm 0.5$ kcal/mol leads to a heat of formation for the iron formyl, $\Delta H_f[(CO)_4FeCHO^-,g] = -194.4 \pm 4.3$ kcal/mol. We may further derive the two homolytic metal–carbon bond energies, $D[(CO)_4Fe{-}CHO^-] = 101.1 \pm 6.1$ kcal/mol and $D[(CO)_4Fe^-{-}CHO] = 43.9 \pm 9.1$ kcal/mol, from these data.[14] The latter value compares well with other iron–acyl bond energies determined in our laboratory.[15]

Several important implications immediately arise from these results. First of all, they confirm earlier ideas from condensed-phase studies concerning the relative stability of the iron formyl and the corresponding hydride,[3,7] viz., decarbonylation is thermodynamically favorable (eq 4).[14]

$$(CO)_4FeCHO^- \rightarrow (CO)_4FeH^- + CO \qquad (4)$$

$$\Delta H = -10.3 \pm 7.4 \text{ kcal/mol}$$

Moreover, the borohydride-based synthesis of $(CO)_4FeCHO^-$ in solution as well as its reported action as a hydride donor must be strongly influenced by solvation, complexation, and counterion effects since hydride transfer *from* trialkylborohydride ions ($HA(R_3B) \geq 70$ kcal/mol)[16] *to* $Fe(CO)_5$ and *from* $(CO)_4FeCHO^-$ *to* simple aldehydes and ketones ($HA)R_2C{=}O) = 40$–46 kcal/mol)[13] are intrinsically endothermic reactions. This implies that, in addition to changing ligands about the metal, the variation of solvents and/or Lewis acid catalysts would be a fruitful approach to the activation of formyl-based reductions.[17] Further thermochemical studies with other metal formyls

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(15) From $\Delta H_f[(CO)_4FeCOOH^-,g] \leq -257$ kcal/mol[6a] a value for $D[(CO)_4Fe^-{-}COOH] \geq 46.1$ kcal/mol may be derived. For other examples, see: Lane, K. R.; Sallans, L.; Squires, R. R., submitted for publication in *J. Phys. Chem.*

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are currently in progress and will be reported in a future publication.

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Registry No. $C_6H_5C\bar{H}CH_3$, 13822-53-2; HCO_2^-, 71-47-6; $(CH_3)_2C^-CN$, 42117-14-6; CH_3C^-HCN, 42117-12-4; $Fe(CO)_5$, 13463-40-6; $(C_6H_6CHCH_3)Fe(CO)_5^-$, 93473-87-1; $(C_6H_5CHCH_3)Fe(CO)_4^-$, 93473-88-2; $HFe(CO)_4^-$, 18716-80-8; $(HCO_2)Fe(CO)_5^-$, 93473-89-3; $((CH_3)_2CCN)Fe(CO)_5^-$, 93473-90-6; $((CH_3)_2CCN)Fe(CO)_4^-$, 93473-91-7; $(CH_3CHCN)Fe(CO)_5^-$, 93473-92-8; $(CH_3CHCN)Fe(CO)_4^-$, 93473-93-9; $(CH_3CHCOCH_3)Fe(CO)_5^-$, 93473-94-0; $(CO)_4FeCHO^-$, 48055-09-0; $CH_3C^-HCOCH_3$, 64723-99-5.

Multidentate Lewis Acids. Halide Complexes of 1,2-Phenylenedimercury Dihalides[1]

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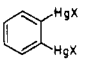
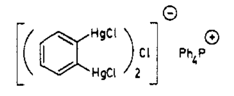
Summary: The bidentate Lewis acids dichloro-1,2-phenylenedimercury (**1**) and dibromo-1,2-phenylenedimercury (**2**) form complexes with halides. These are the first anionic complexes of organomercury compounds which have been isolated and characterized.

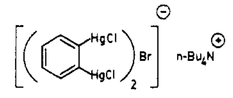
The coordination chemistry of multidentate Lewis bases is a subject of great theoretical and practical importance, and the study of analogous Lewis acids promises to be equally interesting.[2] In this paper, we describe the coordination chemistry of three bidentate Lewis acids, 1,2-phenylenedimercury dihalides **1–3**,[3] and we report the first isolation and structural characterization of anionic complexes of organomercury compounds.



1 (X=Cl)
2 (X=Br)
3 (X=I)



4



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Although inorganic halomercurate anions are well-known, analogous complexes of organomercury compounds have been remarkably elusive.[4,5] Since halogen bridges are a typical structural feature of inorganic halomercurate anions,[6] we suspected that the bidentate organomercury compounds **1–3** might form halide complexes stable enough to be isolated. Dichloro-1,2-phenylenedimercury (**1**) is nearly insoluble in dichloromethane, but the addition of an approximately equimolar amount of tetraphenylphosphonium chloride at 25 °C rapidly produced a homogeneous solution. Addition of hexane precipitated crystals of the 2:1 complex **4** (mp 205–207 °C) in 75% yield.[7] The reactions of other phosphonium and ammonium halides with compounds **1** and **2** yielded similar complexes.[8] However, an analogous experiment with chlorophenylmercury and tetraphenylphosphonium chloride yielded only diphenylmercury and tetraphenylphosphonium tetrachloromercurate, the products of a re-

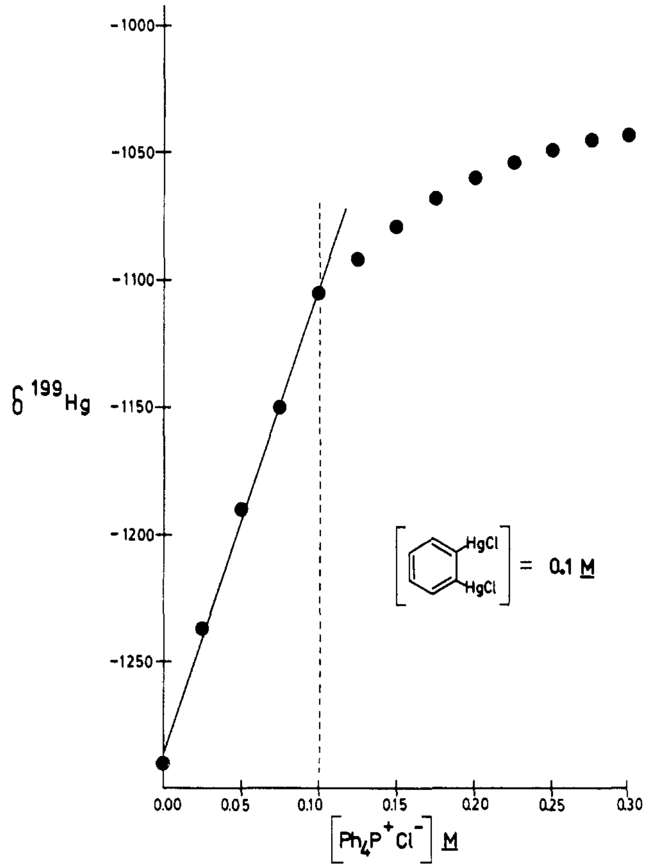

Figure 1. Relationship between the ^{199}Hg chemical shift of dichloro-1,2-phenylenedimercury (**1**; 0.1 M in Me_2SO) and the molar concentration of added tetraphenylphosphonium chloride.[11]

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[8] With a variety of phosphonium and ammonium halides, dichloro-1,2-phenylenedimercury (**1**) yielded 2:1 complexes analogous to **4**. In contrast, elemental analysis suggests that dibromo-1,2-phenylenedimercury (**2**) favors 5:3 complexes. Diiodo-1,2-phenylenedimercury (**3**) and 1,8-naphthalenedimercury dihalides appeared to form related complexes, but these compounds were too unstable to be isolated.

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