that irradiation of the bridging carbonyl signal (δ 220.3) causes a strong decrease in intensity of just one other signal, namely, that due to one of the axial sites (δ 206.5). The other axial carbonyl signal (δ 205.4) is unaffected.[11] The activation energy values obtained from the ^1H and ^{13}C spin saturation transfer experiments are in good agreement, indicating that the exchange is due to a single dynamic process. A mechanism derived from that proposed by Cotton for $Fe_3(CO)_{10}(\mu\text{-}CO)_2$[12] is diagrammed in Scheme I. This mechanism is uniquely consistent with exchange of the bridging carbonyl with only one of the axial carbonyls.

Fluxional processes involving bridge-to-terminal alkylidenes have previously been proposed by Knox[13] for $CpRu_2(\mu\text{-}CO)(\mu\text{-}CMe_2)$, Puddephatt[14] for $Co_2(CO)_4(\mu\text{-}CH_2)_2(\mu\text{-}dppm)$, and Bergman[15] for $Cp_2CoRh(CO)_2(\mu\text{-}CH_2)$. This is the first example supporting such a mechanism in a cluster compound.

Further studies on the reactivity of $Ru_3(CO)_{10}(\mu\text{-}CO)(\mu\text{-}CH_2)$ and its derivatives are in progress.

Acknowledgment. We would like to acknowledge the help and advice provided by D. G. Vander Velde in obtaining the spin saturation transfer results. This work was supported by National Science Foundation Grant CHE84-07233. Instruments supported by Grants NSF CHE79-16100 and NIH GM-27029 were utilized for NMR and mass spectra, respectively.

Registry No. $Ru_3(CO)_{10}(\mu\text{-}CO)(\mu\text{-}CH_2)$, 95123-28-7; $HzRu_3(CO)_9(CCH_2)$, 55802-24-9; $HzRu_3(CO)_9(CCO)$, 90990-76-4; $Ru_3(CO)_{12}$, 15243-33-1; diazomethane, 334-88-3.

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(10) The ^1H NMR experiments were conducted through selective saturation of the signal at δ 7.90 (site A). The decrease in the intensity of the corresponding signal at δ 6.02 (site B) was then measured. The ^{13}C NMR experiments were conducted by placing the transmitter offset on top of the bridging carbonyl resonance (δ 220.3) and delivering a 30-s, low power pulse. The power of the pulse was attenuated until saturation was obtained. $Ru_3(^*CO)_{12}$ was added to the sample as a standard.

(11) A smaller amount of spin saturation transfer to some of the other carbonyls is also observed. These carbonyls seem to be related to the bridging carbonyl by a secondary process, which is under study.

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Oxide-Transfer Reactions within the Monoanionic CO_2 Complex $Li[Fe(\eta\text{-}C_5H_5)(CO)_2(CO_2)]$

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Summary: Oxide transfer from coordinated CO_2 to coordinated CO is facile above −20 °C in the monoanionic CO_2 complex $Li[Fe(\eta\text{-}C_5H_5)(CO)_2(CO_2)]$. The reaction is proposed to involve intramolecular transfer via a metalloanhydride followed by CO or CO_2 exchange. Oxide transfer to excess CO_2 provides at best a minor decomposition pathway for the CO_2 complex.

The η^1-C coordination mode has been established for one member of the important monoanionic class of transition-metal CO_2 complexes,[1] and a similar metallocarboxylate structure can be inferred for $[Fe(\eta\text{-}C_5H_5)(CO)_2(CO_2)]^-$ ($[Fp(CO_2)]^-$),[2-4] consistent with the nucleophilicity of the CO_2 oxygen atoms.[5,6] Our observation that the reactive oxygen atoms in dianionic η^1-C complexes of CO_2 will transfer to excess CO_2 to give CO_3^{2-} and CO[7,8] and will also transfer from CO_2 to CO ligands[8] has led us to examine the nucleophilicity of the oxygen atoms in $[Fp(CO_2)]^-$. We now wish to report that $[Fp(CO_2)]^-$ transfers oxide between CO_2 and CO ligands but does not reductively disproportionate excess CO_2.[3]

The lithium salt of $[Fp(CO_2)]^-$, prepared by slow addition of CO_2 to a 50 mM solution of Li[Fp][9] at −78 °C, was examined in preference to the equally accessible Na^+ or K^+ salts because of its high solubility and relative stability in tetrahydrofuran (THF).[10,11] This stability is probably a consequence of cation coordination of the carboxylate oxygen atoms,[1,12] the effect of which is illustrated by two reactions: treatment of $NBu_4[Fp]$ with CO_2 at −78 or 25 °C gives a ca. 2:1 mixture of $[Fp]^-$ and $[Fp(CO_2)]^-$, and a similar mixture is obtained when dimethylformamide (DMF) is added to a THF solution of $K[Fp(CO_2)]$ (DMF should inhibit contact ion-pairing by competitively complexing K^+).

Oxide transfer within $Li[Fp(CO_2)]$ was studied by monitoring solutions prepared from equimolar mixtures of $^{13}CO_2$[13] and $C^{18}O_2$.[14] The $Li[Fp(CO_2)]$ was converted into volatile $[FpCH_3]$[15] by quenching 10-mL aliquots with CH_3OSO_2F at −78 °C (eq 1),[16] and mass spectroscopy was

$$Li[Fp(CO_2)] + CH_3OSO_2F \rightarrow [FpCH_3] + LiOSO_2F + CO_2 \quad (1)$$

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† Fellow of the Alfred P. Sloan Foundation, 1982–1985.

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(2) The $[Fe(\eta\text{-}C_5H_5)(CO)_2]$ moiety will be abbreviated to [Fp] throughout this paper.

(3) Evans, G. O.; Walter, W. F.; Mills, D. R.; Streit, C. A. *J. Organomet. Chem.* **1978**, *144*, C34.

(4) Prepared by direct reaction of CO_2 with Na[Fp]^3 or $Mg[Fp]_2$^{5}. Phosphine-stabilized derivatives of $[Fp(CO_2)]^-$ have also been prepared from metallocarboxylic acids: Grice, N.; Kao, S. C.; Pettit, R. *J. Am. Chem. Soc.* **1979**, *101*, 1627.

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(10) The 1:1 adduct stoichiometry has been confirmed to within 5% by gas titration using vacuum line techniques for the Li^+, Na^+, and K^+ salts of $[Fp(CO_2)]^-$. There is no evidence for the formation of a subsequent 2:1 CO_2 adduct with Li, Na, or KFp: the carbonyl stretching absorptions of the corresponding $[Fp(CO_2)]^-$ salts and the CO_2 stretching absorption of $Li[Fp(CO_2)]$ (the only salt for which such an absorption can be observed[12]) are unaffected by excess CO_2.

(11) The counterion dependence of stability and solubility parallel each other and vary as $Li^+ > Na^+ > K^+ > NBu_4^+$.

(12) Specific coordination of M^+ to the carboxylate group is indicated by the absence of counterion dependence for the carbonyl stretching absorptions (at 2015 and 1950 cm^{-1} for Li^+, Na^+, K^+, and NBu_4^+ salts), together with the counterion sensitivity of the C–O stretching absorption of the coordinated CO_2. Although partially obscured by solvent, this is at 1510 cm^{-1} in $Li[Fp(CO_2)]$ (ca. 1480 cm^{-1} in $Li[Fp(^{13}CO_2)]$) but is below the solvent cutoff (ca. 1450 cm^{-1}) in THF solutions of $Na[Fp(CO_2)]$, $K[Fp(CO_2)]$, and $NBu_4[Fp(CO_2)]$.

(13) Merck, Sharpe and Dohme: 92.97 atom % ^{13}C.

(14) Prochem: 99.39 atom % ^{18}O.

(15) Piper, T. S.; Wilkinson, G. *J. Inorg. Nucl. Chem.* **1956**, *3*, 104.

(16) This reaction is similar to that reported between $Na[Fp(CO_2)]$ and MeI,[3] but solution IR indicated that the reaction was clean and complete in less than 5 min at −78 °C with $MeOSO_2F$, while more vigorous conditions (>−40 °C), under which scrambling occurred, were required for reaction with MeI.

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Table I. Percentage Isotopic Enrichment of the [$FpCH_3$] Obtained from Aliquots of Li[$Fp(CO_2)$] Solutions in THF Prepared from Mixtures of $^{13}CO_2$[13] and $C^{18}O_2$[14] and Quenched with CH_3OSO_2F[a]

time, h	no label	1 × ^{13}C	1 × ^{18}O and/or 2 × ^{13}C	1 × ^{13}C + 1 × ^{18}O	2 × ^{18}O and/or 2 × ^{13}C + 1 × ^{18}O	1 × ^{13}C + 2 × ^{18}O
	Li[$Fp(CO_2)$] Prepared from 1.02:1 $^{13}CO_2$:$C^{18}O_2$ and Maintained at −40 °C					
24	92.9	2.6	4.5			
	Li[$Fp(CO_2)$] Prepared from 1:1.05 $^{13}CO_2$:$C^{18}O_2$ and Maintained at -20 °C					
0	97.8	0.3	1.9	...	...	...
1	94.3	2.2	3.4	...	...	...
3	89.5	3.7	6.4	0.4	...	...
7	74.5	9.9	13.7	1.3	0.7	...
15	61.1	13.7	20.8	2.9	1.6	...
24	54.8	15.1	23.8	3.9	2.5	...
	Li[$Fp(CO_2)$] Prepared from 1:1.00 $^{13}CO_2$:$C^{18}O_2$ and Maintained at 2 °C					
0	98.6	0.1	1.3	...	...	...
1	57.0	14.4	22.8	3.8	2.1	...
2	52.2	14.6	25.8	4.5	3.1	...
4	51.1	13.6	26.4	5.3	3.0	0.2
8	50.9	13.8	25.6	6.0	3.3	0.5
	Calculated Enrichment after Random Intermolecular Scrambling					
	40.4	14.8	28.1	9.8	5.3	1.6

[a] When label combinations cannot be distinguished, the more probable are listed first. Natural abundance ^{13}C has been listed as "no label".

Table II. Percentage Isotopic Enrichment of the [$FpCH_3$] Obtained by Quenching with CH_3OSO_2F a Solution of Li[$Fp(^{13}CO_2)$][13] in THF Maintained under the Specified Conditions[a]

temp, °C	added reagent	time, h	no label	1 × ^{13}C	2 × ^{13}C
−40	0.1 equiv of LiFp	24	99.6	0.4	...
−40	0.1 equiv of O_2	24	97.4	2.0	0.6
−40	0.1 equiv of CO_2	24	97.9	2.1	...
−40	$h\nu$[b]	4	98.4	1.6	...

[a] Natural abundance ^{13}C has been listed as "no label". [b] Irradiation with a GE 75W sunlamp placed 15 cm from the reaction vessel.

Table III. Percentage Isotopic Enrichment of the [$FpCH_3$] Obtained from Aliquots of a Solution of Li[$Fp(CO_2)$] in THF Maintained at -20 °C under an Atmosphere of "^{13}CO"[a,b]

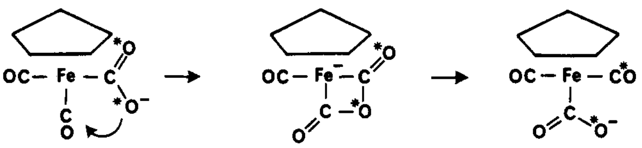
time, h	no label	1 × ^{13}C	2 × ^{13}C and/or 1 × ^{18}O	1 × ^{13}C + 1 × ^{18}O	2 × ^{13}C + 1 × ^{18}O and/or 2 × ^{18}O
0	100	...	...	...	...
2	69.0	21.0	4.5	4.0	1.4
6	44.8	35.2	9.7	6.8	3.6
14	26.6	39.3	18.9	7.7	7.6
24	13.7	36.9	30.9	6.9	11.6

[a] 92.2 atom % ^{13}C and 17.6 atom % ^{18}O. [b] Natural abundance ^{13}C has been listed as "no label".

used to analyze the isotopic composition of the sublimed [$FpCH_3$] as described previously.[8] Since [$FpCH_3$] prepared from freshly labeled mixtures showed negligible label incorporation, enrichment of other samples indicated exchange into the carbonyls of Li[$Fp(CO_2)$].

Data obtained from solutions maintained at −40, −20, and 2 °C (Table I) show that exchange of ^{13}C and ^{18}O into the carbonyls began at −20 °C and was rapid at 2 °C. Exchange was also evinced by the partial replacement of the free $^{13}C^{16}O_2$ and $^{12}C^{18}O_2$ initially observed in solution after quenching (IR[17]) by $^{12}C^{16}O_2$ and $^{12}C^{16}O^{18}O$ as reaction proceeded[18] and was confirmed to involve a thermal reaction by the control experiments in Table II. Absence of exchange within singly labeled Li[$Fp(^{13}CO_2)$], monitored as before, established that scrambling was not induced at -40 °C by 0.1 equiv of Li[Fp], CO_2, or O_2 and could not be driven photochemically at moderate illumination levels.

One process suggested by the observed exchange is oxide transfer from CO_2 to CO via a metalloanhydride (eq 2).[19] Metalloanhydrides have not been previously reported, but there is indirect precedent in the structurally characterized ferraazitidine [$Fe\{C[=C(C_6H_5)_2]N(CH_3)C(OC_2H_5)\}(\eta\text{-}C_5H_5)CO$] formed in a similar intramolecular attack on a coordinated CO.[20]



(2)

Intramolecular transfer can account for the appearance of one ^{13}C or up to two ^{18}O in the carbonyls of a molecule of [$Fp(CO_2)$]$^-$, but the eventual appearance of both ^{13}C and ^{18}O in the same molecule requires an intermolecular exchange. One possibility is that CO_2 dissociation from [$Fp(CO_2)$]$^-$ allows CO_2 exchange, but direct CO exchange could also be occurring, as was demonstrated by monitoring label incorporation into a solution of unlabeled Li[$Fp(CO_2)$] maintained under ^{13}CO (partially ^{18}O labeled) at -20 °C (Table III).

Reductive disproportionation of coordinated CO_2 normally involves oxide transfer to a second CO_2, but facile oxide transfer within Li[$Fp(CO_2)$] does not necessarily imply facile transfer from this species to CO_2, since charge separation may not favor [$Fp(CO)$]$^+$ formation. An al-

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(17) $^{13}C^{16}O_2$, 2270 cm^{-1}; $^{12}C^{18}O_2$, 2298 cm^{-1}; $^{12}C^{16}O_2$, 2337 cm^{-1}; $^{12}C^{16}O^{18}O$, 2318 cm^{-1}.

(18) Splitting of the C≡O stretching absorptions provided direct evidence for ^{13}C and ^{18}O incorporation into the carbonyls of Li[$Fp(CO_2)$] and [$FpCH_3$], but the complexity of the spectra precluded detailed analysis.

(19) Independent evidence for this process has been obtained by G. O. Evans: Evans, G. O., private communication.

(20) Fehlhammer, W. P.; Hirschmann, P.; Stolzenberg, H. *J. Organomet. Chem.* **1982**, *224*, 165. See also: Fehlhammer, W. P.; Christian, G.; Mayr, A. *J. Organomet. Chem.* **1980**, *199*, 87.

ternative disproportionation stoichiometry that gives $[\{Fp\}_2]$ (eq 3) is more reasonable and has been reported for

$$2Na[Fp] + 2CO_2 \rightarrow [\{Fp\}_2] + CO + Na_2CO_3 \quad (3)$$

$Na[Fp(CO_2)]$,[3] but we have not observed an analogous decomposition of $Li[Fp(CO_2)]$ in our laboratory. Decomposition of $Li[Fp(CO_2)]$ in THF becomes significant at ambient temperatures ($t_{1/2}$ = ca. 4 h), giving $[\{Fp\}_2]$ contaminated by <10% [FpH] (IR). Excess CO_2 accelerates decomposition, although it is not consumed and does not affect the course of reaction (IR of solution and of the precipitate). A D_2O solution of the precipitate from the decomposition of $Li[Fp(^{13}CO_2)]$ in the presence of excess $^{13}CO_2$ initially contains (^{13}C NMR) a ca. 1:1:2 ratio of formate, carbonate, and a mixture of unidentified products (possibly iron carbonates) and hydrolyzes after 12 h to a ca. 1:3 mixture of HCO_2^- and CO_3^{2-}. Quantification was complicated by the THF polymerization which invariably accompanied decomposition, but Ba^{2+} precipitation indicated an eventual CO_3^{2-} yield of ca. 60%.

The most remarkable feature of the reaction is that only traces of free[21] CO are formed (0–10%; GC). The formation of labeled $[\{Fp\}_2]$ from $Na[Fp(^{13}CO_2)]$ has previously been taken to imply that the decomposition produces CO,[3] but oxide transfer from CO_2 to CO provides an alternative source of ^{13}CO, and we conclude that the published data do not establish reductive disproportionation of $Na[Fp(CO_2)]$ and that reductive disproportionation provides at best a minor decomposition pathway for $Li[Fp(CO_2)]$.

Formate ion could reasonably be formed in this decomposition by homolysis of the $Fe-CO_2$ bond in $[Fp(CO_2)]^-$ followed by reaction of $CO_2^{-}\cdot$[22] with the solvent, but the observation of CO_3^{2-} formation without CO evolution is less readily interpreted since something other than CO_2 must be reduced. The only obvious possibility is that there is an unidentified organic product derived from the solvent: this is not unreasonable, since the formation of HCO_2^- and, more directly, the invariable THF polymerization observed suggest the intermediacy of solvent-derived radicals.

The experiments above lead to two general conclusions about the organometallic chemistry of CO_2. First, monoanionic CO_2 complexes can be sufficiently nucleophilic to transfer oxide from CO_2 to CO ligands, suggesting that oxide transfer is a general reaction of CO_2 complexes that can be reasonably described as metallocarboxylates; second, carbonate can be formed from CO_2 and transition-metal complexes by routes other than reductive disproportionation: only in cases in which both CO and CO_3^{2-} formation have been established[7] can reductive disproportionation be regarded as proven.

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Registry No. $Li[Fp(CO_2)]$, 33361-37-4; $NBu_4[Fp(CO_2)]$, 95156-00-6; $K[Fp(CO_2)]$, 95156-01-7; $Na[Fp(CO_2)]$, 95156-02-8; Li[Fp], 33361-37-4; $NBu_4[Fp]$, 65836-70-6; $[FpCH_3]$, 12080-06-7; $[\{Fp\}_2]$, 12154-95-9; [FpH], 35913-82-7; K[Fp], 60039-75-0; HCO_2^-, 71-47-6; CO_3^{2-}, 3812-32-6; CO_2, 124-38-9; CH_3OSO_2F, 421-20-5; O_2, 7782-44-7.

Homo- and Heterobimetallic Complexes Connected by Peralkylated Cyclopentadienyl Rings Having a Two-Carbon Bridge between Them

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Summary: The reaction between the tungstenacyclobutadiene complex $W(C_3Et_3)(OCMe_2CMe_2O)(OCMe_3)$ and 3,7-decadiyne yields $[WO_2(OCMe_3)]_2(\eta^5,\eta^5\text{-}Et_4C_5CH_2CH_2C_5Et_4)$ (**1**, $\eta^5,\eta^5\text{-}Et_4C_5CH_2CH_2C_5Et_4$ = $Cp'CH_2CH_2Cp'$). **1** was was treated with HCl and Me_3SiCl to give $[W(O)Cl_3]_2(Cp'CH_2CH_2Cp')$ (**2**). Treatment of **2** with PCl_5 gave $(WCl_4)_2(Cp'CH_2CH_2Cp')$ (**3**). An X-ray structural study of a PMe_3 adduct of **3**, $[(PMe_3)Cl_4W]_2(Cp'CH_2CH_2Cp')$ (**4**), confirmed the presence of the $Cp'CH_2CH_2Cp'$ ring system. **4** crystallizes in the space group $p\bar{1}$ with a = 9.562 (4) Å, b = 10.690 (8) Å, c = 14.007 (13) Å, α = 110.10 (6)°, β = 104.55 (5)°, γ = 95.14 (5)°, and Z = 1. A total of 4458 reflections were collected in the range $3° < 2\theta < 50°$ with the 3371 having $F_o > 4\sigma(F_o)$ being used for refinement by full-matrix least-squares techniques to a final R_1 = 0.046 and R_2 = 0.050. $HC_5Me_4CH_2CH_2C{\equiv}CEt$ was prepared by addition of $LiCH_2CH_2C{\equiv}CEt$ to tetramethylcyclopentenone. $LiC_5Me_4CH_2CH_2C{\equiv}CEt$ was added to $[Rh(CO)_2Cl]_2$ to give $Rh(\eta^5\text{-}C_5Me_4CH_2CH_2C{\equiv}CEt)(CO)_2$ which was subsequently added to $W(C_3Et_3)(OCMe_2CMe_2O)(OMe_3)$ to give $[(Me_3CO)O_2W](\eta^5,\eta^5\text{-}Et_4C_5CH_2CH_2C_5Me_4)[Rh(CO)_2]$.

Approaches to the reduction of carbon monoxide employing complexes in which donor ligands link two metal centers, so far, for the most part, have not anticipated the fact that CO will likely displace donor ligands at the temperatures (≳150 °C) and pressures (≳100 atm of CO/H_2) typically necessary to reduce CO. Bimetallic complexes containing linked cyclopentadienyl rings,[1] on the other hand, may be likely to survive such relatively severe reaction conditions. A molecular model of $Ta_2(\eta^5\text{-}C_5Me_4Et)_2Cl_3(CH_3)H_2$,[2] a relative of compounds of the type $[Ta(\eta^5\text{-}C_5Me_4R)Cl_2H]_2$ which will react with CO to give $[Ta(\eta^5\text{-}C_5Me_4R)Cl_2]_2(\mu\text{-}CHO)(\mu\text{-}H)$ complexes,[3] suggested to us that a two-carbon link between peralkylated rings would distort this bimetallic complex little. We report here some potentially general approaches to bimetallic complexes containing two-carbon-linked peralkylated cyclopentadienyl ring systems in which at least one of the metals is tungsten.

3,7-Decadiyne reacts smoothly with a tungstenacyclobutadiene complex as shown in eq 1.[4,5] The most im-

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[21] We also see no IR evidence for the formation of $[Fe(CO)_5]$ or other potential CO sinks.

[22] This could also give rise to the small amount of CO observed.

[1] Many bimetallic complexes containing cyclopentadienyl rings linked by one or two (or more) units (usually CR_2 or SiR_2) have been prepared. To our knowledge, however, they all contain only one linking unit (often $SiMe_2$), or unalkylated rings, or both.

[2] Belmonte, P. A.; Schrock, R. R.; Day, C. S. *J. Am. Chem. Soc.* **1982**, *104*, 3082.

[3] Belmonte, P. A.; Cloke, F. G. N.; Schrock, R. R. *J. Am. Chem. Soc.* **1983**, *105*, 2643.

[4] (a) The formation of cyclopentadienyl rings from tungstenacyclobutadiene complexes and acetylenes has already been disclosed.[4b,c] (b) Pedersen, S. F.; Schrock, R. R.; Churchill, M. R.; Wasserman, H. J. *J. Am. Chem. Soc.* **1982**, *104*, 6808. (c) Schrock, R. R.; Pedersen, S. F.; Churchill, M. R.; Ziller, J. W. *Organometallics* **1984**, *3*, 1574.

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