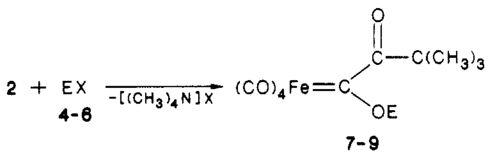
Scheme II



	E	ν_{CO}
7	C_2H_5	2070 m, 2025 s, 2000 vs[a]
8	$Si(CH_3)_3$	2067 m, 2015 s, 1990 vs[b]
9	CH_3CO	2076 m, 2020 s, 1998 vs[b]

The observed molecular geometry of the ferrate anion amply supports that bonding in the Fe–C(O)–C(O)-*t*-Bu framework is largely σ for the Fe–C and C–C bonds, whereas the carbonyl bonds have a large additional π-component, as expected. There is, however, some indication for a minor π-contribution, especially to the Fe–C1 bond. Finally, it should be noted that there are no additional close contacts of the carbonyl oxygen atoms in the crystal structure of 3. In alkali-metal salts of acylmetalates tight ion pair formation with close $M–O_{acyl}$ contacts is observed with subsequent implications for the molecular structure and bonding.[18]

Acylferrates are known to react with electrophiles in two different ways: Addition to the acyl oxygen atom yields neutral oxycarbene complexes according to the original Fischer synthesis.[19,20] Addition to the metal center, on the other hand, is generally followed by reductive elimination leading to carbonyl compounds.[13] The competition of these two pathways depends both on the electrophile and the solvent.[21] The acylferrate 2 reacts with a series of electrophiles such as ethyl fluorosulfonate (4) in ether/HMPT (4/1), chlorotrimethylsilane (5), or acetyl chloride (6) in methylene chloride at low temperature (−50 °C) to give red solutions.[22] The products 7–9 can be characterized by IR spectroscopy (Scheme II), but elude isolation due to their pronounced thermolability which results in decomposition on warming above −40 °C or even on evaporation of the solvent.

The ν_{CO} spectra of the products obtained from the reaction mixtures are characteristic for $(CO)_4FeL$ complexes. They indicate that the electrophile is added to the acyl oxygen functionality and thus are compatible with the formulation of the products as tetracarbonyl pivaloyl carbene complexes.

Acknowledgment. Support of this research from the Deutsche Forschungsgemeinschaft and from the Fonds der Chemischen Industrie is gratefully acknowledged. We are also grateful to Mr. J. Riede for the data collection of 3. G.M. acknowledges helpful discussions with Prof. R. Bau (USC). D.S. acknowledges, with thanks, the award of an Alexander von Humboldt Prize by the Alexander von Humboldt Foundation during the tenure of which this research was initiated.

Supplementary Material Available: Tables of additional crystal structure data, atomic and thermal parameters, and H atom positions (11 pages); a listing of observed and calculated structure factor amplitudes (20 pages). Ordering information is given on any current masthead page.

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[18] Ginsburg, R. E.; Berg, J. M.; Rothrock, R. K.; Collman, J. P.; Hodgson, K. O.; Dahl, L. F. *J. Am. Chem. Soc.* 1979, *101*, 7218 and references cited therein.

[19] Fischer, E. O.; Beck, H. J.; Kreiter, C. G.; Lynch, J.; Müller, J.; Winkler, E. *Chem. Ber.* 1972, *105*, 162.

[20] Conder, H. L.; Darensbourg, M. Y. *Inorg. Chem.* 1974, *13*, 506.

[21] Semmelhack, M. F.; Tamura, R. *J. Am. Chem. Soc.* 1983, *105*, 4099.

[22] In a typical experiment, acylferrate 2 (0.3 g, 0.84 mmol) is dissolved in 10 mL of CH_2Cl_2 and cooled to −55 °C. Addition of $(CH_3)_3SiCl$ (1.72 g, 16 mmol) leads to a dark red solution. After 3 h, the solution is concentrated at −55 °C to a volume of 2 mL while unreacted 2 precipitates and can be removed. Further evaporation of the solvent results in decomposition.

Novel C-Alkylation of a CO-Bridged Dinuclear Iron Carbonyl Anion: A New Synthesis of $Fe_2(CO)_6$ Complexes Containing a Bridging Acyl Ligand

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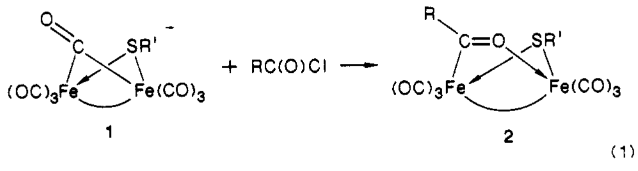
Summary: The reaction of anionic iron carbonyl complexes which contain a bridging CO ligand of type $[Et_3NH][(\mu\text{-}CO)(\mu\text{-}R'S)Fe_2(CO)_6]$ with alkyl- and arylmercuric halides, RHgX, gives products which contain a bridging acyl ligand in place of the bridging CO, $(\mu\text{-}RCO)(\mu\text{-}R'S)Fe_2(CO)_6$. With vinylic and alkynylmercurials, the bridging CO ligand is displaced and products of type $(\mu\text{-}\sigma,\pi\text{-}CH{=}CHR)(\mu\text{-}R'S)Fe_2(CO)_6$ and $(\mu\text{-}\sigma,\pi\text{-}C{\equiv}CR)(\mu\text{-}R'S)Fe_2(CO)_6$ are obtained. In contrast, reaction of $[Et_3NH][(\mu\text{-}CO)(\mu\text{-}R'S)Fe_2(CO)_6]$ with $[Et_3O][BF_4]$ gives a bridging alkoxycarbyne complex, $(\mu\text{-}EtOC)(\mu\text{-}R'S)Fe_2(CO)_6$.

We report here the first examples of what may be a general and potentially useful new reaction: the alkylation and arylation by RHgX *at carbon* of a CO ligand of a dinuclear transition-metal carbonyl anion which contains a bridging CO ligand. The product is a neutral complex which has a bridging acyl group in place of the original bridging CO ligand. The class of transition-metal anions used in this study are of the type $[(\mu\text{-}CO)(\mu\text{-}R'S)Fe_2(CO)_6]^-$ (1) which, as we have reported earlier,[1] are easily prepared by reaction of $Fe_3(CO)_{12}$ with mercaptide ion (best generated in situ by the action of triethylamine on a mercaptan, R'SH, in THF). Among several reactions of 1 which we described were those with allyl chloride, propargyl bromide, and acyl chlorides. In these, the organic electrophile displaced the bridging CO ligand to form a new, neutral dibridged $Fe_2(CO)_6$ complex, e.g., eq 1.



Anions of type 1, we have found, undergo a novel reaction with alkyl- and arylmercuric halides which results in C-alkylation of a CO ligand to give a μ-acyl complex (eq 2). In a typical reaction, a red-brown solution of the

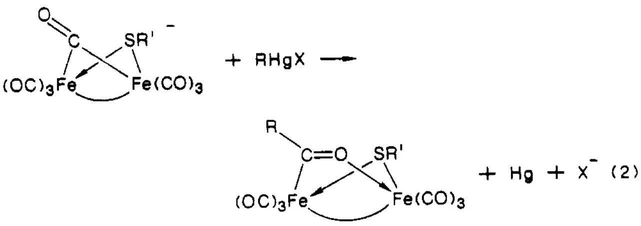


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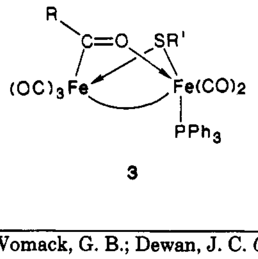
Table I. Reactions of $[Et_3NH][(\mu\text{-}CO)(\mu\text{-}R'S)Fe_2(CO)_6]$ with RHgX

R′	RHgX	product[a] (% yield)
Et	PhHgBr	$(\mu\text{-}PhCO)(\mu\text{-}EtS)Fe_2(CO)_6$ (54)
Et	$p\text{-}FC_6H_4HgBr$	$(\mu\text{-}p\text{-}FC_6H_4CO)(\mu\text{-}EtS)Fe_2(CO)_6$ (53)
Et	$p\text{-}Me_2NC_6H_4\text{-}HgBr$	$(\mu\text{-}p\text{-}Me_2NC_6H_4CO)(\mu\text{-}EtS)Fe_2(CO)_6$ (41)
Et	$MeOCH_2CH_2\text{-}HgCl$	$(\mu\text{-}MeOCH_2CH_2CO)(\mu\text{-}EtS)Fe_2(CO)_6$ (65)
Et	Me_3CHgCl	$(\mu\text{-}Me_3CCO)(\mu\text{-}EtS)Fe_2(CO)_6$ (58)
Ph	EtHgCl	$(\mu\text{-}EtCO)(\mu\text{-}PhS)Fe_2(CO)_6$ (74)
Ph	$c\text{-}C_3H_5HgCl$	$(\mu\text{-}c\text{-}C_3H_5CO)(\mu\text{-}PhS)Fe_2(CO)_6$ (81)
Ph	$MeOCH_2CH_2\text{-}HgCl$	$(\mu\text{-}MeOCH_2CH_2CO)(\mu\text{-}PhS)Fe_2(CO)_6$ (44)
Ph	MeEtCHHgBr	$(\mu\text{-}MeEtCHCO)(\mu\text{-}PhS)Fe_2(CO)_6$ (48)
Ph	$p\text{-}ClC_6H_4HgBr$	$(\mu\text{-}p\text{-}ClC_6H_4CO)(\mu\text{-}PhS)Fe_2(CO)_6$ (82)
Me_3C	n-BuHgCl	$(\mu\text{-}n\text{-}BuCO)(\mu\text{-}Me_3CS)Fe_2(CO)_6$ (44)
Me_3C	Me_3CCH_2HgCl	$(\mu\text{-}Me_3CCH_2CO)(\mu\text{-}Me_3CS)Fe_2(CO)_6$ (66)
Me_3C	PhHgBr	$(\mu\text{-}PhCO)(\mu\text{-}Me_3CS)Fe_2(CO)_6$ (86)

[a] All products were characterized by IR and ^1H and ^{13}C NMR as well as mass spectroscopy and by elemental analysis if they were solids. If they were oils or oily solids, the crystalline mono-Ph_3P substitution product was prepared and analyzed. Characterizing data are provided as supplementary material.

triethylammonium salt of anion 1 ($R' = C_2H_5$) was generated by addition of equimolar amounts of C_2H_5SH and triethylamine to a solution of 2.98 mmol of $Fe_3(CO)_{12}$ in 50 mL of THF at room temperature under nitrogen.[1] To this solution was added, with stirring, 2.8 mmol of solid phenylmercuric bromide. A color change to red with formation of a white precipitate and elemental mercury were observed. After the mixture had been stirred for 3 h, the solvent was evaporated and the residue extracted with pentane. Filtration chromatography (silicic acid, pentane) gave two products: the known[2] $(\mu\text{-}C_2H_5S)_2Fe_2(CO)_6$ (0.58 mmol, 20%) as a mixture of e,a and e,e isomers[3] and a red-orange solid (1.60 mmol, 54%) which was identified as $(\mu\text{-}C_6H_5CO)(\mu\text{-}C_2H_5S)Fe_2(CO)_6$ on the basis of its IR and proton NMR spectra.[1] Further study showed this to be a general reaction for aryl- and alkylmercuric halides (Table I). The product yields were variable, and in most reactions the $(\mu\text{-}R'S)_2Fe_2(CO)_6$ complex was a byproduct. Of interest was that the organic group of an olefin alkoxymercuration product, $CH_3OCH_2CH_2HgCl$, was transferred intact to CO, giving, in the case of the anion $[(\mu\text{-}CO)(\mu\text{-}C_2H_5S)Fe_2(CO)_6]^-$, the complex $(\mu\text{-}CH_3OCH_2CH_2CO)(\mu\text{-}C_2H_5S)Fe_2(CO)_6$ in 65% yield.

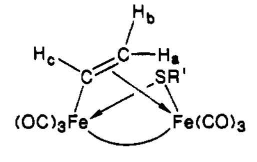
Many of the products listed in Table I are slightly air-sensitive oils which were difficult to purify. In most such cases they were converted to the respective mono(triphenylphosphine) substitution products, usually dark red, crystalline solids which could be recrystallized to give analytically pure products. These complexes were characterized by ^{31}P NMR signals in the δ_P 45–55 range. Since the ^{13}C NMR signal due to the RC=O ligand showed no spin–spin coupling to the phosphorus atom of the $(C_6H_5)_3P$ ligand, we assume that the phosphine substitution products have the structure shown as 3.



3

Unsaturated organomercurials react with the $[(\mu\text{-}CO)(\mu\text{-}R'S)Fe_2(CO)_6]^-$ anion in a different manner. Thus, a reaction of $CH_2{=}CHHgBr$ with $[Et_3NH][(\mu\text{-}CO)(\mu\text{-}C_2H_5S)Fe_2(CO)_6]$, carried out as described for the reaction with C_6H_5HgBr, although the color change and the precipitates observed were the same, did not give $(\mu\text{-}CH_2{=}CHCO)(\mu\text{-}C_2H_5S)Fe_2(CO)_6$. Instead, the product, obtained in 64% yield, was a red-orange oil which was identified as $(\mu\text{-}CH_2{=}CH)(\mu\text{-}C_2H_5S)Fe_2(CO)_6$ (4).[4] A similar reaction of $[Et_3NH][(\mu\text{-}CO)(\mu\text{-}C_6H_5S)Fe_2(CO)_6]$ with vinylmercuric bromide resulted in formation of $(\mu\text{-}CH_2{=}CH)(\mu\text{-}C_6H_5S)Fe_2(CO)_6$, while reaction of $[Et_3NH][(\mu\text{-}CO)(\mu\text{-}C_2H_5S)Fe_2(CO)_6]$ with *trans*-β-styrylmercuric chloride gave $(\mu\text{-}C_6H_5CH{=}CH)(\mu\text{-}C_2H_5S)Fe_2(CO)_6$ (two isomers, a- and e-SC_2H_5), a red crystalline solid, in 47% yield (in addition to $(\mu\text{-}C_2H_5S)_2Fe_2(CO)_6$ in 21% yield).

Bis(phenylethynyl)mercury also reacted with $[Et_3NH][(\mu\text{-}CO)(\mu\text{-}C_2H_5S)Fe_2(CO)_6]$ with displacement of the bridging CO ligand, giving $(\mu\text{-}C_6H_5C{\equiv}C)(\mu\text{-}C_2H_5S)Fe_2(CO)_6$ (5) in 30% yield[5] (and the ubiquitous $(\mu\text{-}C_2H_5S)_2Fe_2(CO)_6$ in 35% yield). It would appear that bridging CO ligand displacement is a general process which occurs when unsaturated mercurials react with $[(\mu\text{-}CO)(\mu\text{-}R'S)Fe_2(CO)_6]^-$ anions, although this remains to be tested more thoroughly with other types of alkenyl- and alkynylmercury compounds.



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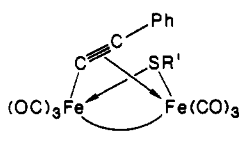

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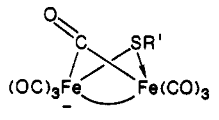
The mechanisms of the two different reactions described above are of interest. In principle, anions of type 1 can be expected to be ambident species, with negatively charged centers at iron and at the oxygen atom of the bridging CO ligand (6 and 7, respectively). We suggest that anions of type 1 are mercurated at iron by the RHgX electrophile, giving an intermediate with a Fe–Hg bond, 8. The latter, we suggest, is not stable and undergoes ready demercuration, giving transient 9. If R in 9 is alkyl or aryl,



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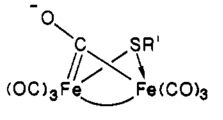

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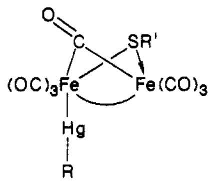
(1) Seyferth, D.; Womack, G. B.; Dewan, J. C. *Organometallics* **1985**, *4*, 398.

(2) (a) Hieber, W.; Spacu, P. *Z. Anorg. Allg. Chem.* **1937**, *233*, 353. (b) Dahl, L. F.; Wei, C.-H. *Inorg. Chem.* **1963**, *2*, 328.

(3) a = axial and e = equatorial isomer, as defined by: Shaver, A.; Fitzpatrick, P. J.; Steliou, K.; Butler, I. S. *J. Am. Chem. Soc.* **1979**, *101*, 1313.

(4) ^1H NMR (90 MHz, $CDCl_3$): δ 1.30 (t, J_{HH} = 7.4 Hz, 3 H, $-SCH_2CH_3$), 2.35 (q, J_{HH} = 7.3 Hz, 2 H, $-SCH_2CH_3$), 2.84 (dd, J_{HH} = 9.2 Hz, 2.1 Hz, 1 H, H_a), 3.79 (dd, J_{HH} = 13.8 Hz, 2.1 Hz, 1 H, H_b), 8.17 (dd, J_{HH} = 9.2 Hz, 13.8 Hz, 1 H, H_c). ^{13}C NMR (67.9 MHz, CD_2Cl_2): δ_C 18.2 (q, J_{CH} = 128.8 Hz, $-SCH_2CH_3$), 34.4 (t, J_{CH} = 145.4 Hz, $-SCH_2CH_3$), 73.9 (t, J_{CH} = 158.7 Hz, $CH_2{=}CH-$), 157.2 (d, J_{CH} = 150.7 Hz, $CH_2{=}CH-$), 209.6, 210.7 (s's, carbonyl C's). IR (CCl_4, NaCl): 3054 (vw), 2996 (w), 2978 (w), 2940 (w), 2879 (vw), 2072 (s), 2035 (vs), 1996 (vs, br), 1960 (s), 1457 (w), 1436 (vs), 1379 (w), 1294 ( ), 1258 (w), 1048 (vw), 974 (vw), 930 (vw), 615 (m), 595 (s), 557 (m), 534 (m), 499 (m) cm^{-1}. Mass spectrum, m/z (relative intensity): 368 (M^+, 11), 340 ($M^+ - CO$, 17), 312 ($M^+ - 2\ CO$, 22), 284 ($M^+ - 3\ CO$, 20), 256 ($M^+ - 4\ CO$, 26), 228 ($M^+ - 5\ CO$, 30), 200 ($M^+ - 6\ CO$, 59), 172 ($Fe_2C_2H_4S^+$, 23), 144 (Fe_2S^+, 100), 112 (Fe_2^+, 56), (Fe^+, 9). Anal. Calcd for $C_{10}H_8Fe_2O_6S$: C, 32.65; H, 2.19. Found: C, 32.42; H, 2.29.

(5) Prepared by an alternate route and fully characterized by J.B. Hoke in these laboratories.



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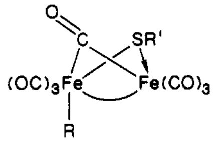
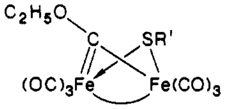


9

then intra- or intermolecular alkylation or arylation of a CO ligand (not necessarily the bridging CO) by the Fe–R takes place and the acyl group thus formed becomes a μ-acyl ligand. Alternatively, if R is a vinyl or an alkynyl group, then it displaces the bridging CO ligand and a neutral μ-σ,π-vinyl or a μ-σ,π-alkynyl complex results.

At this time, our ideas about mechanism are based only on precedent. The formation of transition metal–mercury bonds by the reactions of anionic transition-metal carbonyls with mercuric and organomercuric halides is well-documented,[6] and there are examples of mercury extrusion from an R–Hg–transition-metal system to form a σ-organo-transition-metal complex.[7] Finally, the formation of mononuclear transition-metal acyls via alkyl migration from a transition-metal center to a coordinated CO ligand is a well-known and important process.[8]

Anions of type 1 have shown themselves to be quite versatile in terms of their reactivity. The most common reaction is that with an electrophile of a type which can provide a new bridging group in which the bridging CO ligand is displaced.[1] Then there is the present reaction with RHgX in which we have formal C-alkylation of a CO ligand and no loss of carbon monoxide. A third mode of reaction with electrophiles is that of alkylation of the oxygen atom of the bridging CO ligand in 1, i.e., reaction via hybrid 7. For instance, the action of triethyloxonium tetrafluoroborate on anions of type 1 results in the formation of a neutral $Fe_2(CO)_6$ complex with a bridging ethoxycarbyne ligand, **10**.[9] This reaction has precedent in other examples of the O-alkylation of bridging CO ligands in di- and polynuclear metal carbonyl anions to give μ-alkoxycarbyne ligands.[10-12] These three types of reactivity will make possible the development of a broad chemistry of dinuclear iron carbonyl complexes containing organosulfur and other types of bridging ligands, but we expect also that synthetic applications to di- and polynuclear metal carbonyl anions of other elements should be possible.



10

Acknowledgment. We are grateful to the National Science Foundation for generous support of this work.

Registry No. **1**·$[Et_3NH]$ ($R' = Et$), 93530-38-2; **1**·$[Et_3NH]$ ($R' = Ph$), 105040-75-3; **1**·$[Et_3NH]$ ($R' = Me_3C$), 105040-77-5; **2** ($R = Ph$, $R' = Et$), 105118-48-7; **2** ($R = p\text{-}FC_6H_4$, $R' = Et$), 105040-78-6; **2** ($R = p\text{-}Me_2NC_6H_4$, $R' = Et$), 105040-79-7; **2** ($R = MeOCH_2CH_2$, $R' = Et$), 105040-80-0; **2** ($R = Me_3C$, $R' = Et$), 105040-81-1; **2** ($R = Et$, $R' = Ph$), 105040-82-2; **2** ($R = c\text{-}C_3H_5$, $R' = Ph$), 105040-83-3; **2** ($R = MeOCH_2CH_2$, $R' = Ph$), 105040-84-4; 2 ($R = MeEtCH$, $R' = Ph$), 105040-85-5; **2** ($R = p\text{-}ClC_6H_4$, $R' = Ph$), 105040-86-6; **2** ($R = n\text{-}Bu$, $R' = Me_3C$), 105040-87-7; **2** ($R = Me_3CCH_2$, $R' = Me_3C$), 105040-88-8; **2** ($R = Ph$, $R' = Me_3C$), 105040-89-9; **4** ($R' = Et$), 105040-90-2; **4** ($R' = Ph$), 105040-91-3; **5** ($R' = Et$), 105064-13-9; **10** ($R' = Me_3C$), 105040-93-5; (e,a)-$(\mu\text{-}C_2H_5S)_2Fe(CO)_6$, 18771-06-7; (e,e)-$(\mu\text{-}C_2H_5S)_2Fe(CO)_6$, 18771-07-8; (*E*)-(a)-$(\mu\text{-}C_6H_5CH{=}CH)(\mu\text{-}C_2H_5S)Fe_2(CO)_6$, 105040-92-4; (*E*)-(e)-$(\mu\text{-}C_6H_5CH$<<dbe$CH)(\mu\text{-}C_2H_5S)Fe_2(CO)_6$, 105118-49-8; $Fe_3(CO)_{12}$, 17685-52-8; PhHgBr, 1192-89-8; $p\text{-}FC_6H_4HgBr$, 2146-77-2; $p\text{-}Me_2NC_6H_4HgBr$, 20733-22-6; $MeOCH_2CH_2HgCl$, 123-88-6; Me_3CHgCl, 38442-51-2; EtHgCl, 107-27-7; $c\text{-}C_3H_5HgCl$, 88947-44-8; MeEtCHHgBr, 868-82-6; *n*-BuHgCl, 543-63-5; $p\text{-}ClC_6H_4HgBr$, 13351-54-7; Me_3CCH_2HgCl, 10284-47-6; C_2H_5SH, 75-08-1; $CH_2{=}CHHgBr$, 16188-37-7; (*E*)-$PhCH{=}CHHgCl$, 36525-03-8; Fe, 7439-89-6; bis(phenylethynyl)mercury, 6077-10-7.

Supplementary Material Available: Spectroscopic (IR, ^1H and ^{13}C NMR, 70-eV EI mass) and analytical data for the products of Table I and, in some cases, of their mono-Ph_3P substitution products (18 pages). Ordering information is given on any current masthead page.

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(6) Selected recent examples: (a) RHgX + $Fe(CO)_4^{2-}$, $Co(CO)_4^-$, $CpW(CO)_3^-$: Glocking, F.; Mahale, V. B.; Sweeney, J. J. *J. Chem. Soc., Dalton Trans.* **1979**, 767. (b) RHgX + $Mn(CO)_5^-$, $Re(CO)_5^-$: Suleimanov, G. Z.; Sokolov, V. I.; Reutov, O. A. *Izv. Akad. Nauk SSSR, Ser. Khim.* **1978**, 2837. (c) PhHgBr + $[\eta^5\text{-}CH_3C_5H_4Mn(CO)_2SiMe_2Ph]^-$; Schubert, U.; Kunz, E. *J. Organomet. Chem.* **1986**, *303*, C1.

(7) (a) Examples from organopalladium and -platinum chemistry: Sokolov, V. I.; Reutov, O. A. *Coord. Chem. Rev.* **1978**, *27*, 89. Bashilov, V. V.; Sokolov, V. I.; Reutov, O. A. *Izv. Akad. Nauk SSSR, Ser. Khim.* **1982**, 2069 (engl. transl. p 1825). (b) Organonickel chemistry: Isaeva, L. S.; Morozova, L. N.; Bashilov, V. V.; Petrovskii, P. V.; Sokolov, V. I.; Reutov, O. A. *J. Organomet. Chem.* **1983**, *243*, 253. (c) $(PhC{\equiv}C)_2Hg$ + $[M_2(CO)_{10}]^{2-}$ ($[PhC{\equiv}CM(CO)_5]^-$; M = Cr, Mo, W): Schlientz, W. J.; Ruff, J. K. *Synth. Inorg. Met.-Org. Chem.* **1971**, *1*, 215.

(8) (a) Collman, J. P.; Hegedus, L. S. *Principles and Applications of Organotransition Metal Chemistry*; University Science Books: Mill Valley, CA, 1980; Chapter 5. (b) Collman, J. P.; Rothrock, R. K.; Finke, R. G.; Rose-Munch, F. *J. Am. Chem. Soc.* **1977**, *99*, 7381.

(9) Complex **10** ($R' = Me_3C$): 58% yield; mp 107–108 °C. ^1H NMR (90 MHz, $CDCl_3$): δ 1.03 (s, 9 H, $-SC[CH_3]_3$), 1.66 (t, $J_{HH} = 7.3$ Hz, 3 H, $-OCH_2CH_3$), 4.92 (q, $J_{HH} = 7.3$ Hz, 2 H, $-OCH_2CH_3$). ^{13}C NMR (67.9 MHz, C_6H_6): δ_C 14.2 (q, $J_{CH} = 130.4$ Hz, $-OCH_2CH_3$), 32.3 (q, $J_{CH} = 126.2$ Hz, $-SC[CH_3]_3$), 47.0 (s, $-SC[CH_3]_3$), 86.7 (t, $J_{CH} = 150.3$ Hz, $-OCH_2CH_3$), 210.1 (s, br, carbonyl C's), 383.3 (s, carbyne C). Mass spectrum, m/z (relative intensity): 426 (M^+, 30), 398 ($M^+ - CO$, 54), 370 ($M^+ - 2\,CO$, 46), 342 ($M^+ - 3\,CO$, 42), 314 ($M^+ - 4\,CO$, 42), 286 ($M^+ - 5\,CO$, 99), 258 ($M^+ - 6\,CO$, 100), 229 ($Fe_2[CO][SC_4H_9]^+$, 65), 202 ($Fe_2[COEt][SH]^+$, 22), 201 ($Fe_2[SC_4H_9]^+$, 22), 145 (Fe_2SH^+, 85), 144 (Fe_2S^+, 96), 56 (Fe^+, 63). Anal. Calcd for $C_{13}H_{14}Fe_2O_7S$: C, 36.66; H, 3.31. Found: C, 36.81; H, 3.42.

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