Journal of Organometallic Chemistry, 407 (1991) 81–89
Elsevier Sequoia S.A., Lausanne
JOM 21549

Unexpected reactions of simple iron alkyls with a thiosulfinate ester capable of serving as a disulfur monoxide source

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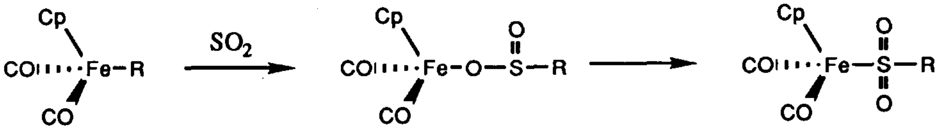
(Received September 10th, 1990)

Abstract

Attempts to use 4,5-diphenyl-3,6-dihydro-1,2-dithiin-1-oxide as a disulfur monoxide (S_2O) source for S_2O insertion into the metal carbon bond of $CpMe_nFe(CO)_2R$ (n = 0, 5; R = Me, Et) yielded unexpected iron thioester complexes, $CpMe_nFe(CO)_2SC(O)R$. The characterization and independent syntheses of these thioester complexes are reported as well as the results of experiments designed to yield some information about the mechanism of formation of these thioester complexes.

Introduction

We recently initiated a research program to develop new synthetic routes to S_2O coordination complexes and to study the reactions of S_2O with a variety of transition metal complexes [1]. As part of this program we wanted to investigate the possibility of using 4,5-diphenyl-3,6-dihydro-1,2-dithiin-1-oxide (4) as a S_2O source for insertion of S_2O into metal carbon bonds. We then hoped to be able to use S_2O insertion products (5) in thiosulfinate ester synthesis [2]. We chose to initiate this



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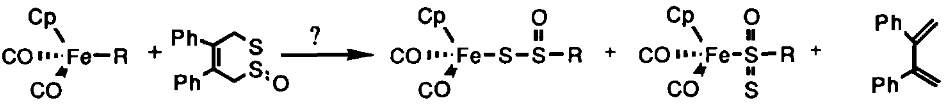

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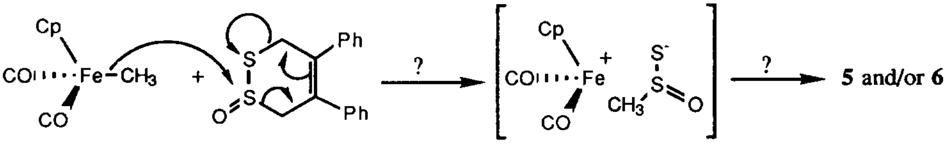
study with the iron alkyls (**1**) because of their ready accessibility from inexpensive starting materials [3], and because sulfur dioxide (SO_2) was known to insert rapidly into the iron carbon bond of these complexes [4]. These SO_2 insertions have been shown to proceed through the intermediacy of *O*-bonded sulfinates (**2**) which isomerize to the isolable *S*-sulfonate products (**3**) [5]. Since we had previously shown that **4** can serve as a S_2O source [1], we postulated that when iron alkyls (**1**) were treated with **4**, we would isolate metallothiosulfinate esters (**5**) and/or metal *S*-thiosulfonates (**6**). The details of our experiments toward this end are presented here.

Results and discussion

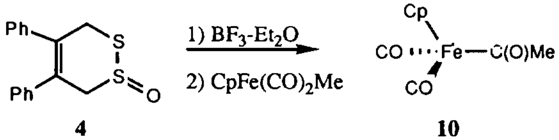
From our earlier work [1b], we knew that **4** was susceptible to attack from transition-metal nucleophiles, so we postulated that when **4** was treated with iron methyl complex (**8**) reaction would proceed through an intermediate of structure (**9**) to yield **5** or **6**. However, when **8** (0.05 *M* in CH_3CN, CH_3NO_2, or THF) was treated with **4**, there was no evidence for reaction by ^1H NMR or TLC after 24 h at 25 ° C. Heating these solutions resulted only in the decomposition of **4** into **7**.



8 **4** **9**

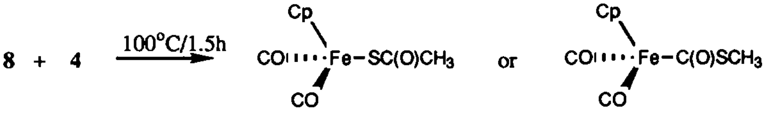
$$\mathbf{8} + \mathbf{4} \xrightarrow[\text{CH}_3\text{CN or CH}_3\text{NO}_2]{\text{THF,}} \text{No reaction at 25 °C; decomposition upon heating}$$

We next attempted to activate **4** to nucleophilic attack by treatment of **4** with Lewis acids prior to addition of **8**. Wojcicki had shown previously that SO_2 insertion could be accelerated by Lewis acids [6], and we knew that **4** interacted with "hard" Lewis acids [7] through oxygen complexation and that retro Diels–Alder reaction of **4** was slow in the presence of "hard" Lewis acids [1c]. When **4** was treated with $Eu(FOD)_3$ followed by addition of **8**, monitoring of the reaction by ^1H NMR showed slow decomposition of **4** into **7** over a period of 48 h but **8** remained unaffected in solution. Treatment of **4** with BF_3–Et_2O followed by addition of **8** lead only to isolation of **7** (51%) and iron acetyl (**10**) (32%). The Lewis acid here is simply serving to accelerate CO insertion for **8** rather than nucleophilic attack on **4** [8].



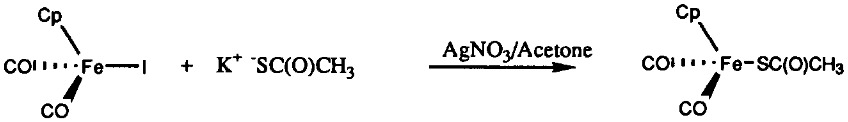
4 **10**

Our last attempt at S_2O insertion with iron alkyls involved mixing **8** with **4** and simply melting them together at 100 ° C under nitrogen for 1.5 h. To our surprise, we isolated a complex of molecular formula $C_9H_8O_3SFe$ from this reaction. This material exhibited 2 metal carbonyl IR absorptions (2044, 1996 cm^{-1}, $CDCl_3$) and 1 other carbonyl absorption (1629 cm^{-1}, $CDCl_3$). Its ^1H NMR spectrum ($CDCl_3$) contained only 2 singlets at δ4.99 (5H) and 2.42 (3H). On the basis of this initial information, we formulated the product of this reaction as having either structure **11** or **12**. The isolated yield of this material is reproducibly 40–45%, 50% being the maximum possible without a CO source other than **8**. The isolated yield (based on **4**) of **11**/**12** improved slightly to 55% when two equivalents of **8** were used.

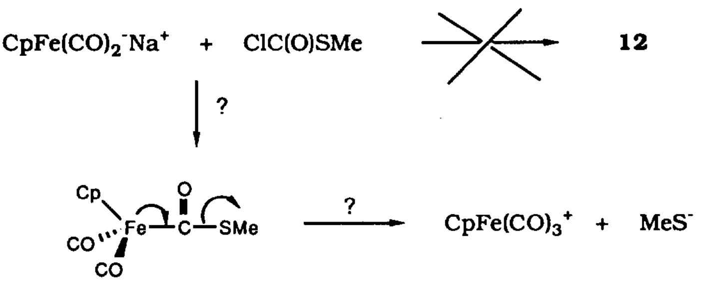


11 **12**

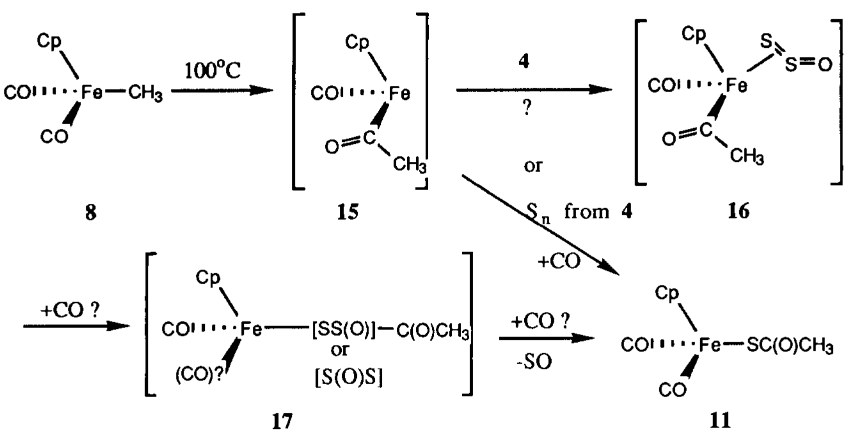
To prove the structure of this material as **11** or **12**, we sought to independently synthesize both **11** and **12** and then compare spectral properties with the material from the attempted insertion reaction. We synthesized **11** by a slight modification of a known procedure [9] (82%) and it proved to be identical by spectroscopic and chromatographic comparison to the material isolated from the thermal reaction of **8** with **4**. A ^1H NMR spectrum of a mixture of the pyrolysis product and the thioacetate reaction product was identical to that of the individual components. Several attempts (reaction at 25, 0, and −78 °C) to synthesize the other possible regioisomer (**12**) from $NaCpFe(CO)_2$ [3] and methylchlorothioformate [10] resulted only in the production of $[CpFe(CO)_2]_2$. This may not be surprising since other iron acetyls with poorer leaving groups than the thiomethoxide anion are also known to be unstable [11].



13 **14** **11**



The origin of **11** from the reaction of **8** and **4** is puzzling (Scheme 1). Analysis of the pyrolysis reaction by ^1H NMR at short reaction times showed only **8** and **11**. Due to the position of the sulfur in the final product one has to postulate that methyl migration, to yield an intermediate of structure **15** occurs before sulfur incorporation. Evidence for an intermediate of structure **15** comes from photolysis of iron acetyl **10** in the presence of **4**. This photochemical reaction, which is known to proceed through **15** [12], produced **8** (the deinsertion product) and **11** in a 2 : 1 ratio.



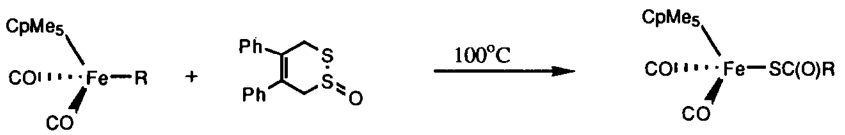
Scheme 1

Incorporation of the last CO could occur at any one of a number of points in Scheme 1. We believe it occurs after methyl migration and sulfur incorporation since heating acetyl **10** in the presence of **4** under conditions identical to those used for the reaction of **8** and **4** yielded only unreacted **10** and diene **7**. Proposing a mechanism involving S_2O coordination which accounts for the production of **11** from **15** is difficult. $CpFe(CO)(C(O)CH_3)(S_2O)$ (**16**) could be involved as an intermediate because our earlier studies had shown that **4** undergoes a rapid retro Diels–Alder reaction in the presence of coordinatively unsaturated transition metal complexes [1c]. However, loss of the elements S and O from **16** (as SO?) is also required to give the final product **11**. Analysis of the gas phase (by IR) above the pyrolysis of **8** and **4** was performed in hopes of gaining some information relevant to sulfur and oxygen loss. Heating **8** and **4** in an evacuated Schlenk tube followed by periodic analysis (10–40 min after pyrolysis) showed a large amount of sulfur dioxide and a trace of carbon monoxide [13]. Sulfur dioxide should be the detectable product of any pyrolysis reaction where SO or S_2O are generated because sulfur monoxide has a halflife of about 2 msec and is known to disproportionate into S_2O and SO_2 and S_2O is known to rapidly disproportionate into sulfur and SO_2 upon heating [14]. Analysis of the gas produced by pyrolysis of **4** alone under identical conditions also yielded only SO_2.

$$10 \quad + \quad 4 \quad \xrightarrow{h\nu/25^{\circ}C} \quad 8 \quad + \quad 11$$

$$10 \quad + \quad 4 \quad \xrightarrow{100^{\circ}C} \quad 10 \quad + \quad 7$$

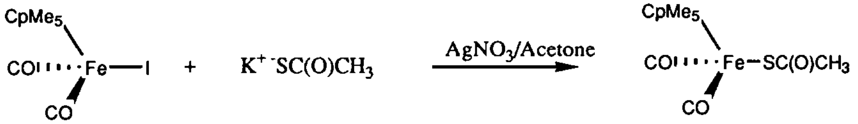
The simplest way to account for the production of **11** in the pyrolysis reaction would be to postulate that **15** simply reacts with a sulfur fragment (S_n) produced by thermal decomposition of **4**. To test this hypothesis, **8** and sulfur were heated under conditions identical to those used in the pyrolysis of **8** and **4**. This reaction did indeed produce **11** (36%, 90% based on **8** consumed) in addition to some recovered unreacted **8** (22%). Based on this result and our previous experiments it seems most likely that thermal decomposition of **4** is producing sulfur which reacts with **15** to produce **11**.



18 R=Me
19 R=Et

4

20 R=Me, 34%
21 R=Et, 40%



22

14

20 62%

We next attempted to examine the insertion of S_2O into the metal carbon bond of $Me_5CpFe(CO)_2R$ complexes (**18**, **19**) since SO_2 insertion had been shown by Wojcicki to be accelerated by substituting the more electron releasing pentamethylcyclopentadienyl ligand for the unsubstituted cyclopentadienyl ligand [4c]. No reaction was observed at 25 °C and once again only decomposition of **4** into **7** was noted upon heating in solution. Heating and stirring **18** or **19** with **4** under nitrogen produced the thioester complexes **20** and **21** analogous to what we had observed for the cyclopentadienyl complexes. Once again, we confirmed that the pyrolysis product **20** had the FeSC(O)Me rather than the FeC(O)SMe bond linkage through independent synthesis. Treatment of $Me_5CpFe(CO)_2I$ (**22**) [21] with potassium thioacetate (**14**) in acetone yielded a product **20** identical by spectroscopic and chromatographic comparison with that isolated from the pyrolysis of **18** and **4**. Attempts to synthesize the other possible regioisomer from the reaction of $Me_5CpFe(CO)_2Na$ [15] and methylchlorothioformate [10] were unsuccessful. Instead the major product of this reaction was the dimer $[Me_5CpFe(CO)_2]_2$.

Conclusions

In summary, attempted insertion of S_2O into the iron–carbon bond of cyclopentadienyl iron dicarbonyl alkyl complexes has yielded unexpected iron thioester complexes. Two characteristics of the $CpFe(CO)_2R$ complexes may be responsible for their failure to react with **4** to yield SO_2 insertion products: 1) their known abilities to participate in thermal CO insertion (alkyl migration) reactions [16] and 2) their relatively low nucleophilicity due to the presence of the π-acid CO ligands. We are attempting to circumvent these problems by investigating insertion of S_2O into the metal carbon bond of $CpNi(PR_3)R$ complexes [17]. The results of these experiments will be reported in due course.

Experimental

General comments

All infrared spectra were recorded on Perkin–Elmer 1330 or 1620 infrared spectrophotometers. Nuclear magnetic resonance spectra were obtained on a Varian

VXR-200. All ^1H and ^{13}C absorptions were expressed in parts per million (δ) relative to residual $CHCl_3$. Melting points were determined on a Mel-Temp melting point apparatus and were reported uncorrected. Combustion analyses were performed by Atlantic Microlab, Inc. and mass spectra were recorded at the Midwest Center for Mass Spectrometry. Tetrahydrofuran and diethyl ether were distilled from sodium/benzophenone under nitrogen immediately prior to use. Dichloromethane was distilled from calcium hydride under nitrogen immediately prior to use. All reactions were carried out under an atmosphere of dry nitrogen unless otherwise noted. $NaCpFe(CO)_2$ [3], $CpFe(CO)_2Me$ [3] (**8**), $CpFe(CO)_2$-C(O)Me [18] (**10**), $Me_5CpFe(CO)_2Me$ [15] (**18**), $Me_5CpFe(CO)_2Et$ [19] (**19**), CpFe-$(CO)_2I$ [20] (**13**), $Me_5CpFe(CO)_2I$ [21] (**22**), 4,5-diphenyl-3,6-dihydro-1,2-dithiin-1-oxide [1c] (**4**) and methylchlorothioformate [10] were all synthesized according to reported procedures.

Reaction of $CpFe(CO)_2Me$ (8) with 4 in the presence of BF_3–Et_2O

S_2O precursor **4** (0.055 g, 0.19 mmol) was dissolved in dichloromethane (5 mL) and cooled to 0 °C under nitrogen. BF_3–Et_2O (0.021 mL, 0.17 mmol) was added via syringe and the solution was stirred for 0.25 h prior to the dropwise addition of iron methyl **8** (0.033 g, 0.17 mmol) in dichloromethane (1 mL). The solution was stirred for 4 h at 0 °C and then water (20 mL) was added. Extraction with dichloromethane (2 × 15 mL), drying ($MgSO_4$), followed by solvent removal by rotary evaporation yielded a crude product which by ^1H NMR contained only diene **7**, iron acetyl **10** and a trace of unreacted **8**. Chromatography (silica gel, 230–400 mesh, 2 : 1 CH_2Cl_2 : petroleum ether) yielded diene **7** (0.020 g, 51%) and iron acetyl **10** (0.012 g, 32%) identical to authentic samples by spectral comparison [22,18].

Thermal reaction of iron methyl (8) with 4

Cyclopentadienyl iron dicarbonyl methyl complex [3] **8** (0.100 g, 0.52 mmol) and dithiin-1-oxide (**4**) (0.149 g, 0.52 mmol) were placed in a 1 mL round bottom flask under N_2 and heated (100 °C) with stirring for 1.5 h. Periodic TLC analysis (silica gel, 1 : 1 petroleum ether : diethyl ether) during the heating period showed gradual disappearance of **8** ($R_f = 0.9$) and appearance of a new material ($R_f = 0.5$). After heating, the crude product was dissolved in acetone and chromatographed on silica gel (230–400 mesh). Elution with petroleum ether yielded 2,3-diphenylbutadiene (**7**) (0.041 g, 38%) and a trace of unreacted **8**. Elution with 2 : 1 petroleum ether : diethyl ether yielded **11** (0.054 g, 41%). Use of 2 equivalents of **8** (0.200 g) under identical conditions yielded **11** (0.072 g, 55%). Spectral data for **11**: m.p.: (petroleum ether/diethyl ether) 60–62 °C (lit. [9] 59–60 °C); IR ($CDCl_3$, cm^{-1}): 2919, 2044, 1996, 1629; ^1H NMR ($CDCl_3$): 4.99 (s, 5H), 2.42, (s, 3H); ^{13}C NMR ($CDCl_3$): 212.0, 205.0, 85.3, 34.0; HRMS found: 251.9545; $M-CO$: 223,9601; $M-2CO$: 195.9652; $C_9H_8FeO_3S$ calcd.: 251.9553.

Reaction of $CpFe(CO)_2I$ (13) with potassium thioacetate

Iron iodide [20] (**13**) (0.260 g, 0.856 mmol) and silver nitrate (0.250 g, 1.47 mmol) were dissolved in acetone (70 mL) and stirred for 2 h under N_2. The solution was then filtered and potassium thioacetate (0.133 g, 1.16 mmol) was added to the filtrate. The solution was then refluxed (2 h) and cooled and filtered. Rotary evaporation was used to remove the solvent and chromatography (silica gel, 230–400 mesh; 3 : 2 petroleum ether : diethyl ether) yielded **11** (0.216 g, 82%) m.p.: (petroleum ether) 62–63 ° C (lit. [9] 59–60 ° C), IR (CDCl$_3$, cm^{-1}): 2040, 1990, 1629; ^{1}H NMR (CDCl$_3$): 4.99 (s, 5H), 2.42, (s, 3H); ^{13}C NMR (CDCl$_3$): 212.0, 205.0, 85.3, 34.0. Anal. Found: C, 42.84; H, 3.22; S, 12.65. $C_9H_8FeO_3S$ calcd.: C, 42.88; H, 3.20; S, 12.72%.

*Attempted thermal reaction of iron acetyl **10** and **4***

Dithiin-1 oxide [1c] (**4**) (0.052 g, 0.182 mmol) and **10** (0.030 g, 0.136 mmol) were stirred and heated (100 ° C) under nitrogen for 1 h. Filtration of the crude product through silica gel (CDCl$_3$) and analysis by ^{1}H NMR showed only diene **7** and unreacted acetyl **10**.

*Photolysis of iron acetyl **10** and **4***

Iron acetyl **10** (0.020 g, 0.091 mmol) and **4** (0.029 g, 0.10 mmol) were dissolved in CDCl$_3$ (0.5 mL) along with 1,2-dichloroethane (5 μL, 0.063 mmol) as an internal standard. These materials were photolyzed (450W water cooled Hanovia lamp, quartz filter) in an inverted, sealed 5 mm NMR tube (positioned 12 inches from the lamp) and the progress of this photolysis was monitored by ^{1}H NMR. Ratios of **10**, **8**, and **11** present at different times are presented below. The relative amounts of **10**, **8**, and **11** present remained constant after 40 min and there was no **4** present. However, decomposition was significant upon further photolysis.

Time after photolysis	**10**	**8**	**11**
10 min	66%	28%	6%
20 min	42%	46%	12%
30 min	29%	49%	22%
40 min	22%	53%	25%

*Thermal reaction of **8** with sulfur*

Cyclopentadienyl iron dicarbonyl methyl complex **8** [3] (0.050 g, 0.26 mmol) and sulfur (0.075 g, 0.26 mmol) were placed in a small Schlenk tube under nitrogen. The mixture was stirred and heated (100 ° C) for 1 h after which time the solid had turned black. The residue was extracted with small portions of acetone until the extracts were colorless. The solution was then concentrated and chromatographed (silica gel, preparative TLC, petroleum ether: diethyl ether, 1 : 1) to yield unreacted **8** (0.011 g, 22%) and **11** (0.012 g, 36%) both of which were identical by TLC and ^{1}H NMR comparison with authentic samples.

*Thermal reaction of $Me_5CpFe(CO)_2Me$ (**18**) with **4***

Dithiin-1-oxide (**4**) (0.109 g, 0.381 mmol) and iron methyl complex [15] **18** (0.100 g, 0.381 mmol) were heated (100 ° C) and stirred for 1.5 h under N$_2$. The crude product was dissolved in acetone and chromatographed (silica gel, 230–400 mesh). Elution with pentane yielded 2,3-diphenylbutadiene (**7**) (0.048 g, 61%) (identical to an authentic sample by spectral comparison) [22]. Elution with 5 : 1 petroleum ether : diethyl ether yielded **20** (0.042 g, 34%) as an orange solid; m.p. 94–95 ° C (petroleum ether/diethyl ether); IR (CDCl$_3$, cm^{-1}): 2023, 1979, 1620; ^{1}H NMR (CDCl$_3$): 2.41 (s, 3H), 1.78 (s, 15H); ^{13}C NMR (CDCl$_3$) (<200 ppm): 96.1, 34.1, 9.6; HRMS: found: 322.0319. $C_{14}H_{18}FeO_3S$ calcd.: 322.0325. Elution with 1 : 1 ethanol : diethyl ether yielded (0.007 g) of an additional iron complex which was not present in the crude product NMR and which may be a product of reaction of **20** with the adsorbent.

Reaction of $Me_5CpFe(CO)_2I$ (22) with potassium thioacetate

$Me_5CpFe(CO)_2I$ [21] (**22**) (0.300 g, 0.802 mmol) and silver nitrate (0.163 g, 0.962 mmol) were dissolved in acetone (70 mL) and stirred for 4 h under N_2. The resulting orange solution was filtered and potassium thioacetate (0.128 g, 1.12 mmol) was added. This solution was then refluxed (2 h) under nitrogen. After cooling, the solution was filtered and the solvent removed by rotary evaporation. The crude product was chromatographed (silica gel, 230–400 mesh, 1 : 1 diethyl ether : petroleum ether) to yield **20** (0.160 g, 62%) as an orange solid; m.p.: (petroleum ether/diethyl ether) 94–95 °C; IR ($CDCl_3$, cm^{-1}): 2024, 1974, 1624; ^1H NMR ($CDCl_3$): 2.41 (s, 3H), 1.78 (s, 15H); ^{13}C NMR ($CDCl_3$): 214.5, 200.0, 96.1, 34.1, 9.6; Anal. Found: C, 52.27; H, 5.66; S, 10.01. $C_{14}H_{18}FeO_3S$: calcd.: C, 52.19; H, 5.63; S, 9.95%.

Thermal reaction of iron ethyl complex 19 with 4

Iron ethyl complex [19] **19** (0.100 g, 0.362 mmol) and **4** (0.114 g, 0.398 mmol) were heated (100 °C) and stirred under nitrogen for 1.5 h. ^1H NMR analysis of the crude product showed 1 new cyclopentadienyl resonance in addition to a small amount of unreacted **19**. The crude product was dissolved in acetone and chromatographed (silica gel, 230–400 mesh). Elution with 10 : 1 petroleum ether : diethyl ether yielded **7** followed by unreacted **19** (0.016 g, 16%). Elution with 1 : 1 petroleum ether : diethyl ether yielded **21** (0.048 g, 40%) m.p.: (petroleum ether) 77–79 °C; IR ($CDCl_3$, cm^{-1}): 2983, 2918, 2023, 1974, 1617; ^1H NMR ($CDCl_3$): 2.68 (q, $J = 7$ Hz, 2H); 1.74 (s, 15H); 1.09 (t, $J = 7$ Hz, 3H); ^{13}C NMR ($CDCl_3$): (< 200 ppm) 96.2, 40.5, 11.4, 9.7; HRMS Found: $M^+ - CO$ 208.0547. $C_{15}H_{20}FeO_3S$ calcd.: 336.0475, $M^+ - CO$ 308.0526.

Acknowledgements

We are grateful to the North Carolina Board of Science and Technology, the donors of the Petroleum Research Fund, administered by the American Chemical Society, and the National Science Foundation (CHE 8817985) for support of this work. The Midwest Center for Mass Spectrometry, a National Science Foundation regional instrumentation facility (CHE 8211164), performed mass spectrometric analyses.

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