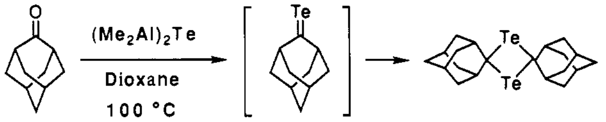
involve the initial coordination of aluminum of **1** to carbonyl oxygen followed by intramolecular transfer of the tellurium group to the electropositive carbonyl carbon center. Thermodynamically stable Al–O–Al bond formation then leads to the construction of the carbon–tellurium double bond.

In the absence of a diene, **1** reacted with adamantanone to give a dimer of telluroadamantanone in 28% yield (eq 3). The mass



(3)

spectrum of this compound supported the dimeric molecular formula of $C_{20}H_{28}Te_2$ with a molecular ion at m/e = 528 (^{130}Te) having the expected isotope pattern for Te_2.

Thus, it becomes possible for the first time to study the chemistry of telluroaldehydes and -ketones.

Acknowledgment. This work was supported in part by a Grant-in-Aid for Scientific Research (63750830) from the Ministry of Education, Science and Culture of Japan. We also thank Professors Noboru Sonoda and Shinji Murai (Osaka University) for their helpful discussions and comments.

Supplementary Material Available: IR, ^{1}H and ^{13}C NMR, and mass spectral data for all cycloadducts described (4 pages). Ordering information is given on any current masthead page.

A Reactive Organometallic Oxo Intermediate, $Cp^*_2Zr{=}O$: Generation and Subsequent Trapping Reactions Forming Alkyne and Nitrile Addition Products

Michael J. Carney, Patrick J. Walsh, Frederick J. Hollander, and Robert G. Bergman*

Department of Chemistry, University of California
Berkeley, California 94720

Received July 24, 1989

The metal oxo linkage (M=O) in high-valent group 5–8 metal complexes is relatively well-known[1] but is normally quite robust.[2] The apparent lack of monomeric group 4 oxometallocene species ((η^5-$C_5R_5)_2$M=O, M = Ti, Zr, Hf) in the literature led us to believe that if these could be generated they would indeed display greater reactivity than that of metal oxo species already known. In addition, the recent successful isolation and characterization of a reactive monomeric zirconium imido complex, Cp_2(THF)-Zr=N(t-Bu) (Cp = η^5-C_5H_5), in these laboratories[3,4] indicated to us that perhaps the isoelectronic Zr=O complex could be generated in a similar α-elimination reaction.[5] We report here evidence supporting the successful generation of Cp^*_2Zr=O (Cp* = η^5-C_5Me_5), its subsequent trapping with various unsaturated organic molecules, and the unusual rearrangement reactions of some of these trapped products.

The chemistry observed is outlined in Scheme I. Heating Cp^*_2ZrPh(OH)[6] (**1**) in toluene or benzene at 160 °C in the presence of 3–4 equiv of diphenyl- or di-p-tolylacetylene leads to loss of benzene (observed by ^{1}H NMR spectrometry) and generation of the orange zirconium oxametallacycles **2** (68% isolated yield;[7] 92% yield by ^{1}H NMR) and **3** (78% isolated yield), respectively. Isolation of **3** indicates that the phenyl group initially bound to Zr in **1** is liberated (vide infra). Complex **2** has been characterized by X-ray diffraction;[8] an ORTEP diagram is included in Scheme I and clearly shows that the phenyl ring is orthometalated.

Heating **1** in the presence of excess 1,4-diphenyl-1,3-butadiyne leads to the formation of a deep red compound, **4** (84% isolated yield), in which a Cp* ligand is now bonded to the coordinated enolate (see Scheme I). A single-crystal X-ray diffraction study[8] confirmed this description of **4**; an ORTEP diagram is included in Scheme I. Heating **1** at 160 °C in the presence of 3–4 equiv of benzonitrile (PhCN) provides an isolable compound that we formulate as the PhCN insertion product, **5**, in 74% yield. Here 2 equiv of PhCN have added to give the six-membered metallacycle, as illustrated in Scheme I. Once again, an X-ray structure determination of **5** was carried out[8] and confirms the proposed connectivity.

The following experiments provide information about the mechanism of thermal decomposition of hydroxyphenylzirconium complex **1**. (1) GC–MS analysis of the volatile materials formed upon separate thermolysis of Cp^*_2ZrPh(OH) and Cp^*_2ZrPh(OD)[9] (160 °C in toluene in the presence of PhCCPh) indicates that C_6H_6 and C_6H_5D, respectively, are the exclusive (≥97%)[10] elim-

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[1] (a) Nugent, W. A.; Mayer, J. A. *Metal-Ligand Multiple Bonds*; Wiley-Interscience: New York, 1988. (b) Holm, R. H. *Chem. Rev.* **1987**, *87*, 1401 and references therein.

[2] Apparent exceptions among organometallic oxo compounds include certain Cp*Re oxo complexes (Herrmann, W. A. *J. Organomet. Chem.* **1986**, *300*, 111) and the recently reported Cp^*_2W=O (Parkin, G.; Bercaw, J. E. *J. Am. Chem. Soc.* **1989**, *111*, 391).

[3] Walsh, P. J.; Hollander, F. J.; Bergman, R. G. *J. Am. Chem. Soc.* **1988**, *110*, 8729.

[4] For a related zirconium imido complex, see: Cummins, C. C.; Baxter, S. M.; Wolczanski, P. T. *J. Am. Chem. Soc.* **1988**, *110*, 8731.

[5] Pertinent references concerning α-elimination to generate isolobal alkylidene complexes include the following: (a) McDade, C.; Green, J. C.; Bercaw, J. E. *Organometallics* **1982**, *1*, 1629. (b) Bulls, A. R.; Schaefer, W. P.; Serfas, M.; Bercaw, J. E. *Organometallics* **1987**, *6*, 1219. (c) Gibson, C. P.; Dabbagh, G.; Bertz, S. H. *J. Chem. Soc., Chem. Commun.* **1988**, 603. (d) Bertz, S. H.; Dabbagh, G.; Gibson, C. P. *Organometallics* **1988**, *7*, 563.

[6] Schock, L. E.; Marks, T. J. *J. Am. Chem. Soc.* **1988**, *110*, 7701.

[7] Compounds **2–5** were fully characterized by ^{1}H and ^{13}C{^{1}H} NMR, IR, and EI mass spectrometry and elemental analysis; data are provided as supplementary material. All yields are for recrystallized complexes.(7) The X-ray diffraction studies were carried out at the University of California, Berkeley, X-ray diffraction facility (CHEXRAY) with Mo Kα (I = 0.71073 Å) radiation at 25 °C. Crystal data for **2**: $P2_1/n$, V = 2908.9 (9) Å^3, μ = 3.9 cm^{-1}, D_{calcd} = 1.27 g cm^{-3}, a = 9.4243 (10) Å, b = 18.7513 (17) Å, c = 16.8210 (17) Å, β = 101.882 (9)°, Z = 4; the final residuals for 325 variables refined against the 3169 data for which $F^2 > 3\sigma(F^2)$ were R = 0.0272, R_w = 0.0398, and GOF = 2.05. The R value for all 3797 data was 4.81%. Crystal data for **4**: $P2_1/n$, V = 2974.8 (12) Å^3, μ = 3.8 cm^{-1}, D_{calcd} = 1.24 g cm^{-3}, a = 9.6706 (17) Å, b = 10.2703 (11) Å, c = 30.132 (3) Å, β = 96.285 (12)°, Z = 4; the final residuals for 343 variables refined against 3176 data for which $F^2 > 3\sigma(F^2)$ were R = 2.82%, R_w = 3.96%, and GOF = 1.99. The R value for all 3882 data was 4.13%. Crystal data for **5**$\cdot{}^1/_2C_6H_5CH_3$: $P2_1/c$, V = 3330.1 (17) Å^3, μ = 3.5 cm^{-1}, D_{calcd} = 1.26 g cm^{-3}, a = 9.4840 (20) Å, b = 17.5041 (19) Å, c = 20.670 (1) Å, β = 103.960 (15)°, Z = 4; the final residuals for 389 variables refined against 3598 data for which $F^2 > 3\sigma(F^2)$ were R = 3.13%, R_w = 4.25%, and GOF = 2.09. The R value for all 4337 data was 4.28%. The six-membered metallacycle in **5** is disordered about a pseudo-two-fold axis containing the zirconium atom and the central nitrogen atom of the ring. Details of the structure determinations are included as supplementary material.

[8] The X-ray diffraction studies were carried out at the University of California, Berkeley, X-ray diffraction facility (CHEXRAY) with Mo Kα (I = 0.71073 Å) radiation at 25 °C. Crystal data for **2**: $P2_1/n$, V = 2908.9 (9) Å^3, μ = 3.9 cm^{-1}, D_{calcd} = 1.27 g cm^{-3}, a = 9.4243 (10) Å, b = 18.7513 (17) Å, c = 16.8210 (17) Å, β = 101.882 (9)°, Z = 4; the final residuals for 325 variables refined against the 3169 data for which $F^2 > 3\sigma(F^2)$ were R = 0.0272, R_w = 0.0398, and GOF = 2.05. The R value for all 3797 data was 4.81%. Crystal data for **4**: $P2_1/n$, V = 2974.8 (12) Å^3, μ = 3.8 cm^{-1}, D_{calcd} = 1.24 g cm^{-3}, a = 9.6706 (17) Å•, b = 10.2703 (11) Å, c = 30.132 (3) Å, β = 96.285 (12)°, Z = 4; the final residuals for 343 variables refined against 3176 data for which $F^2 > 3\sigma(F^2)$ were R = 2.82%, R_w = 3.96%, and GOF = 1.99. The R value for all 3882 data was 4.13%. Crystal data for **5**$\cdot{}^1/_2C_6H_5CH_3$: $P2_1/c$, V = 3330.1 (17) Å^3, μ = 3.5 cm^{-1}, D_{calcd} = 1.26 g cm^{-3}, a = 9.4840 (20) Å, b = 17.5041 (19) Å, c = 20.670 (3) Å, β = 103.960 (15)°, Z = 4; the final residuals for 389 variables refined against 3598 data for which $F^2 > 3\sigma(F^2)$ were R = 3.13%, R_w = 4.25%, and GOF = 2.09. The R value for all 4337 data was 4.28%. The six-membered metallacycle in **5** is disordered about a pseudo-two-fold axis containing the zirconium atom and the central nitrogen atom of the ring. Details of the structure determinations are included as supplementary material.

[9] Cp^*_2ZrPh(OD) was prepared analogously to Cp^*_2ZrPh(OH) (ref 6) with D_2O used in place of H_2O. Integration (^{1}H NMR) of the residual OH versus the Cp* or phenyl resonances indicated that Cp^*_2ZrPh(OD) was ca. 98% deuterated.

[10] The percentage is based on a comparison of the GC–MS data (fragmentation patterns and peak heights) of these products with those of authentic samples of C_6H_6 and C_6H_5D (the latter prepared from PhLi and D_2O).

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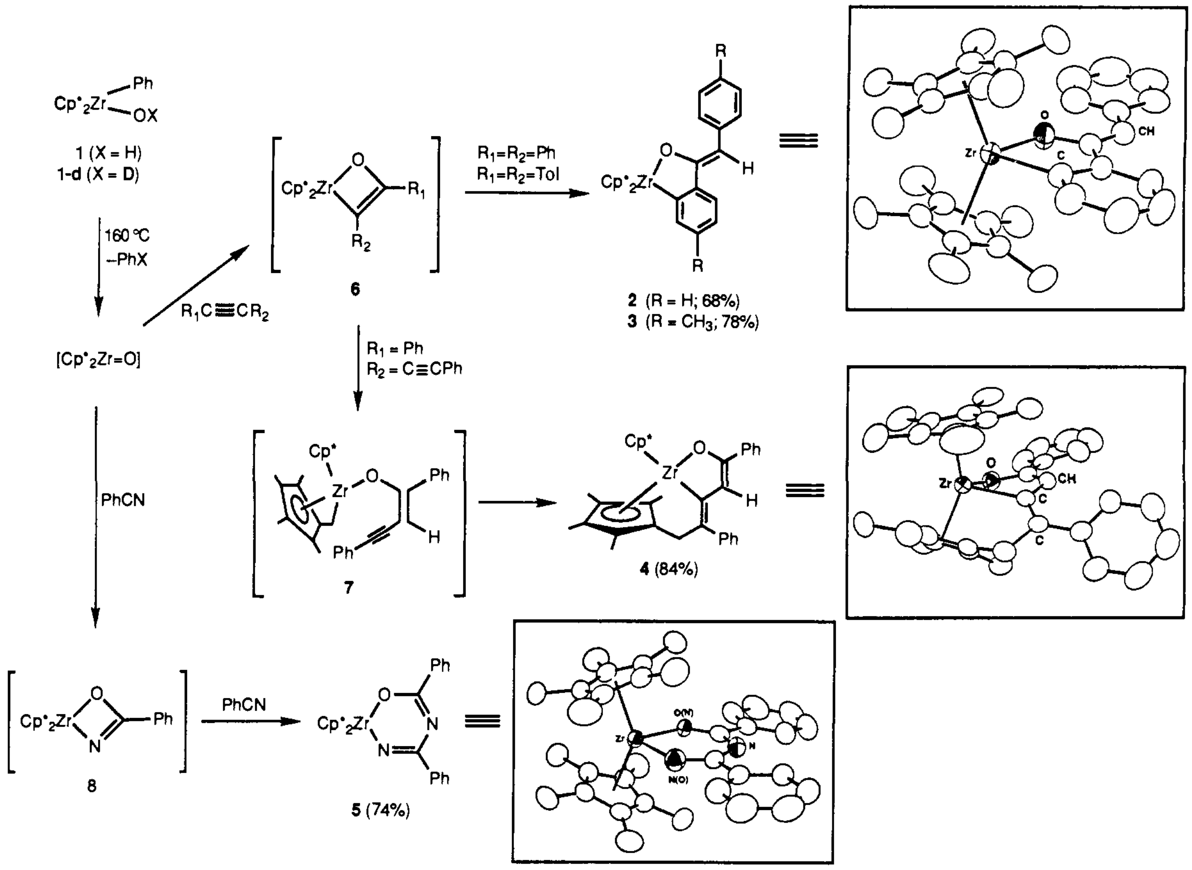
Scheme I



ination products. (2) Kinetic evidence favors initial loss (α-elimination) of benzene from **1**. When **1** is heated at 160 °C in C_6D_6[11] in the presence of 3.5, 7.5, or 11.0 equiv of ditolylacetylene or in the presence of 1,4-diphenyl-1,3-butadiyne or benzonitrile, the rate of disappearance of **1** (monitored by ^1H NMR spectrometry) is clearly first order ($k_{obsd} = (3.5 \pm 0.2) \times 10^{-5}\ s^{-1}$) and is *independent of the type or concentration of trapping ligand.* (3) Studies of the rate of thermal decomposition of $Cp^*_2ZrPh(OD)$ at 160 °C in C_6D_6 reveal a substantial deuterium isotope effect ($k_H/k_D = 4.3 \pm 0.1$), indicating that an O-H (or O-D) bond undergoes cleavage in the transition state of the rate-determining step.[12]

These observations are consistent with our initial hypothesis that thermolysis of **1** proceeds by first-order, rate-determining elimination of benzene, leading to $Cp^*_2Zr{=}O$. By analogy to the behavior of $Cp_2Zr{=}N(t\text{-}Bu)$, we propose that alkynes react initially with the oxo complex to give oxametallacycles of type **6**.[13] However, under the conditions necessary to generate $Cp^*_2Zr{=}O$, these oxametallacycles are unstable and rearrange to cyclometalated products **2**–**4**. Fortunately, Hillhouse and coworkers have recently prepared the oxametallacycle **6** ($R_1 = R_2$ = Ph) upon treatment of the zirconocene diphenylacetylene complex $Cp^*_2Zr(PhCCPh)$ with N_2O.[14] These workers have kindly provided us with a sample of this metallacycle, and we find that its thermolysis at 160 °C (40 min) leads to **2** (86% by ^1H NMR). This provides strong additional evidence that **6** is an intermediate in the overall reaction of **1** with diphenylacetylene.

The details of the conversion of **6** to the final alkyne-trapped and rearranged products **2**–**4** are not yet clear. The formation of **4** from the diacetylene suggests that Cp*-mediated cleavage of the Zr–C bond in **6**[15] can occur, leading in this case to intermediate **7**; rearrangement involving the remaining C≡C triple bond would then give **4**. Preliminary experiments suggest that a somewhat different process operates in the conversion of **6** to **2** and **3**: thermolysis of **1** in the presence of diphenylacetylene-d_{10} gives product **2**-d_{10} in which approximately 25% of the deuterium atom removed from the ortho position of the aromatic ring has been incorporated into a Cp* methyl and 75% is located in the exocyclic vinyl position. Thus, although part of the product may arise from initial Cp* metalation (followed by "σ-bond metathesis"[16]), the majority is produced by hydrogen (deuterium) transfer from the phenyl, rather than the Cp*, ring. Possible mechanisms that may account for this involve cleavage of the Zr—C bond in **6**, leading to a zwitterionic or diradical intermediate followed by hydrogen transfer, or reversion of **6** to $Cp^*_2Zr(O)(ArC{\equiv}CAr)$, with subsequent addition of an ortho arene C—H bond across the Z=O linkage and then addition of the resulting O—H bond across the alkyne. In the reaction of $Cp^*_2Zr{=}O$ with nitriles, by analogy to its reaction with alkynes,

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[11] Similar rate constants are obtained when **1** is heated (160 °C) with excess ditolylacetylene in THF-d_8 ($k = 3.6 \times 10^{-5}\ s^{-1}$) and when **1** and ditolylacetylene are thermolyzed in C_6D_6 with 1 equiv of pyridine present ($k = 3.6 \times 10^{-5}\ s^{-1}$). This latter result suggests that these reactions are not base-catalyzed.

[12] The kinetic and isotope labeling studies performed here cannot distinguish between $Cp^*_2Zr{=}O$ and $(\eta^5\text{-}Cp^*)(\eta^5,\eta^1\text{-}C_5(CH_3)_4CH_2)Zr(OH)$ as the reactive intermediate involved in the alkyne and nitrile addition reactions. Indeed, these are isomers and may be in equilibrium under the reaction conditions employed. However, when **1** is heated in the absence of any trapping ligand, one does not obtain $(\eta^5\text{-}Cp^*)(\eta^5,\eta^1\text{-}C_5(CH_3)_4CH_2)Zr(OH)$ nor any single product cleanly.

[13] Similar cycloaddition chemistry has been observed for the isoelectronic titanium methylidene complex $Cp_2Ti{=}CH_2$. (a) Tebbe, F. N.; Parshall, G. W.; Reddy, G. S. *J. Am. Chem. Soc.* **1978**, *100*, 3611. (b) Howard, T. R.; Lee, J. B.; Grubbs, R. H. *J. Am. Chem. Soc.* **1980**, *102*, 6876. (c) Finch, W. C.; Anslyn, E. V.; Grubbs, R. H. *J. Am. Chem. Soc.* **1988**, *110*, 2406 and references therein.

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[15] For apparent Cp* mediated cleavages of group 4 metal–carbon bonds, see ref 5a,b as well as the following: (a) Parkin, G.; Bercaw, J. E. *Organometallics* **1989**, *8*, 1172. (b) Schock, L. E.; Brock, C. P.; Marks, T. J. *Organometallics* **1987**, *6*, 232.

[16] A similar "σ-bond metathesis" forming a hafnium–benzyl metallacyclobutene from $(\eta^5\text{-}Cp^*)(\eta^5,\eta^1\text{-}C_5(CH_3)_4CH_2)Hf(CH_2Ph)$ has been reported (ref 5b).

we propose initial formation of azaoxametallacycle **8**, followed by subsequent insertion of a second nitrile into the Zr—N bond of this molecule. Investigations designed to resolve these mechanistic questions are continuing and will be reported in a full paper.

Acknowledgment. We thank the National Institutes of Health (Grant No. GM-25457) for financial support of this work. M.J.C. gratefully acknowledges a postdoctoral (NRSA) fellowship from the NIH. We also thank Prof. Gregory Hillhouse and his colleagues for their generous gift of Cp*$_2$Zr(OCPh=CPh) and Drs. J. O'Connor, K. H. Theopold, and D. M. Roddick for helpful mechanistic suggestions.

Supplementary Material Available: Spectroscopic and analytical data for complexes **2–5** and details of the structure determinations for **2**, **4**, and **5** including experimental descriptions, ORTEP drawings showing full atomic numbering, crystal and data collection parameters, general temperature factor expressions (*B*'s), positional parameters and their estimated standard deviations, and intramolecular distances and angles (51 pages); tables of structure factors for **2**, **4**, and **5** (63 pages). Ordering information is given on any current masthead page.

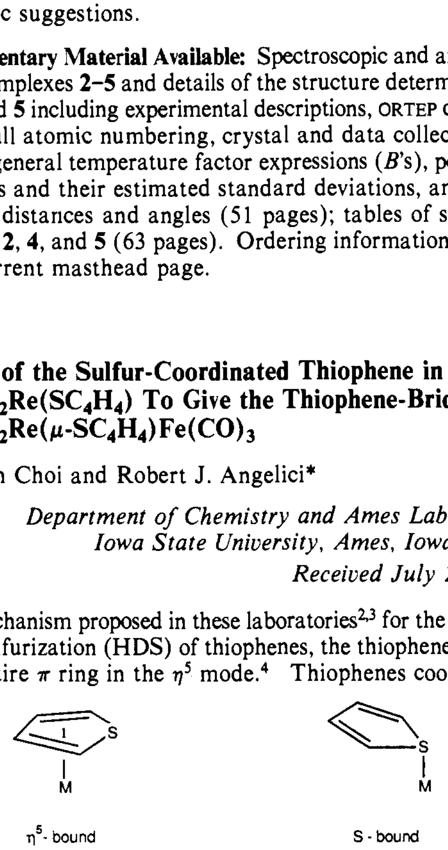
Reaction of the Sulfur-Coordinated Thiophene in Cp*(CO)$_2$Re(SC$_4$H$_4$) To Give the Thiophene-Bridged Cp*(CO)$_2$Re(μ-SC$_4$H$_4$)Fe(CO)$_3$

Moon-Gun Choi and Robert J. Angelici*

Department of Chemistry and Ames Laboratory[1]
Iowa State University, Ames, Iowa 50011

Received July 27, 1989

In a mechanism proposed in these laboratories[2,3] for the catalytic hydrodesulfurization (HDS) of thiophenes, the thiophene adsorbs via the entire π ring in the η^5 mode.[4] Thiophenes coordinated



in this manner in Mn and Ru complexes[5] are susceptible to attack by hydride sources, and this reactivity is the basis for the proposed HDS mechanism.[2,3] Another mechanism[6] assumes initial coordination of the thiophene through only the sulfur atom, and this coordination is presumed to activate the thiophene so as to give the H$_2$S and C$_4$ hydrocarbon products. However, in model complexes with S-bound thiophene (T) ligands such as CpFe(CO)$_2$(T)$^+$,[7] or even (C$_5$H$_4$CH$_2$C$_4$H$_3$S)Ru(PPh$_3$)$_2^+$,[8] where the thiophene is part of a cyclopentadienyl chelate ligand, the thiophene is so weakly coordinated to the metal that all attempted reactions of the ligand have simply led to thiophene dissociation from the metal. Thus, there is no evidence that S-coordinated thiophene is activated to react. In this communication, we describe the synthesis of an unusually stable S-bound thiophene complex Cp*Re(CO)$_2$(T) (Cp* = η^5-C$_5$Me$_5$) and its subsequent reaction with "Fe(CO)$_3$" to give a dinuclear complex Cp*(CO)$_2$Re(μ-T)Fe(CO)$_3$ in which the thiophene is S-bound to the Re and η^4-bound through the four carbons to the Fe(CO)$_3$.

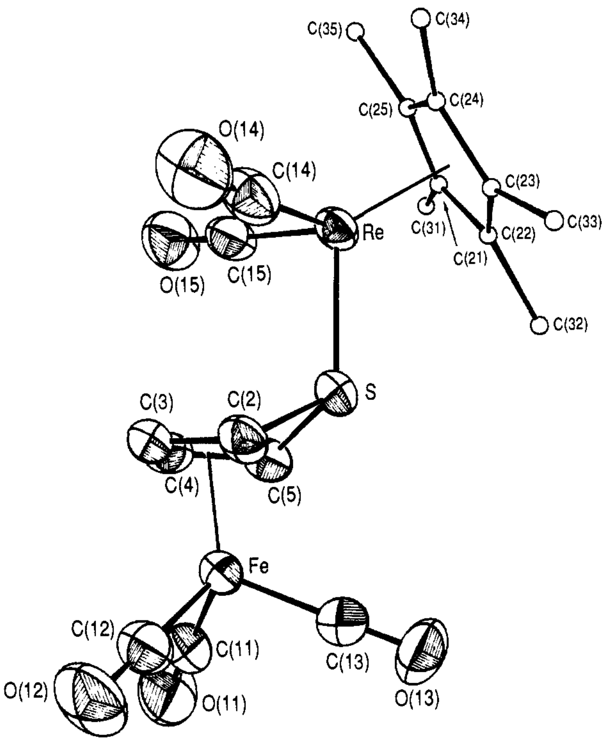


Figure 1. ORTEP drawing of Cp*Re(CO)$_2$(μ-T)Fe(CO)$_3$ (**2**). Selected bond distances (Å) and angles (deg) are Re–S = 2.330 (1), Re–C(14) = 1.884 (7), Re–C(15) = 1.883 (6), Fe–C(2) = 2.086 (6), Fe–C(3) = 2.044 (6), Fe–C(4) = 2.048 (6), Fe–C(5) = 2.099 (6), Fe–C(11) = 1.769 (8), Fe–C(12) = 1.789 (7), Fe–C(13) = 1.798 (6), S–C(2) = 1.807 (6), S–C(5) = 1.802 (5), C(2)–C(3) = 1.458 (8), C(3)–C(4) = 1.379 (9), C(4)–C(5) = 1.427 (8), C(2)–S–C(5) = 82.3 (3), C(11)–Fe–C(12) = 91.4 (3), C(11)–Fe–C(13) = 100.9 (3), C(12)–Fe–C(13) = 99.0 (3), S–Re–C(14) = 93.4 (2), S–Re–C(15) = 93.0 (2), C(14)–Re–C(15) = 87.5 (3), C(2)–C(3)–C(4) = 110.1 (5), and C(3)–C(4)–C(5) = 110.4 (5).

A solution of Cp*(CO)$_2$Re(THF), prepared by UV irradiation[9] of a THF solution (120 mL) of Cp*Re(CO)$_3$ (0.74 mmol), is stirred with thiophene (5.0 mL, 62 mmol) at room temperature for 6–8 h. After removal of the solvent in vacuo, the residue is chromatographed in CH$_2$Cl$_2$/hexanes (1:4) on neutral alumina. Slow evaporation of the solvent from the yellow band gives light yellow, air-stable crystals of Cp*(CO)$_2$Re(T) (**1**) (38% yield), which is characterized by its elemental analyses and IR, ^{1}H NMR, ^{13}C NMR, and mass spectra;[10] these data support its formulation as a complex with an S-coordinated thiophene. Although there are no crystal structures of simple thiophene complexes, the sulfur in **1** is presumed to be pyramidal, as found in the chelated thiophene complex[8] (C$_5$H$_4$CH$_2$C$_4$H$_3$S)Ru(PPh$_3$)$_2^+$ and in several thiaporphyrin complexes,[11] and the thiophene may be folded slightly (12–15°)[11] along a line from C(2) to C(5).

When a solution of Cp*(CO)$_2$Re(T) (**1**) (0.076 mmol) in THF (15 mL) is treated with Fe$_2$(CO)$_9$ (0.274 mmol) at −40 °C and then slowly warmed to room temperature and stirred for 20 h,

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[1] Ames Laboratory is operated for the U.S. Department of Energy by Iowa State University under Contract No. W-7405-Eng-82. This research is supported by the Office of Basic Energy Sciences, Chemical Sciences Division.

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[10] **1**: IR (hexanes) ν(CO) 1934, 1874 cm^{-1}; ^{1}H NMR (CDCl$_3$) δ 7.19 (br, s, 2 H, T), 7.11 (br s, 2 H, T), 1.95 (s, 15 H, Cp*); ^{13}C NMR (CDCl$_3$) δ 205.2 (CO), 138.9 and 129.9 (T), 96.0 (C of Cp*), 10.5 (Me of Cp*); EIMS (70 eV) *m/e* 462 (M$^+$), 404 (M$^+$ − C$_2$H$_2$S), 376 (M$^+$ − (C$_2$H$_2$S + CO)), 348 (M$^+$ − (C$_2$H$_2$S + 2CO)), 84 (T$^+$), 58 (C$_2$H$_2$S$^+$). Anal. Calcd for C$_{16}$H$_{19}$O$_2$ReS: C, 41.63; H, 4.15. Found: C, 41.70; H, 4.14.

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