structure has the equatorial chlorides and TMEDA nitrogens bent away from the axial multiply-bonded nitride, as expected.[9] V(1) resides in an approximately octahedral environment defined by three chlorides, two TMEDA nitrogens, and the bridging nitride. The V(1)–N(1) distance (2.092 (4) Å) is consistent with a single bond, although shortened compared to other vanadium(III)–nitrogen donor distances[10,11] (also compare V(1)–N(2) and V(1)–N(3), Figure 1) as might be expected[12,13] due to the sp hybridization of the nitride ion (V(1)–N(1)–V(2) = 172.0 (3)°). A closely related asymmetrically bridged $V^{IV}(N)V^{III}$ derivative has recently been reported.[14]

The structure indicates that **2** is best viewed as consisting of a neutral vanadium(V) nitride, $V(N)Cl_2(TMEDA)$ (**3**), coordinated to a vanadium(III) center via a dative interaction, $V^{V}{\equiv}N{:}{\rightarrow}V^{III}$.[15] The magnetic properties of this compound further support this picture. Molar susceptibilities for **2** measured between 6 and 300 K on a SQUID susceptometer show Curie–Weiss behavior consistent with an isolated d^2 vanadium(III) center ($g = 1.81$, $\mu_{eff} = 2.54\ \mu_B$ at 300 K).[16] We can prepare **3** independently by the fast reaction of **1** with TMEDA in benzene and find that it reacts immediately with $VCl_3(THF)_3$ plus TMEDA, producing **2** (Scheme I). This suggests that formation of **2** from **1**, $VCl_3(THF)_3$, and TMEDA proceeds via **3** rather than by a condensation reaction between the silylimido ligand of **1** and a chloride ligand of a VCl_3 derivative.

In the absence of TMEDA, **1** and VCl_3THF_3 react in a 2:1 ratio, producing a black crystalline solid, $V_3(N)_2Cl_7(THF)_2{\cdot}x$THF (**4**·$x$THF) ($x = 0.33$–$0.50$, on the basis of elemental analyses). Compound **4** is very labile in solution, decomposing on attempts to purify by recrystallization and reacting rapidly with TMEDA to form **2** plus **3** (Scheme I) and with pyridine to form $[V(N)Cl_2(py)_2]_n$[1,17] together with $VCl_3(py)_3$.[11] On the basis of this reactivity and by analogy to **2**, we propose that **4** consists of two vanadium(V) nitrido complexes coordinated to a vanadium(III) center (Scheme I) in a structure reminiscent of that observed for $VCl_3(NMe_3)_2$.[10]

For the variety of vanadium-containing nitride-bridged compounds that we have studied, the propensity of vanadium(V) to form strong short triple bonds to the nitride ion dominates the observed chemistry. This is again the case for the mixed-valence vanadium nitrido compounds described here. We now need to explore whether these asymmetrically substituted, asymmetrically bridged metallonitrides can be converted to symmetrically substituted, symmetrically bridged species via manipulation of the vanadium coordination environment.

Acknowledgment. We gratefully acknowledge support of this work by the Air Force Office of Scientific Research, Air Force Systems Command, USAF (Grant No. AFOSR-87-0362). Acknowledgment is made to the donors of the Petroleum Research Fund, administered by the American Chemical Society, for partial support of this research. We express our thanks to Professor Frank J. Feher for his assistance with the magnetic studies and to Professor Christopher Reed for providing access to a SQUID susceptometer at the University of Southern California. We also thank Dr. Susan C. Critchlow for a preliminary X-ray study of **2**. N.M.D. thanks the Alfred P. Sloan Foundation for a fellowship (1990–1992).

Supplementary Material Available: A textual presentation of the spectroscopic and analytical data for **2**–**4** and magnetic data for **2** (including experimental and simulation procedures), a plot of experimental and simulated magnetic data and a table of SQUID data for **2**, and X-ray crystal data for **2**, including a textual description of experimental procedures and tables of crystal data, atomic coordinates and equivalent isotropic displacement coefficients, interatomic distances, interatomic angles, anisotropic displacement coefficients, and hydrogen atom coordinates and isotropic displacement coefficients (12 pages); a listing of observed and calculated structure factors for **2** (14 pages). Ordering information is given on any current masthead page.

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(15) NMR spectra for **2** have been obtained, but provide only limited information. In CD_2Cl_2, we observe equally broad ^1H and ^{13}C NMR signals for paramagnetic **2** and diamagnetic **3**, with no resolvable H–H coupling in the ^1H NMR spectra of either compound. Additionally, decomposition of **2** (faster) and **3** (slower) is observed in dichloromethane solution, as well as in all other solvents in which these compounds are soluble. These observations preclude meaningful assignment of the NMR spectra for **2**.

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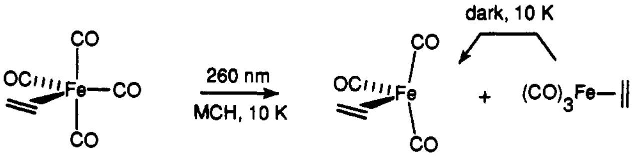
Isomerism in Coordinatively Unsaturated $Fe(CO)_3(\eta^2$-ethene) Complexes

Despite continuing experimental[1-5] and theoretical[3,6-8] interest in the structure and reactivity of $Fe(CO)_3L$ complexes, the geometry of $Fe(CO)_3(\eta^2$-ethene) remains uncertain. Hayes and Weitz recently reported evidence that $Fe(CO)_3(\eta^2$-ethene) possesses a triplet ground state, but they could not address the issue of molecular geometry.[2] We now report direct observation of geometric isomerism in coordinatively unsaturated $Fe(CO)_3(\eta^2$-ethene) complexes (**2** and **3**) using matrix-isolation spectroscopy.

Photolysis (260 ± 10 nm, 30 min) of $Fe(CO)_4(\eta^2\text{-}C_2H_4)$ (**1**),[9,10] matrix-isolated in either argon or methylcyclohexane at 10 K,[11]



1 **2** **3**

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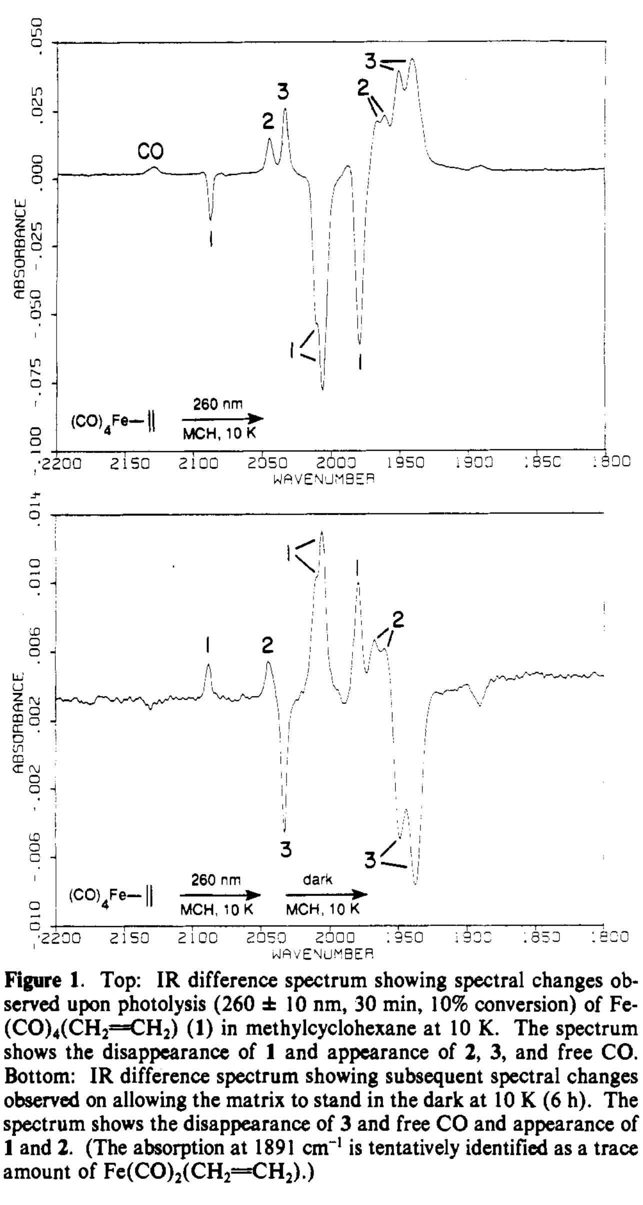

Figure 1. Top: IR difference spectrum showing spectral changes observed upon photolysis (260 ± 10 nm, 30 min, 10% conversion) of $Fe(CO)_4(CH_2{=}CH_2)$ (**1**) in methylcyclohexane at 10 K. The spectrum shows the disappearance of **1** and appearance of **2**, **3**, and free CO. Bottom: IR difference spectrum showing subsequent spectral changes observed on allowing the matrix to stand in the dark at 10 K (6 h). The spectrum shows the disappearance of **3** and free CO and appearance of **1** and **2**. (The absorption at 1891 cm^{-1} is tentatively identified as a trace amount of $Fe(CO)_2(CH_2{=}CH_2)$.)

results in a decrease in intensity of the carbonyl infrared absorptions of **1**, appearance of free CO (2132 cm^{-1}), and appearance of six new carbonyl infrared absorptions (Figure 1).[12] The following spectral changes occur upon either warming the matrix or allowing the matrix to stand in the dark at 10 K: increase in intensity of the four carbonyl absorptions of **1**, decrease in intensity of free CO, total disappearance of three of the new carbonyl absorptions, and growth of the other three new carbonyl absorptions (Figure 1). These observations suggest that (a) photolysis of **1** gives rise to two species, **2** and **3** (b) **2** and **3** each exhibit three carbonyl absorptions, and (c) **3** thermally reacts to give **2** at 10 K. The ratio of **2**:**3** depends strongly on the photolysis conditions and on the matrix material. Monitoring the photolysis (260 nm) at low conversion of starting material reveals that **2** and **3** are formed in a constant ratio (ca. 1:1.1) during the early stages of the reaction. This result implies that both **2** and **3** are primary photoproducts. Isomer **3** is a transient intermediate under all of our conditions. In contrast, **2** is stable as long as the matrix is intact. At $T > 140$ K, the methylcyclohexane glass melts, and **2** rapidly recombines with CO to regenerate **1** in nearly quantitative yield.

We infer the basic formulation $Fe(CO)_3(\eta^2\text{-}C_2H_4)$ for **2** and **3**, on the basis of the information presented above. We exclude $Fe(CO)_4$[13,14] and $Fe(CO)_3$[15] as possible structures for **2** or **3**, by comparison with the known infrared spectra of these species. We exclude $Fe(CO)_2(C_2H_4)$ as a possible structure for **2** or **3**, on the basis of the fact that both species contain (at least) three carbonyl groups and that both are primary photoproducts.[16] We cannot exclude a vinyl hydride structure[17] (e.g. $HFe(CO)_3(CH{=}CH_2)$) on the basis of spectroscopic evidence, but the presence of such a species would be difficult to reconcile with the reactivity displayed by the corresponding propene complex.[18]

Two lines of evidence argue against a scenario in which one species is the "free" coordinatively unsaturated complex and the other species is a weak solvent adduct. First, *both* **2** and **3** are observed in argon matrices, where effects of coordination are minimal.[19,20] Second, we expect a solvent adduct (which would exhibit a lower carbonyl stretching frequency[21]) to be formed upon annealing the matrix at elevated temperature. In fact, we find the opposite behavior in our experiment; the ***higher*** frequency carbonyl absorptions grow, and the lower frequency carbonyl absorptions disappear.

Several theoretical studies address the structure of ML_4 fragments. Extended Hückel calculations predict either D_{4h} (square planar) or D_{2d} geometries for low-spin d^8 ML_4[7,8] and C_{2v} geometry for high-spin d^8 ML_4.[7] Ab initio calculations predict a D_{2d} geometry for low-spin d^8 (singlet) $Ru(CO)_4$ and a C_{2v} geometry for high-spin d^8 (triplet) $Ru(CO)_4$.[22] $Fe(CO)_4$ possesses a C_{2v} geometry and a high-spin d^8 (triplet) electronic ground state.[13] Recent studies concerning the gas-phase reactivity of $Fe(CO)_3(\eta^2\text{-}C_2H_4)$ suggest that it possesses a triplet electronic ground state.[2] Thus, we postulate that $Fe(CO)_3(\eta^2\text{-}C_2H_4)$ also possesses local C_{2v} symmetry at iron. We consider two possible types of structural features that could give rise to geometric isomerism. Two distinct coordination sites exist in C_{2v} symmetry; we refer to these as pseudoequatorial and pseudoapical. Structures A–D represent four limiting geometries generated by replacing a carbonyl ligand in $Fe(CO)_4$ (**4**) with an alkene ligand. The alkene may occupy a pseudoequatorial site (lying either in the equatorial plane (A) or perpendicular to the equatorial plane (B)) or a pseudoapical site (lying either in the apical plane (C) or per-

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(12) IR data for relevant compounds [ν_{CO}, cm^{-1} (relative OD)]: **1** (Ar, 10 K), 2093 (1.0), 2019 (3.1), 2013 (4.4), 2010 (3.7), 1991 (4.1), 1983 (1.6); **1** (MCH, 10 K), 2088 (1.0), 2011 (3.3), 2006 (4.7), 1980 (3.7); **2** (Ar, 10 K), 2055 (1.0), 1975 (2.0), 1970 (1.6); **2** (MCH, 10 K), 2043 (1.0), 1967 (1.6), 1961 (1.7); **3** (Ar, 10 K), 2039 (1.0), 1960 (1.5), 1949 (2.0); **3** (MCH, 10 K), 2033 (1.0), 1952 (2.0), 1942 (2.0).

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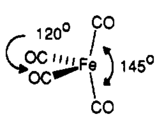
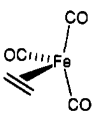
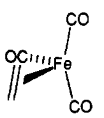


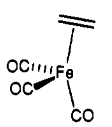
4



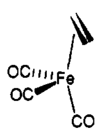
A



B



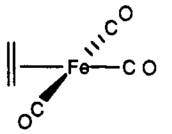
C



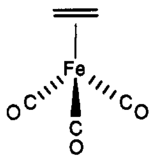
D

pendicular to the apical plane (D)). Each of the structures A–D possesses C_s symmetry and would be expected to exhibit three carbonyl stretching frequencies in the infrared spectrum.[23] Our experimental spectra are thus consistent with any of these structures.[24]

Extended Hückel calculations concerning $Fe(CO)_3(\eta^2\text{-}C_2H_4)$ predict a square-planar geometry with the alkene perpendicular to the plane of the $Fe(CO)_3$ fragment (E).[3,6] In addition, Albright et al. find that trigonal-pyramidal $Fe(CO)_3(\eta^2\text{-}C_2H_4)$ (F) lies 4 kcal/mol higher in energy than square-planar E and is separated from E by a barrier of 4 kcal/mol.[6] Although E and F represent possible structures for our observed intermediates, we hestitate to make these assignments for two reasons. First, neither report specifies the electronic configuration (high-spin or low-spin) used in the calculation. We presume a low-spin configuration was used in both instances, since the results predict a square-planar geometry. Thus, the calculations were likely performed on the wrong electronic manifold (vide supra). Second, our experiments require that 3 rearranges to 2 thermally at 10 K. The rearrangement of F to E requires a substantial geometry change, which seems unreasonable for a thermal process occurring at 10 K. Thus, structures E and F seem unlikely to us, but we cannot rigorously exclude them at this time.



E



F

We employed Fenske–Hall calculations to assess the bonding in $Fe(CO)_4$ and in structures A–D.[25,26] On the basis of these approximate calculations, it is not possible to unambiguously determine whether $Fe(CO)_4$ or $Fe(CO)_3(\eta^2\text{-}C_2H_4)$ possesses a singlet or a triplet ground state.[27] However, the following arguments are independent of whether an open-shell or closed-shell model is employed. The Mulliken population of the alkene π-orbital is the smallest for structure A (1.644), reflecting the fact that the alkene acts as the strongest σ-donor in this geometry. The Mulliken population of the alkene π^*-orbital is the largest for structure A (0.389), reflecting the fact that the alkene acts as the strongest π-acceptor in this geometry. In both instances, the population differences between A and the other three structures are quite substantial, particularly when one considers that they arise due to small variations in geometry. Both effects (better σ-donor, better π-acceptor) serve to strengthen the bond between the metal and the alkene in structure A. On this basis, we assign structure A as the thermodynamically more stable isomer (2) that is formed upon rearrangement of 3 at 10 K. The preference of the alkene to lie in the equatorial plane thus exists not only in coordinatively saturated $Fe(CO)_4(\eta^2\text{-}C_2H_4)$[28,29] but also in coordinatively unsaturated $Fe(CO)_3(\eta^2\text{-}C_2H_4)$. The population of the carbonyl π^*-orbitals is the smallest for structure A, which is consistent with our observation that 2 exhibits the higher-frequency infrared carbonyl absorptions.

Neither our experimental results nor our theoretical calculations provide enough information to make a definitive structural assignment for 3. At the present time, we favor formulation as an apical isomer (C or D), on the basis of the precedent of equatorial/apical isomerism in isotopomers of $Fe(CO)_4$. The thermal rearrangement of 3 to 2 at 10–50 K explains the inability of previous workers to observe 3 in frozen organic glasses at 90 K[4] or in the gas phase at room temperature.[2] Comparison of our results with an earlier matrix isolation study of 1 reveals a discrepancy. Ellerhorst et al. reported only a single photoproduct exhibiting IR absorptions at 2039, 1976, and 1950 cm^{-1}.[5] Two bands (2039 and 1950 cm^{-1}) coincide with absorptions we assigned to 3. The origin of the third band (1976 cm^{-1}), however, remains unclear. Differences in photolysis conditions can account for differences in the ratio of 2:3, but we cannot account for the presence of an absorption at 1976 cm^{-1} and the absence of an absorption at 2055 cm^{-1} in the earlier experiments. Wrighton et al. previously noted similarities between the IR absorptions observed upon photolysis of 1 in MCH at 90 K (2041, 1963, 1957 cm^{-1}) and in Ar at 10 K (2039, 1976, 1950 cm^{-1}).[4] Our results establish that these similarities are fortuitous; the bands in MCH are due to 2, while the bands in Ar are due to 3.

In summary, photolysis of $Fe(CO)_4(\eta^2\text{-}C_2H_4)$ (1) in various matrices yields geometric isomers of $Fe(CO)_3(\eta^2\text{-}C_2H_4)$. The thermodynamically more stable isomer (2, A) bears the alkene ligand in the equatorial plane. The thermodynamically less stable isomer (3) likely bears the alkene in an apical position. 3 slowly rearranges to 2 in the dark at 10 K. Our ability to spectroscopically characterize these highly reactive species sets the stage for detailed investigations into the role of similar intemediates in various organometallic rearrangement mechanisms, including metal–carbonyl-catalyzed alkene isomerization.[18]

Acknowledgment. We thank Professors Mark S. Wrighton and Charles P. Casey for helpful discussions. We express our appreciation to various organizations that supported this research program: Wisconsin Alumni Research Foundation, National Science Foundation (Presidential Young Investigator Program CHE-8957529, Project Grant CHE-9003319), 3M Corp., Rohm and Haas Corp., DuPont Corp., and Nicolet Instrument Corp.

Supplementary Material Available: A table containing Mulliken populations of selected orbitals computed for structures A–D using Fenske–Hall calculations (1 page). This material is contained in many libraries on microfiche, immediately follows this article in the microfilm version of the journal, and can be ordered from the American Chemical Society. Ordering information is given on any current masthead page.

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