tion of Dr. J.-P. Mariot (Université du Maine) in recording Mössbauer data at the preliminary state of this work are greatly appreciated. Financial support by the CNRS is gratefully acknowledged.

Note Added in Proof. This series of articles on the functionalization of the complexes Fe(arene)$_2$, which includes ref 11g–i, is dedicated to the memory of Dr. Bianka Tchoubar (deceased April 24, 1990), who hosted D.A. in Sivergue (Provence, France) when ref 11g–i were written up in July 1988. Dr. Tchoubar, an outstanding soul in the French chemistry of this century, has inter alia contributed a book on salt effects[19a] and many stimulating discussions.

Registry No. **1**, 53382-63-1; **2**, 55280-55-2; **3a**, 89278-40-0; **3b**, 53382-61-9; **4**, 89278-41-1; **5**, 127355-88-8; **6**, 81971-77-9; **7**, 81971-78-0; **8**, 89278-52-4; **9**, 127355-91-3; **10**, 127355-95-7; **11**, 65296-91-5; **12**, 127355-97-9; **13**, 127355-93-5; **14**, 127382-92-7; **15**, 125844-92-0; **16**, 127355-89-9; **17**, 127400-45-7; [FeCp(C$_6$Me$_6$)], 70414-92-5; [FeCp(C$_6$Me$_6$)]$^+$PF$_6^-$, 53702-66-2.

Notes

Highly Specific, Interligand Triple Hydrogen Migrations Involved in the Formation of C_3H_6 from 1-Heptyne and Bare Iron(I) Cations. A Gas-Phase Analogue of the Crabtree–Felkin Type of CH Bond Activation†

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Summary: Fe$^+$-mediated formation of C$_3$H$_6$ from 1-heptyne constitutes a gas-phase analogue of the Crabtree–Felkin mechanism which accounts for the transition-metal-induced activation of alkanes. Extensive labeling studies in conjunction with tandem mass spectrometry and CIDI experiments demonstrate that the C$_3$H$_6$ neutral species originates $>91\%$ from the *unsaturated* C$_3$ part of 1-heptyne; in the course of the reaction three hydrogen atoms are transferred from the C$_4$H$_9$ alkyl ligand to the C$_3$H$_3$ fragment to generate a Fe(C$_3$H$_6$)(C$_4$H$_6$)$^+$ intermediate, which eventually dissociates to C$_3$H$_6$ and Fe(1,3-butadiene)$^+$.

There exists ample experimental as well as theoretical evidence that many unimolecular reactions of organic cations in the gas phase proceed via multistep processes involving ion/dipole complexes as central intermediates,[2] and in the last decade it became obvious that a similar principle is operative in the transition metal ion mediated activation of CH and CC bonds.[3] A typical, well-studied example from our own laboratory[4] is shown in Scheme I: Loss of C$_2$H$_4$ from 4-octyne was demonstrated to proceed via complex 3. While the metal ion M$^+$ (M = Cr, Fe, Co, Ni, Cu) exerts a pronounced effect in determining which of the steps depicted in Scheme I corresponds to the rate-determining step (RDS),[4,5] common to all the organometallic systems 1 studied is the fact that step 2 → 3 is irreversible. Obviously, olefin detachment from 3 is so fast that insertion of ethylene in the M$^+$–H bond, which would bring about H/D exchange in an isotopomer labeled in the ethyl group, cannot compete with the dissociation of 3. To put this finding in a different, more general, context, the ligands "anchored" at the metal centers of the intermediates 2 and 3 do not communicate with each other.

However, this situation is not always encountered. For example, Jacobson and Freiser[6] demonstrated that the bisligated complex formed in the ion/molecule reaction

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† In memoriam Professor J. Charles Morrow, Chapel Hill, NC.

[1] Present address: Department of Chemistry, University of Oslo, P.O. Box 1033, Blindern, N-0315 Oslo 3, Norway.

[2] For pertinent reviews and leading articles, see: (a) Morton, T. H. *Tetrahedron* **1982**, *38*, 3195. (b) McAdoo, D. J. *Mass Spectrom. Rev.* **1988**, *7*, 363. (c) Heinrich, N.; Schwarz, H. In *Ion and Cluster Ion Spectroscopy and Structure*; Maier, J. P., Ed.; Elsevier: Amsterdam, 1989; p 329. (d) Heinrich, N.; Louage, F.; Lifshitz, C.; Schwarz, H. *J. Am. Chem. Soc.* **1988**, *110*, 8183. (e) Hammerum, S. *J. Chem. Soc., Chem. Commun.* **1988**, 858. (f) For an elegant and convincing study on the effects of ion/dipole complexes on the rates of unimolecular dissociation of cations, see: Shao, J.-D.; Baer, T.; Morrow, C. J.; Fraser-Monteiro, M. L. *J. Chem. Phys.* **1987**, *87*, 5242.

[3] For reviews, see: (a) Beauchamp, J. L.; Stevens, A. E.; Corderman, R. R. *Pure Appl. Chem.* **1979**, *51*, 967. (b) Freiser, B. S. *Talanta* **1985**, *32*, 697. (c) Allison, J. *Prog. Inorg. Chem.* **1986**, *34*, 627. (d) Armentrout, P. B. In *Structure/Reactivity and Thermochemistry of Ions*; Ausloos, P., Lias, S. G.; Eds.; D. Reidel: Dordrecht, The Netherlands, 1987; Vol. 193, p 97. (e) Ridge, D. P. *Ibid.*, p 165. (f) Armentrout, P. B. In *Gas Phase Inorganic Chemistry*; Russel, D. H.; Ed.; Plenum: New York, 1989; p 1. (g) Squires, R. R. *Chem. Rev.* **1987**, *87*, 623. (h) Buckner, S. W.; Freiser, B. S. *Polyhedron* **1988**, *7*, 1583. (i) Armentrout, P. B. Georgiadis *Ibid.* **1988**, *7*, 1573. (j) Eller, K.; Schwarz, H. *Oganometallics* **1989**, *8*, 1820. (k) Schwarz, H. *Acc. Chem. Res.* **1989**, *22*, 282. (l) Eller, K.; Schwarz, H. *Chimia* **1989**, *43*, 371. (m) Armentrout, P. B.; Beauchamp, J. L. *Acc. Chem. Res.* **1989**, *22*, 315.

[4] Schulze, C. Ph.D. Thesis, Technical Univserity of Berlin, D 83, 1989.

[5] A recent, detailed study (Schulze, C.; Schwarz, H. *Angew. Chem., Int. Ed. Engl.*, in press) reveals the following: For Co$^+$ and Cu$^+$ the RDS corresponds to the β-hydrogen transfer 2 → 3; in distinct contrast, for Fe$^+$ it is the ligand detachment that is subject to an isotope effect, and for M$^+$ = Co$^+$ and Ni$^+$ either step 2 → 3 or 3 → C$_2$H$_4$ is associated with a kinetic isotope effect.

[6] Jacobson, D. B.; Freiser, B. S. *J. Am. Chem. Soc.* **1983**, *105*, 7484.

Scheme I

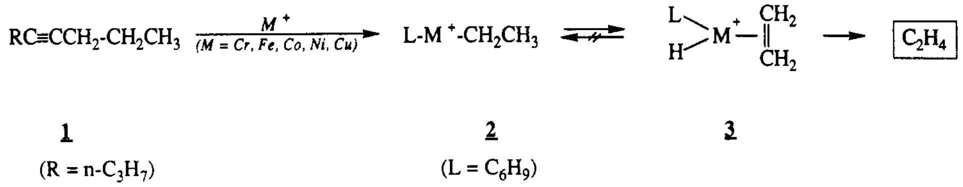


Table I. Isotopic Distribution (Percent) for the Unimolecular Generation of C_3H_6 Isotopomers from Metastable Complexes of Fe(1-heptyne)$^+$ Isotopomers 4a–4h[a]

	precursor							
	4a	4b	4c	4d	4e	4f	4g	4h
$^{12}C_3H_6$	5	91						
$^{13}C^{12}C_2H_6$	95	9						
C_3H_6			23	5	54	14	15	10
C_3H_5D			77	31	38	79	81	65
$C_3H_4D_2$				64	8	7	4	22
$C_3H_3D_3$								3

[a] Data are normalized to $\sum(C_3H_6 \text{ isotopomers}) = 100\%$.

of M(propene)$^+$ (M = Fe, Co, Ni) with C_2D_4 is capable of exchanging *one* H/D per collision (eq 1).

$$M(C_3H_6)^+ + C_2D_4 \rightarrow M(C_3H_6)(C_2D_4)^{+*} \rightarrow M(C_3H_5D)^+ + C_2HD_3 \quad (1)$$

Here, we describe a highly specific *intramolecular triple* hydrogen transfer[7] between two hydrocarbon fragments anchored at a transition-metal ion. As will be demonstrated, the reaction is not of pure academic interest; rather, it has a direct bearing on a fundamental process in catalysis, i.e. the selective dehydrogenation of alkanes by transition-metal complexes.

Chart I

$HC\equiv CCH_2CH_2CH_2CH_2CH_3$ **4**

$HC\equiv C\text{-}^{13}CH_2CH_2CH_2CH_2CH_3$ **4a**

$HC\equiv CCH_2CH_2CH_2CH_2\text{-}^{13}CH_3$ **4b**

$DC\equiv CCH_2CH_2CH_2CH_2CH_3$ **4c**

$HC\equiv CCD_2CH_2CH_2CH_2CH_3$ **4d**

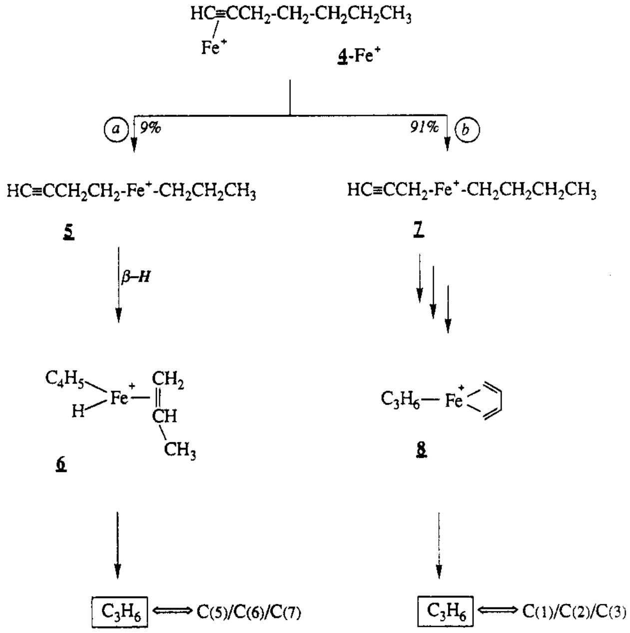
$HC\equiv CCH_2CD_2CH_2CH_2CH_3$ **4e**

$HC\equiv CCH_2CH_2CD_2CH_2CH_3$ **4f**

$HC\equiv CCH_2CH_2CH_2CD_2CH_3$ **4g**

$HC\equiv CCH_2CH_2CH_2CH_2CD_3$ **4h**

Scheme II



Both the metastable ion (MI) and the collisional activation (CA) mass spectra[8] of Fe(1-heptyne)$^+$ (4-Fe$^+$) are dominated by a signal due to the loss of 42 amu (78 and 37% of the total fragment ion currents[9]). MS/MS experiments[10] on the fragment ions generated by loss of either H_2 or C_3H_4 from 4-Fe$^+$ demonstrate that the signal due to the elimination of 42 amu is not the result of consecutive losses of H_2/C_3H_4 or vice versa[11] but rather cor-

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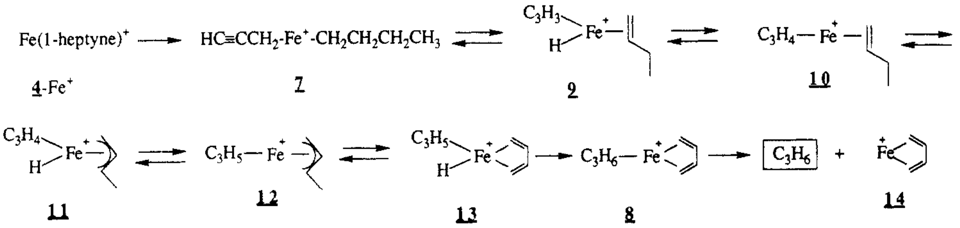
[7] In an earlier study (Steinrück, N.; Schwarz, H. *Organometallics* **1989**, *8*, 759) we described reactions in which up to four (!) hydrogen atoms were *intramolecularly* shifted from one alkyl chain across the Fe$^+$-complexed C=C=C unit of 1,2- and 2,3-octadienes to the other alkyl chain. However, the complexity of the system did not permit us to propose a mechanistic rationale for this quite remarkable rearrangement.

[8] The 1-heptyne/Fe$^+$ complexes were generated in the ion source of a VG Instruments ZAB-HF-3F mass spectrometer by reacting 1-heptyne with Fe$^+$; the latter was generated by 100-eV electron impact ionization of $Fe(CO)_5$: emission current 0.5 mA; repeller voltage 0 V; ion source temperature 200 °C; ion source pressure 10^{-6} mbar. The organometallic complexes of interest were accelerated to 8-keV kinetic energy, mass selected by means of B(1)E (B stands for magnetic and E for electric sector) at a resolution sufficient to separate isobaric species. The MI spectra were recorded by scanning B(2). Collision-induced dissociations were brought about by admitting He as a collision gas (70% transmission). MS/MS/MS experiments, performed in order to prove that C_3H_6 is indeed formed as a genuine molecule, were conducted by mass-selecting the 1-heptyne/Fe$^+$ complex by means of B(1); the products due to unimolecular losses of C_3H_4 or H_2 were "isolated" by using E; fragment ions originating from further dissociations of these primary daughter ions were recorded by scanning B(2). CIDI experiments (see text and further below) were performed on neutral C_3H_6 generated from metastable mass-selected ions, dissociating in the second field-free region. Ionic species were deflected away by applying 1 keV to a deflector electrode situated between B(1) and E in front of a collision cell. Fast-moving neutral molecules entering the collision cell are reionized by collision with helium. Ionic products formed are recorded by scanning E. For further details of CIDI experiments, see: Drewello, T.; Schwarz, H. *Int. J. Mass Spectrom. Ion Proc.* **1989**, *93*, 177. All data reported correspond to an average of 10–100 spectra accumulated by using the VG 11/250 data system. 1-Heptyne and its various isotopomers were synthesized by standard laboratory procedures, purified by preparative gas chromatography, and characterized by spectroscopic means.

[9] The remaining signals (percent abundance of MI/CA data are given in parentheses) are due to the losses of H_2 (5/6), H_2/H_2 (<1/3), CH_4 (1/1), CH_4/H_2 (<1/2), C_2H_4 (8/8), C_3H_4 (6/27), C_3H_6 (78/37), C_4H_6 (<1/1), and C_4H_8 (2/15).

[10] For reviews, see: (a) McLafferty, F. W., Ed. *Tandem Mass Spectrometry*; Wiley: New York, 1983. (b) Busch, K. L.; Glish, G. L.; McLuckey, S. A. *Mass Spectrometry/Mass Spectrometry: Techniques and Applications of Tandem Mass Spectrometry*; VCH: Weinheim, FRG, 1988.

Scheme III



responds to the formation of an intact C_3H_6 molecule. This is evidenced by the following findings: While mass-selected $[4\text{-}Fe^+ - 2]^+$ ions give rise to loss of H_2, a signal for the loss of C_3H_4 is not present; in addition, mass-selected $[4\text{-}Fe^+ - C_3H_4]^+$ ions do not decompose at all in the absence of collision gas. Direct evidence for the generation of an *intact* C_3H_6 molecule from 4-Fe$^+$ is provided by a CIDI (collisionally induced dissociative ionization) experiment.[12] As shown in Figure 1a, the CIDI spectrum of 4-Fe$^+$ contains a clearly discernible ion at *m/z* 42 ($C_3H_6^{\bullet+}$). In addition, the fact that the CIDI spectra shown in parts a and b of Figure 1 are quite distinct indicates that the C_3H_6 species generated in the two experiments are structurally different. In view of the very close resemblance of Figure 1b to a 70-eV EI spectrum of authentic $CH_3CH{=}CH_2$, we conclude that the C_3H_6 neutral species formed in the dissociation of $C_7H_{14}^{\bullet+}$ corresponds indeed to $CH_3CH{=}CH_2$; this implies that at least part of the C_3H_6 neutral species generated from $Fe(C_7H_{12})^+$ is due to a second isomer, which quite likely corresponds to c-C_3H_6.[13]

Further detailed insight into the mechanism for the generation of C_3H_6 from Fe(1-heptyne)$^+$ is provided by the study of the 1-heptyne isotopomers shown in Chart I.

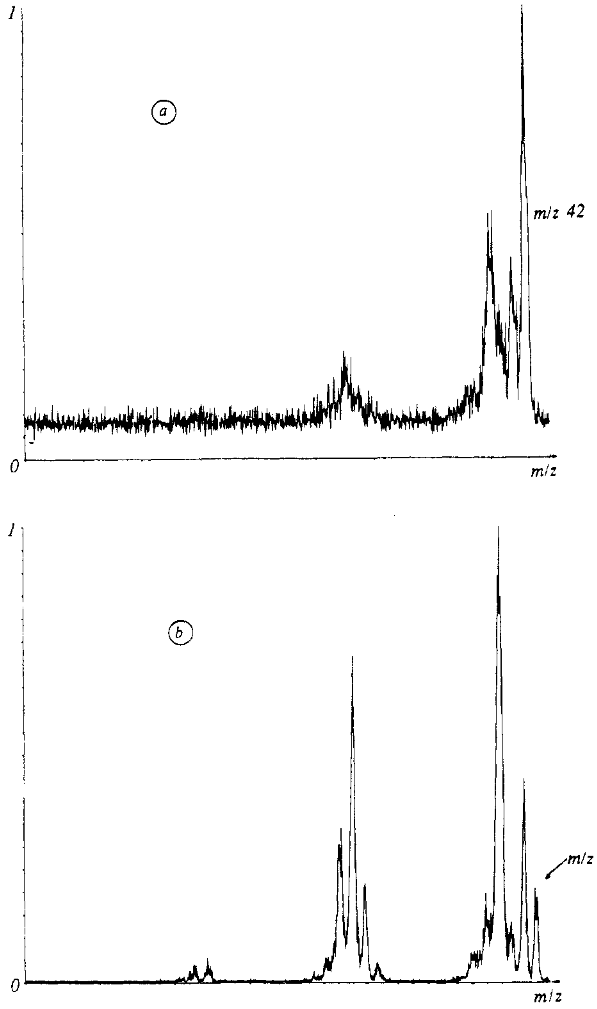


Figure 1. CIDI mass spectra of 42 amu (C_3H_6) (a) generated from 8 keV energy Fe(1-heptyne)$^+$ (4-Fe$^+$) and (b) generated from (1-heptene)$^{\bullet+}$ ($C_7H_{14}^{\bullet+} \rightarrow C_4H_{10}^{\bullet+} + CH_3CH{=}CH_2$) (reionization gas He (ca. 80% transmission)).

Intuitively one might be inclined to describe the formation of C_3H_6 as involving the terminal part of the saturated alkyl chain of 4, i.e. assuming that activation of the C(4)–C(5) bond (Scheme II; 4-Fe$^+$ → 5) is followed by β-hydrogen transfer to generate the propene-containing complex 6, which eventually splits off a C_3H_6 fragment.[14] However, the study of the ^{13}C-labeled substrates **4a** and **4b** reveals that this mode of reaction (Scheme II, path a) contributes no more than 9% to the formation of C_3H_6. At least 91% of the C_3H_6 neutral species contains the carbon atom C(3) of 1-heptyne (path b); thus, it is the *unsaturated* C_3 fragment of 1-heptyne that provides the carbon skeleton for the formation of neutral C_3H_6. Obviously, in the course of the reaction *three* hydrogen atoms have to be transferred from the *n*-butyl chain of 7 to the C_3H_3 fragment in order to generate 8, from which C_3H_6 can be eliminated, thus generating a $Fe(C_4H_6)^+$ fragment.[15]

Inspection of the data shown in Table I[16] clearly reveals that the three hydrogen atoms transferred originate preferentially from positions C(5), C(6), and C(7), respectively. Noteworthy is the finding that the isotopomers **4f** and **4g** result in practically the same isotopic distribu-

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[11] There is compelling evidence for the operation of consecutive losses of C_nH_{2n}/H_2 from organometallic species in the gas phase. Mechanistically quite remarkable systems are reported in: (a) Czekay, G.; Eller, K.; Schröder, D.; Schwarz, H. *Angew. Chem., Int. Ed. Engl.* **1989**, *28*, 1277. (b) Czekay, G.; Drewello, T.; Eller, K.; Zummack, W.; Schwarz, H. *Organometallics* **1989**, *8*, 2439. (c) Schröder, D.; Schwarz, H. *J. Am. Chem. Soc.*, in press.

[12] For leading references and a detailed description of the methodology of CIDI, a variant of neutralization–reionization mass spectrometry (NRMS), see: (a) Burgers, P. C.; Holmes, J. L.; Mommers, A. A.; Terlouw, J. K. *Chem. Phys. Lett.* **1983**, *102*, 1. (b) Wesdemiotis, C.; McLafferty, F. W. *Chem. Rev.* **1987**, *87*, 485. (c) Terlouw, J. K.; Schwarz, H. *Angew. Chem., Int. Ed. Engl.* **1987**, *26*, 805. (d) Terlouw, J. K. *Adv. Mass Spectrom.* **1989**, *11*, 984. (e) Schwarz, H. *Pure Appl. Chem.* **1989**, *61*, 685. (f) Holmes, J. L. *Mass Spectrom. Rev.* **1989**, *8*, 513. (g) McLafferty, F. W. *Science (Washington, D.C.)* **1990**, *247*, 925.

[13] It should be recalled that there exist precedents for the preferred generation of energy-rich hydrocarbon isomers from organometallic systems. For example, Fe$^+$-induced decarbonylation of cyclobutanone is suggested to give rise to c-C_3H_6, and decomposition of Fe(1-pentene)$^+$ is reported to cogenerate c-C_3H_6 and $CH_3CH{=}CH_2$: van Koppen, P. A. M.; Jacobson, D. B.; Illies, A.; Bowers, M. T.; Hanratty, M.; Beauchamp, J. L. *J. Am. Chem. Soc.* **1989**, *111*, 1991 and references cited therein. Similarly, cyclopropane and propene are cogenerated in the unimolecular dissociation of the complex formed directly from Co$^+$ and 1-pentene: van Koppen, P. A. M.; Bowers, M. T.; Beauchamp, J. L. *Organometallics* **1990**, *9*, 625.

[14] This kind of remote functionalization is, for example, the preferred mode by which neutral C_2H_4 and C_3H_6 are formed from the complex Fe(2-octyne)$^+$, involving C(7)/C(8) and C(6)/C(7)/C(8), respectively: Schulze, C.; Schwarz, H.; Peake, D. A.; Gross, M. L. *J. Am. Chem. Soc.* **1987**, *109*, 2368.

[15] In an earlier study (Peake, D. A.; Gross, M. L. *Organometallics* **1986**, *5*, 411) the structure of $Fe(C_4H_6)^+$ was already assigned to an Fe-(η^4-1,3-butadiene)$^+$ complex.

[16] The data given in Table I are corrected for the contribution of C_3H_4. (For details, see ref. 4.)

tion of $C_3H_{6-x}D_x$, which points to an intermediate in which C(5) and C(6) of 1-heptyne are rendered equivalent. In addition, the minor contributions of multiple hydrogen transfers involving the *same* carbon atom (i.e. loss of 8, 7, and 4% of $C_3H_4D_2$ from **4e**, **4f**, and **4g**, respectively) indicate that the vinylic CH bonds of the various intermediates generated en route to C_3H_6 are indeed quite "inert". Recently, evidence was presented showing that the rate of exchange of the central vinylic CH group of propene is orders of magnitude slower than that of the methyl hydrogen atoms,[17] which presents indirect support for the involvement of hydrido–metal–allyl complexes in the course of H/D exchange of olefins. Not surprisingly, the data also reveal that the hydrogen-transfer processes are, to some extent, reversible (see, for example, loss of C_3H_6 from **4c** or of C_3H_5D from **4d**). The reaction depicted in Scheme III is, at least in a qualitative sense, in keeping with the experimental findings. We note, in passing, that the overall reaction is an *intramolecular* variant of the well-known *Crabtree–Felkin* system,[18] which is of prime importance in the study of CH activation processes in the condensed phase.[19] In the Crabtree–Felkin system, an olefin is added externally to a ligand-deficient metal fragment and then severs as a hydrogen acceptor. The hydrogen atoms are provided by an alkane ligand; the latter, in the course of the reaction, is transformed by the transition metal to an alkylhydridometal complex. In the present case, all structural features typical of the Crabtree–Felkin system are generated in an *intramolecular* fashion, presumably involving **7** as the central intermediate (Scheme III). It is the "butyl" fragment of **7** that serves as the hydrogen atom source, and the C_3H_3 ligand of **7** acts as a hydrogen acceptor. The driving force for the reaction, quite likely, corresponds to the formation of the Fe^+ (η^4-1,3-butatiene) complex 14 (Scheme III).[20]

Acknowledgment. We gratefully acknowledge financial support of our work by the Fonds der Chemischen Industrie, the Volkswagen-Stiftung, and the Gesellschaft von Freunden der Technischen Universität Berlin.

Registry No. 4-Fe^+, 127383-14-6; **5**, 127383-15-7; **7**, 127383-16-8; Fe^+, 14067-02-8; 1-heptyne, 628-71-7.

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(17) Jacobson, D. B. *Organometallics* **1988**, *7*, 578.

(18) For reviews, see: (a) Crabtree, R. H. *Chem. Rev.* **1985**, *85*, 245. (b) Baudry, D.; Ephritikine, M.; Felkin, H.; Fillebeen-Khan, T.; Gault, Y.; Holmes-Smith, R.; Yingrui, L.; Zakrzewski, J. In *Organic Synthesis*; Streith, J., Prinzbach, H., Schill, G., Eds.; Blackwell Scientific Publications: Oxford, U.K., 1985; p 25.

(19) For selected reviews, see: (a) Shilov, A. E. *Pure Appl. Chem.* **1978**, *50*, 725. (b) Bergman, R. D. *Science (Washington, D.C.)* **1984**, *223*, 902. (c) Crabtree, R. H. *Chem. Rev.* **1985**, *85*, 245. (d) Brookhart, M.; Green, M. L. H.; Wong, L.-L. *Prog. Inorg. Chem.* **1988**, *36*, 1. (e) Jones, W. D.; Feher, F. J. *Acc. Chem. Res.* **1989**, *22*, 91.

(20) Recently, multiple interligand hydrogen migration was reported (Hettich, R. L.; Freiser, B. S. *Organometallics* **1989**, *8*, 2447) for the reaction $Co(C_2H_4)^+$ + 1-butene → Co (η^4-1,3-butadiene)$^+$ + C_2H_6. However, lack of labeling studies precluded any mechanistic conclusions.

Regiospecific Allyl–Alkyne Coupling on an Organotriruthenium Carbonyl Cluster. Crystal Structure of $Ru_3(CO)_8(\mu\text{-}\eta^1{:}\eta^1{:}\eta^4\text{-}PhC{=}C(Ph)C(Me){=}CCH_2NMe_2)$

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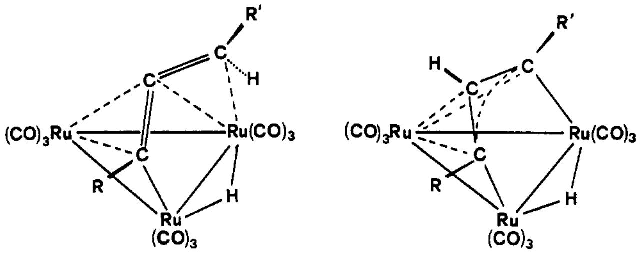
Received October 30, 1989

Summary: The reaction of $Ru_3(H)(CO)_9(MeCCHCNMe_2)$, having an allyl moiety σ–π bonded to the triruthenium frame, with diphenylacetylene yields $Ru_3(CO)_8(PhC_2PhMeC_2CH_2NMe_2)$, containing the 1-(dimethylamino)-3-methyl-4,5-diphenylpentadiene ligand coordinated to an intact triruthenium triangle. The organic ligand acts as an overall 8e donor to the metal triangle via σ–π metal–carbon bonds and a σ metal–nitrogen bond.

The oxidative addition of nonterminal alkynes, having a methylenic group α to the triple bond ($RC{\equiv}CCH_2R'$), to $Ru_3(CO)_{12}$ gives two isomeric products, namely $Ru_3(H)(CO)_9(RC{=}C{=}CHR')$ (1) and $Ru_3(H)(CO)_9(RC{\cdots}CH{\cdots}CR')$ (2)[1] (chart I).

Chart I



1 **2**

The kinetically favored isomer **1**, which contains a 1-η^1:1-2-η^2-allyl ligand, isomerizes on heating to the thermodynamically more stable $\eta^1{:}\eta^1{:}\eta^3$-allyl isomer **2**; similar behavior is found for osmium.[2]

The allylic derivative **2** has been employed as starting material for a highly selective, one-step synthesis of the ruthenacyclopentadienyl derivatives **3**[3] (Scheme I).

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(1) Aime, S.; Milone, L.; Osella, D.; Valle, M. *J. Chem. Res., Synop.* **1978**, *77*; *J. Chem. Res., Miniprint* **1978**, 0785–0797.

(2) Deeming, A. J. *Adv. Organomet. Chem.* **1986**, *26*, 1.