in this work reveals a remarkably significant correlation ($r = 0.981$, $p < 0.001$), suggesting that differential solvation within this series is unimportant.

A quantitative measure of the nonadditivity can be obtained for the alkylation reaction by defining $S = k_{rel}/k_{calcd}$ for each α-substituted compound (Table II). The definition of S leads to a value of $S = 1$ for 2-picoline, consistent with DeTar's recent criterion for a sterically unstrained standard.[13] A value of $S >$ 1 implies less steric hindrance relative to 2-picoline, and $S < 1$ indicates the converse.

To relate S to structural properties, we performed complete geometry optimizations by using the MINDO/3 program of Rinaldi[14,15] for **1**-**13**. The bond lengths and angles calculated for pyridine compare very favorably with those determined by microwave spectroscopy and electron diffraction studies.[16] Experimental information relevant to the structure of **2**-**13** is rare,[17] but some data exist for their benzene analogues.[18] We have therefore performed complete MINDO/3 energy minimization for benzene, toluene, the xylenes *o*-di-tert-butylbenzene, indane, and tetralin.[19] Comparison of the available experimental data[18] for these nonheterocyclic aromatic compounds with the semiempirical results indicates that the MINDO/3 method does predict appropriate trends in the bond angles and lengths calculated.

For the 2-substituted pyridines, the key geometric parameters appear to be θ, the N-C_2-$C_{2\alpha}$ bond angle, and d_{NH}, the distance from nitrogen to the closest hydrogen on the α carbon (see Table II). A wide range in both θ and d_{NH} is found, spanning 13.00° and 0.451 Å, respectively. 2,3-Lutidine (**5**) iodomethylates with one-half the rate of 2,5-lutidine. That **5** fails to satisfy a LFER due to a buttressing effect is seen in Table II as a 3.13° *decrease* in θ and a 0.064 Å *decrease* in d_{NH} relative to 2,5-lutidine. At the other extreme, tying the methyl groups together with a methylene unit in 2,3-cyclopentenopyridine (**11**) *increases* both θ (by 10.29°) and d_{NH} (by 0.334 Å) relative to 2,5-lutidine; **11** iodomethylates almost five times faster than does **5**.

Excellent correlations are found for the nonadditivity factor S as a function of both θ ($S = -17.700 + 0.160\theta$; $r = 0.970$, $p = 0.001$) and d_{NH} ($S = -11.844 + 4.950d_{NH}$; $r = 0.996$, $p < 0.001$). The parameters θ and d_{NH} are highly correlated with one another ($r = 0.971$, $p = 0.001$) and are not independent.

That θ (and d_{NH}) so strongly correlate with the observed kinetics is a unique demonstration of the relationship between substrate equilibrium geometry and reaction rate. The coupling of chemical kinetics with theoretical treatments for compounds which fail to follow LFER will lead to a better understanding of non-additive substituent effects.

Acknowledgment. We thank Dr. Neal Nunnally for developing the automated conductivity analysis instrumentation, Henry V. Secor, Dianne DeNagel, William Stevenson, and Jean Lawson for valuable technical assistance, and Drs. Charles G. Chavdarian and William Dwyer for helpful discussions during the conceptualization of this project.

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Formation of an Iridium Ethyl Complex by Methyl Migration to a Coordinated Methylene Group

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Contribution No. 2907
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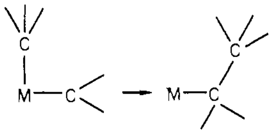
Despite the importance of migratory reactions in organometallic chemistry,[1] examples of one potentially useful class of migrations—alkyl migration from a metal atom to the carbon atom of a coordinated carbene or alkylidene ligand (equation 1)—have only recently been encountered.[2-5] While a large number of



(1)

alkylalkylidene complexes of the earlier transition metals are known, they apparently show no tendency to undergo this reaction, owing to the pronounced nucleophilicity of the alkylidene carbon atom.[6] In contrast, electrophilic carbene complexes of the later transition metals often readily react with external nucleophilic reagents,[7-9] and it is in such complexes that the reaction illustrated in equation 1 is most likely to be facile. In this communication we report the synthesis and characterization of a compound with an alkyl group cis to an electrophilic carbene precursor ligand, the iridium methyl(methoxymethyl) complex $IrBr(CH_3)(CH_2OCH_3)(P(CH_3)_3)_3$ (**1**). Compound **1** is readily transformed into a transient methylmethylene complex, which subsequently undergoes methyl migration to the methylene group. A stable ethyl complex has been isolated, and its structure has been confirmed by a single-crystal X-ray diffraction study.

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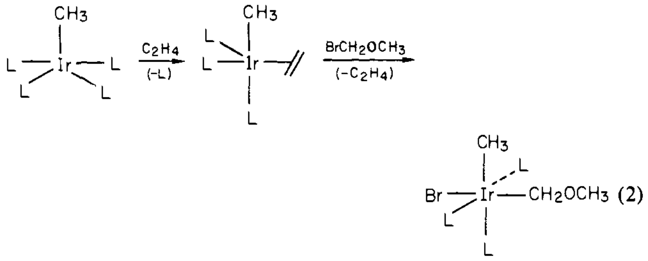
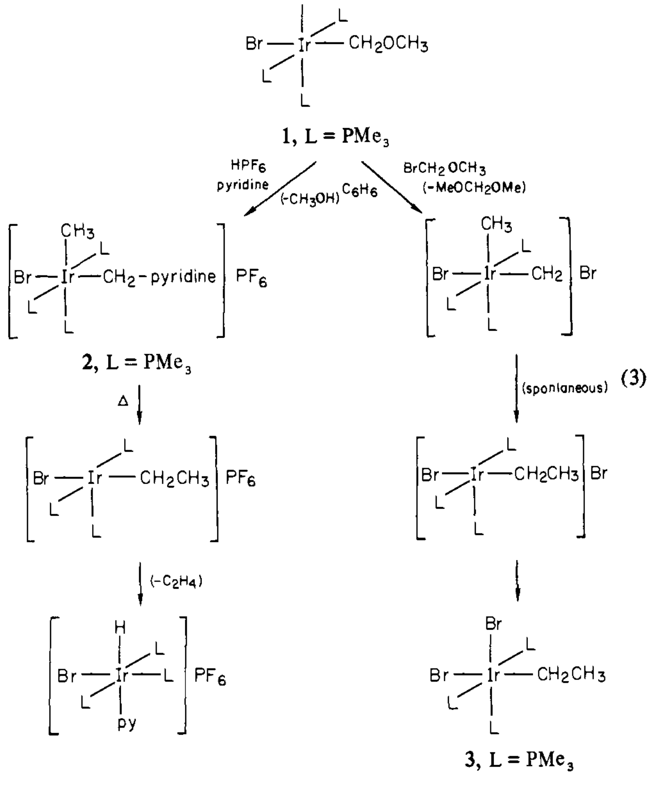
Compound **1**[10] is prepared in ca. 60% yield according to the reaction sequence of equation 2,[11] starting from the iridium(I) methyl complex $Ir(CH_3)(P(CH_3)_3)_4$.[12] While stable at room



1, L = PMe$_3$

temperature in the absence of air and other impurities, compound **1** reacts rapidly with electrophilic reagents, resulting in cleavage of a C–O bond of the methoxymethyl group and formation of a transient, cationic methylene complex.[13] The fate of this methylene complex is determined by the solvent and reagent used for the C–O bond cleavage of compound **1**. Two examples are discussed below and summarized in equation 3.



1, L = PMe$_3$

2, L = PMe$_3$

3, L = PMe$_3$

When compound **1** reacts with a stoichiometric amount of HPF_6 in pyridine, methanol is immediately formed together with the "pyridine-trapped" methylene complex $[IrBr(CH_3)(CH_2\text{-}NC_5H_5)(P(CH_3)_3)_3][PF_6]$ (**2**)[14,15] in ≥80% yield. Compound **2** is stable in pyridine solution at room temperature but upon warming to 60 °C the trapping pyridine is displaced by the migrating methyl group (equation 3). The 5-coordinate ethyl complex which must result from this migration then decomposes by β-hydrogen elimination; ethylene and the hydrido complex $[IrHBr(P(CH_3)_3)_3(py)][PF_6]$[16] are the major products in the reaction solution (^{1}H NMR spectroscopy).

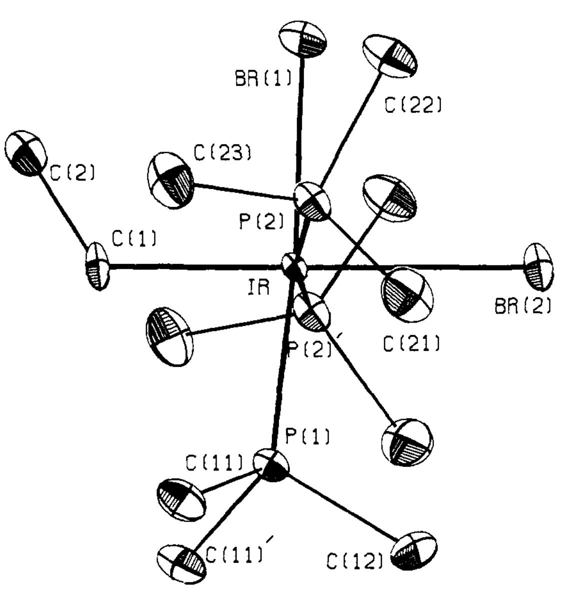


Figure 1. ORTEP drawing of a molecule of *mer*-$IrBr_2(C_2H_5)(P(CH_3)_3)_3$ (**3**). Thermal ellipsoids are drawn at the 50% probability level. The molecule possesses crystallographically imposed mirror symmetry.[18] Selected bond distances (Å): Ir–Br(1), 2.584 (1); Ir–Br(2), 2.636 (1); Ir–P(1), 2.263 (1); Ir–P(2), 2.344 (1); Ir–C(1), 2.123 (5); C(1)–C(2), 1.501 (9). Selected bond angles (deg): Br(1)–Ir–Br(2), 88.11 (2); Br(1)–Ir–P(1), 175.41 (4); Br(1)–Ir–P(2), 86.94 (3); Br(1)–Ir–C(1), 91.7 (2); Br(2)–Ir–C(1), 179.8 (2); P(2)–Ir–P(2)′, 169.15 (5); Ir–C(1)–C(2), 121.3 (4).

When a concentrated benzene solution of compound **1** is treated with excess bromomethyl methyl ether[10], the ethyl complex *mer*-$IrBr_2(C_2H_5)(P(CH_3)_3)_3$ (**3**)[17] precipitates in 50% yield after ca. 24 h. The structure of this ethyl complex **3**, determined by a single-crystal X-ray diffraction study, is illustrated in Figure 1.[18] The expected organic product, dimethoxymethane, has been detected in the reaction solution (^{1}H NMR spectroscopy). The ethyl complex resulting from the reaction of $IrBr(^{13}CH_3)(CH_2OCH_3)(P(CH_3)_3)_3$ with excess bromomethyl methyl ether contains the ^{13}C label evenly distributed between α and β sites (^{13}C NMR).[17] This scrambling most likely occurs by a β-hydrogen elimination reaction of the ethyl complex, followed by olefin

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[10] ^{1}H NMR of **1** (pyridine-d_5): Ir–CH$_3$, d (J = 5.2 Hz) of t (J = 7.8 Hz) at δ 0.22; PMe$_3$, d (J = 7.6 Hz) at δ 1.33, t (J(apparent) = 3.4 Hz) at δ 1.30; $-CH_2OCH_3$, s, δ 2.96; $-CH_2OCH_3$, d (J = 3.4 Hz) of t (J = 5.6 Hz) at δ 3.60. ^{13}C NMR (C_6D_6) of $IrBr(^{13}CH_3)(CH_2OCH_3)(P(CH_3)_3)_3$: d ($J$ = 79.1 Hz) of t (J = 8.8 Hz) at δ −11.35. Anal. Calcd: C, 25.75, H, 6.29. Found: C, 25.49, H, 6.43.

[11] Full details will be published separately. Caution: Owing to the possibly carcinogenic nature of bromomethyl methyl ether, the toxicity of trimethylphosphine, and the air and moisture sensitivity of the iridium compounds, we have carried out all reactions in a nitrogen-filled dry box.

[12] Thorn, D. L. *J. Am. Chem. Soc.* **1980**, *102*, 7109–7110. Cobalt analogues of some of the compounds are known: Klein, H.-F. *Angew. Chem.* **1980**, *92*, 362–375. *Angew. Chem., Int. Ed. Engl.* **1980**, *19*, 362–375; Klein, H.-F.; Hammer, R.; Gross, J.; Schubert, U. *Angew. Chem.* **1980**, *92*, 835–836. *Angew. Chem., Int. Ed. Engl.* **1980**, *19*, 809–810.

[13] Carbene formation by this reaction was first reported by: Jolly, P. W.; Pettit, R. *J. Am. Chem. Soc.* **1966**, *88*, 5044–5045. This has been successfully employed by, e.g.: Brookhart, M.; Tucker, J. R.; Flood, T. C.; Jensen, J. *Ibid.* **1980**, *102*, 1203–1205. See also ref 7 and 8.

[14] ^{1}H NMR of **2** (pyridine-d_5): Ir–CH$_3$, d (J = 4.8 Hz) of t (J = 7.8 Hz) at δ 0.10; PMe$_3$, d (J = 7.2 Hz) at δ 1.41, t (J(apparent) = 3.3 Hz) at δ 1.39; "Ir–CH$_2$", pseudo q (J = 6 Hz) at δ 5.10. Analytically pure samples of this compound have not yet been obtained.

[15] A pyridine-trapped methylene complex of Re was reported by: Wong, W.-K.; Tam, W.; Gladysz, J. A. *J. Am. Chem. Soc.* **1979**, *101*, 5440–5442. Pyridine–ylide Pt complexes have been reported. See: Puddephatt, R. J. *Coord. Chem. Rev.* **1980**, *33*, 149–194 and references therein.

[16] ^{1}H NMR of $[IrHBr(P(CH_3)_3)_3(py)][PF_6]$ (pyridine-d_5): Ir–H, d (J = 20 Hz) of t (J = 13 Hz) at δ −20.5; PMe$_3$, d (J = 10.5 Hz) at δ 1.59, t (J(apparent) = 3.8 Hz) at δ 1.15. This compound is the only hydride-containing complex present and comprises ca. 80% of the total iridium–phosphine species.

[17] ^{1}H NMR of **3** (pyridine-d_5): ethyl, br, δ 1.1; PMe$_3$, d (J = 9.5 Hz) at δ 1.33, t (J(apparent) = 3.8 Hz) at δ 1.52. ^{13}C NMR of **3** (C_6D_6): C(1), q (J = 4.5 Hz) at δ −14.73; C(2), pseudo q (J = 2 Hz) at δ 21.55. Anal. Calcd: C, 21.68; H, 5.29. Found: C, 21.84, H, 5.37.

[18] Crystal data for **3**: Space group $P2_1/m$ (C_{2h}^2); cell dimensions (at −100 °C): a = 10.105 (1), b = 11.358 (2), c = 8.381 (1) Å; β = 93.21 (1)°; V = 960.4 (4) Å^3; Z = 2. On the basis of refinement of 88 variables (all nonhydrogen atoms with anisotropic thermal parameters) using 1973 unique reflections with $F_0^2 > 3\sigma(F_0^2)$, R = 0.026, R_w = 0.030. Full details will be published separately.

rotation and reinsertion,[19] after the initial methyl migration[20] is complete.

These results suggest alkyl migration to a carbene ligand may be a general and useful method for forming carbon–carbon bonds under very mild conditions.[21] In some respects it resembles the well-known reductive elimination of *cis*-dialkyl groups. However, closely related *cis*-dialkyl complexes of iridium do not undergo reductive elimination except possibly under extreme conditions.[22] Indeed, the stability of iridium (III) dialkyl complexes has permitted the isolation and study of compound **1**. Related compounds which may undergo similar migratory reactions are under active investigation.

Acknowledgment. We thank L. Lardear and W. Gray for skilled technical assistance and Professors B. Trost and M. Brookhart for helpful discussions.

Supplementary Material Available: Tables of positional and thermal parameters and observed and calculated structure amplitudes (16 pages). Ordering information is given on any current masthead page.

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(19) Rapid scrambling by a similar mechanism was proposed by: Yamamoto, A.; Yamamoto, T.; Saruyama, T.; Nakamura, Y. *J. Am. Chem. Soc.* **1973**, *95*, 4073–4074. This was ruled out by: Cotton, F. A.; Frenz, B. A.; Hunter, D. L. *Ibid.* **1974**, *96*, 4820–4825.

(20) We prefer to regard the reaction as a methyl migration rather than as a methylene insertion, owing to the stereochemistry observed for the final ethyl compound **3**. However, mechanistic information is lost if a fluxional 5-coordinate compound is an intermediate in the reaction or is in equilibrium with the product.

(21) Methyl migration to a methylene ligand was predicted to be facile by: Berke, H.; Hoffmann, R. *J. Am. Chem. Soc.* **1978**, *100*, 7224–7236.

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*Book Reviews**

Reminiscences of Los Alamos, 1943–1945. Edited by Lawrence Badash (University of California, Santa Barbara), Joseph O. Hirshfelder (University of Wisconsin), and Herbert P. Broida (University of California, Santa Barbara). D. Reidel Publishing Co., Dordrecht, Holland. 1980. xii + 188 pp. $26.50, cloth; $9.95, paper.

This book joins a number of other fascinating accounts of the Los Alamos years. It is the outgrowth of a lecture series held at UCSB in 1975 and focuses not on the technical aspects of the Los Alamos years 1943–1945, but on the personal reminiscences of a diverse group of people associated with Los Alamos during that period. In its own way, the book is a very powerful one, raising all the "Faustian" questions about whether we have sold our souls to the devil. It, like many of the other books on Los Alamos, will haunt many readers.

The whole experience haunts many of its participants. Some of the reminiscences are little more than attempts by the contributors to explain, perhaps rationalize, their actions during this period. It is interesting to speculate about whether this is more a matter of conscience or a reaction to present-day criticism of all things nuclear.

Other participants provide us with many rich details of the life at Los Alamos, including the civilian/military "standoff", the Sante Fe/Los Alamos relationships, and the many interesting interactions with the Native Americans and Spanish Americans of the vicinity. There are many poignant and dramatic passages in this book, particularly some by Elsie McMillan. There is humor, especially in Feynman's chapter chronicling his escapades.

Every chapter is interesting and worthwhile; the contributors are as follows: John D. Dudley, Edward M. McMillan, John H. Manley, Elsie McMillan, George G. Kistiakowsky, Joseph O. Hirshfelder, Laura Fermi, Richard P. Feynman, Bernice Brode, and Norris Bradbury. Everyone who chooses to read this book will find some fascinating aspect of these Los Alamos years.

R. Damrauer, *University of Colorado at Denver*

Petroleum Geochemistry and Geology. By John M. Hunt (Woods Hole Oceanographic Institution). W. H. Freeman and Co., San Francisco. 1979. xii + 617 pp. $30.00.

This book is intended for students with "basic courses in geology and chemistry and also for oil company operating personnel who are interested in the application of geochemistry to petroleum exploration". Abundant use of organic structural formulas are used throughout, so some grounding in the fundamentals of organic chemistry would also seem desirable for the reader. The author suggests that the book can serve not only as a text but as a reference book; this is a fully justified claim as the book is well-organized and contains a wealth of information. A number of subjects with which the reader is either unfamiliar or out-of-touch are discussed in detail before being utilized; these include discussions of isotopes and isotopic dating, the effect of temperature on reaction rates, fluid expulsion models, geothermal gradients, etc.

The book is divided into four main sections: (1) an introduction with historical information, a description of the primative earth and the origin of life, the petroleum potential of various types of rocks, and a description of petroleum and its products, (2) an origin and migration section with a detailed description of how oil and gas form, how they migrate, and how they accumulate, (3) a habitat section discussing source rocks, the quantity of organic materials in various types of rock, types of organic matter, techniques for measuring the state of maturation of kerogen, and the petroleum reservoir and its characteristics, and (4) an applications section discussing seeps and surface prospecting, subsurface prospecting, crude oil correlations, and prospect evaluations.

In all, this would seem to be an excellent book not only for its intended use, but also for the general chemist seeking a readable, yet thoroughly comprehensive treatment of the important area of petroleum geochemistry.

R. Damrauer, *University of Colorado at Denver*

Infrared Interferometric Spectrometers. By A. E. Martin. Elsevier Scientific Publishing Company, Amsterdam and New York. 1980. XI + 292 pp. $73.25.

Dr. A. E. Martin has been designing infrared instrumentation for many years and was, until recently, associated with a company which has produced infrared interferometers for many years. Thus, this book is written by an expert in the field. It is not primarily concerned with the use of the rapid-scanning infrared interferometers that are in widespread use in analytical and research laboratories today, although the principles and details that it explains are basic to these spectrometers.

The book explains the physical principles and practical details that are required to understand infrared interferometers in detail. It is written from the point of view of a man interested in slow-scanning interferometers, and includes many details that are usually transparent to the user of rapid-scan instruments.

The book contains nine chapters and 13 appendices. The second chapter, on Theoretical Considerations, occupies just under a third of the book, and other major chapters, which together constitute a second third of the book, discuss Optical Considerations, Interferometer Details, and Computation. The remaining chapters describe Commercial Instruments (inevitably not quite up to date), Measurement of Refractive Index, Modifications of the Basic Michelson Optical System, and Other Interferometers and Allied Instruments.

Dr. Martin's style is to present the fundamental principles of a topic and then to immediately explore details that are important but are not essential to the novice. Thus, this book is probably not well suited to the reader who is totally new to the subject, although the level of presentation is such that such a reader would understand most of it. It is very well suited to the reader who knows about these instruments in general and wishes to learn about the details. Thus, the first nine pages of Chapter II contain the basic principles of the Michelson interferometer, then an account of negative frequencies, then quite a detailed account of the troubles that can arise from the constant term in the interferogram. The chapter then goes on to Asymmetric Interferograms, Gaussian Functions and Resolution, and Sampling Considerations, Scanning Functions and Apodization, all of which are presented simply but with an unusual amount of detail that is of interest even to people experienced in the field.

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*Unsigned book reviews are by the Book Review Editor.