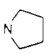
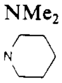
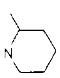
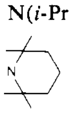
Table I. Comparison of Rates (log k_{obsd}) with Expected Rates of Single-Electron Transfer (log k_{SET}) for Reactions of 9-Dialkylaminofluorenide Ions, 9-R_2N-Fl^-, with F_3CCH_2I, Ph_2CHCl, and $PhCH_2Cl$

		F_3CCH_2I[c]		Ph_2CHCl[f]		$PhCH_2Cl$[g]	
NR_2[a]	$E_{ox}(A^-)$[b]	log k_{obsd}[d]	log k_{SET}[e]	log k_{obsd}[d]	log k_{SET}[e]	log k_{obsd}[d]	log k_{SET}[e]
	−0.865	fast	+1.57	+0.91	+1.37	+1.35	−0.50
	−0.805	fast	+1.11	+0.54	+0.92	+0.46	−0.99
NMe_2	−0.660	+0.75	−0.03	−0.57	−0.17	+0.27	−2.2
	−0.643	−0.28	−0.17	−0.57	−0.31	+0.11	−2.4
	−0.598	−1.43	−0.56	−1.21	−0.66	−1.14	−2.8
N(*i*-Pr)$_2$	−0.485	−1.50	−1.53	−1.45	−1.61	−1.00	−3.8
	−0.438	−2.25	−1.91	−1.55	−1.99	−2.49	−4.2

[a] The 9-R_2N-FlH compounds were prepared by the reaction of 9-BrFlH with the appropriate amine; pK_a data and physical properties will be reported separately. [b] Measured by cyclic voltammetry in Me_2SO with Ag/AgI reference electrode;[9] referenced to the aqueous standard hydrogen electrode (SHE) by subtracting 0.125 V. [c] $E_{rd} = 0.4 \times (-1.425) = -0.57$; $\lambda = 60$.[10] [d] Measured spectrophotometrically by monitoring the disappearance of 9-R_2N-Fl^- ion absorbance.[5] [e] Calculated by using an equation derived from the Marcus equation by Eberson:[3] $\log k_{SET} = \log k_d - \log \{1 + 0.2 \exp [{}^{\lambda}/_{4}(1 + (\Delta G°/4))^2/RT]\}$, where k_d is the diffusion rate in Me_2SO (3.3×10^9);[11] λ is the intrinsic barrier chosen from literature values[3b,4] to give the best fit, and $\Delta G°$ is estimated from the redox potentials.[3b] [f] $E_{rd} = 0.4 \times (-1.185) = -0.474$ (vs SHE);[10] $\lambda = 65$; Eberson[3b] reports $\lambda = 63$ for the reaction of Ph_2CH^- with $PhCH_2Cl$. [g] $E_{rd} = -0.72$ (Eberson[12]); $\lambda = 65$.

the reactions of members of the 9-R_2N-Fl^- ion family with F_3CCH_2I, a known electron acceptor[6] (Figure 1; Table I).

The kinetics with F_3CCH_2I were first order in each reactant but remained so for only about 1 half-life, which is typical of the nonchain SET reactions that we have studied earlier.[6] Reaction of the 9-R_2N-Fl^- ion family with Ph_2CHCl, which has a slightly more positive reduction potential (E_{rd}) than F_3CCH_2I (−1.2 vs −1.4), also gave a linear Marcus-type plot (Figure 2), but here the kinetics remained second order for several half-lives, and substitution products were formed in high yield with no indication of the presence of radical products.[13] Comparison of log k_{obsd} with log k_{SET}, calculated according to the method of Eberson (Table I), shows a close correspondence of values for reactions of both F_3CCH_2I and Ph_2CHCl. This evidence suggests that both are reacting by SET mechanisms, F_3CCH_2I giving radical-type products[6] and Ph_2CHCl giving S_N2-type products.

Surprisingly, $PhCH_2Cl$, which has a 0.8 V less positive E_{rd} (E_p) than does Ph_2CHCl, also gave a linear Marcus-type plot for reactions with 9-R_2N-Fl^- ions, and the correspondence between k_{obsd} and k_{SET} (Table I) is close enough to fall in the range that Eberson designates as "SET feasible".[3b] A similar appearing Marcus-type plot was obtained with *n*-BuBr ($E_{rd} = -2.1$[3b]) and the log k_{obsd} − log k_{SET} values were 3–4, which is still in the "SET feasible" range.[3b]

We conclude that the reactions of 9-R_2N-Fl^- ions with Ph_2CHCl are occurring by SET involving rapid coupling of radical pair intermediates, whereas those with $PhCH_2Cl$ and *n*-BuBr are probably in the hybrid[2a] or merged[2b] region of a SET–S_N2 spectrum. Application of this test to other electrophiles is in progress.

Acknowledgment. We thank Tsuei-Yun Lynch for rate data with F_3CCH_2I and Craig A. Wilson for rate data with $PhCH_2Cl$. This research was sponsored by a grant from the National Science Foundation.

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(6) Bordwell, F. G.; Wilson, C. A. *J. Am. Chem. Soc.* **1987**, *109*, 5470–5474.

(7) Marcus plots log k_{obsd} vs $\Delta G°'$; Figure 1 is a Marcus-type plot in the sense that, since E_{rd} is constant, changes in $E_{ox}(A^-)$ are proportional to changes in $\Delta G°'$. Over large ranges of $\Delta G°'$ the Marcus equation predicts curvature, but over relatively small ranges in the endergonic region the curve is flat and essentially linear.[8]

(8) Klinger, R. J.; Kochi, J. *J. Am. Chem Soc.* **1982**, *104*, 4186–4196.

(9) Bordwell, F. G.; Bausch, M. J. *J. Am. Chem. Soc.* **1986**, *108*, 1979–1985.

(10) The observed E_{rd} measured as a peak potential (E_p) has been multiplied by 0.4 to correct for the anodic shift caused by the rapid follow reaction.[2a,3b]

(11) Gordon, A. J.; Ford, R. A. In *The Chemist's Companion, A Handbook of Practical Data, Techniques and References*; Wiley: New York, 1972; pp 6–7 and 137–138.

(12) Eberson[3a] reports $E_{rd} = -0.52$ V vs SHE_{Me_2SO}. By applying $-\Delta G°_{tr}(H_2O \rightarrow Me_2SO)$ for the proton, we converted this value to E_{rd} vs SHE_{aq} to be consistent with our E_{ox} values. The resulting value is ~40% of our measured E_{rd}.

(13) In the reaction of 9-(i-Pr)$_2$N-Fl^- ion with Ph_2CHCl, the presence of the persistent 9-(i-Pr)$_2$N-Fl$^\bullet$ radical ($\lambda_{max} = 450$ nm)[6] was not observed.

Molecular Hydrogen Complexes. 5. Electronic Control of η^2-H_2 versus Dihydride Coordination. Dihydride Structure of $MoH_2(CO)(R_2PC_2H_4PR_2)_2$ for R = Et, *i*-Bu versus η^2-H_2 for R = Ph

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Received May 26, 1987

Since their discovery,[1] new examples of transition-metal molecular-hydrogen (M–η^2-H_2) complexes have been established at an increasingly rapid pace,[2] including several complexes previously formulated as classical hydrides (e.g., $FeH_4(PEtPh_2)_3$).[3] Equilibrium between dihydrogen and dihydride ligands has been observed in solution,[2a,2d] emphasizing that η^2-H_2 complexes can be considered *tautomers* of hydrides and not merely arrested

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(1) Kubas, G. J.; Ryan, R. R.; Swanson, B. I.; Vergamini, P. J.; Wasserman, H. J. *J. Am. Chem. Soc.* **1984**, *106*, 451.

(2) (a) Kubas, G. J.; Unkefer, C. J.; Swanson, B. I.; Fukushima, E. *J. Am. Chem. Soc.* **1986**, *108*, 7000 and references therein. (b) Kubas, G. J.; Ryan, R. R.; Wroblewski, D. *J. Am. Chem. Soc.* **1986**, *108*, 1339. (c) Kubas, G. J. *Acct. Chem. Res.*, in press. (d) Chinn, M. S.; Heinekey, D. M. *J. Am. Chem. Soc.* **1987**, *109*, 5865. (e) Bianchini, C.; Mealli, C.; Peruzzini, M.; Zanobini, F. *Ibid.* **1987**, *109*, 5548. (f) Bautista, M.; Earl, K. A.; Morris, R. H.; Sella, A. *Ibid.* **1987**, *109*, 3780.

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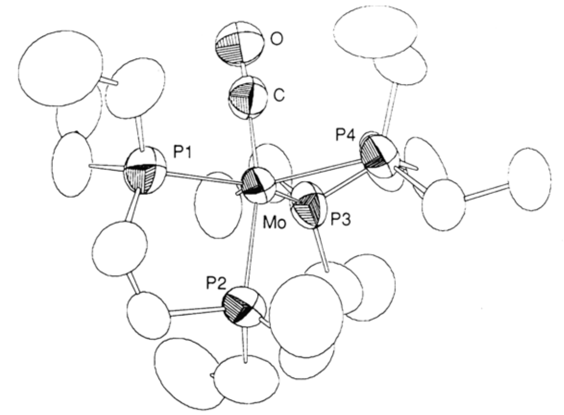

Figure 1. ORTEP drawing of $MoH_2(CO)(R_2PC_2H_4PR_2)_2$ for R = Et. Distances in angstroms, angles in degrees. Distances: Mo–P1 = 2.388 (3), Mo–P2 = 2.513 (3), Mo–P3 = 2.443 (3), Mo–P4 = 2.445 (3), Mo–C = 1.91 (1). Angles: P1–Mo–P_2 = 80.4 (1), P1–Mo–P_3 = 144.7 (1), P1–Mo–P4 = 135.7 (1), P2–Mo–P3 = 103.1 (1), P2–Mo–P4 = 103.7 (1), P3–Mo–P4 = 78.3 (1), C–Mo–P1 = 86.7 (3), C–Mo–P2 = 166.9 (3), C–Mo–P3 = 86.2 (3), C–Mo–P4 = 87.1 (3).

intermediates in hydride formation. Much remains to be learned about these complexes which represent the first examples of coordination of a σ bond to a metal. In particular, the electronic (e.g., ligand basicity) and steric (ligand bulk) factors which stabilize H_2 binding are not yet completely understood. We report here the preparation of a series of complexes in which the fate of hydrogen addition (η^2-H_2 versus dihydride binding) rests primarily upon the basicity of the metal center as dictated by the electron-donating ability[4] of ancillary phosphine ligands.

The complexes $MoH_2(CO)(R_2PC_2H_4PR_2)_2$ (**1**) have been synthesized by H_2 addition to unsaturated 16-electron precursors, $Mo(CO)(R_2PC_2H_4PR_2)_2$ (**2**).[5] Complex **1-Ph** had previously been established to be a dihydrogen complex,[2b] and new IR data have located ν_{HH} at 2650 cm^{-1}, one of the lowest values observed for an H_2 complex. Bonding models for η^2-H_2 coordination show that the primary interaction is donation of electrons from the σ molecular orbital of H_2 to a vacant metal orbital, combined with metal $\rightarrow$ H_2 σ^* back bonding, much as in metal–olefin binding.[2c,6] Increasing the basicity of ancillary ligands should thereby result in more effective back bonding to H_2 σ^*, ultimately leading to H–H bond scission. Thus, precursors **2** containing more basic[4] *alkyl*phosphines were prepared in order to determine if H_2 addition would result in a dihydride complex. Complexes **2** with R = Et, *i*-Bu, and with the mixed phosphine, $Ph_2PC_2H_4PEt_2$ (Ph–Et), were synthesized by refluxing *trans*-$Mo(N_2)_2(R_2PC_2H_4PR_2)_2$ in 10:1 toluene/ethyl acetate for 40 min under argon (a modification of the method for R = Ph[5]). The bis(N_2) complex abstracted CO from the ester, and the remaining labilized N_2 ligand dissociated at reflux temperature to give a dark-colored solution of **2**. Reduction of solvent volume in vacuo, addition of nonane, and further solvent removal yielded solid air-sensitive **2**,[7] which range in color from brick red (R = Et) to deep brown (Ph–Et). Complexes **2** possibly contain "agostic" M···H—C interactions as in $W(CO)_3(PR_3)_2$[8] and reversibly add N_2 to give yellow to orange $Mo(CO)(N_2)(R_2P{-}PR_2)_2$, (**3**),[7] analogous to the known[5] **3-Ph**. Addition of H_2 (1 atm) to hydrocarbon solutions of **2** rapidly gave **1**, isolable as highly soluble pale yellow (R = Et, *i*-Bu) or yellow-orange (Ph–Et) crystals upon solvent removal. In vacuo, **1** readily loses H_2 in solution to give **2**, but for R = Et, H_2 loss is much slower. The X-ray crystal structure[9] of **1-Et** showed a *7-coordinate pentagonal bipyramidal* structure, similar to $MoH_2(PMe_3)_5$,[10] with the CO and one phosphorus atom (P2) in axial sites (Figure 1). As for $MoH_2(PMe_3)_5$, the Mo–P distances show a considerable variation. In the equatorial plane the distances between Mo and P3 or P4 are significantly longer than the distance to P1, presumably due to the trans influence of the two *hydride* ligands which, no doubt, occupy distal sites in the plane. The Mo–P2 distance, approximately trans to CO, is longer still, and the three equatorial phosphine atoms are distorted toward the axial

Scheme I

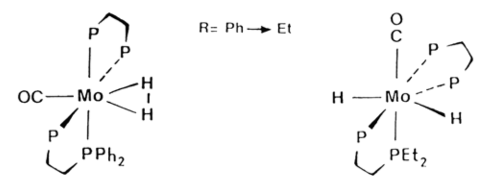

Table I. Spectral Parameters for $MoH_2(CO)(R_2P{-}PR_2)_2$

R	δ [J_{PH}, Hz]	J_{HD}, Hz	T_1,[a] ms	ν_{CO}[b]	ν_{MoH}[b]
Ph	−4.70 vbr	34	20	1815	875[c]
Et	−5.40 quint [34]	<2	370[d]	1790	1647
i-Bu	−5.11 quint [34]	<2	200	1780	1658
Ph–Et	−4.30 br			1788	

[a] At −70 °C. [b] In hexane except for R = Ph [Nujol], cm^{-1}. [c] Symmetric Mo–H_2 stretch. [d] 1 s at 25 °C.

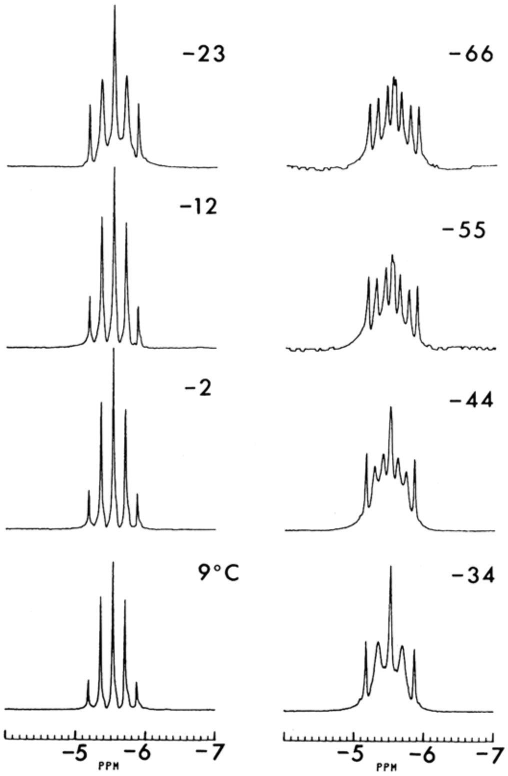

Figure 2. Variable temperature ^1H NMR spectrum of $MoH_2(CO)(Et_2PC_2H_4PEt_2)_2$ in toluene-d_8 at 200 MHz.

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(7) ν_{CO}, cm^{-1} [Nujol]: **2-Ph**, 1723; **2-Et**, 1748; **2-*i*-Bu**, 1725. ν_{NN}, ν_{CO}: **3-Et**, 2050, 1776; **3-*i*-Bu**, 2060, 1797; **3-(Ph–Et)**, 2060, 1782.

(8) Wasserman, H. J.; Kubas, G. J.; Ryan, R. R. *J. Am. Chem. Soc.* **1986**, *108*, 2294.

(9) The complex with R = Et crystallizes in the space group $P2_1/a$ with cell constants of *a* = 15.340 (14) Å, *b* = 10.804 (2) Å, *c* = 18.386 (6) Å, β = 109.44 (5)°, *Z* = 4, D_x = 1.30 g/cm^3. Solution of the structure by Patterson and Fourier techniques was followed by full-matrix least-squares refinements including anisotropic thermal parameters on all atoms heavier than hydrogen converged to an unweighted *R* value of 5.5% for 2497 intensity values for which $I > 2\sigma(I)$. Hydrogen atom positions could not be reliably determined. Additional details are presented as supplementary material.

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CO. Other distortions are understandable in terms of the bite angle of the phosphine. The overall geometry contrasts with that for R = Ph which was octahedral with an H_2 ligand *trans* to the CO. Thus, electronic control of H_2 activation on metal complexes has been achieved.

IR spectra of **1-Et** in Nujol showed a broad, medium-intensity Mo–H stretch at 1647 cm^{-1} shifting to 1194 cm^{-1} for the dideuteride. ^1H NMR {200 MHz, toluene-d_8} was consistent with a stereochemically nonrigid, 7-coordinate dihydride structure. At 25 °C, a sharp binomial quintet was observed at −5.40 ppm for the hydride ligands, in contrast to the broad singlets observed for the η^2-H_2 in **1-Ph**[2b] and the apparent η^2-H_2 in **1-(Ph–Et)** (Table I). Below −25 °C the hydride multiplet of **1-Et** broadened (Figure 2), behavior resembling that of $CrH_2[P(OMe)_3]_5$, which has been shown by NMR to be fluxional and possess the distal pentagonal bipyramidal structure.[11] In the slow exchange limit (< −66 °C) an A_2BCX_2 multiplet pattern (J_{PH} = 23, 49, 64 Hz) resulted, consistent with a pentagonal bipyramidal structure. ^1H NMR of $MoHD(CO)(Et_2P\text{–}PEt_2)_2$ displayed no observable HD coupling at 25 or −90 °C, while the Ph complex gave J_{HD} = 34 Hz, diagnostic[1,2a] of H_2 coordination.

The cone angles[12] of $P(i\text{-}Bu)_3$ and PPh_3 are similar (~145°) and both are larger than that for PEt_3 (132°). Thus the bulkiness of $R_2PC_2H_4PR_2$ should follow the same order, while the basicities of $R_2PC_2H_4PR_2$ for R = Et and *i*-Bu should be comparable but greater than that for R = Ph. Therefore, **1-*i*-Bu** provides an opportunity for separating steric and electronic factors. IR and NMR data (Table I) for **1-*i*-Bu** and its D_2 and HD isotopomers were similar to those for **1-Et**, indicating that **1-*i*-Bu** is also a dihydride. Since **1-*i*-Bu** is of comparable steric encumbrance to the H_2 complex **1-Ph**, it must follow that steric effects are of much less consequence than electronic influences in stabilizing H_2 coordination.

Several solution properties of **1-*i*-Bu**, including facile loss of H_2 in vacuo, relaxation time (T_1) of the hydride NMR signal, and collapse of the multiplet NMR pattern to a broad singlet below −55 °C, possibly indicate the presence of some η^2-H_2 tautomer. Crabtree has found that T_1 < 125 ms is characteristic of H_2 ligands while T_1 > 300 ms corresponds to hydride ligands.[4a,b,13] The T_1 for **1-Et** is 370 ms at −70 °C, consistent with a dihydride structure, while that for **1-Ph** is 20 ms, consistent with the known H_2 coordination. However, the T_1 for **1-*i*-Bu** (200 ms) is in the "gray area" between the values for H_2 and hydride complexes. Thus bulky ligands may favor H_2 ligation to a minor extent. Whether or not bulky ligands contribute to the *thermal* stability of η^2-H_2 complexes remains to be determined.

As in $Mo(CO)_3(PR_3)_2(H_2)$ and most other H_2 complexes, N_2 will displace the H_2 ligand in **1** to form the corresponding N_2 complexes **3** (the hydrides in **1-Et** and **1-*i*-Bu** are also displaceable). As a measure of the basicity of the metal center, Morris[14] has proposed that when ν_{NN} of N_2 complexes is in the range 2060–2160 cm^{-1}, H_2 complexes should result (upon "replacement" of the N_2 by H_2) versus hydrides for ν_{NN} <2060 cm^{-1} (electron-rich metal center). Interestingly, ν_{NN} for **3-Ph** is 2090 cm^{-1}, within the dihydrogen region, while ν_{NN} for **3-Et** (2050 cm^{-1}) and **3-*i*-Bu** (2060 cm^{-1}) are on the borderline (cf. 1950 cm^{-1} for the N_2 analogue of $MoH_2(PMe_3)_5$).

Further experiments are in progress to take advantage of this unique opportunity to map out the reaction coordinate for σ-bond activation at a metal center and to compare the chemistry of H_2 complexes with that of closely related dihydrides.

Acknowledgment. This work was performed under the auspices of the U. S. Department of Energy, Division of Chemical Sciences, Office of Basic Energy Sciences.

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Supplementary Material Available: Details of data collection, the structure determination, and refinement and tables of crystal data (Table II) and final coordinates and thermal parameters (Tables III and IV) (4 pages); listing of observed and calculated structure factors (Table V) (17 pages). Ordering information is given on any current masthead page.

A Synthetic Route to Forskolin[†]

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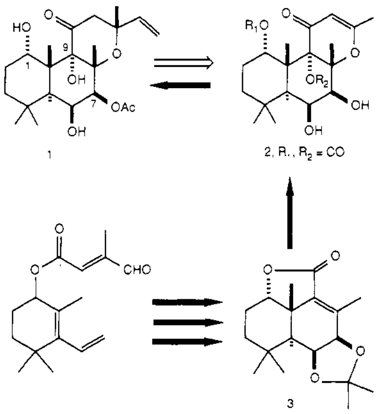
Received August 4, 1987

Forskolin **1**, isolated from the Indian plant *Coleus forskohlii*,[2] has been the subject of intense medicinal and chemical interest[3] owing to its pronounced inotropic,[4] antihypertensive,[4] and bronchospasmolytic[5] activity and its ability to effect adenylate cyclase activation in the absence of the guanine nucleotide-binding protein.[6] Forskolin and its derivatives lower intraocular pressure in humans by topical application.[7] In this communication, we report the formal synthesis of forskolin by the transformation of racemic lactone **3**, which had been previously synthesized by an



1

2, R₁, R₂ = CO

3

[†] Dedicated to Professor Kenneth B. Wiberg on the occasion of his 60th birthday.

[1] Recipient of a Dox Fellowship, 1987.

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