






Table I. Comparison of Rates ($\log k_{\text{obsd}}$) with Expected Rates of Single-Electron Transfer ($\log k_{\text{SET}}$) for Reactions of 9-Dialkylaminofluorenone Ions, 9-R₂N-FI⁻, with F₃CCH₂I, Ph₂CHCl, and PhCH₂Cl

NR ₂ ^a	E _{ox} (A ⁻) ^b	F ₃ CCH ₂ I ^c		Ph ₂ CHCl ^f		PhCH ₂ Cl ^g	
		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>i</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-R₂N-FIH compounds were prepared by the reaction of 9-BrFIH with the appropriate amine; p*K*_a data and physical properties will be reported separately. ^b Measured by cyclic voltammetry in Me₂SO with Ag/AgI reference electrode;⁹ referenced to the aqueous standard hydrogen electrode (SHE) by subtracting 0.125 V. ^c E_{rd} = 0.4 × (-1.425) = -0.57; λ = 60.¹⁰ ^d Measured spectrophotometrically by monitoring the disappearance of 9-R₂N-FI⁻ ion absorbance.⁵ ^e Calculated by using an equation derived from the Marcus equation by Ebersson:³ log k_{SET} = log k_d - log [1 + 0.2 exp {λ²/4(1 + (Δ*G*^o/4))²/RT}], where k_d is the diffusion rate in Me₂SO (3.3 × 10⁹);¹¹ λ is the intrinsic barrier chosen from literature values^{3b,4} to give the best fit, and Δ*G*^o is estimated from the redox potentials.^{3b} ^f E_{rd} = 0.4 × (-1.185) = -0.474 (vs SHE);¹⁰ λ = 65; Ebersson^{3b} reports λ = 63 for the reaction of Ph₂CH⁻ with PhCH₂Cl. ^g E_{rd} = -0.72 (Ebersson¹²); λ = 65.

the reactions of members of the 9-R₂N-FI⁻ ion family with F₃C-CH₂I, a known electron acceptor⁶ (Figure 1; Table I).

The kinetics with F₃CCH₂I 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-R₂N-FI⁻ ion family with Ph₂CHCl, which has a slightly more positive reduction potential (E_{rd}) than F₃CCH₂I (-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 Ebersson (Table I), shows a close correspondence of values for reactions of both F₃CCH₂I and Ph₂CHCl. This evidence suggests that both are reacting by SET mechanisms, F₃CCH₂I giving radical-type products⁶ and Ph₂CHCl giving S_N2-type products.

Surprisingly, PhCH₂Cl, which has a 0.8 V less positive E_{rd} (E_p) than does Ph₂CHCl, also gave a linear Marcus-type plot for reactions with 9-R₂N-FI⁻ ions, and the correspondence between k_{obsd} and k_{SET} (Table I) is close enough to fall in the range that Ebersson 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-R₂N-FI⁻ ions with Ph₂CHCl are occurring by SET involving rapid coupling of radical pair

intermediates, whereas those with PhCH₂Cl 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 F₃CCH₂I and Craig A. Wilson for rate data with PhCH₂Cl. This research was sponsored by a grant from the National Science Foundation.

Molecular Hydrogen Complexes. 5. Electronic Control of η²-H₂ versus Dihydride Coordination. Dihydride Structure of MoH₂(CO)(R₂PC₂H₄PR₂)₂ for R = Et, *i*-Bu versus η²-H₂ for R = Ph

Gregory J. Kubas,* R. R. Ryan, and Clifford J. Unkefer

Los Alamos National Laboratory
University of California
Los Alamos, New Mexico 87545

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Since their discovery,¹ new examples of transition-metal molecular-hydrogen (M-η²-H₂) complexes have been established at an increasingly rapid pace,² including several complexes previously formulated as classical hydrides (e.g., FeH₄(PEtPh₂)₃).³ Equilibrium between dihydrogen and dihydride ligands has been observed in solution,^{2a,2d} emphasizing that η²-H₂ complexes can be considered *tautomers* of hydrides and not merely arrested

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(7) Marcus plots log k_{obsd} vs Δ*G*^o; 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 Δ*G*^o. Over large ranges of Δ*G*^o the Marcus equation predicts curvature, but over relatively small ranges in the endergonic region the curve is flat and essentially linear.⁸

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(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) Ebersson^{3a} reports E_{rd} = -0.52 V vs SHE_{Me₂SO}. By applying -Δ*G*^o_{ir}(H₂O → Me₂SO) 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)₂N-FI⁻ ion with Ph₂CHCl, the presence of the persistent 9-(*i*-Pr)₂N-FI⁻ radical (λ_{max} = 450 nm)⁶ was not observed.

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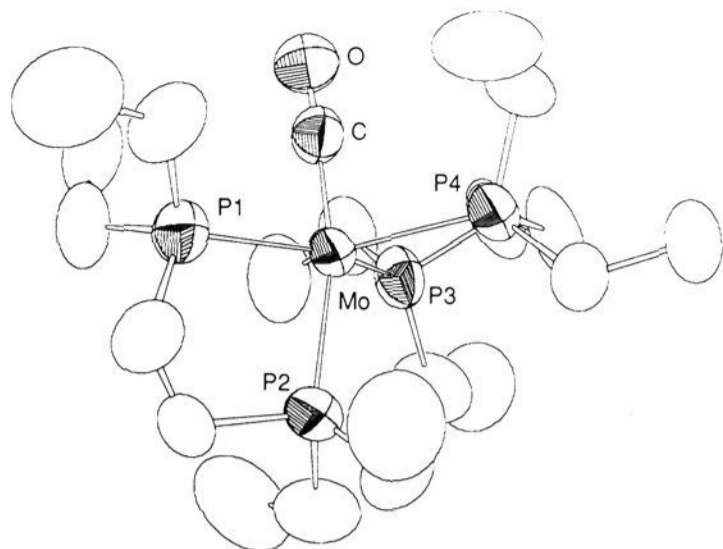
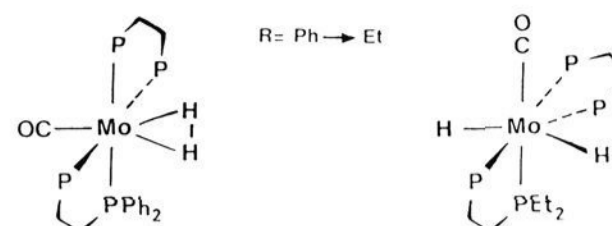


Figure 1. ORTEP drawing of $\text{MoH}_2(\text{CO})(\text{R}_2\text{PC}_2\text{H}_4\text{PR}_2)_2$ for $\text{R} = \text{Et}$. Distances in angstroms, angles in degrees. Distances: $\text{Mo-P1} = 2.388$ (3), $\text{Mo-P2} = 2.513$ (3), $\text{Mo-P3} = 2.443$ (3), $\text{Mo-P4} = 2.445$ (3), $\text{Mo-C} = 1.91$ (1). Angles: $\text{P1-Mo-P2} = 80.4$ (1), $\text{P1-Mo-P3} = 144.7$ (1), $\text{P1-Mo-P4} = 135.7$ (1), $\text{P2-Mo-P3} = 103.1$ (1), $\text{P2-Mo-P4} = 103.7$ (1), $\text{P3-Mo-P4} = 78.3$ (1), $\text{C-Mo-P1} = 86.7$ (3), $\text{C-Mo-P2} = 166.9$ (3), $\text{C-Mo-P3} = 86.2$ (3), $\text{C-Mo-P4} = 87.1$ (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 ($\eta^2\text{-H}_2$ versus dihydride binding) rests primarily upon the basicity of the metal center as dictated by the electron-donating ability⁴ of ancillary phosphine ligands.

The complexes $\text{MoH}_2(\text{CO})(\text{R}_2\text{PC}_2\text{H}_4\text{PR}_2)_2$ (**1**) have been synthesized by H_2 addition to unsaturated 16-electron precursors, $\text{Mo}(\text{CO})(\text{R}_2\text{PC}_2\text{H}_4\text{PR}_2)_2$ (**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 $\eta^2\text{-H}_2$ 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 $\rightarrow \text{H}_2 \sigma^*$ 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 $\text{H}_2 \sigma^*$, ultimately leading to H-H bond scission. Thus, precursors **2** containing more basic⁴ alkylphosphines were prepared in order to determine if H_2 addition would result in a dihydride complex. Complexes **2** with $\text{R} = \text{Et}$, *i*-Bu, and with the mixed phosphine, $\text{Ph}_2\text{PC}_2\text{H}_4\text{PEt}_2$ (Ph-Et), were synthesized by refluxing *trans*- $\text{Mo}(\text{N}_2)_2(\text{R}_2\text{PC}_2\text{H}_4\text{PR}_2)_2$ in 10:1 toluene/ethyl acetate for 40 min under argon (a modification of the method for $\text{R} = \text{Ph}$).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 ($\text{R} = \text{Et}$) to deep brown (Ph-Et). Complexes **2** possibly contain "agostic" $\text{M}\cdots\text{H}-\text{C}$ interactions as in $\text{W}(\text{CO})_3(\text{PR}_3)_2$ ⁸ and reversibly add N_2 to give yellow to orange $\text{Mo}(\text{CO})(\text{N}_2)(\text{R}_2\text{P-PR}_2)_2$, (**3**),⁷ analogous to the known⁵ **3-Ph**. Addition of H_2 (1 atm) to hydrocarbon solutions of **2** rapidly gave **1**, isolable as highly soluble pale yellow ($\text{R} = \text{Et}$, *i*-Bu) or yellow-orange (Ph-Et) crystals upon solvent removal. In vacuo, **1**

Scheme I

Table I. Spectral Parameters for $\text{MoH}_2(\text{CO})(\text{R}_2\text{P-PR}_2)_2$

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>i</i> -Bu	-5.11 quint [34]	<2	200	1780	1658
Ph-Et	-4.30 br			1788	

^aAt -70°C . ^bIn hexane except for $\text{R} = \text{Ph}$ [Nujol], cm^{-1} . ^cSymmetric Mo-H_2 stretch. ^d1 s at 25°C .

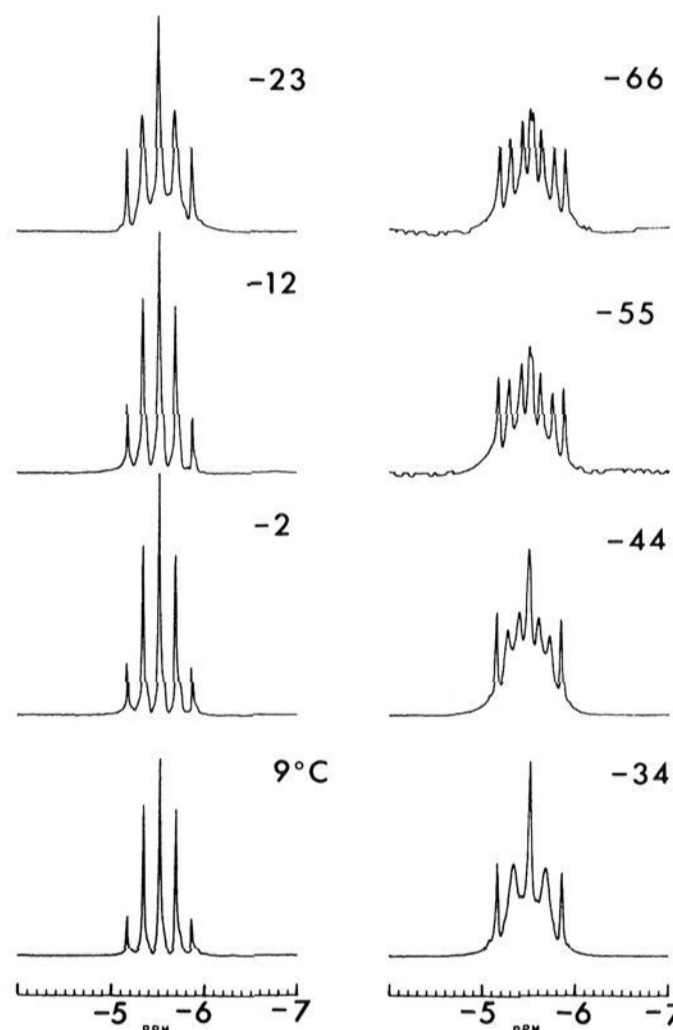


Figure 2. Variable temperature ^1H NMR spectrum of $\text{MoH}_2(\text{CO})(\text{Et}_2\text{PC}_2\text{H}_4\text{PEt}_2)_2$ in toluene- d_8 at 200 MHz.

readily loses H_2 in solution to give **2**, but for $\text{R} = \text{Et}$, H_2 loss is much slower. The X-ray crystal structure⁹ of **1-Et** showed a 7-coordinate pentagonal bipyramidal structure, similar to $\text{MoH}_2(\text{PMe}_3)_5$,¹⁰ with the CO and one phosphorus atom (P2) in axial sites (Figure 1). As for $\text{MoH}_2(\text{PMe}_3)_5$, 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

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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.

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(9) The complex with $\text{R} = \text{Et}$ 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, $\beta = 109.44$ (5)°, $Z = 4$, $D_x = 1.30 \text{ g/cm}^3$. 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 H₂ ligand *trans* to the CO. Thus, electronic control of H₂ 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 deuteride. 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 η²-H₂ in **1-Ph**^{2b} and the apparent η²-H₂ in **1-(Ph-Et)** (Table I). Below -25 °C the hydride multiplet of **1-Et** broadened (Figure 2), behavior resembling that of CrH₂[P(OMe)₃]₃, 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 A₂BCX₂ multiplet pattern (*J*_{PH} = 23, 49, 64 Hz) resulted, consistent with a pentagonal bipyramidal structure. ¹H NMR of MoHD(CO)(Et₂P-PEt₂)₂ displayed no observable HD coupling at 25 or -90 °C, while the Ph complex gave *J*_{HD} = 34 Hz, diagnostic^{1,2a} of H₂ coordination.

The cone angles¹² of P(*i*-Bu)₃ and PPh₃ are similar (~145°) and both are larger than that for PEt₃ (132°). Thus the bulkiness of R₂PC₂H₄PR₂ should follow the same order, while the basicities of R₂PC₂H₄PR₂ 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 D₂ 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 H₂ complex **1-Ph**, it must follow that steric effects are of much less consequence than electronic influences in stabilizing H₂ coordination.

Several solution properties of **1-*i*-Bu**, including facile loss of H₂ 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 η²-H₂ tautomer. Crabtree has found that *T*₁ < 125 ms is characteristic of H₂ 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 H₂ coordination. However, the *T*₁ for **1-*i*-Bu** (200 ms) is in the "gray area" between the values for H₂ and hydride complexes. Thus bulky ligands may favor H₂ ligation to a minor extent. Whether or not bulky ligands contribute to the thermal stability of η²-H₂ complexes remains to be determined.

As in Mo(CO)₃(PR₃)₂(H₂) and most other H₂ complexes, N₂ will displace the H₂ ligand in **1** to form the corresponding N₂ complexes **3** (the hydrides in **1-Et** and **1-*i*-Bu** are also displaceable). As a measure of the basicity of the metal center, Morris¹⁴ has proposed that when ν_{NN} of N₂ complexes is in the range 2060-2160 cm⁻¹, H₂ complexes should result (upon "replacement" of the N₂ by H₂) 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 N₂ analogue of MoH₂(PMe₃)₃).

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 H₂ 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.

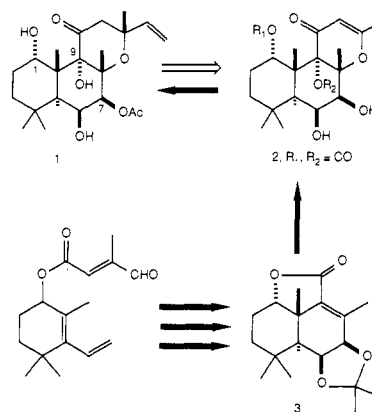
A Synthetic Route to Forskolin[†]

Frederick E. Ziegler,* Burton H. Jaynes,¹ and Manohar T. Saindane

*Sterling Chemistry Laboratory, Yale University
New Haven, Connecticut 06511-8118*

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Forskolin **1**, isolated from the Indian plant *Coleus forskohlii*,² has been the subject of intense medicinal and chemical interest³ owing to its pronounced inotropic,⁴ antihypertensive,⁴ and bronchospasmodic⁵ 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

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

(1) Recipient of a Dox Fellowship, 1987.

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