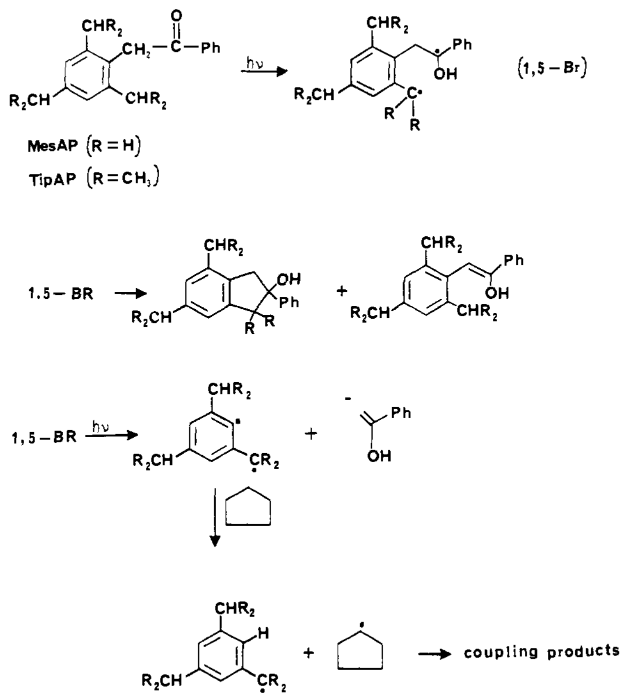
J. Am. Chem. Soc. **1984**, *106*, 4627-4629

Scheme I



process produces any new permanent products, we subjected 2-mL samples of deaerated cyclopentane solutions 0.02 M in ketone to 10 min of 308-nm laser excitation (6000 pulses). Capillary GC-MS analysis revealed much different product distributions from those obtained at low intensities. Several lower molecular weight products were formed from both ketones, most of which were easily identified from their MS spectra. Acetophenone was formed from both ketones. Triisopropylbenzene, tetraisopropylbicumyl, and a $C_{15}H_{22}$ product (probably 3,5-diisopropyl-1-propenylbenzene) were the major new products formed from TipAP, in total yields of 30% relative to indanol. Mesitylene, bimesityl, and mesitylcyclopentane were formed from MesAP in yields of 1-2% relative to indanol.

As summarized in Scheme I, we believe that these additional products all result from excitation and cleavage of the 1,5-biradical. It is not surprising that a biradical with an extra 90 kcal/mol of excitation energy might undergo fragmentation reactions inaccessible to the ground-state biradical. The actual fragmentation products appear to arise from the same sort of radical β-cleavage that gives Norrish type II elimination products from 1,4-biradicals.[12] Acetophenone enol presumably is formed in both cases. However, elimination of a benzyl-aryl 1,3-biradical from these 1,5-biradicals requires substantial additional excitation energy beyond that needed for a 1,4-biradical to eliminate a ground-state alkene. Given the high reactivity of aryl radicals,[13] the resulting 1,3-biradicals would be expected to abstract hydrogen rapidly from solvent to give substituted cumyl or benzyl radicals which then disproportionate and couple as usual. Our laser flash experiments indeed demonstrate the formation of benzyl radicals.

We ascribe the ability of these ketones to undergo this laser-specific photochemistry primarily to their short triplet lifetimes. The duration of the excimer laser pulses is 4-5 ns. With the triplet ketones reacting in less than 1 ns, and with the biradical products absorbing strongly at 308 nm, many of the biradicals formed by each laser pulse are also excited by the same pulse. These results are the first solution photochemistry of which we are aware in which a single light pulse sequentially excites first the reactant and then the metastable product formed from that reactant. As such, it exploits a combination of several characteristics unique to pulsed lasers, unlike other "laser vs. lamp" comparisons which utilize only high intensity[14] or monochromaticity.[15]

With MesAP, several apparent radical coupling products containing duryl, cyclopentyl, and benzoyl fragments are formed in 1% total yield with low-intensity lamps. This yield rises to 10% under excimer laser excitation. It appears that the laser pulse also excites triplet MesAP to an upper triplet which undergoes Norrish type I cleavage more efficiently than the lowest triplet. Biphotonic photochemistry involving upper excited states of reactants is well-known[16] and distinct from biradical excitation.

We are now studying other biradical systems to see how general this phenomenon may be. Two obvious questions involve the nature of the excitation and the necessity for a second benzylic center. We are also attempting to trap the postulated 1,3-biradicals, as Closs was able to do with the intermediates observed upon irradiation of 3*H*-indazoles.[17]

Acknowledgment. PJW thanks the National Science Foundation for continuing support of his research, the John Simon Guggenheim Foundation for a fellowship which facilitated this collaboration, and the National Research Council of Canada for its hospitality.

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[12] (a) Yang, N. C.; Yang, D.-H. *J. Am. Chem. Soc.* **1958**, *80*, 2913. (b) Wagner, P. J.; Kelso, P. A.; Zepp, R. G. *Ibid.* **1972**, *94*, 7480.

[13] Scaiano, J. C.; Stewart, L. C. *J. Am. Chem. Soc.* **1983**, *105*, 3609.

[14] (a) Turro, N. J.; Aikawa, M.; Butcher, J. A., Jr.; Griffin, G. W. *J. Am. Chem. Soc.* **1980**, *102*, 5127. (b) Turro, N. J.; Aikawa, M.; Gould, I. R. *Ibid.* **1982**, *104*, 856. (c) Adam, W.; Hannemann, K.; Hossel, P. *Tetrahedron Lett.* **1984**, *25*, 181.

[15] (a) Malatesta, V.; Willis, C.; Hackett, P. A. *J. Am. Chem. Soc.* **1981**, *103*, 6781. (b) Dauben, W. G.; Phillips, R. B. *Ibid.* **1982**, *104*, 355, 5780. (c) Wilson, R. M.; Wunderley, S. W.; Walsh, T. F.; Musser, A. K.; Outcalt, R.; Geiser, F.; Gee, S. K.; Brabender, W.; Yerino, L., Jr. *Ibid.* **1982**, *104*, 4429.

[16] (a) Siegel, S.; Eisenthal, K. *J. Chem. Phys.* **1965**, *42*, 2494. (b) Brocklehurst, B.; Gibbons, W. A.; Lang, F. T.; Porter, G.; Savadatti, M. I. *Trans. Faraday Soc.* **1966**, *62*, 1793. (c) Muralidharan, S.; Ferraudi, G. *J. Phys. Chem.* **1983**, *87*, 4877.

[17] Closs, G. L.; Riemenschneider Kaplan, L.; Bendall, V. I. *J. Am. Chem. Soc.* **1967**, *89*, 3376.

Structure of Phenyllithium in Solution

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Two X-ray crystallographic structures have been reported for phenyllithium. The first report is for the structure of the tetramethylethylenediamine (TMEDA) solvate, which is found to be a dimer with a planar arrangement of the C(1) and Li atoms.[1] Very recently, Hope and Power[2] have shown the existence of a tetrameric diethyl ether solvate in which the C(1) and Li atoms are at the eight corners of a distorted cube. It has been claimed,[3-5] on the basis of colligative methods, that the species in diethyl ether and tetrahydrofuran solution are dimers although there may be some indication of more extensive aggregation at high concentration (~0.8 M) in the former solvent.[6] Kinetic analyses of certain reactions of phenyllithium in the concentration range 0.005-2 M in tetrahydrofuran are consistent with a dimer, rather than a higher aggregate, in equilibrium with low concentrations of monomers.[7] Unequivocal evidence for the existence of the dimer in THF at −120 °C comes from the multiplicity of the

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[1] Thoennes, D.; Weiss, E. *Chem. Ber.* **1978**, *111*, 3157.

[2] Hope, H.; Power, P. P. *J. Am. Chem. Soc.* **1983**, *105*, 5321.

[3] Wittig, G.; Meyer, F. J.; Lange, G. *Justus Liebigs Ann. Chem.* **1951**, *571*, 167.

[4] Talalaeva, T. V.: Rodionov, A. N.; Kocheshkov, K. A. *Dokl. Chem.* (*Engl. Transl*) **1964**, *154*, 47.

[5] West, P.; Waack, R. *J. Am. Chem. Soc.* **1967**, *89*, 4395.

[6] West, P. In "MTP International Reviews of Science"; Vol. 4, B. Aylett, J., Ed.; University Park Press: Baltimore, 1972; Inorganic Chemistry, Ser. One, p 1, and references cited therein.

[7] Waack, R.; West, P.; Doran, M. A. *Chem. Ind.* (*London*) **1966**, 1035.

0002-7863/84/1506-4627$01.50/0

Table I. Spin-Lattice Relaxation Times (s) at 26 °C and Internuclear Distances (pm)

compd nucleus, (isotope)	$T_1^{\text{obsd }a}$	$T_1^{\text{DD }a}$	$T_1{}^{\text{tetramer}}{}_{\text{calcd}}$	$T_1{}^{\text{dimer}}{}_{\text{calcd}}$	X-ray		r_{obsd}	
					r_{dimer}	r_{tetramer}	r_{dimer}	r_{tetramer}
1 C(1), (^{7}Li)	15.4 ± 0.97							
						230	212 ± 4	227 + 5^b
		27.6 ± 2.43	36.4	45.6	221	230		
					228	234		
2 C(1), (^{6}Li)	31.8 ± 1.30							
3 H(2), (^{7}Li)	4.82 ± 0.08							
						241		
		7.48 ± 0.25	7.57	21.6	300	324	270 ± 1	270 ± 1^c
					341	401		
4 H(2), (^{6}Li)	11.9 ± 0.23							
		11.0 ± 0.30	12.9	>140	>400	270	262 ± 2	262 ± 2^d
5 H(2), (^{6}Li)	6.22 ± 0.11							

^{a}See ref 11. $^b r$[C(1)–Li]. $^c r$[H(2)–Li]. $^d r$[H(2)–H(2′)].

^{13}C(1) signal arising from coupling with ^{6}Li.[8] We have observed that solutions of phenyllithium in TMEDA and ether solvents at room temperature slowly generate benzene, an observation that casts some doubt of conclusions based on colligative properties. Solutions in diethyl ether–cyclohexane (1:2), however, are stable for long periods, and we now report studies of the detailed structure of phenyllithium by a novel method which should prove generally useful for investigating the solution structures of organolithium compounds.

We have determined the internuclear distances r[Li–C(1)] and r[Li–H(2)] from the contributions of ^{7}Li to the spin–lattice relaxation times of C(1) and H(2) using the isotope substitution method[9,10] with ^{7}Li and ^{6}Li. This procedure takes advantage of the fact that ^{7}Li is almost (0.7:1) as effective as ^{1}H in causing dipole–dipole relaxation and that the readily available ^{6}Li is very much less effective (^{6}Li/^{7}Li = 0.08). The rotational correlation times required for the calculation of r were evaluated from the T_1 of C(4), which arises solely from dipole–dipole interaction with the attached proton and is unaffected by possible internal rotation of the phenyl rings. The cubic tetramer must undergo isotropic rotational diffusion, and inspection of a molecular model of the dimer indicates that the assumption of a single correlation time for such a species is also justified. The vibrationally averaged bond distance of 110.7 pm for r[C(4)–H(4)][9] has been used. The interring ortho proton internuclear distance has also been determined by using the deuterium-substitution method.[9,10]

Measurements of r[Li–C(1)] were made using the ^{7}Li and ^{6}Li isotopomers of [2,3,5,6-^{2}H$_4$]phenyllithium (**1** and **2**, respectively) in order to eliminate contributions to T_1C(1) from the ortho protons. It was also necessary to measure the T_1's at 2.35T in order to reduce relaxation by chemical shift anisotropy.[12] Two solvent systems, diethyl ether–cyclohexane (1:2) and TMEDA–cyclohexane (1:9),[15,16] which favor the tetramer and dimer, respectively, were used. The values of r[Li–C(1)] (Table I) are in excellent agreement with those found in the X-ray structures of the corresponding crystalline solvates. A comparison of the predicted and observed relaxation contributions of ^{7}Li are also included in Table I.

Table II. ^{13}C Chemical Shifts at 26 °C^a

	δ ^{13}C(3)	^{13}C shifts relative to C(3)		
		C(1)	C(2)	C(4)
1.1 M Ph^{7}Li, THF-d_8^{b}	125.00	+63.2	+19.2	−1.8
1.5 M Ph^{7}Li-2,3,5,6-d_4, TMEDA/C$_6$H$_{12}$, 1:9	124.92 (t)	+59.6	+18.9	−1.4
0.2 M Ph^{7}Li, Et$_2$O	127.45	+53.4	+16.6	−1.1
6 M Ph^{7}Li, Et$_2$O	127.92	+48.3	+15.0	−0.82
5^c	126.54 (t)	+48.3	+15.1	−0.66
2	126.05 (t)	+47.6	+14.7	−0.64

^{a}C$_6$H$_{12}$ = 27.300. ^{b}THF-d_8 = 25.300. ^{c}C$_6$D$_{12}$ = 26.400.

In order to measure accurately the contribution of ^{7}Li to T$_1$ of H(2), it was necessary to reduce contributions from the solvent protons,[17] the ortho protons of adjacent rings, and, of course, meta protons on the same rings. Accordingly, samples (**3** and **4**) of the ^{7}Li and ^{6}Li isotopomers of [3,5-^{2}H$_2$]phenyllithium diluted 9-fold with [2,3,5,6-^{2}H$_4$]phenyllithium in [^{2}H$_{12}$]cyclohexane/[^{2}H$_{10}$]diethyl ether were used. In both the tetramer and dimer, H(2) is relaxed by two lithium nuclei and our measurements yield an effective distance ($\langle 1/r^6\rangle^{1/6}$) of 263 pm/Li. This clearly indicates that the structure is not that of the dimer for which r[Li–H(2)] distances of 341 and 300 pm are found in the crystalline TMEDA solvate. On the other hand, the value is in very good agreement with the two distances of 242 and 324 pm ($\langle 1/r^6\rangle^{1/6}$ = 265 pm) found for the tetrameric etherate in the solid state.

Comparison of the relaxation times of the ortho protons of **4** with those of pure [3,5-^{2}H$_2$,^{6}Li]phenyllithium in the fully deuterated solvent mixture **5** leads to an interring r[H(2)–H(2′)] of 262 pm, which is also in complete agreement with the tetrameric structure (r = 270 pm). The corresponding distance in the dimer is >400 pm.

Clearly, these results establish the structure of phenyllithium in cyclohexane/diethyl ether as being tetrameric and having essentially the same geometry as in the solid state.[18] Examination of ^{13}C chemical-shift data (Table II) for various solutions indicates, however, that the tetrameric and dimeric structures coexist in dilute diethyl ether solution. The data further indicate that phenyllithium is dimeric in THF at room temperature as well as at −120 °C.[19]

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(8) Seebach, D.; Hässig, R.; Gabriel, J. *Helv. Chim. Acta* **1983**, *66*, 308.

(9) Jackman, L. M.; Trewella, J. C. *J. Am. Chem. Soc.* **1976**, *98*, 5712.

(10) Jackman, L. M.; Trewella, J. C. *J. Am. Chem. Soc.* **1979**, *101*, 6428.

(11) The experimental values of T_1C(4) varied from sample to sample depending on concentration and also solvent composition but were generally in the range 0.5–1.0 s. Values of all other T_1's given in Table I are normalized to T_1C(4) = 1.00, and their standard deviations include the propagated error in T_1C(4).

(12) A sizable chemical-shift anisotropy has been evoked to account for the anomalous chemical shifts of C(1) and H(2) in phenyllithium.[13,14] We have measured T_1C(1) at 2.35, 4.7, and 8.5T for a 1.0 M cyclohexane/ether solution (sample **2**) and have found that $1/T_1^{\text{CSA}}$ contributes 34.3%, 67.6%, and 90.4%, respectively, to $1/T_1^{\text{total}}$. C(4) exhibited full nOe.

(13) (a) Fraenkel, G.; Adams, D. G.; Dean, R. R. *J. Phys. Chem.* **1968**, *72*, 944. (b) Fraenkel, G.; Dayagi, S.; Kobayashi, S. *Ibid.* **1968**, *72*, 953. (c) Jones, A. J.; Grant, D. M.; Russell, J. G.; Fraenkel, G. *Ibid.* **1969**, *73*, 1624.

(14) Grutzner, J. B., private communication.

(15) Phenyllithium in this solvent, as in pure ether, slowly generates benzene.

(16) r_{obsd}[C(1)—Li] = 218.8 ± 2.2 pm.

(17) T_1H(2) for 1.0 M [3,5-^{2}H$_2$,^{6}Li]phenyllithium in [^{2}H$_{12}$]cyclohexane/diethyl ether at 26 °C increased from 4.33 to 7.72 s[11] when the ether was replaced by its ^{2}H$_{10}$ isotopomer.

(18) The effect of internal rotation about the C(1)–C(4) axis may cause *shortening* of the r[H(2)–Li] and r[H(2)–H(2′)] by <18 pm and does not invalidate our conclusions regarding the existence of a cubic tetramer. All r_{obsd} measurements are the vibrationally averaged internuclear distances.[9]

(19) **Note Added in Proof**: Dr. G. Fraenkel has informed us that PhLi·TMEDA in diethyl ether at −60 °C exists mainly as the dimer.[20]

(20) Fraenkel, G.; Hsu, H.; Su, B. M., Lithium Chemistry Symposium, ACS S.E. Regional Meeting, Charlotte, N.C. Nov. 1983.

Acknowledgment. We thank the National Science Foundation (CHE 81-03037) for support of this research.

Registry No. 1, 91083-39-5; **2**, 91083-40-8; **3**, 91083-41-9; **4**, 91083-42-0; **5**, 91083-43-1; LiC$_6$H$_5$, 591-51-5.

Highly Diastereocontrolled Reduction of Ketones by means of Hydrosilanes. Practical Synthesis of Optically Active 1,2-Diols and 2-Amino Alcohols of Threo or Erythro Configuration

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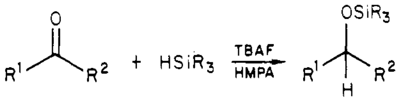
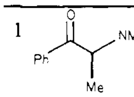
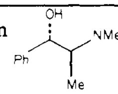
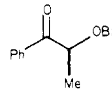
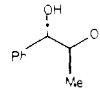
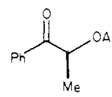
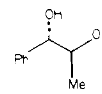
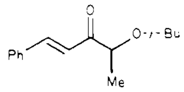
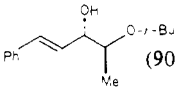
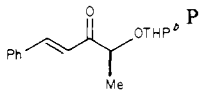
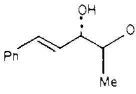
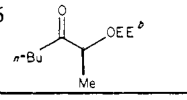
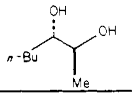
In spite of numerous studies on the stereoselective transformations carried out during last two decades,[1,2] the reduction of 2-amino or 2-hydroxy ketones remains unexplored in a practical point of view.[3,4] Since the resulting 2-amino alcohols and 1,2-diols constitute important structural moieties of pharmacologically useful substances, research on this subject seems to be warranted. In this communication we report a new convenient method for such transformation, wherein diastereometric threo or erythro alcohols can be selectively produced by merely changing the catalyst with almost complete stereochemical control.

Of many hydride reagents, hydrosilanes are outstanding in regard to easy handling. We have found that hydrolsilanes reduce aldehydes and ketones in the presence of a catalytic amount[5] of tetrabutylammonium fluoride (TBAF) in hexamethylphosphoric triamide (HMPA).[6] A solution of an aldehyde or a ketone (1 mmol) and phenyldimethylsilane (1.2 mmol) in HMPA (1–2 mL) was treated with TBAF (0.5 M tetrahydrofuran (THF) solution, 2–5 mol %). After the reaction at 0 °C or at room temperature for 0.5–12 h and subsequent workup, the yield of the resulting silyl ether was estimated by GLC assay. The results are given in the order of substrate and yield (%): n-C$_{10}$H$_{21}$CHO, 91; PhCHO, 91; PhCH=CHCHO, 100;[8] n-C$_6$H$_{13}$COMe, 87; CH$_2$=CHCH$_2$CH$_2$COMe, 57. In addition to the regio- and chemoselectivity,[5,7,8] the reaction shows remarkably high stereoselectivity. 2-Methylcyclohexanone was reduced to give *cis*-2-methylcyclohexanol with 94% selectivity. The cis selectivity was found to depend on the bulkiness of the silicon ligands (hydrosilane and % cis): PhMe$_2$SiH, 76; Ph$_2$SiH$_2$, 86; Ph$_2$MeSiH, 94; Ph$_3$SiH, 95 (yields 40–99%). The results are consistent with the fact that the cis selectivity becomes higher with the increasing steric bulk of the hydride reagent.[9]



$$R^1C(=O)R^2 + HSiR_3 \xrightarrow[HMPA]{TBAF} R^1C(OSiR_3)(H)R^2$$

Table I. TBAF-Catalyzed Threo-Selected Ketone Reduction with Hydrosilane[a]

run	ketone	hydrosilane and conditions	product (% yield)[c]	threo: erythro
1		PhMe$_2$SiH, room temp, 12 h	 (83)	>99:1[d]
2		PhMe$_2$SiH, 0 °C, 6 h	 (82)	96:4[d]
3		PhMe$_2$SiH, 0 °C, 20 h	 (95)	95:5[d]
4		PhMe$_2$SiH, 0 °C, 20 h	 (90)	93:7[e]
5		Ph$_2$MeSiH, 0 °C, 16 h	 (77)	87:13[e]
6		PhMe$_2$SiH, 0 °C, 12 h	 (86)[f]	84:16[f]

[a] Generally, 1.1–1.2 mol of hydrosilane and 5–10 mol % of TBAF in HMPA were employed. [b] Bz = benzoyl, EE = 1-ethoxyethyl, THP = tetrahydropyran-2-yl. [c] The *O*-silyl group and the protecting group were removed under acidic (1 M HCl, room temperature) or basic (1 M KOH/MeOH, room temperature) conditions, and the total yield is given. The isolated major product is shown. [d] Estimated by ^{1}H NMR analysis. The *threo*-amino alcohol showed a peak at δ 4.15 (d, J = 10 Hz, C*H*–OH) and the *threo*-glycol at δ 4.28 (d, J = 7 Hz), whereas each erythro isomer gave a peak at δ 4.88 (d, J = 4 Hz) and δ 4.57 (d, J = 4 Hz), respectively. [e] Determined by HPLC assay. [f] Determined by GLC analysis.

The stereoselective reduction finds further applications in acyclic system. When 2-(dimethylamino)-1-phenyl-1-propanone was treated with dimethylphenylsilane (1.2 mol) in HMPA in the presence of TBAF (5 mol %) (room temperature, 12 h), *N*-methylpseudoephedrine with exclusively threo configuration[10] was produced after desilylation. The similar threo-selective reduction was found applicable to 2-hydroxy ketone derivatives (Table I). It should be emphasized that racemization of the starting ketone does not take place to any measurable extent. For example, (*S*)-2-acetoxy-1-phenyl-1-propanone of 88% ee was reduced to give (1*S*,2*S*)-1-phenyl-1,2-propanediol of 88% ee (Table I, run 3). The stereochemical outcome[11] is attributed to the bulky

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(1) (a) Morrison, J. D.; Mosher, H. S. "Asymmetric Organic Reactions"; Prentice-Hall: Englewood Cliffs, NJ, 1971; Chapter 3. (b) Tramomtini, M. *Synthesis* **1982**, 605.

(2) (a) Yamada, S.; Koga, K. *Tetrahedron Lett.* **1967**, 1711. (b) Katzenellenbogen, J. A.; Bowlus, S. B. *J. Org. Chem.* **1973**, *38*, 627. (c) Bowlus, S. B.; Katzenellenbogen, J. A. *Ibid.* **1974**, *39*, 3309. See also (3): Keck, G. E.; Boden, E. P. *Tetrahedron Lett.* **1984**, *25*, 265 and references cited therein.

(3) Threo-selective reduction of 2-hydroxy ketones having a sophisticatedly bulky hydroxyl protecting group: (a) Nakata, T.; Tanaka, T.; Oishi, T. *Tetrahedron Lett.* **1983**, *24*, 2653. (b) Nakata, T.; Fukui, M.; Oishi, T. *Ibid.* **1983**, *24*, 2657. (c) Nakata, T.; Fukui, M.; Ohtsuka, H.; Oishi, T. *Ibid.* **1983**, *24*, 2661. (d) Overman, L. E.; McCready, R. J. *Ibid.* **1982**, *23*, 2355.

(4) Buckley, T. F., III; Rapoport, H. *J. Am. Chem. Soc.* **1981**, *103*, 6157.

(5) Use of fluoride salts such as KF and CsF effects the hydrosilylation of aldehydes, ketones, and esters as shown by Corriu and his co-workers. For efficient reduction they had to employ appropriately activated hydrosilanes such as di- or trihydrosilanes and alkoxysilanes. Boyer, J.; Corriu, R. J. P.; Perz, R.; Reye, C. *J. Organomet. Chem.* **1979**, *172*, 143. Corriu, R. J. P.; Perz, R.; Reye, C. *Tetrahedron* **1983**, *39*, 999.

(6) Other aprotic polar solvents such as 1,3-dimethyl-3,4,5,6-tetrahydro-2(1*H*)-pyrimidinone (DMPU) and *N*,*N*-dimethylformamide were also applicable, whereas less polar ones, e.g., tetrahydrofuran (THF) and dichloromethane, were inefficient.

(7) Nitriles and esters were not reduced.

(8) A parallel reaction with RhCl(PPh$_3$)$_3$ as the catalyst afforded a mixture of 1,2- and 1,4-hydrosilylated products in a ratio of 47:53. Regioselectivity in the RhCl(PPh$_3$)$_3$ catalyzed hydrosilylation of α,β-unsaturated compounds is discussed in detail: Ojima, I.; Kogure, T. *Organometallics* **1982**, *1*, 1390.

(9) Wigfield, D. C. *Tetrahedron* **1979**, *35*, 449 and references cited therein.

(10) The relative stereochemical nomenclature proposed by Noyori (Noyori, R.; Nishida, I.; Sakata, J. *J. Am. Chem. Soc.* **1983**, *105*, 1598, footnote 32) is particularly pertinent. This notation applies not only to recently studied aldols and β-hydroxy esters but also to classical 1,2-diols, 2-amino alcohols, and sugars (Streitwieser, A., Jr.; Heathcock, C. H. "Introduction to Organic Chemistry"; Macmillan: New York, 1976; Chapters 24 and 25), and we need not change the conventional notation. Compare other notations: (a) Masamune, S.; Ali, S. A.; Snitman, D. L.; Garvey, D. S. *Angew. Chem., Int. Ed. Engl.* **1980**, *19*, 557. (b) Seebach, D.; Prelog, V. *Ibid.* **1982**, *21*, 654. (c) Carey, F. A.; Kuehne, M. E. *J. Org. Chem.* **1982**, *47*, 3811.

(11) The configuration of the products was determined by comparison of their NMR spectra with those of an authentic sample (run 1) or with the reported values (runs 2 and 3: Zioudrou, C.; Chrysochou, P. *Tetrahedron* **1977**, *33*, 2103). Other products were transformed into known compounds or judged as such on the basis of the empirical rule of J(threo) > J(erythro) (see footnote *d* of Table I).

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