Potassium Triphenylborohydride. A New Reducing Agent for the Reduction of Carbonyl Compounds with an Exceptional Stereo- and Chemoselectivity

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Potassium triphenylborohydride (KTPBH), a highly hindered potassium triarylborohydride prepared from triphenylborane and potassium hydride, exhibits remarkable stereo- and chemoselectivity for the reduction of carbonyl compounds. KTPBH reduces 2-methylcyclohexanone to give *cis*-2-methylcyclohexanol with an excellent stereospecificity (98.5:1.5), approaching that of lithium tri-*sec*-butylborohydride. KTPBH also shows a remarkable chemoselectivity. It shows 97:3 selectivity between cyclohexanone and cyclopentanone and 99.4:0.6 selectivity between cyclohexanone and 4-heptanone. This chemoselectivity is comparable to those achieved by lithium di-*n*-butyl-9-BBN and *tert*-butylamine–borane, the best two reagents reported for such purposes. KTPBH is stable over 2 months at room temperature, and this reagent possesses a practical advantage in the isolation of the alcohol product without oxidation and distillation.

The reaction of potassium hydride with weak Lewis acids such as hindered trialkylboranes[1] and trialkoxyboranes[2] proceeds readily with the formation of the corresponding trialkyl- and trialkoxyborohydrides. Among these, K-*sec*-Bu$_3$BH[1a] and K(*i*-PrO)$_3$BH[2c] are reported to show very good stereoselectivities for the reduction of carbonyl compounds.
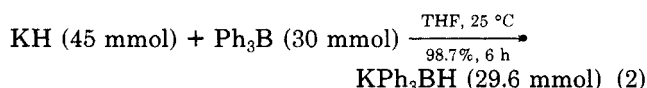
The reaction of saline hydride and triarylborane has been reported,[3] but application of this reagent to the reduction of carbonyl compounds has been reported only with lithium dimesitylborohydride–bis(dimethoxyethane) (LDMBH$_2$·2DME).[3b] In 1949, potassium triphenylborohydride was reported by Wittig and co-workers.[3a] But, they prepared this reagent from triphenylborane and 1,4-dipotassio-1,1,4,4-tetraphenylbutane instead of potassium hydride. Now, we prepared potassium triphenylborohydride (KTPBH) from triphenylborane and potassium hydride in tetrahydrofuran (THF). This re-

$$\text{KH} + \text{Ph}_3\text{B} \xrightarrow[\text{25 °C, 6 h}]{\text{THF}} \text{KPh}_3\text{BH} \qquad (1)$$
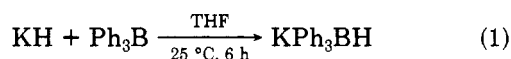
action provides a convenient synthesis of KTPBH in high yield under very mild conditions. Now, we wish to report that KTPBH shows high stereo- and chemoselectivity in the reduction of carbonyl compounds in THF.

Results and Discussion

Reaction of Triphenylborane with Potassium Hydride. KTPBH was prepared by the addition of 37.5 mL of 0.80 M solution of triphenylborane in THF (30 mmol) to 1.8 g of potassium hydride (45 mmol, 50% excess, free from oil) and stirring vigorously to provide a 39.0 mL of 0.76 M solution (29.6 mmol) of the product in 98.7% yield.

$$\text{KH (45 mmol)} + \text{Ph}_3\text{B (30 mmol)} \xrightarrow[\text{98.7\%, 6 h}]{\text{THF, 25 °C}} \text{KPh}_3\text{BH (29.6 mmol)} \qquad (2)$$

The reaction was carried out under dry nitrogen for 6 h at 25 °C. The course of the reaction was monitored by

withdrawing aliquots of the clear reaction mixture at appropriate intervals and observing its hydride concentration. As shown in Figure 1, potassium hydride reacts with triphenylborane somewhat slower than with tri-*sec*-butylborane, however, much faster than trisiamylborane. The rate difference may be attributed to the steric and electronic effects.[1b]

In the presence of excess potassium hydride, triphenylborane appears to undergo simple Lewis acid–base reaction in 1/1 ratio to form a molecular addition compound. Based on this stoichiometry, the ratio of K/B/H in solution should be 1/1/1. We analyzed solution of KTPBH for potassium, boron, and hydride.[1b] Indeed, such analyses revealed a 1/1/1 ratio. Concentration of potassium was determined by hydrolyzing a known aliquot of the solution with water and titrating the base formed against standard acid. Boron was determined by GLC analysis of the phenol produced by oxidation of KTPBH. Hydride concentration was estimated by measuring the hydrogen evolved on hydrolysis of the known aliquot of the solution.

IR and ^{11}B NMR Spectra. Solution of KTPBH in THF displayed a typical absorption in the IR. A strong, broad absorption is observed around 2200 cm^{-1}, attributed to the B–H stretching vibration in the borohydride anion.[4] ^{11}B NMR showed a clean sharp doublet (J_{B-H} = 78.7 Hz) due to boron–hydrogen coupling centered at δ −7.96 relative to BF$_3$·OEt$_2$. Similar observation was reported for the tetraethylammonium triphenylborohydride (2198 cm^{-1} for B–H stretching vibration, J_{B-H} = 79 ± 1 Hz).[4]

Stability of Potassium Triphenylborohydride. Under dry nitrogen, THF solution of KTPBH was maintained at room temperature, and the hydride concentration was checked periodically by hydrolysis of aliquots. The hydride concentration of KTPBH remained constant after 2 months at room temperature. This results clearly indicate that under dry nitrogen this reagent was stable over 2 months at room temperature.

Stereoselectivities in the Reduction of Cyclic Ketones. In order to test the stereoselectivity of KTPBH in the ketone reduction and compare it with those of stereoselective hydrides, we have chosen a representative group of cyclic ketones, namely, 2-methylcyclohexanone, 4-*tert*-butylcyclohexanone, norcamphor, and *d*-camphor. The first three ketones were readily reduced quantitatively

(1) (a) Brown, C. A. *J. Am. Chem. Soc.* **1973**, *95*, 4100. (b) Brown, C. A.; Krishnamurthy, S. *J. Organomet. Chem.* **1978**, *156*, 110.

(2) (a) Brown, H. C.; Nazer, B.; Sikorski, J. A. *Organometallics* **1983**, *2*, 634. (b) Brown, H. C.; Cha, J. S.; Nazer, B. *Inorg. Chem.* **1984**, *23*, 2929. (c) Brown, H. C.; Cha, J. S.; Kim, S. C.; Brown, C. A. *J. Org. Chem.* **1984**, *49*, 885.

(3) (a) Wittig, G.; Keicher, G.; Rückert, A.; Raff, P. *Justus Liebigs Ann. Chem.* **1949**, *563*, 110. (b) Hooz, J.; Akiyama, S.; Cedar, F. J.; Bennett, M. J.; Tuggle, R. M. *J. Am. Chem. Soc.* **1974**, *96*, 274.

(4) Burlitch, J. M.; Burk, J. H.; Leonowicz, M. E.; Hughes, R. E. *Inorg. Chem.* **1979**, *18*, 1702.

Table I. Stereoselective Reduction of Cyclic and Bicyclic Ketones with Potassium Triphenylborohydride (KTPBH) in THF[a,b]

ketone	temp, °C	less stable isomer	ratio of less stable isomer, %		
			KTPBH	LDMBH₂·2DME[e]	Li-sec-Bu₃BH[f]
2-methylcyclohexanone	0	cis	95.4	99.0	98.0 (99.0)[g]
	−78		98.5		
4-tert-butylcyclohexanone	0	cis	78.5	94.0	93.0 (88.0)[g,h]
	−78		92.8		
norcamphor	0	endo	91.2		99.6
	−78		98.1		
d-camphor	0	exo			99.6
	25		c	99.8	
	65		96.5[d]		

[a] Reaction mixtures were 0.25 M in ketone at 0 °C, and those at −78 °C were 0.125 M. A 1.1:1 ratio for reagent–ketone was used. Reactions at 0 °C were run for 1 h and those at −78 °C for 3 h. [b] The yields of alcohols (GLC) were quantitative. [c] A 25% reduction to the corresponding alcohol in 24 h at 25 °C. [d] A 91% reduction to the corresponding alcohol in 6 h. [e] Lithium dimesitylborohydride-bis(dimethoxyethane); Hooz, J.; Akiyama, F. J.; Cedar, M. T.; Tuggl, R. M. *J. Am. Chem. Soc.* 1974, 96, 274. [f] L-Selectride: Krishnamurthy, S.; Brown, H. C. *J. Am. Chem. Soc.* 1972, 94, 7159. [g] K-Selectride: Brown, C. A. *J. Am. Chem. Soc.* 1973, 95, 4100. [h] The data of 4-methylcyclohexanone.
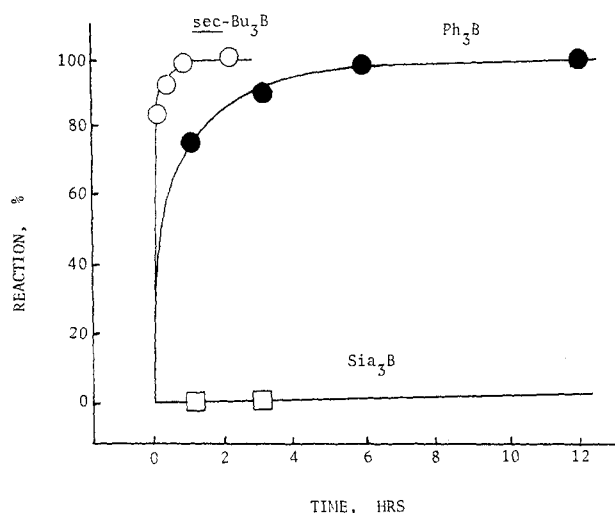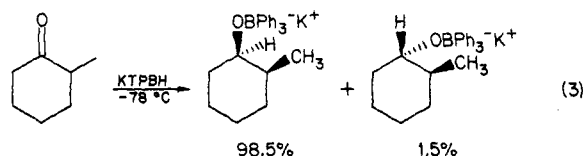

Figure 1. Reaction of triphenylborane with potassium hydride in THF at 25 °C. Comparable data for the tri-sec-butylborane and trisiamylborane with potassium hydride have been published (cf. ref 1b).

with an equimolar KTPBH (10% excess) in 1 h at 0 °C and in 3 h at −78 °C. However, d-camphor, to our surprise,


was reduced only 25% in 24 h even at 25 °C. However, we could reduce it in 6 h at 65 °C in a 91% yield. The results are summarized in Table I.

Generally, KTPBH showed an excellent stereoselectivity toward the monocyclic and bicyclic ketones examined, comparable to other stereoselective hydride reducing agents, such as K-sec-Bu₃BH,[1] LDMBH₂·2DME,[3b] and Li-sec-Bu₃BH.[5] Apparently the three phenyl groups in KTPBH exert a similar degree of steric effect as the three sec-butyl groups in L- or K-Selectride (Aldrich), Li-sec-Bu₃BH or K-sec-Bu₃BH. These results are summarized in Table I.

Chemoselectivities in the Competitive Reduction between Carbonyl Compounds. Selective reduction of one carbonyl group in the presence of other such groups is a frequent synthetic problem. In recent years, various reagents have been developed for such selective reduc-

tions.[6] These reagents are mostly capable of reducing aldehydes in the presence of ketones, and only a few reagents have been reported for the selective reduction between ketones.[7] The unexpected slow reduction of d-camphor prompted us to examine the chemoselectivity of KTPBH. The chemoselectivity of KTPBH was studied by competitively reducing the equimolar mixture of two carbonyl compounds with an equimolar KTPBH. And, it was tested for seven representative ketone–ketone pairs and three aldehyde–ketone pairs. The results are summarized in Table II. As shown in Table II, KTPBH exhibits a very good chemoselectivity especially between ketones. Thus, 2-heptanone and acetophenone can be selectively reduced in the presence of 4-heptanone (eq 4).
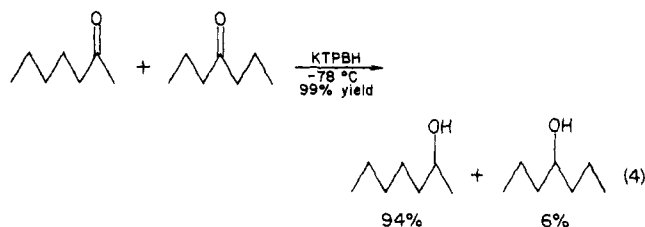
The structural difference is only methyl vs. n-propyl (entries 1 and 2). KTPBH shows a much better selectivity between n-propyl vs. tert-butyl showing 98.9:1.1 selectivity (entry 3). Cyclohexanone was reduced much more readily than 2-heptanone and 4-heptanone showing 88.7:11.3 and 99.4:0.6 selectivities. These chemoselectivities are comparable with those of tert-butylamine–borane[7b] but far better than those of Li(n-Bu)₂-9-BBN[7a] (entries 4 and 5). The 95:5 selectivity between 4-tert-butylcyclohexanone and 2-methylcyclohexanone is surprising. Since, KTPBH attack 4-tert-butycyclohexanone and 2-methylcyclohexanone both from the equatorial site (Table I), the effect of methyl group should be minimal. KTPBH also differenciate cyclohexanone and cyclopentanone excellently with a 97:3 selectivity. tert-Butylamine–borane is reported to show 100:10 selectivity for the same pair of keones (entry 7). As shown in entries 8–10, KTPBH also shows a good selectivity between aldehyde and ketone with preferential reduction of aldehyde. The selectivity of KTPBH is equal to or slightly lower than that of previously reported se-

(5) Brown, H. C.; Krishnamurthy, S. *J. Am. Chem. Soc.* 1972, 94, 7159.

(6) (a) Brown, H. C.; Kulkarni, S. U. *J. Org. Chem.* 1977, 42, 4169. (b) Yoon, N. M.; Cha, J. S. *J. Korean Chem. Soc.* 1978, 22, 259. (c) Krishnamurthy, S. *J. Org. Chem.* 1981, 46, 4628. (d) Yoon, N. M.; Park, G. B.; Gyoung, Y. S. *Tetrahedron Lett.* 1983, 5367.

(7) (a) Yamamoto, T.; Toi, T.; Sonoda, A.; Murahashi, S. I. *J. Am. Chem. Soc.* 1976, 98, 1965. (b) Andrews, G. C. *Tetrahedron Lett.* 1980, 697.

### Table II. Relative Reactivities of Carbonyl Compounds toward Potassium Triphenylborohydride in THF[a]

entry	starting mixture	temp, °C	time, h	ratio of redn products[b]	other reducing reagents
1	2-heptanone/4-heptanone	0	2	90.0:10.0	91.0:9.0[c]
		-78	6	94.0:6.0	
2	acetophenone/4-heptanone	0	1	97.8:2.2	
3	4-heptanone/pinacolone	0	1	91.3:8.7	
		-78	5	98.9:1.1	
4	cyclohexanone/2-heptanone	0	2	81.6:18.4	100:5[d]
		-78	6	88.7:11.3	100:78[c]
5	cyclohexanone/4-heptanone	0	1	99.4:0.6	100:4[d]
6	4-tert-butylcyclohexanone/2-methylcyclohexanone	0	2	87.0:13.0	
		-78	6	95.0:5.0	
7	cyclohexanone/cyclopentanone	0	2	72.3:27.7	100:10[d]
		-78	6	97.0:3.0	
8	hexanal/2-heptanone	0	1	96.0:4.0	95:5[c]
		-78	5	99.1:0.9	100:0[e]
9	hexanal/cyclohexanone	0	1	65.0:35.0	
		-78	5	90.6:9.4	99.6:0.4[e]
10	benzaldehyde/acetophenone	0	1	82.0:18.0	100:0[f]
		-78	5	91.4:8.6	

[a] Reaction mixtures were 0.25 M in substrates at 0 °C, and those at -78 °C were 0.125 M. 1.1 equiv of reagent was utilized for the competitive reduction of equimolar mixture of two carbonyl components. [b] Normalized ratio determined by GLC with appropriate internal standard; the total yields of product alcohols were ≥95%. [c] Li(n-Bu)$_2$-9-BBN; Yamamoto, Y.; Toi, H.; Sonoda, A.; Murahashi, S. I. *J. Am. Chem. Soc.* 1976, 98, 1965. [d] t-BuNH$_2$·BH$_3$: Andrews, G. C. *Tetrahedron Lett.* 1980, 697. [e] Li(Et$_3$CO)$_3$AlH: Krishnamurthy, S. *J. Org. Chem.* 1981, 46, 4628. [f] BH$_3$-LiCl (1:0.1): Yoon, N. M.; Cha, J. S. *J. Korean Chem. Soc.* 1978, 22, 259.

lective reagents,[6] such as 9-BBN–pyridine,[6a] BH$_3$·LiCl system,[6b] lithium tris(3-ethyl-3-pentyl)oxy aluminium hydride,[6c] and borohydride exchange resin (BER).[6d]

Simplified Product Isolation. Hindered trialkylborohydrides such as lithium and potassium tri-sec-butylborohydrides are very valuable reagents, achieving exceptional stereoselectivities. Although these reagents are very useful for such reductions and have been widely applied, the reaction byproducts, tri-sec-butylborane, can cause difficulties with the isolation of the reduction product, the desired alcohol. A common solution is the oxidation of the organoborane at the end of the reduction with alkaline hydrogen peroxide.

The new reducing reagent, KTPBH possesses a practical advantage in facilitating the recovery of the alcohol product. Thus it was found that the procedure[8] developed for K-9-OThx-9-BBNH could also be applied to KTPBH. Controlled addition of water to the reaction mixture converts the triphenylborane moiety to the "ate" complex (eq 5). The THF solvent is removed and pentane is added.

$$[Ph_3BOR]^-K^+ + H_2O \xrightarrow{\text{pentane}} [Ph_3BOH]^-K^+\downarrow + ROH \tag{5}$$
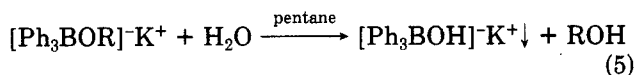
The "ate" complex precipitates. Indeed, we experienced no difficulty in isolating a 95% yield of cis-2-methylcyclohexanol in 98.5% isomeric purity following removal of Ph$_3$B moiety from the reaction of 2-methylcyclohexanone with KTPBH (eq 3).

Conclusion

Potassium triphenylborohydride (KTPBH) is readily prepared by treating the triphenylborane with potassium hydride in THF at 25 °C and is stable over 2 months at room temperature. This reagent shows excellent stereoselectivity and chemoselectivity in the reduction of carbonyl compounds at 0 and -78 °C. In the reduction of cyclic ketones, the stereoselectivity approaches to those achieved with LDMBH$_2$·2DME[3b] and Li-sec-Bu$_3$BH.[5] This reagent also exhibits a remarkable chemoselectivity between ketones that are only slightly different in structure. The chemoselectivity is better than those of lithium di-

n-butyl-9-BBN[7a] and tert-butylamine–borane,[7b] the best two reagents reported for such purpose. Also this reagent possessed a significant advantage, an easy removal of the byproduct from the reaction mixture, facilitating isolation of the reduction product.

Experimental Section

General Methods. All glassware was dried in an oven, assembled hot, and cooled in a stream of dry nitrogen. All reactions were carried out under a dry nitrogen atmosphere. All additions of reagent and solvents were carried out with oven-dried, nitrogen-purged hypodermic syringes fitted with stainless steel needle.

Materials. THF was distilled from benzophenone–sodium ketyl and stored under dry nitrogen. Triphenylborane (8–10% sodium hydroxide adduct) was obtained from Du Pont. It was separated from the sodium hydroxide adduct by using the Du Pont method.[9] Potassium hydride was used as received from Fluka and was separated from the mineral oil by washing three times with dry n-pentane.

Spectra. Infrared spectra were recorded on a Shimatzu IR-440 spectrophotometer by using sealed liquid cells.[10] ^{11}B NMR spectra were recorded with a Varian FT-80A instrument. The chemical shifts reported are in δ (ppm) relative to BF$_3$·OEt$_2$.

GLC Analyses. GLC analyses were carried out with a Varian Model 3700 TCD chromatograph. The alcohol products were analyzed with a 6 ft × 0.125 in. column of 10% Carbowax 20M on 100/200 mesh Chromosorb W and 10% diglycerol on 100/120 mesh Chromosorb W. All GLC yields were determined by using a suitable internal standard and an authentic sample.

Preparation of Potassium Triphenylborohydride (KTPBH) in THF at Room Temperature. An oven-dried, 100-mL, round-bottom flask with a side arm, a condenser, and an adaptor was attached to a mercury bubbler. The flask was flushed with dry nitrogen and maintained under a static pressure of nitrogen. To this was added 1.8 g of potassium hydride (45 mmol, 50% excess) as an oil suspension by using a double-ended needle. The mineral oil was removed with pentane (3 × 10 mL). To this pure potassium hydride was added 37.5 mL of 0.80 M solution of triphenylborane in THF (30 mmol) while the mixture was stirred at room temperature. The reaction was monitored by withdrawing aliquots of the reaction mixture at appropriate time intervals (1.0, 3.0, 6.0, and 12.0 h) and determining its hydride concentration.

(8) Brown, H. C.; Cha, J. S.; Nazer, B. *J. Org. Chem.* 1985, 50, 549.

(9) Isolation of triphenylborane from the adduct solution (ca. 8% Ph$_3$B as Ph$_3$B·NaOH) involves neutralization with concentrated acid (38 wt % HCl) and then drying the isolated triphenylborane in a vacuum oven.
(10) Brown, H. C.; Kramer, G. W.; Levy, A. B.; Midland, M. M. "Organic Syntheses via Boranes"; Wiley-Interscience: New York, 1975.

In another run, after 6 h of reaction, the total reaction mixture was centrifuged, and it was found the total volume of clear solution increased from 37.5 to 39 mL. At the appropriate periods of time, an aliquot of the reaction mixture was removed with a syringe, excess KH was removed by using a centrifuge, and then the sample was injected into a hydrolyzing mixture containing a 1:1 mixture of 4 N H_2SO_4–THF. The hydride concentration was determined from the corrected volume of hydrogen evolved, and was found to be 0.52 M (20.3 mmol, 67.6%, 1 h), 0.68 M (26.5 mmol, 88.4%, 3 h), 0.76 M (29.6 mmol, 98.7%, 6 h), and 0.76 M (29.6 mmol, 98.7%, 12 h). The ^{11}B NMR spectrum of the resulting clear solution showed a clean doublet centered at δ –7.96 (J_{B-H} = 78.7 Hz), and the IR spectrum showed a strong absorption at 2200 cm^{-1} attributed to the B–H stretching vibration in the borohydride anion. This KTPBH solution (0.76 M in hydride) was analyzed for boron and potassium. The boron concentration was estimated to be 0.76 M from the amount of phenol produced by oxidation of an aliquot of the solution with NaOH–H_2O_2. The potassium content was measured as potassium hydroxide, after the solution of reagent was quenched with water. Titration with standard acid indicated the concentration of potassium ion to be 0.76 M. From these results, the relative ratio of potassium, boron, and hydrogen of this reagent was proved to be 1:1:1. Consequently, the identity of the reagent was firmly established. A 0.76 M solution of KTPBH in THF was maintained under dry nitrogen at room temperature and analyzed for the hydride concentration periodically (0, 12, 24 h, etc.) by hydrolysis of aliquots. The hydride concentration of KTPBH remained constant (0.76 M) over 2 months at room temperature.

Stereoselective Reductions. The following procedure for the reduction of 2-methylcyclohexanone is representative. In a 50-mL round-bottom flask was placed 2.9 mL of a 0.76 M solution of KTPBH in THF (2.2 mmol in hydride), followed by 3.1 mL of THF. The flask was kept at 0 °C with the aid of an ice–water bath. To this flask was added 2.0 mL of precooled 2-methylcyclohexanone solution in THF (2.0 mmol in ketone), and the reaction mixture was stirred at 0 °C for 1 h (3 h at –78 °C). This makes the reaction mixture 0.25 M in ketone at 0 °C; however, the concentration of ketone was lowered to 0.125 M for the reaction at –78 °C, because of the decreased solubility of KTPBH and to keep the molar ratio 1.1:1 for KTPBH–ketone. It was then hydrolyzed with 1 mL of water, and then, the triphenylborane formed was oxidized with 1 mL of 2 N NaOH solution and 1 mL of 30% hydrogen peroxide and stirred for 2 h at 30–35 °C in order to complete the oxidation. After oxidation, dodecane was added as an internal standard. The aqueous layer was saturated with anhydrous potassium carbonate. The GLC analysis of the THF layer showed the presence of 99.4% 2-methylcyclohexanols containing 95.4% (98.5% at –78 °C) of the cis isomer. The results are summarized in Table I.

Chemoselective Reductions. The following competitive reduction between 2- and 4-heptanone is representative. In a 50-mL round-bottom flask was placed 2.9 mL of a 0.76 M solution of KTPBH in THF (2.2 mmol in hydride), followed by 3.1 mL of THF. The flask was kept at 0 °C with the aid of an ice–water bath. To this flask was added 2.0 mmol each of 2- and 4-heptanone solution in THF, and the reaction mixture was stirred at 0 °C for 2 h (6 h at –78 °C). It was then hydrolyzed with 1 mL of water, and then, the triphenylborane formed was oxidized with 1 mL of 2 N NaOH and 1 mL of 30% hydrogen peroxide and stirred for 2 h at 30–35 °C in order to complete the oxidation. After oxidation, 1-octanol was added as an internal standard, and the aqueous layer was saturated with anhydrous potassium carbonate. The GLC analysis of the THF layer showed a 97.2% yield of 2-heptanol and a 10.8% yield of 4-heptanol. The normalized ratio, 90:10 (94:6 at –78 °C), is shown in Table II.

The simplified isolation procedure was also applied to the competitive reduction of cyclohexanone and 2-heptanone. In the usual assembly, in a 50-mL round-bottom flask was placed 5.3 mL of a 0.76 M solution of KTPBH in THF (4.0 mmol), followed by 22.7 mL of THF at –78 °C. To this flask was added 4.0 mmol each of cyclohexanone and 2-heptanone solution in THF (1 M, 4 mL). After 6 h of reaction at –78 °C, the reaction mixture was hydrolyzed with 0.2 mL (11 mmol) of water for 0.5 h at room temperature. All THF was then pumped off by using an aspirator. Then, 32 mL of pentane was added to the residue, and the mixture was stirred out. A white solid precipitated out. The GLC analysis of the pentane solution showed a 84.3% yield of cyclohexanol and a 10.7% yield of 2-heptanol. The normalized ratio, 88.7:11.3, was same as that shown in Table II, which was obtained by oxidative workup from the reaction utilizing 1.1 equiv of KTPBH. The results are summarized in Table II.

Product Isolation from a Preparative Run. In a large-scale reaction, the simplified isolation procedure was followed. In a 500-mL round-bottom flask, was placed 44 mL of a 0.76 M solution of KTPBH in THF (33 mmol), followed by 166 mL of THF. The flask was kept at –78 °C with the aid of an acetone–dry ice bath. To this flask was added 30 mL of 2-methylcyclohexanone solution in THF (30 mmol), and the reaction was complete in 3 h. The reaction mixture was then hydrolyzed with 0.9 mL of water for 0.5 h at room temperature. All THF was then pumped off by using an aspirator. Pentane (30 mL) was added to the residue. A white solid precipitated as the mixture was stirred for 1 h, and then after filtration the pentane was pumped off by using an aspirator. Then, we gained 3.2 g (95% yield) of essentially pure 2-methylcyclohexanol. GLC examination revealed the presence of 98.5% cis-2-methylcyclohexanol and 1.5% trans-2-methyl-cyclohexanol.