

1 H), 4.20–4.92 (m, 1 H), 2.38–3.68 (m, overlapping a doublet at 3.38, $J = 6$ Hz, 4 H).

Anal. Calcd for $C_8H_7IO_2$: C, 30.28; H, 2.96. Found: C, 30.49; H, 2.91.

10-Oxa-8-methylenebicyclo[5.3.0]dec-2-en-9-one (24g). To a solution of **23g** (0.394 g, 1.35 mmol) in benzene (2.5 mL) at 25 °C under nitrogen was added DBU (0.27 mL, 1.8 mmol). After being stirred at 62 °C for 9 h, the mixture was filtered to remove white solid, and the filtrate was washed with 0.5 N hydrochloric acid, dried (anhydrous magnesium sulfate), and concentrated by rotary evaporation. Bulb-to-bulb distillation (oven temperature 160 °C, 0.02 torr) of the residue afforded 0.172 g (78%) of **24g** as a clear, colorless liquid: IR (neat) 3060, 1765, 1660 cm^{-1} ; 1H NMR ($CDCl_3$) 6.24 (d, $J = 2.6$ Hz, 1 H), 5.0–5.9 (m, 4 H), 3.18 (m, 1 H), 1.2–2.5 (m, 6 H); mass spectrum, m/e 164.0842 (M^+), 164.0837 calcd for $C_{10}H_{12}O_2$. The following compounds were obtained by similar procedures.

24e: prepared from **23e** (3.54 g, 13.4 mmol); 88% yield (isolated); IR (neat) 3058, 1755, 1658, 1615 cm^{-1} ; 1H NMR ($CDCl_3$) 5.33–6.71 (m, overlapping doublets at 6.29, $J = 2.9$ Hz, and 5.66, $J = 2.5$ Hz, 5 H), 3.58 (m, 1 H), 2.13–3.33 (m, 2 H); mass spectrum, m/e 136.0546 (M^+), 136.0524 calcd for $C_8H_8O_2$.

Anal. Calcd for $C_8H_8O_2$: C, 70.58; H, 5.92. Found: C, 69.64; H, 5.94.

24f: prepared from **23f** (0.476 g, 1.72 mmol); 94% yield (isolated); IR (neat) 3040, 1765, 1650 cm^{-1} ; 1H NMR ($CDCl_3$) 5.67–6.43 (m, overlapping doublets at 6.25, $J = 2.6$ Hz, 5.60 $J = 2.2$ Hz, 4 H), 4.87 (dd, $J = 7.2, 2.2$ Hz, 1 H), 2.93–3.83 (m, 1 H), 1.34–2.37 (m, 4 H).³³

Anal. Calcd for $C_9H_{10}O_2$: C, 71.98; H, 6.71. Found: C, 71.80; H, 6.85.

28: prepared from **27** (0.407 g, 1.71 mmol); 26% yield (isolated by bulb-to-bulb distillation; oven temperature 25 °C, 0.005 torr); IR (neat) 1770, 1652, 1612 cm^{-1} ; 1H NMR ($CDCl_3$) 7.17 (m, 1 H), 5.15 (d, $J = 2.5$ Hz, 1 H), 4.82 (d, $J = 2.5$ Hz, 1 H), 2.07 (s, 3 H); mass spectrum, m/e 110.0379 (M^+), 110.0368 calcd for $C_6H_6O_2$.

8-Oxa-10-methylenebicyclo[5.3.0]decan-9-one (25g). To a solution of **23g** (0.894 g, 3.10 mmol) and benzene (15 mL) at 20 °C under nitrogen was added tri-*n*-butyltin hydride (0.83 mL, 3.14 mmol). After being stirred at 55 °C for 15 h, the solution was concentrated by rotary evaporation, and the residue was chromatographed on alumina (methylene chloride) to give 0.40 g (80%) of **25g** as a clear, colorless oil: IR (neat) 3100, 1760, 1660 cm^{-1} ; 1H NMR ($CDCl_3$) 6.28 (d, $J = 3.5$ Hz, 1 H), 5.53 (d, $J =$

3.5 Hz, 1 H), 4.67 (dt, $J = 9, 2$ Hz, 1 H), 2.9–3.5 (m, 1 H), 0.9–2.3 (m, 10 H); mass spectrum, m/e 166.0976 (M^+), 166.0994 calcd for $C_{10}H_{14}O_2$.³⁴

The following compounds were similarly obtained.

25e: prepared from **23e** (0.498 g, 1.88 mmol); 68% yield (GLC with *n*-pentadecane as the internal standard); purified by chromatography on silica gel (35% ethyl acetate/hexane, R_f 0.35); IR (neat) 1760, 1658 cm^{-1} ; 1H NMR ($CDCl_3$) 6.24 (d, $J = 2.5$ Hz, 1 H), 5.64 (d, $J = 2.2$ Hz, 1 H), 4.99 (m, 1 H), 3.41 (m, 1 H), 1.00–2.25 (m, 6 H).

Anal. Calcd for $C_8H_{10}O_2$: C, 69.55; H, 7.29. Found: C, 69.49; H, 7.31.

25f: prepared from **23f** (0.347 g, 1.25 mmol); 60% yield (isolated by chromatography on silica gel, 1:1 hexane-ether); IR (neat) 1767, 1655 cm^{-1} ; 1H NMR ($CDCl_3$) 6.18 (d, $J = 2.5$ Hz, 1 H), 5.53 (d, $J = 2.5$ Hz, 1 H), 4.53 (q, $J = 5.5$ Hz, 1 H), 3.02 (m, 1 H), 0.7–2.3 (m, 8 H).^{20d,35}

29: prepared from **27** (0.778 g, 3.27 mmol); 18% yield (GLC); purified by preparative GLC; IR (neat) 1770, 1670 cm^{-1} ; 1H NMR ($CDCl_3$) 6.30 (t, $J = 2$ Hz, 1 H), 5.73 (t, $J = 2$ Hz, 1 H), 4.73 (sextet, $J = 7$ Hz, 1 H), 2.17–3.40 (m, 2 H), 1.47 (d, $J = 7$ Hz, 3 H); mass spectrum, m/e 112.0524 (M^+), 112.0524 calcd for $C_6H_8O_2$.

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Registry No. 17, 3853-06-3; 18, 78804-62-3; 19a, 10205-34-2; 19b, 69637-59-8; 19c, 69637-60-1; 19d, 69637-61-2; 19e, 78804-63-4; 19f, 69637-62-3; 19g, 78804-64-5; 20a, 33016-24-9; 20c, 69637-63-4; 20d, 78804-65-6; 20e, 78804-66-7; 20f, 69637-65-6; 20g, 78822-60-3; 21a, 80-62-6; 21c, 3070-68-6; 21d, 51122-89-5; 21e, 78804-67-8; 21f, 69637-66-7; 21g, 78804-68-9; 22e, 78804-69-0; 22f, 54109-55-6; 22g, 78804-70-3; 23e, 78804-71-4; 23f, 54109-56-7; 23g, 78804-72-5; 24e, 78804-73-6; 24f, 60916-75-8; 24g, 78804-74-7; 25e, 61747-55-5; 25f, 16822-06-3; 25g, 3725-04-0; 26, 4743-96-8; 27, 78804-75-8; 28, 61892-54-4; 29, 62873-16-9; 1-iodobutane, 542-69-8; 1-bromobutane, 109-65-9; iodomethane, 74-88-4; iodoethane, 75-03-6; allyl bromide, 106-95-6; 3-bromocyclopentene, 36291-48-2; 3-bromocyclohexene, 1521-51-3; 3-bromocycloheptene, 36291-49-3.

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Addition Compounds of Alkali-Metal Hydrides. 21. Rapid Reaction of Dialkyl- and Monoalkylboranes with Lithium Aluminum Hydride in the Presence of Triethylenediamine. A Facile and Quantitative Synthesis of Lithium Dialkyl- and Monoalkylborohydrides¹

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Dialkylboranes react rapidly with lithium aluminum hydride in diethyl ether in the presence of triethylenediamine (TED) at 0 °C to form the corresponding lithium dialkylborohydrides and aluminum hydride: $R_2BH + LiAlH_4 \rightarrow LiR_2BH_2 + AlH_3$. The aluminum hydride precipitates as its triethylenediamine adduct. In a similar manner, monoalkylborane-triethylenediamine adducts ($TED \cdot BH_2R$) react with lithium aluminum hydride at 65 °C in tetrahydrofuran (THF) to give the corresponding lithium monoalkylborohydrides with concomitant precipitation of the triethylenediamine-aluminum hydride adduct ($TED \cdot AlH_3$). The reaction is quantitative and is applicable to a wide variety of di- and monoalkylboranes. Consequently, the present procedure provides a general, convenient synthesis of lithium di- and monoalkylborohydrides of greatly varying steric requirements.

It has been well established that the trialkylborohydrides are exceptionally powerful selective reducing agents³ and

very versatile synthetic intermediates.⁴ From our preliminary exploration of different alkyl-substituted boro-

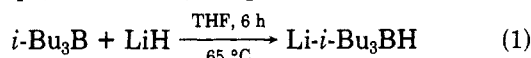
Table I. Reaction of TED·BH₂Thx with Various Olefins in THF at 65 °C^a

olefin	time, h	TME ^b displaced, mmol	ROH ^{c,d} found, mmol	TED·BH ₂ R ^{d,e} isolated, %	mp, ^f °C
cyclopentene	2	9.7	9.9	85	74-76
cyclohexene	2	9.7	9.9	92	109-111
norbornene	2	9.6	9.7	94	140-141
2-methyl-2-butene	2	9.6	9.8	80	70-72

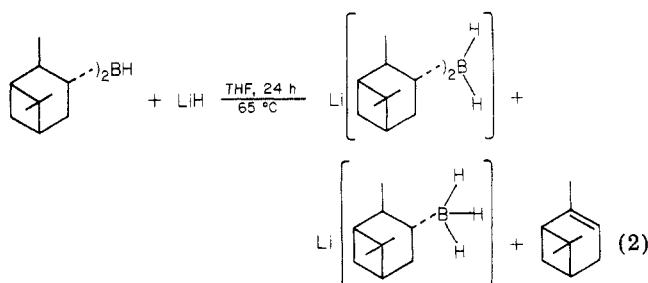
^a 10-mmol scale. ^b Analysis after methanolysis via GLC on SE-30 and CW-20M columns.¹² ^c Analysis via GLC on CW-20M column after alkaline hydrogen peroxide oxidation of the reaction mixture.¹² ^d R corresponds to the olefin used. ^e The crude adduct is isolated by pumping off the volatiles from the reaction mixture at 12 mmHg. ^f Melting points are uncorrected and were obtained in sealed capillary tubes.

hydrides, it was clearly evident that the reactivity of these derivatives and the stereochemical course of the reactions involving them were strongly influenced by the nature of the alkyl substituent on boron. In recent years, several important routes to hindered and highly hindered trialkylborohydrides have been developed.⁵⁻⁷ Surprisingly, little effort has been devoted to the synthesis of borohydrides with less than three alkyl groups on boron.¹ Hence, it appeared highly desirable to explore the possibility of synthesizing lithium di- and monoalkylborohydrides through the routes well established for the preparation of trialkylborohydrides.

The most direct way to prepare lithium trialkylborohydrides is the reaction of trialkylboranes with lithium hydride (eq 1).⁵ However, this procedure is not favorable



for the preparation of certain lithium di- and monoalkylborohydrides. Unlike the trialkylboranes, the di- and monoalkylboranes tend to be both thermally unstable and sensitive to redistribution.⁸ Consequently, they often undergo this reaction to yield a mixture of products (eq 2).¹ It was clear that a satisfactory synthesis of the di-



and monoalkylborohydrides required a fast transfer of hydride from the reagent to boron, a transfer sufficiently fast as to minimize the relatively rapid thermal decomposition and redistribution reactions of the di- and monoalkylboranes.

(1) Part 20: Brown, H. C.; Singaram, B.; Mathew, C. P. *J. Org. Chem.* 1981, 46, in press.

(2) (a) Postdoctoral research associate on Grant GM 10937 from the National Institutes of Health. (b) Postdoctoral research associate on Grant DAAG 29-79-C-0027 from the U.S. Army Research Office.

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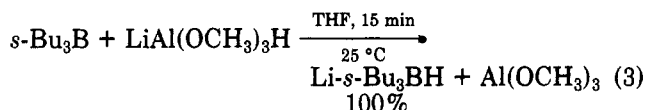
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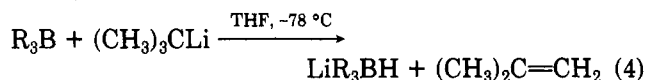
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A general synthesis of lithium trialkylborohydride has been developed based on the transfer of hydride from lithium trimethoxyaluminum hydride (LTMA) (eq 3).⁶



The analogous reaction involving di- and monoalkylboranes, while promising, suffers from the difficulty of separating the product from the aluminum methoxide formed concurrently.

Perhaps the most elegant synthesis of lithium trialkylborohydrides is the reaction of *tert*-butyllithium with trialkylboranes (eq 4).⁷ Unfortunately, dialkylboranes

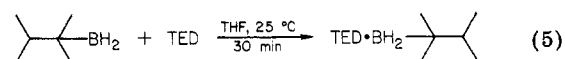


react with *tert*-butyllithium to yield a mixture of products.⁹

Recently we reported a successful synthesis of lithium trialkylborohydrides.¹⁰ Trialkylborane reacts with lithium aluminum hydride in the presence of triethylenediamine (TED) at 0 °C to form the corresponding lithium trialkylborohydride and aluminum hydride. The 1:1 adduct, TED·AlH₃, readily precipitates from solution, providing the desired lithium trialkylborohydride free of other products. This reaction is very general and is applicable to a wide variety of trialkylboranes. This prompted us to explore the possibility that the reaction of lithium aluminum hydride with representative di- and monoalkylboranes might provide a suitable route to the desired lithium di- and monoalkylborohydrides in a state of satisfactory purity.

Results and Discussion

The following dialkylboranes of considerably different steric requirements were selected for this study: 9-borabicyclo[3.3.1]nonane (9-BBN), dicyclohexylborane (Ch₂BH), disiamylborane (Sia₂BH), and diisopinocampheylborane (IPC₂BH). All of the monoalkylboranes selected for the present work were prepared and used as their triethylenediamine adducts (TED·BH₂R). The thexylborane-triethylenediamine adduct (TED·BH₂Thx) was prepared by combining thexylborane and the appropriate quantity of TED (eq 5).



Thexylborane-triethylenediamine reacts with olefin with the facile displacement of tetramethylethylene (TME) and the formation of the corresponding monoalkylborane-

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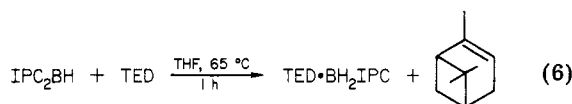
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Table II. Infrared and ^{11}B NMR Spectral Data of the Products from Reactions of Lithium Aluminum Hydride with Representative Dialkylboranes in the Presence of Triethylenediamine

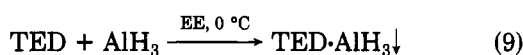
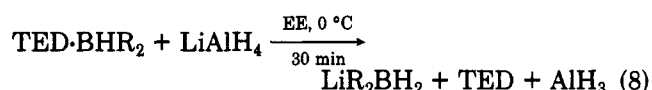
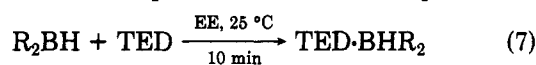
dialkylborane	$\nu_{\text{B-H}}$, cm^{-1}	chemical shift, ^a δ (multiplicity)	$J_{\text{B-H}}$, Hz
9-borabicyclo-[3.3.1]nonane (9-BBN)	2148	-14.15 (t)	69
dicyclohexylborane (Chx_2BH)	2108	-9.28 (t)	67
disiamylborane (Sia_2BH)	2120	-12.21 (t)	67
diisopinocampheylborane (IPC_2BH)	2100	-5.69 (t)	68

^a All chemical shifts are relative to $\text{Et}_2\text{O}\cdot\text{BF}_3$ with those upfield assigned as negative.

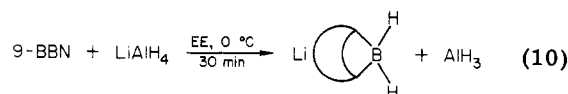
triethylenediamine adducts in nearly quantitative yield (Table I). The reaction of α -pinene with $\text{TED}\cdot\text{BH}_2\text{Thx}$ is very sluggish. Hence, monoisopinocampheylborane-triethylenediamine ($\text{TED}\cdot\text{BH}_2\text{IPC}$) was prepared by displacement of α -pinene from diisopinocampheylborane (eq 6).



For preparation of the lithium dialkylborohydrides, the reactions were generally run by adding a solution of TED in diethyl ether (EE) to the dialkylborane in EE with constant stirring. The resulting clear solution was then treated at 0°C with a solution of lithium aluminum hydride in diethyl ether (EE). A voluminous white precipitate of $\text{TED}\cdot\text{AlH}_3$ formed rapidly. Initially, the precipitate was present as a voluminous gel. However, on standing for approximately 12 h, it became granular, readily centrifuged from the reaction mixture. By washing the precipitate with EE, >95% of the lithium dialkylborohydride was generally recovered. The resulting solutions contained the products in essentially pure form (Table II). Apparently the reaction proceeds as shown in eq 7-9.



In the case of 9-BBN, the formation of the TED complex took more than 1 h for completion. (The reaction proceeds through a slow rate-determining dissociation of 9-BBN dimer.) If the lithium aluminum hydride was added before the complete formation of $\text{TED}\cdot\text{9-BBN}$, a mixture of products was obtained. Alternatively, 9-BBN can be reacted directly with an EE solution of LiAlH_4 and the resulting solution treated with TED in EE to remove AlH_3 (eq 10). This reaction establishes that, unlike many



trialkylborohydrides examined,¹⁰ this LiR_2BH_2 does not undergo exchange reactions with free AlH_3 .

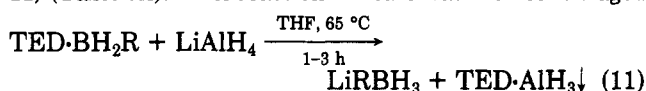
The reaction between $\text{TED}\cdot\text{BH}_2\text{R}$ and LiAlH_4 was relatively slow at 25°C . Consequently, the reaction of LiAlH_4

Table III. Reaction of $\text{TED}\cdot\text{BH}_2\text{R}$ with Lithium Aluminum Hydride in THF at 65°C

monoalkylboranes ^a	time, h	^{11}B NMR characteristics of the product	
		chemical shift, ^b δ (multiplicity)	$J_{\text{B-H}}$, Hz
monocyclopentylborane	3	-26.6 (q)	74
monocyclohexylborane	2	-25.4 (q)	74
mononorbonylborane	2	-25.7 (q)	75
monosiamylborane	1	-27.5 (q)	75
monoisopinocampheylborane	1	-23.5 (q)	76
thexylborane	1	-24.4 (q)	77

^a Used as their triethylenediamine adducts. ^b All chemical shifts relative to $\text{Et}_2\text{O}\cdot\text{BF}_3$ with those upfield assigned as negative.

with $\text{TED}\cdot\text{BH}_2\text{R}$ was examined in refluxing THF. Under those conditions, the reaction was complete in 1-3 h (eq 11) (Table III). The reaction mixture was then centrifuged



to remove the precipitated $\text{TED}\cdot\text{AlH}_3$.

Infrared Spectra. Alkali-metal alkylborohydride exhibits a very broad and strong absorption in the infrared region due to the B-H stretch in the borohydride anion. Solution of all lithium di- and monoalkylborohydrides, prepared by the present procedure, exhibit this characteristic absorption around 2100 and 2200 cm^{-1} , respectively.¹ There were no absorptions attributable to the Al-H frequency in the infrared spectrum.¹⁰

^{11}B NMR Spectra. All of the lithium dialkylborohydrides exhibited triplets in the ^{11}B NMR spectrum. The lithium monoalkylborohydrides displayed sharp quartets in the ^{11}B NMR spectrum. The results are summarized in Tables II and III.

Conclusion

Lithium aluminum hydride reacts rapidly and quantitatively in the presence of triethylenediamine with all of the dialkylboranes examined to give the corresponding lithium dialkylborohydrides and aluminum hydride. The aluminum hydride precipitates as $\text{TED}\cdot\text{AlH}_3$. Lithium aluminum hydride also reacts with $\text{TED}\cdot\text{BH}_2\text{R}$ in refluxing THF to give the corresponding lithium monoalkylborohydrides with concurrent precipitation of $\text{TED}\cdot\text{AlH}_3$. The present procedure is the only route currently available for the preparation of pure lithium di- and monoalkylborohydrides. The present study also describes a general method for the preparation of the triethylenediamine-monoalkylborane adducts, $\text{TED}\cdot\text{BH}_2\text{R}$, a valuable means for stabilizing the monoalkylborane intermediates.

Experimental Section

The reaction flasks and other glass equipment used for experiments were oven-dried and assembled in a stream of dry nitrogen gas. The special techniques for the manipulation of air-sensitive materials are described elsewhere.^{11,12}

Tetrahydrofuran and diethyl ether were stored under nitrogen in a Teflon stopcock protected ampule. Tetrahydrofuran and diethyl ether were distilled from excess lithium aluminum hydride.

(11) Unpublished research of Brown, H. C.; Wang, K. K.

(12) Brown, H. C.; Kramer, G. W.; Levy, A. B.; Midland, M. M. "Organic Syntheses via Boranes"; Wiley-Interscience: New York, 1975; Chapter 9.

The standard solution of lithium aluminum hydride was prepared, filtered, and stored as described earlier.¹³ 9-BBN (Aldrich) was used after recrystallization from monoglyme. All other dialkylboranes were prepared by the hydroboration of corresponding olefins with borane-methyl sulfide (BMS).¹⁴

The spectra were obtained under an inert atmosphere. The infrared spectra were obtained with a Perkin-Elmer 700 spectrometer, using sealed cells and a two-syringe technique. The ¹¹B NMR and ²⁷Al NMR spectra were recorded on a Varian FT-80A instrument. The ¹¹B chemical shifts are in δ relative to EE-BF₃ with chemical shifts downfield from EE-BF₃ assigned as positive.

Reaction of Dialkylboranes with Lithium Aluminum Hydride in the Presence of Triethylenediamine. The following procedure for the preparation of lithium dicyclohexylborohydride is representative. A slurry in EE of dicyclohexylborane was prepared in a 50-mL centrifuge vial by adding 2.0 mL of cyclohexene (20 mmol) to a solution of 1.0 mL of 10.0 M BMS (10 mmol) in EE (7.0 mL) at 0 °C.¹³ The reaction mixture was maintained for 3 h.¹³ To the product was added 10 mL of a 1.0 M solution of TED in EE. The solid dissolved and a clear solution formed. The clear solution was maintained at 0 °C and 10 mL of a 1.0 M solution of LiAlH₄ in EE was added slowly with vigorous stirring. A voluminous precipitate of TED·AlH₃ separated out. The reaction mixture was stirred as efficiently as possible with an ovoid Teflon-coated magnetic stirring bar. The reaction mixture was then set aside for 12 h at 25 °C and centrifuged to remove the granulated TED·AlH₃. A 1.0-mL aliquot of the clear solution containing the product, LiChx₂BH₂, gave on hydrolysis 0.66 mmol (100%) of hydrogen. The supernatant liquid, ~27 mL (~90%), was withdrawn for further examination. ¹¹B NMR δ -9.28 (t, J = 67 Hz); IR ν 2108 cm⁻¹ (B-H). No signals attributable to the presence of AlH₃ in the solution could be detected either in the ²⁷Al NMR or in the IR.

Reaction of 9-Borabicyclo[3.3.1]nonane with Lithium Aluminum Hydride. An oven-dried, 50-mL centrifuge vial was charged with 20 mL of a 0.5 M solution of 9-BBN in EE. To this solution was added with stirring at 25 °C for 30 min 10 mL of a 1.0 M solution of LiAlH₄ in EE. ¹¹B NMR δ -13.3 (t, J = 69 Hz); ²⁷Al NMR δ +119 (br s); IR ν 2130 (B-H), 1760 cm⁻¹ Al-H.

To the reaction mixture was added 10 mL of a 1.0 M solution of TED in EE. The TED·AlH₃ precipitated immediately from solution and was removed by centrifugation. The supernatant solution contained the product, Li-9-BBNH, in pure form. ¹¹B NMR δ -14.15 (t, J = 69 Hz); IR ν 2148 cm⁻¹ (B-H).

Preparation of Thexylborane-Triethylenediamine. Thexylborane (10.0 mmol) was prepared according to the literature procedure.¹⁴ To this product at 25 °C was added 10 mL of a 1.0 M solution of TED in THF. Following completion of the vigorous reaction (15 min), THF was removed under reduced pressure (12

mmHg). The solid adduct was washed with cold *n*-pentane and dried. There was obtained 1.98 g (94%) of the 1:1 adduct: mp 97-99 °C; ¹H NMR (CDCl₃) δ 0.80 (s, 6 H), 0.90 (d, J = 6 Hz, 6 H), 1.40 (septet, J = 6 Hz, 1 H), 2.93 (s, 12 H); ¹¹B NMR δ +0.78 (br t).

Reaction of Thexylborane-Triethylenediamine with Various Olefins in THF at 65 °C. The following procedure is representative. A solution of TED·BH₂Thx (10 mmol) in THF was prepared as described above. The THF solvent was refluxed (65 °C) and 10 mL of a 1.0 M solution of norbornene in THF was added. The reaction mixture was stirred at 65 °C for 2 h. It was then cooled to 25 °C and the THF was removed under reduced pressure (12 mmHg). The heavy white solid was washed with cold *n*-pentane and dried. There was obtained 2.1 g (94% yield) of a 1:1 adduct: mp 140-142 °C; ¹H NMR (CDCl₃) δ 0.50-2.05 (m, 11 H), 2.92 (s, 12 H); ¹¹B NMR δ -0.1 (br t).

Preparation of Monoisopinocampheylborane-Triethylenediamine Adduct (TED·BH₂IPC). A slurry of IPC₂BH (10 mmol) in THF was prepared in the usual manner.¹⁴ To this slurry was added 10 mL of a 1.0 M solution of TED in THF. The THF solvent was heated under reflux (65 °C) for 1 h and then cooled to 25 °C. The solvent was then removed under reduced pressure (12 mmHg). The solid was next washed with cold *n*-pentane and dried. There was obtained 2.2 g of TED·BH₂IPC: mp 126-127 °C; ¹H NMR (CDCl₃) δ 0.98 (d, 3 H, J = 7 Hz), 1.10 (s, 3 H), 1.16 (s, 3 H), 1.67-2.3 (m, 7 H), 2.97 (s, 12 H); ¹¹B NMR δ +1.46 (br s).

Reaction of the Monoalkylborane-Triethylenediamine Adducts with Lithium Aluminum Hydride in THF at 65 °C. The following procedure for the preparation of lithium monocyclohexylborohydride, LiChx₂BH₂, is typical. An oven-dried, 50-mL flask, equipped with a reflux condenser, was charged with 10 mL of a 1.0 M solution of monocyclohexylborane-triethylenediamine in THF. The THF solvent was brought to reflux (65 °C) and 10 mL of a 1.0 M solution of LiAlH₄ in THF was added dropwise. The reaction mixture was stirred at 65 °C for 2 h. It was then cooled to 25 °C and transferred to a centrifuge vial. The TED·AlH₃ was removed by centrifugation. ¹¹B NMR δ -25.4 (q, J = 74 Hz); IR ν 2230 cm⁻¹ (B-H).

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Registry No. TED·BH₂Thx, 78919-47-8; TED·BH₂ cyclopentyl, 78891-24-4; TED·BH₂Chx, 78891-25-5; TED·BH₂ norbornyl, 78891-26-6; TED·BH₂Sia, 78891-27-7; 9-BBN, 280-64-8; Chx₂BH, 1568-65-6; Sia₂BH, 1069-54-1; IPC₂BH, 57456-97-0; Li-9-BBNH, 76448-08-3; LiChx₂BH₂, 67813-27-8; LiSia₂BH₂, 67813-43-8; LiIPC₂BH₂, 76430-47-2; TED·BH₂IPC, 78891-28-8; LiBH₃ cyclopentyl, 78891-64-2; LiChx₂BH₃, 76430-52-9; LiBH₃ norbornyl, 78891-65-3; LiSia₂BH₃, 76430-53-0; LiIPC₂BH₃, 78891-66-4; LiThx₂BH₃, 76430-48-3; LiAlH₄, 16853-85-3; cyclopentene, 142-29-0; cyclohexene, 110-83-8; norbornene, 498-66-8; 2-methyl-2-butene, 513-35-9.

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