Reaction of Lithium (2,3-Dimethyl-2-butyl)-*t*-butoxyborohydride with Selected Organic Compounds Containing Representative Functional Groups

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The general reducing characteristics of a newly synthesized reducing agent, lithium (2,3-dimethyl-2-butyl)-*t*-butoxyborohydride (Li Thx^{t}BuOBH$_2$, **1**), in tetrahydrofuran (THF) toward selected organic compounds containing representative functional groups under practical conditions has been examined. The reagent revealed an interesting and unique reducing characteristics. Especially, the stereoselectivity in the reduction of cyclic ketones was extraordinary. Thus, the introduction of bulky alkyl and alkoxy groups into the parent borohydride affords a high stereoselectivity. In general, the reducing power of the reagent is somewhere between the dialkylborohydride and the parent borohydride. This permits the reagent to be a reagent of choice for selective reduction of organic compounds with an improved selectivity.

Keywords : Li Thx^{t}BuOBH$_2$, Reduction, Organic compounds.

Introduction

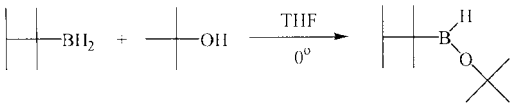
In contrast to the reducing characteristics of lithium borohydride[1], its derivative lithium trialkylborohydride[2-5] and dialkylborohydride[6] are remarkably powerful and selective reducing agents. The introduction of alkyl groups accelerates the hydride donor activity tremendously. However, situation changes when alkoxy groups are introduced to lithium borohydride. For example, the reducing power of lithium triisopropoxyborohydride[7] is much milder than that of lithium borohydride[1]. Therefore, it appeared of interest to explore a mixed derivative, monoalkylmonoalkoxyborohydride, for its reducing characteristics.

Because of easy preparation of thexyl^{t}butoxyborane (Thx-^{t}BuOBH) and its simple conversion into lithium thexyl-^{t}butoxyborohydride (Li Thx^{t}BuOBH$_2$, **1**), this derivative attracted our attention. Being a monoalkylmonoalkoxyborohydride, this reducing agent was anticipated to have different characteristics from those of dialkylborohydride such as lithium 9-boratabicyclo[3.3.1]nonane (Li 9-BBNH).[6]

We undertook a detailed study of the stoichiometry and products of the reaction of Li Thx^{t}BuOBH$_2$ with selected organic compounds containing representative functional groups under the practical conditions.

Results and Discussion

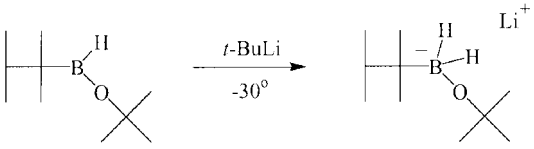
Preparation of Lithium Thexyl^{t}butoxyborohydride (Li Thx^{t}BuOBH$_2$, 1) Solutions. Thexylborane (2,3-dimethyl-2-butylborane) has been prepared by the hydroboration of 2,3-dimethyl-2-butene with borane-THF in the ratio of 1 : 1.[8] The reaction of thexylborane with excess *tert*-butyl alcohol at 0 and 25 °C was examined and the results are summarized in Table 1. As shown in the table, although thexylborane possesses two B-H bonds, it reacts only 1 equiv of *tert*-butyl alcohol even in the presence of excess amount at 0 or 25 °C (Eq. 1).



(1)

The ^{11}B NMR spectrum of a solution of thexyl^{t}butoxyborane in THF showed a doublet (J_{B-H} = 390 Hz) centered at δ 48.3 ppm relative to $BF_3 \cdot OEt_2$.

A solution of lithium thexyl^{t}butoxyborohydride (Li Thx^{t}Bu-



(2)

1

OBH$_2$, **1**) was prepared by adding a solution of *tert*-butyllithium[9] in pentane to a solution of thexyl^{t}butoxyborane in THF at -30 °C (Eq. 2). The ^{11}B NMR of a solution of **1** in

Table 1. Reaction of Thexylborane with Excess *tert*-Butyl Alcohol in Tetrahydrofuran[a]

Temperature (°C)	Time (min)	Hydrogen evolution[b]
0	1	0.56
	5	0.89
	15	0.91
	30	1.00
	6	1.01
25	1	0.95
	5	1.00
	15	1.00
	30	1.00

[a]Two equiv of alcohol reacted. [b]Mmol of per mmol of thexylborane.

Table 2. Reaction of Lithium Thexyl^{t}butoxyborohydride with Representative Active Hydrogen Compounds at 0 °C[a,b]

Compound	Time (h)	Hydrogen evolved[c]	Hydride used[c]	Hydride used for reduction[c]
1-hexanol	0.25	0.17	0.17	0.00
	6	0.36	0.36	0.00
	24	0.40	0.40	0.00
benzyl alcohol	0.25	0.36	0.36	0.00
	6	0.38	0.38	0.00
	24	0.38	0.38	0.00
3-hexanol	0.25	0.12	0.12	0.00
	6	0.38	0.38	0.00
	24	0.39	0.39	0.00
3-ethyl-3-pentanol	0.25	0.06	0.06	0.00
	6	0.29	0.29	0.00
	24	0.30	0.30	0.00
phenol	0.25	0.98	0.98	0.00
	1	1.01	1.01	0.00
	6	1.01	1.01	0.00
n-hexylamine[d]	0.25	0.02	0.02	0.00
	6	0.17	0.17	0.00
	24	0.18	0.18	0.00
1-hexanethiol	0.25	0.62	0.62	0.00
	1	0.73	0.73	0.00
	24	0.76	0.76	0.00
benzenethiol	0.25	0.99	0.99	0.00
	1	1.00	1.00	0.00
	6	1.00	1.00	0.00

[a]In a mixed solvent of THF and pentane. [b]Reagent to compound is 1 : 1. [c]Mmol of hydride per mmol of compound. [d]Two equiv of reagent utilized.

a mixed solvent of THF and pentane showed a triplet ($J_{B\text{-}H}$ = 219 Hz) centered at δ -7.6 ppm[10] relative to $BF_3{\cdot}OEt_2$.

Alcohols, Phenols, Amines, and Thiols (Active Hydrogen Compounds). Among the alcohols examined, only phenol liberated hydrogen relatively fast and quantitatively. All the primary, secondary and tertiary alcohols examined liberated hydrogen very showly and finally the hydrogen evolution was incomplete within 24 hours under the experimental conditions. The primary amine, *n*-hexylamine, also liberated hydrogen very slowly. The thiols examined appeared to be relatively reactive toward the reagent. Thus, 1-hexanethiol evolved about 70% hydrogen in 1 h and benzenethiol liberated a quantitative hydrogen within 1 h. The results are summarized in Table 2.

Table 3. Reaction of Lithium Thexyl^{t}butoxyborohydride with Representative Aldehydes and Ketones at 0 °C[a]

Compound	Time (h)	Ratio of rgt/cmpd	Product	Yield (%)[b]
caproaldehyde	0.5	0.5	1-hexanol	99
benzaldehyde	0.5	0.5	benzyl alcohol	99.5
	1.0	0.5	benzyl alcohol	99.0
2-heptanone	0.5	0.5	2-heptanol	98.5
norcamphor	0.5	0.5	norborneol	99
acetophenone	3	0.5	1-phenylethanol	81
	0.5	1.0	1-phenylethanol	100
benzophenone	3	0.5	benzhydrol	38
	0.5	1.0	benzhydrol	78
	0.5	2.0	benzhydrol	100
cinnamaldehyde	0.5	0.5	cinnamyl alcohol	99.5

[a]See the corresponding footnote in Table 2. [b]Analyzed by GC with a suitable internal standard.

Aldehydes and Ketones. Simple aldehydes and ketones listed in Table 3 were cleanly reduced to the corresponding alcohols with the stoichiometric amount (a half equivalent) of **1**. However, the reaction of aromatic ketones, such as acetophenone and benzophenone, with the stoichiometric

Table 4. Stereoselectivity in the Reduction of Cyclic Ketones with Lithium Thexyl^{t}butoxyborohydride[a,b]

Ketone	Temperature (°C)	Time (h)	Conversion yield (%)[c]	Less stable isomer	Selectivity (%)
2-methylcyclopentanone	0	0.5	100	*cis*	>99.5
	-20	0.5	100		>99.5
2-methylcyclohexanone	0	0.5	100	*cis*	>99.5
	-20	0.5	100		>99.9
2-*t*-butylcyclohexanone	0	6	100	*cis*	>99.5
	-20	12	100		>99.9
3-methylcyclohexanone	0	0.5	100	*trans*	96
	-20	0.5	100		98
4-methylcyclohexanone	0	0.5	100	*cis*	92
	-20	0.5	100		94
4-*t*-butylcyclohexanone	0	0.5	100	*cis*	95
	-20	0.5	100		97
3,3,5-trimethylcyclohexanone	0	0.5	100	*trans*	>99.5
	-20	0.5	100		>99.9
norcamphor	0	0.5	100	*endo*	98
	-20	0.5	100		99.5
camphor	0	48	56	*exo*	>99.5
	-20	72	100		>99.5

[a]See the corresponding footnote in Table 2. [b]A 2 : 1 ratio for reagent to compound was utilized. [c]Analyzed by GC.

Table 5. Comparison of Stereoselectivity in the Reduction of Cyclic Ketones with Representative Reagents at 0 °C

Ketone	Selectivity (%)[a]				
	Li Thx^{t}BuOBH$_2$	K 9-OThx-9-BBNH[b]	Li^{s}Bu$_3$BH[c]	Li Sia$_3$BH[b]	K 9-^{t}Bu-9-BBNH[d]
2-methylcyclohexanone	>99.5	98.5	99.3	99.4	99.5
3-methylcyclohexanone	96	90	85	98	96
4-methylcyclohexanone	92	85.5	80.5	93	94
4-*t*-butylcyclohexanone	95	87	87.5	96.5	98.5
3,3,5-trimethylcyclohexanone	>99.5	>99.9	99.8	99	99
norcamphor	98	95	99.6	99	95.5
camphor	>99.5	97.5	99.6	>99.9	99.9

[a]Analyzed by GC. The figures are ratio of the thermodynamically less stable epimers.[b]Data taken from ref. 11. [c]Data taken from ref. 13. [d]Data taken from ref. 12.

amount was relatively slow, requiring a higher concentration of the reagent. In practice, one or two equivalents of reagent was needed to reduce such ketones completely to the corresponding alcohols. In the case of cinnamaldehyde, the reagent in a stoichiometric amount reduced it cleanly in a fashion of 1,2-reduction to provide cinnamyl alcohol in a yield of 99.5%.

Stereoselectivity Study. The stereoselectivity of the reagent **1** in the reduction of cyclic ketones is summarized in Table 4 and the compared data achieved by other representative reagents are listed in Table 5.

The reagent revealed an excellent stereoselectivity in the reduction of representative cyclic ketones both at 0 or -20 °C. Its stereoselectivity is comparable to the results previously achieved with lithium trisiamylborohydride (Li Sia$_3$BH)[11] and potassium 9-^{t}butyl-9-boratabicyclo[3.3.1]nonane (K 9-^{t}Bu-9-BBNH),[12] even higher than the selectivity shown by lithium tri^{s}butylborohydride (Li ^{s}Bu$_3$BH)[13] and potassium 9-(2,3-dimethyl-2-butoxy)-9-boratabicyclo[3.3.1]nonane (K 9-OThx-9-BBNH)[11] at 0 °C.

Quinones. Two examples for quinones were examined with 2 equiv of the reagent (H^-/compd = 4) at 0 °C. The reaction of *p*-benzoquinone produced hydroquinone and 1,4-dihydroxycyclohexadiene in a 50 : 50 distribution ratio, along with 0.5 equiv of hydrogen evolution, whereas anthraquinone was reduced completely to give a quantitative yield of 9,10-dihydro-9,10-anthracenediol. The experimental data are summarized in Table 6.

Carboxylic Acids and Derivatives. Carboxylic acids and their derivatives were examined with an excess amount of the reagent at 0 °C. The acids reacted with the reagent to evolve a quantitative amount of hydrogen immediately but the further reduction was not followed at all, whereas the primary and tertiary amides were inert to this reagent without any evolution of hydrogen. However, the acid chlorides, esters and nitriles were reduced readily and cleanly to the corresponding alcohols. The results are summarized in Table 7.

Table 6. Reaction of Lithium Thexyl^{t}butoxyborohydride with Representative Quinones at 0 °C[a,b]

Compound	Time (h)	Product	Yield (%)[c]
p-benzoquinone[d]	12	hydroquinone	48
		1,4-dihydroxycyclohexadiene	51
Anthraquinone[e]	12	9,10-dihydroxyanthracene	0
		9,10-dihydro-9,10-dihydroxyanthracenediol	99.9

[a]See the corresponding footnote in Table 2. [b]Two equiv of reagent utilized. [c]Analyzed by GC. [d]Along with 0.5 equiv of hydrogen evolution. [e]No hydrogen evolved.

Table 7. Reaction of Lithium Thexyl^{t}butoxyborohydride with Representative Carboxylic Acids and Their Derivatives at 0 °C[a]

Compound	Time (h)	Ratio of rgt/cmpd	Product	Yield (%)[b]
caproic acid[c]	60	2.0	none	
	24[d]	2.0	none	
benzoic acid[c]	60	2.0	none	
	24[d]	2.0	none	
caproyl chloride	3	1.5	1-hexanol	98
benzoyl chloride	3	1.5	benzyl alcohol	99
ethyl caproate	3	2.0	1-hexanol	98
ethyl benzoate	3	2.0	benzyl alcohol	98
capronitrile	3	2.0	1-hexanol	98
benzonitrile	3	2.0	benzyl alcohol	97
caproamide[e]	60	2.0	none	
	24[d]	2.0	none	
benzamide[e]	60	2.0	none	
	24[d]	2.0	none	
N,*N*-dimethylcaproamide	60	2.0	none	
	24[d]	2.0	none	
N,*N*-dimethylbenzamide	60	2.0	none	
	24[d]	2.0	none	

[a]See the corresponding footnote in Table 2. [b]Analyzed by GC. [c]Immediately 1 equiv of hydrogen evolved. [d]At 25 °C. [e]No hydrogen evolved.

Sulfur Compounds. Among the sulfur compounds listed in Table 8, only the aliphatic disulfide, di-*n*-butyl disulfide, underwent quantitative reduction to *n*-butanethiol, along with 1 equiv of hydrogen evolution. Other sulfur compounds, such as aromatic disulfides, sulfoxides and sulfones, were inert to this reagent. Although methanesulfonic acid evolved 1 equiv of hydrogen immediately, no reduction was followed.

Epoxides. The results of reducing three epoxides are

Table 8. Reaction of Lithium Thexyl^{t}butoxyborohydride with Representative Sulfur Compounds at 0 °C[a]

Compound	Time (h)	Ratio of rgt/cmpd	Product	Yield (%)[b]
di-*n*-butyl disulfide[c]	6	1.5	*n*-butanethiol	99
diphenyl disulfide	24	1.5	none	
dimethyl sulfoxide	24	1.5	none	
diphenyl sulfone	24	1.5	none	
methanesulfonic acid[d]	24	2.0	none	

[a]See the corresponding footnote in Table 2. [b]Analyzed by GC. [c]Along with 1 equiv of hydrogen evolution. [d]Immediately 1 equiv of hydrogen evolved.

Table 9. Reaction of Lithium Thexyl^{t}butoxyborohydride with Representative Epoxides at 0 °C[a]

Compound	Time (h)	Ratio of rgt/cmpd	Product	Yield (%)[b]
1,2-epoxyoctane	0.5	0.5	1-octanol	1
			2-octanol	98
	0.5	1	1-octanol	0.5
			2-octanol	99.4
styrene oxide	0.5	0.5	1-phenylethanol	96
			2-phenylethanol	4
	0.5	1	1-phenylethanol	98.5
			2-phenylethanol	0.5
cyclohexene oxide	0.5	0.	cyclohexanol	99.9

[a]See the corresponding footnote in Table 2. [b]Analyzed by GC.

summarized in Table 9. The aliphatic epoxides examined were rapidly reduced with a quantitative amount of the reagent to give the S_N2-type of ring-opened products exclusively, whereas the reaction of styrene oxide, an aromatic one, with a theoretical amount of the reagent afforded a mixture of 96% 1-phenylethanol and 4% 2-phenylethanol. However, the reagent in an excess amount (H^-/compd = 2) reduced styrene oxide to give an almost single product, 1-phenylethanol, in a yield of 98.5%.

Conclusion

The reducing power of a newly synthesized monoalkylmonoalkoxyborohydride, lithium (2,3-dimethyl-2-butyl)-*t*-butoxyborohydride (lithium thexyl^{t}butoxyborohydride, Li Thx^{t}BuO BH_2, **1**), in a mixed solvent of THF and pentane toward organic compounds containing representative functional groups under practical conditions has been investigated. The results reveal that the reducing power of the reagent is somewhere between the dialkylborohydride and the parent borohydride. The reducing power of the lithium borohydride derivatives appears in the order of LiR_3BH > LiR_2BH_2 > $LIR(RO)BH_2$ > $LiBH_4$ > $Li(RO)_3BH$. The introduction of alkyl and alkoxy groups into the parent borohydride affords an interesting and unique reducing characteristics. Consequently, this reagent should find a valuable role as a reagent of choice in organic synthesis.

Experimental Section

All glassware used in the experiments was predried thoroughly in a drying oven and cooled under a dry nitrogen atmosphere. Hypodermic syringes were used to transfer solutions. All reactions were carried out under a static pressure of nitrogen in flasks fitted with septum-covered sidearms with use of standard techniques for handling air-sensitive materials.[14]

Materials. Most of the organic compounds utilized in this study were commercial products of the highest purity. They were further purified by distillation or recrystallization when necessary. Tetrahydrofuran (THF) was distilled from sodium benzophenone ketyl and stored under dry nitrogen. A solution of *tert*-butyllithium was purchased from the Aldrich Chemical Co. A solution of BH_3 in THF was synthesized by the reaction of sodium borohydride and dimethyl sulfate.[1]

Instruments. GC analyses were carried out on a Varian 3300 FID with capillary columns of DB-WAX (30 m) and HP-FFAP (25 m). All GC yields were determined with use of a suitable internal standard and authentic mixtures. NMR spectrometer used was a Bruker AMX 300.

Preparation of a Solution of Thx^{t}BuOBH. An oven-dried, 1-L, round-bottom flask with a sidearm, equipped with a magnetic stirring bar and an adapter, was attached to a mercury bubbler. The flask was flushed with dry nitrogen and then maintained under a static pressure of nitrogen. The flask was charged with 329 mL of a 1.52 M solution of BH_3-THF (500 mmol) and maintained at 0° with the aid of ice-water bath. Into the flask 44.2 g of 2,3-dimethyl-2-butene (525 mmol) was injected slowly and the mixture was stirred for 3 h. The concentration of a solution of thexylborane was estimated gasometrically to indicate 1.25 M. The ^{11}B NMR spectrum showed a triplet (J_{B-H} = 359 Hz) centered at δ 24.4 ppm relative to $BF_3{\cdot}OEt_2$.

Into the flask was charged with 320 mL of a solution of thexylborane (400 mmol) thus prepared above and the flask was immersed into an ice-water bath. To this 31.4 g of *tert*-butyl alcohol (420 mmol) was injected dropwise. After the complete evolution of hydrogen, the reaction mixture was stirred for additional 1 h. The concentration of the solution was estimated by hydrolysis to indicate 1.07 M. The ^{11}B NMR spectrum exhibited a doublet (J_{B-H} = 390 Hz) centered at δ 48.3 ppm.

Preparation of a Solution of Li Thx^{t}BuOBH$_2$, 1. Into a 100-mL flask was placed 40 mL of a solution of Thx-^{t}BuOBH (40.3 mmol) thus prepared above and the flask was cooled to -30° by use of a refrigerating bath circulator. To this flask was added 26 mL of a precooled 1.7 M solution of *tert*-butyllithium (44 mmol) in pentane dropwise with vigorous stirring. After the reaction mixture was stirred for additional 1 h at -30°, the flask was brought to 0°. The concentration of the reagent was estimated gasometrically by hydrolyzing an aliquot to give 0.60 M of Li ThxtBuOBH$_2$: ^{11}B NMR (THF and pentane) δ 7.6 ppm (*t*, J_{B-H} = 219 Hz).[10]

General Procedure Used for Reductions. The following

procedure was used for general reductions. The reduction of benzaldehyde is described as an example of the experimental procedure. The reagent **1** solution, 16.7 mL of 0.60 M (10 mmol of the reagent, 20.0 mmol of hydride), was introduced into a dried, 50-mL flask fitted with a rubber syringe cap on an inlet port, a magnetic stirring bar, and a bent adapter connected to a gas burette through a reflux condenser and a dry ice vapor trap. The flask was immersed in an ice-water bath, the stirred solution was maintained at 0°, and 1.07 g of benzaldehyde (10.0 mmol) in 2 mL of THF and tridecane (5 mmol) as an internal standard was injected. After 30 min, a 4.0-mL aliquot of the reaction mixture was removed and injected into a buffer solution (pH 7). After then, the solution was treated with 2 mL of 3 N NaOH and 2 mL of 30% H_2O_2 with stirring at room temperature for 2 h. The aqueous layer was saturated with K_2CO_3. The separated organic layer was washed with saturated NaCl solution and dried over anhydrous $MgSO_4$. The gas chromatographic analysis showed 99.5% benzyl alcohol. Aliquot was also removed after 1.0 h of the reaction time and worked up for GC analysis. The yield appeared to be 99%. Obviously, the reaction was complete within 0.5 h.

General Procedure for Stereoselectivity Studies. The reduction of 2-methylcyclohexanone is described as representative. To a 50-mL flask was added 8.4 mL of 0.60 M solution of the reagent **1** (5.0 mmol). The flask was cooled to -20° with use of a refrigerating bath circulator and to this was added 1.0 mL of a precooled solution of 2-methylcyclohexanone (0.29 g, 2.5 mmol) in THF. After 30 min, the reaction mixture was quenched with 3 N NaOH and oxidized with 30% H_2O_2 for 2 h at room temperature. The aqueous layer was saturated with K_2CO_3. The separated organic layer was washed with saturated NaCl and dried over anhydrous $MgSO_4$. The gas chromatographic analysis with use of a 30 m capillary column of DB-WAX showed no presence of the starting ketone (*i.e.*, 100% conversion) and >99.5% *cis*-2-methylcyclohexanol along with only trace amount of the *trans*-isomer. The results are summarized in Table 4.

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