original axes (Figure 1) and considering nodal planes with respect to the y axis gives σ, π_x, and π_z labels. Note that orbitals of the triiron fragment of π_z symmetry will include a contribution from the terminal H atom that lies in the yz plane (Chart I and Figure 9).[42]

Acknowledgment. The support of the National Science Foundation under Grant No. CHE 81-09503 is gratefully acknowledged. We also thank the Notre Dame Computing Center for computing time, Professor Roger DeKock for his help with the transformations, and Reynaldo Barreto for his help in running the Fenske–Hall calculations.

Registry No. I, 74792-06-6; II, 62361-87-9; III, 89414-14-2.

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[42] It would be more usual to denote the triiron fragment orbital symmetries with respect to the C_3 axis of the Fe_3C tetrahedron. The nomenclature chosen here allows the independent orbital analyses of $HFe_4(CO)_{12}CH$ to be compared more easily.

Addition Compounds of Alkali-Metal Hydrides. 25. Rapid Reaction of Boronic Esters and Acids with Lithium Aluminum Hydride. A Novel and Quantitative Synthesis of Lithium Monoorganylborohydrides[1]

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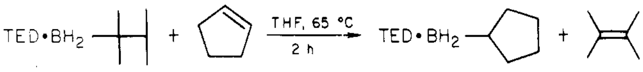
Received January 20, 1984

Boronic esters readily react with lithium aluminum hydride in diethyl ether–pentane at 0 °C to form the corresponding lithium monoorganylborohydrides and dialkoxyalanes. Under these reaction conditions, the dialkoxyalanes generally precipitate quantitatively from solution. The reaction is essentially quantitative and is broadly applicable to a representative variety of boronic esters. Boronic acids can also be readily converted to the corresponding lithium monoorganylborohydrides by treatment with lithium aluminum hydride. The reaction is quantitative and, like the boronic esters, is broadly applicable to a wide variety of boronic acids. These procedures provide a general, convenient synthesis of lithium monoorganylborohydrides with organic groups of greatly varying steric requirements.

Trialkylborohydrides have been demonstrated to be powerful selective reducing agents[3] and versatile synthetic intermediates.[4] Recently, several important routes to trialkylborohydrides have been developed.[5] In contrast, little effort has been devoted to the chemistry of monoorganylborohydrides, principally the result of the absence of satisfactory synthetic routes for monoorganylboranes. We reported one synthetically useful preparation of pure lithium monoalkylborohydrides[6] by treating triethylenediamine–monoalkylborane complexes ($TED{\cdot}BH_2R$) with lithium aluminum hydride ($LiAlH_4$), giving the borohydride in quantitative yield (eq 1). However, this procedure is limited to those monoalkylborane complexes that can be readily prepared from the reaction of relatively hindered olefins with triethylenediamine–thexylborane adduct ($TED{\cdot}BH_2Th$) (eq 2).

$$\mathrm{TED{\cdot}BH_2R + LiAlH_4 \xrightarrow[1\ h]{THF,\ 65\ ^\circ C} LiRBH_3 + TED{\cdot}AlH_3}\downarrow \qquad (1)$$



(2)

The reaction of borane–dimethyl sulfide (BMS) with various organolithium reagents has been reported to give the corresponding lithium monoorganylborohydrides (eq 3).[7,8] However, experimental work in our laboratory has shown that this reaction produces a mixture of products.[9]

$$\mathrm{Me_2S{\cdot}BH_3 + RLi \rightarrow LiRBH_3 + Me_2S} \qquad (3)$$

$$\mathrm{R = Me,}\ n\text{-}\mathrm{Bu}$$

More recently, Nöth and co-workers[10] have reported similar results in their systematic investigation of the reaction of organolithium reagents with various borane donors: $THF{\cdot}BH_3$, $Me_2S{\cdot}BH_3$, and $Me_3N{\cdot}BH_3$. Irrespective of the reaction conditions and reagents, all members of the series $LiR_{(4-n)}BH_n$ are present in the product. In few cases were pure lithium monoorganylborohydrides isolated from the reaction mixture in only modest yield.

In the course of our study of the synthesis of dialkylboranes from borinic esters (R_2BOMe), we discovered that

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Table I. Reaction of $LiAlH_4$ with Various Boronic Acid Derivatives[a]

boronic esters $RB(OR')_2$	^{11}B NMR chem shift, δ (multiplicity)	J_{BH}, Hz	^{27}Al NMR chem shift, δ
dimethylene methylboronate	−30.8 (q)	72	*b*
methylboroxine	−30.7 (q)	72	*b*
dimethyl *n*-hexylboronate	−28.1 (q)	74	*b*
diisopropyl *n*-hexylboronate	−28.1 (q)	74	*c*
di-*tert*-butyl *n*-hexylboronate	−28.1 (q)	74	+103

[a] Equimolar amount of 1.0 M $LiAlH_4$ in EE was added to 0.5 M solution of the boronic acid derivative at 0 °C. Clear supernatant solution was analyzed by ^{11}B and ^{27}Al NMR spectroscopy. [b] No signal was observed due to aluminum-containing compounds, indicating quantitative precipitation of the dialkoxyalane. [c] A weak signal at +103 ppm, relative to $Al(NO_3)_3$, was observed.

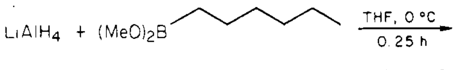
$LiAlH_4$ and R_2BOMe in a 1:1 molar ratio cleanly produced the corresponding lithium dialkylborohydrides (eq 4).[11]

$$R_2BOMe + LiAlH_4 \xrightarrow[0.5\ h]{THF,\ 25\ °C} LiR_2BH_2 + AlH_2(OMe) \quad (4)$$

Surprisingly, little is known concerning the reaction of boronic esters, $RB(OR')_2$, with $LiAlH_4$ in a 1:1 molar ratio.[12,13] The recent reports of simple and convenient syntheses of boronic esters[14,15] generated interest in a systematic investigation of the reaction of $LiAlH_4$ with representative boronic esters as a potential general route to lithium monoorganylborohydride.

Results and Discussion

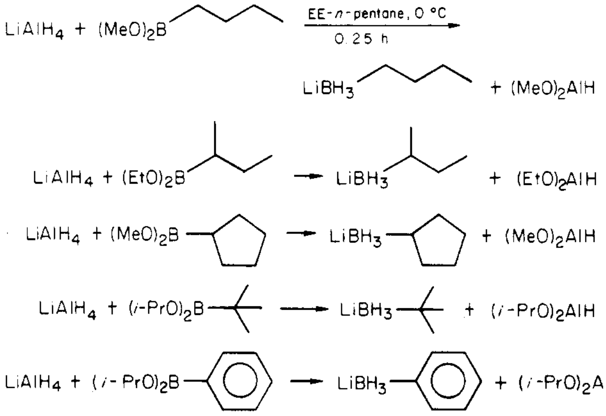
Addition of a tetrahydrofuran (THF) solution of $LiAlH_4$ to dimethyl *n*-hexylboronate in THF at 0 °C resulted in a moderately exothermic reaction with the concurrent formation of a white precipitate. A ^{11}B NMR spectrum of the clear, colorless supernatant solution showed only a quartet (δ −28.1 (J_{BH} = 74 Hz)) attributed to the clean formation of lithium *n*-hexylborohydride. The same solution gave a signal in the ^{27}Al NMR spectrum attributed to the dimethoxyalane. Evidently, the reaction proceeds as shown in eq 5. Encouraged by this result, we investigated the role of the following solvents diethyl ether (EE), THF, and EE–*n*-pentane (1:2). The reaction was equally clean in all cases; however, the EE–*n*-pentane (1:2) solvent mixture emerged as the solvent of choice for achieving quantitative precipitation of the dimethoxyalane. Addition of an ethereal solution of $LiAlH_4$ (1.0 M) to an equivalent amount of dimethyl *n*-hexylboronate in *n*-pentane (0.5 M) at 0 °C gave a white viscous mixture. The mixture was centrifuged and the clear supernatant solution transferred to another flask. The solid dimethoxyalane was washed twice with *n*-pentane. The clear centrifuged washings were combined with the supernatant. This solution contained pure lithium *n*-hexylborohydride, free of any detectable aluminum material. The purity of monoorganylborohydride by ^{11}B NMR spectrum directly reflects the purity of the boronic ester. Under these reaction conditions, borinic esters and borates are cleanly converted to the corresponding borohydride. Consequently, the presence of such borinic esters and borates as impurities in the original boronic ester results in the presence of the derived borohydrides as impurities in the final product.



(5)

Various alkoxy groups were studied to determine their effects on the solubility of the dialkoxyalane. Using the above solvent system, all boronic esters studied, except the *tert*-butyl ester, gave satisfactory results (Table I). The solubility of the dialkoxyalanes increases in the following order: $(MeO)_2AlH < (EtO)_2AlH < (i\text{-}PrO)_2AlH < (t\text{-}BuO)_2AlH$.

Having established satisfactory reaction conditions, we applied this reaction to a series of boronic esters (eq 6–10).



(6) (7) (8) (9) (10)

The isolated borohydrides were dissolved in a known amount of solvent, and their concentrations were estimated by hydrolysis.[16] From these values the yields were determined. The purity of the borohydrides was examined by ^{11}B and ^{27}Al NMR and shown to be free of any boron- and aluminum-containing impurities. These results are summarized in Table II. It is significant that oxidation of the lithium *tert*-butylborohydride to the alcohol, followed by analysis by gas chromatography, showed no isomerization of the relatively labile *tert*-butyl group.

The addition of one equivalent of *N,N,N′,N′*-tetramethylethylenediamine (TMED) to the pentane solution of lithium *tert*-butylborohydride provided the crystalline 1:1 TMED adduct in 80% isolated yield.[10] In a similar fashion, the TMED addition compounds for lithium methylborohydride and lithium phenylborohydride were isolated in 92% and 93% yields, respectively. On the other hand, lithium cyclopentylborohydride formed a TMED addition compound that proved to be highly soluble in *n*-pentane. Evaporation of the solvent gave the desired crystalline TMED adduct. However, the TMED addition product of lithium *n*-hexylborohydride proved to be a liquid at 25 °C.

The boronic esters preferred for the conversion to the corresponding borohydride, methyl or ethyl, are sometimes not those which are most readily prepared,[15] and they are sometimes difficult to isolate in high yields. However, isolation of the crystalline boronic acid from hydrolysis of esters can usually be achieved in practically quantitative

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Table II. Lithium Monoorganylborohydrides

boronic esters $RB(OR')_2$	lithium monoorganyl borohydride $LiRBH_3$ R	isolated yield,[a] %	^{11}B NMR chem shift, δ (multiplicity)	^{11}B NMR J_{BH}, Hz	TMED·$LiRBH_3$ mp, °C
methyl boroxine	methyl	63	−30.9 (q)	73	168–170
dimethyl *n*-butylboronate	*n*-butyl	75	−28.1 (q)	75	
diethyl *sec*-butylboronate	*sec*-butyl	80	−24.7 (q)	75	
diisopropyl *tert*-butylboronate	*tert*-butyl	70	−20.4 (q)	74	194–196
dimethyl 2-methyl-1-pentylboronate	2-methyl-1-pentyl	69	−29.3 (q)	74	
dimethyl *n*-hexylboronate	*n*-hexyl	86	−28.1 (q)	74	*b*
dimethyl cyclopentylboronate	cyclopentyl	85	−26.3 (q)	75	110–112[c]
diisopropyl phenylboronate	phenyl	85	−26.0 (q)	76	180–182

[a] Determined by hydride analysis. See Experimental Section. [b] Forms a TMED addition compound which is a liquid at 25 °C. [c] Forms a TMED addition compound which is soluble in *n*-pentane.

Table III. Lithium Monoorganylborohydrides Prepared from Boronic Acids

boronic acids $RB(OH)_2$	lithium monoorganyl borohydride $LiRBH_3$ R	isolated yield, %	^{11}B NMR chem shift, δ (multiplicity)	^{11}B NMR J_{BH}, Hz	IR ν_{B-H}, cm^{-1}
methylboronic acid	methyl	87	−30.8 (q)	72	2167
thexylboronic acid	2,3-dimethyl-2-butyl	86	−29.0 (q)	73	2172
trans-2-methylcyclopentylboronic acid	*trans*-2-methylcyclopentyl	77	−27.4 (q)	74	2190
cyclohexylboronic acid	cyclohexyl	75	−24.5 (q)	73	2164
phenylboronic acid	phenyl	85	−26.4 (q)	76	2241, 2189

yield. In order to effect the highest possible yield from valuable boronic compounds, it seems desirable to be able to utilize boronic acids in place of boronic esters. We discovered that the use of a greater than 1 equiv of $LiAlH_4$ readily converted the boronic acid quantitatively to the corresponding borohydride. Experimentally a molar ratio of $LiAlH_4$ to boronic acid of ca. 1.7 gave the highest yield of the monoorganylborohydride, free of any aluminum materials. The excess $LiAlH_4$, 0.7 equiv, is required to deprotonate the boronic acid and to react with water associated with the boronic acid. The amounts of water may vary, thus requiring additional amounts of $LiAlH_4$. Larger ratios of $LiAlH_4$ can be used, but this procedure then requires that the residual $LiAlH_4$ be removed.[17] More conveniently, we found several indicators that are capable of detecting $LiAlH_4$ in the presence of a borohydride. 2,2′-Bipyridine and 2,2′-biquinoline readily form orange and deep blue solutions with $LiAlH_4$ in EE, respectively. The slow addition of an ethereal solution of the boronic acid to the above at 25 °C results in the evolution of hydrogen and the formation of a granular precipitate. When approximately 0.6 equiv of boronic acid has been added, the solution becomes colorless. Analysis of the supernatant solution by ^{27}Al NMR at this point confirms the complete disappearance of all aluminum material. The generality of this procedure for preparing borohydrides from the corresponding boronic acids is indicated by the data in Table III. The use of these indicators can be extended to the conversion of boronic esters to the corresponding borohydrides. This avoids the need to prepare carefully standardized solutions of $LiAlH_4$.

Conclusion

Lithium aluminum hydride reacts rapidly and quantitatively with all boronic esters examined to give the corresponding lithium monoorganylborohydrides with concurrent formation of the dialkoxyalane. In a similar fashion, boronic acids can be converted to the borohydrides by using a larger ratio of $LiAlH_4$ to boron derivatives. These lithium monoorganylborohydrides are the only boron-containing compound in solution and can be isolated by simple evaporation. This procedure is the first general method for preparing lithium monoorganylborohydrides from either the corresponding boronic esters or the boronic acids in essentially quantitative yield.

Experimental Section

All operations were carried out under a nitrogen atmosphere with oven-dried glassware.[16] Gas chromatographic analyses were carried out with a Hewlett-Packard 5750 chromatograph with a TC detector. The spectra were obtained in an inert atmosphere. The infrared spectra were obtained with a Perkin-Elmer 1420 spectrometer, with sealed cells and a two-syringe technique. The ^{11}B NMR and ^{27}Al NMR spectra were recorded on a Varian FT-80A instrument. The ^{11}B NMR chemical shifts are in δ relative to EE·BF_3 with chemical shifts downfield from EE·BF_3 assigned as positive. The ^{27}Al NMR chemical shifts are in δ relative to $Al(NO_3)_3$.

Materials. Tetrahydrofuran (THF) was distilled from $LiAlH_4$ and stored under nitrogen. Anhydrous diethyl ether (EE) was purchased from Mallinckrodt, Inc., and used directly. *N,N,-N′,N′*-Tetramethylethylenediamine (TMED) was distilled from calcium hydride. $LiAlH_4$ (1.0 M) in EE was purchased from Aldrich Chemical Co. All boronic esters and acids were prepared by procedures described in the literature.[14,15]

Reaction of Boronic Esters with $LiAlH_4$. The following procedure for the preparation of lithium *n*-hexylborohydride is representative. A 50-mL centrifuge vial fitted with a rubber septum and magnetic stirring bar was charged with 20 mL of a 0.5 M solution of dimethyl *n*-hexylboronate (10 mmol) in *n*-pentane and cooled to 0 °C. A 1.0 M solution of $LiAlH_4$ in EE (10 mL, 10 mmol) was added with vigorous stirring. A voluminous precipitate of $(MeO)_2AlH$ separated. The reaction mixture was stirred as efficiently as possible for 0.25 h at 0 °C. The reaction mixture was then centrifuged, and the clear supernatant liquid was transferred via a double-ended needle to another vial. The solid $(MeO)_2AlH$ was washed with *n*-pentane (2 × 10 mL), and the washings were combined with the supernatant solution. The solvent was evaporated at 25 °C under reduced pressure (gradually reduced to 12 mmHg). The solid residue (1.5 g) was dissolved in EE (18.5 mL) and estimated by hydride analysis:[16] 0.43 M, 8.6 mmol, 86% yield; ^{11}B NMR δ −28.1 (q, J_{BH} = 74 Hz); IR ν 2200 cm^{-1}. No signal attributable to the presence of aluminum compounds in the solution could be detected either in the ^{27}Al

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[17] Excess $LiAlH_4$ can be removed from the isolated product by dissolving the borohydride in *n*-pentane. The residual $LiAlH_4$ is insoluble and can be removed by decantation. Care should be exercised in destroying the residual $LiAlH_4$ and dialkoxyalane.

NMR spectrum or in the IR spectrum.

In a separate experiment, the borohydride solution was quenched with methanol and then oxidized. The product alcohol was analyzed by GC using a 6 ft × 0.25 in column packed with 10% Carbowax 20M on Chromosorb W (60–80 mesh) with *n*-undecane as the internal standard. There was obtained 8.3 mmol of *n*-hexanol (83% yield).

Preparation of *N,N,N′,N′*-Tetramethylethylenediamine Addition Compounds of Lithium Monoorganylborohydrides. The following procedure for the preparation of the TMED addition compound of lithium phenylborohydride is typical. A 50-mL centrifuge tube fitted with a rubber septum and a magnetic stirring bar was charged with 10 mL of a 0.5 M solution of lithium phenylborohydride (5 mmol) in *n*-pentane. It was then treated with 5 mmol of TMED at 25 °C with stirring. An exothermic reaction set in and the addition compound crystallized from the reaction mixture at 25 °C. The reaction mixture was centrifuged and the supernatant liquid transferred by using a double-ended needle. The crystalline solid was washed with *n*-pentane (2 × 3 mL) and dried at 25 °C under reduced pressure (12 mmHg): 1.0 g (93% yield); mp 180–182 °C (lit.[10] mp 120 °C dec);[18] ^{11}B NMR δ −26.0 (q, J_{BH} = 76 Hz).

The TMED addition compound of lithium *tert*-butylborohydride was prepared similarly in 80% yield: mp 194–196 °C; ^{11}B NMR δ −20.3 (q, J_{BH} = 74 Hz).

The TMED addition compound of lithium methylborohydride was prepared similarly in 92% yield: mp 168–170 °C; ^{11}B NMR δ −30.2 (q, J_{BH} = 72 Hz).

Lithium cyclopentylborohydride formed a TMED addition compound which was highly soluble in *n*-pentane at 25 °C. The solvent was evaporated under reduced pressure (12 mmHg) to give the crystalline adduct in quantitative yield: mp 110–112 °C; ^{11}B NMR δ −26.3 (q, J_{BH} = 75 Hz).

Reaction of Boronic Acids with $LiAlH_4$. The following procedure for the synthesis of lithium phenylborohydride is representative. A 50-mL centrifuge vial containing a magnetic stirring bar was fitted with a rubber septum and was charged with 0.002 g of 2,2′-biquinoline and 10 mL of a 1.0 M solution of $LiAlH_4$ in EE. After being stirred for a short period of time, the solution became deep blue. A solution of phenylboronic acid (0.8 g, 6.6 mmol) in 15.2 mL of EE was slowly added at 25 °C. There was a rapid evolution of hydrogen and formation of a granular precipitate. After the addition of 11.8 mL (5.9 mmol) of the boronic acid, the supernatant solution became colorless. The reaction mixture was centrifuged, and the clear solution was decanted from the blue solid into another vial. The solid was washed with EE (2 × 10 mL), and the washings were combined with the supernatant solution. The solvent was evaporated under reduced pressure (12 mmHg). The residue, 0.6 g, was dissolved in 9.4 mL of EE and estimated by hydride analysis: 0.5 M, 5.0 mmol (85% yield); ^{11}B NMR δ −26.4 (q, J_{BH} = 76 Hz); IR ν 2241, 2189 cm^{-1}. No detectable signal was observed in the ^{27}Al NMR spectrum.

Acknowledgment. We thank the National Science Foundation for support by Grant CHE-7918881.

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[18] The melting point of TMED·$LiBH_3Ph$ differs considerably from that reported by Nöth.[10] The analytical and spectral data obtained for this compound agreed with the data reported by Nöth. At present we are unable to account for this large difference in the melting point.

Transition-Metal Ketenes. 21.[1] Conversion of η^2-Ketenyl Complexes into Novel Cations with a Tungstaphosphabicyclo[1.1.0]butanone Unit. Synthesis and Structure

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Received October 27, 1983

The reaction of η^2-ketenyl complexes η^5-$C_5H_5(CO)[P(CH_3)_3]\overline{WC(R)C}O$ (R = CH_3, $C_6H_4CH_3$) with halophosphines, R_2PCl (R = CH_3, C_6H_5), provides at −78 °C high yields of salts of novel tungstaphosphabicyclo[1.1.0]butanone cations. These yellow, temperature-sensitive solids are interesting intermediates in the synthesis of phosphine ketene complexes. Selected nucleophiles (CH_3^-, OCH_3^-) cause cleavage of the phosphorus–carbon bond in the cyclic system to afford η^1- and subsequently η^2-ketenyl complexes. The structures of the new bicyclic complexes were determined by spectroscopic investigations and, in the case of the tris(methylene chloride) solvate of **2a**, additionally by X-ray analysis. Crystal data for **2a**·$3CH_2Cl_2$ are as follows: space group $P2_1/c$, a = 15.447 (3) Å, b = 10.908 (3) Å, c = 17.603 (4) Å, β = 106.08 (2)°, Z = 4.

Introduction

Mild carbonylation of carbyne complexes leads via CC bond formation to η^1- and η^2-ketenyl complexes.[2-5] These react with Lewis acids such as alkyl cations, halides, or alkyls of the third main group to give alkyne π-complexes.[1,6] Toward Lewis bases, however, the η^2-ketenyl compounds show a pronounced reactivity to afford η^1-ketenyl,[4] anionic η^2-ketenyl complexes,[7] and σ-bonded alkyne derivatives,[8,9] depending upon the base and the reaction

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