tones. The ability of the polymer to reduce 3-methylcyclohexanone but not 3-methyl-2-cyclohexen-1-one is an interesting and potentially useful feature.

Borane complexes of poly(4-vinylpyridine) and poly(4-vinylpyridine-*co*-styrene) react sluggishly with carbonyls. In an effort to alleviate this situation, we modified the surface of these polymers by partial alkylation. Thus, the poly(4-vinylpyridine) or its copolymer was treated with dodecyl bromide in 1:1 nitromethane/nitroethane at 65 °C for 24 h. The extent of alkylation was determined by elemental analysis and by NMR (i.e., by integrating the aromatic proton signals from quaternized and nonquaternized pyridine rings). Most polymer samples had 48 ± 5% of their residues derivatized. The polymer was then reacted with BH_3·SMe_2 in the usual manner such that 44 ± 5% of the total pyridines was associated with BH_3. It was found that the modified polymer reduced octanal under standard conditions (see Experimental Section) with an average yield of 86% in 5 min. Nonalkylated polymer gave only 66% yield in 8 h. Clearly, alkylation with dodecyl groups produces a remarkably effective polymeric reducing agent.

Two reasonable explanations exist for the efficient reduction by "laurylated" polymer: (1) The octanal substrate adsorbs more readily on polymer bearing long hydrophobic chains. (2) The additional positive charge on the polymer facilitates reduction. The validity of the second alternative is suggested by our finding that polymer which is 50% alkylated with ethyl groups reacts as rapidly as the "laurylated" material. Very likely, therefore, the enhanced positive charge on the polymer chain causes it to assume less compact configurations,[14] thereby exposing the reactive sites to external aldehyde. The *N*-alkylpyridinium ions could also promote the release of borane or stabilize the transition state for the reductive process.

In summary, we have developed a new polymer reducing agent which converts aldehydes and ketones into alcohols under mild conditions, with high yield and particularly easy workup. The potential of surface-modified polymers in selective reductions of only one group in a symmetrically bifunctional molecule (a feat not possible in the absence of adsorption) remains to be determined.

Experimental Section

Materials. Commercially available polymers were used exclusively; it was felt that a methodology requiring the synthesis of polymers would lack general utility. Thus, poly(2-vinylpyridine) was purchased from Aldrich and poly(4-vinylpyridine) from Polysciences. Aldrich poly(4-vinylpyridine-*co*-styrene) with a 10% styrene content seemed to have more favorable solubility characteristics than the poly(4-vinylpyridine) but behaved similarly in the alkylation experiments.

Procedure A. A solution of 10 g of poly(2-vinylpyridine) in 100 mL of purified THF was added dropwise to 50 mL of BH_3·SMe_2 in THF (2 M) cooled in an ice bath. The mixture was stirred for 20 h (which is believed to be longer than necessary; this point was not checked). Hexane (100 mL) was then added and the stirring continued for another 12 h. The resulting precipitate was removed by filtration, washed with hexane, and dried under reduced pressure for 24 h at 45 °C to give 11.3 g of polymer–borane complex. Polymer prepared by this procedure requires 1 h in refluxing benzene for optimal reducing power. Its shelf-life at room temperature is at least 4 weeks.

Procedure B. A solution of 5.0 g of poly(2-vinylpyridine) in 50 mL of dry THF was added dropwise to 25 mL of BH_3·SMe_2 in THF (2 M) cooled in an ice bath. The mixture was stirred for 1 h after which 50 mL of dry benzene was added and stirring continued for another 12 h. The suspension was filtered to collect the polymer which was then washed with dry benzene (3 × 50 mL). After being dried for 5 h in vacuo at 40 °C, the polymer weighed 5.6 g. This material need not be pretreated with hot benzene.

The products from methods A and B are fine powders (mainly 60 mesh or smaller) that were used without grinding. If one adds the borane to the polymer (reversing the procedures described above), then the polymer forms an undesirable coagulate.

Reduction of Octanal. Polymer–borane complex from procedure A (0.25 g) was exposed to 3 mL of refluxing benzene for 1 h. The benzene was cooled, and 0.128 g (1 mmol) of octanal and 1 mmol of BF_3·OEt_2 were added (along with an internal standard in 2 mL of dry benzene). After 3 h at room temperature, the reaction mixture was shaken with saturated aqueous $NaHCO_3$ and filtered through Celite. Analysis of the filtrate by GLC showed that octanol was produced in 98% yield.

Reduction of Benzaldehyde. Dry benzene (25 mL) over 1.50 g of polymer–borane complex from procedure A was boiled under reflux for 1 h. The benzene was cooled, 1.28 g of benzaldehyde and 1.41 g of BF_3·OEt_2 in 5 mL of benzene were added, and the mixture was stirred for 1 h. Saturated aqueous $NaHCO_3$ (2 mL) was mixed with the benzene, the solids were removed by filtration, and the filtrate was washed with aqueous $NaHCO_3$ and 1 N HCl. After the benzene was removed, the product was distilled under reduced pressure to give 0.85 g (65%) of benzyl alcohol.

Polymer Alkylation. Poly(4-vinylpyridine-*co*-styrene) (2.22 g) was dissolved in 56 mL of 1:1 nitromethane/nitroethane and traces of insoluble material were removed by filtration. Redistilled dodecyl bromide (2.30 mL) was then added, and the solution was heated at 65 °C for 24 h. Alkylated polymer, isolated by precipitating the solid from small aliquots of the reaction mixture with 5 volumes of hexane, was subjected to elemental and NMR analyses. The remainder of the polymer solution was added slowly to 20 mL of borane–methyl sulfide complex in THF (2 M) cooled in an ice bath. After the solution was stirred for 1 h, 20 mL of benzene were added and the stirring was continued for another 48 h. The final polymeric product was precipitated with excess hexane, washed several times with hexane, and dried under reduced pressure at 50 °C for 24 h. The borane content was determined by volumetric measurement of H_2 in the presence of strong acid. Ethylated polymer was made in much the same manner.

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Registry No. 4-*tert*-Butylcyclohexanone, 98-53-3; cyclohexanone, 108-94-1; 3-methylcyclohexanone, 591-24-2; 3-methyl-2-cyclohexen-1-one, 1193-18-6; octanal, 124-13-0; benzaldehyde, 100-52-7; benzeneacetaldehyde, 122-78-1; 3-phenyl-2-propenal, 104-55-2; benzoic acid, 65-85-0; methyl benzoate, 93-58-3; benzoyl chloride, 98-88-4; acetophenone, 98-86-2; poly(2-vinylpyridine), 25014-15-7; poly(4-vinylpyridine-*co*-styrene), 24980-54-9; poly(4-vinylpyridine), 25232-41-1.

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Synthesis of 4-Deoxy-D-*lyxo*-hexose

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In connection with a program to investigate the effects of sugar analogues on glycoprotein biosynthesis, a convenient and efficient synthesis of 4-deoxy-D-*lyxo*-hexose (7) (4-deoxy-D-mannose) was required. This deoxy sugar was first prepared by Černý and co-workers via a multistep sequence that employed 1,6-anhydro-β-D-glucopyranose as starting material.[1] In this report a six-step synthesis of

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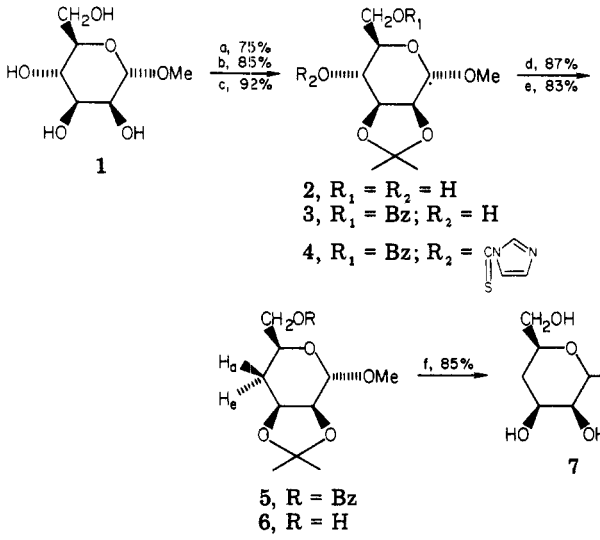
Scheme I[a]



[a] a, $CH_3C(OMe)_2CH_3$, H^+; b, PhC(O)Cl; c, TCDI; d, $SnBu_3H$; e, MeO^-; f, H_2O, H^+.

4-deoxy-D-*lyxo*-hexose from methyl α-D-mannopyranoside (**1**) in 35% overall yield is described (Scheme I).

Methyl 6-*O*-benzoyl-2,3-*O*-isopropylidene-α-D-mannopyranoside (**3**), available in two steps from **1**,[2] provided a readily accessible sugar derivative that was appropriately protected for substitution at C-4. Attempts to deoxygenate the 4-hydroxyl group of **3** by any of the standard methods that involve an S_N2 reaction[3] seemed unlikely to succeed because of steric hindrance and neighboring-group participation.[4] Deoxygenation by formation of the thiocarbonylimidazolide **4** and reduction with tributylstannane[5] appeared to be more promising. The latter reaction occurs via a free-radical intermediate[5] and thus avoids the problems associated with S_N2 processes.

Treatment of **3** with *N,N'*-thiocarbonyldiimidazole (TCDI) in 1,2-dichloroethane afforded crystalline methyl 6-*O*-benzoyl-2,3-*O*-isopropylidene-4-*O*-thiocarbonylimidazoyl-α-D-mannopyranoside (**4**) in 92% isolated yield. Reduction of **4** with tributylstannane in refluxing toluene cleanly produced the deoxygenated product methyl 6-*O*-benzoyl-4-deoxy-2,3-*O*-isopropylidene-α-D-*lyxo*-hexopyranoside (**5**). The ^1H NMR spectrum of **5** confirmed the presence of two C-4 protons (H_a, δ 1.65, J = 13.5, 9.3, 10.2 Hz; H_e, δ 2.00, J = 13.5, 6.6, 3.0 Hz). Deprotection of the deoxy sugar proceeded uneventfully. Base-catalyzed debenzoylation followed by acid hydrolysis yielded 4-deoxy-D-*lyxo*-hexose as a syrup. After azeotropic removal of water with ethanol, the deoxy sugar crystallized as an amorphous solid when triturated with hot ethyl acetate. The ^1H and ^{13}C NMR spectra and elemental analysis (see the Experimental Section) support the identity of **7**. The *p*-nitrobenzenesulfonylhydrazone[1] of **7** had mp 107–110 °C dec (lit.[1] mp 113–120 °C dec).

In summary, these results describe a convenient, high-yield procedure for the synthesis of 4-deoxy-D-*lyxo*-hexose and confirm the usefulness of thiocarbonyldiimidazole/tributylstannane treatment[5] as a method for the deoxygenation of hindered secondary hydroxyl groups.

Experimental Section

Melting points were determined with a Thomas-Hoover capillary melting-point apparatus and are uncorrected. NMR spectra were recorded on a Varian EM 390 or CFT 20 spectrometer. Chemical shifts are expressed in parts per million downfield from Me_4Si. Optical rotations were measured on a Perkin-Elmer Model 141 polarimeter. Elemental analyses were performed by Galbraith Laboratories.

Methyl 6-*O*-Benzoyl-2,3-*O*-isopropylidene-4-*O*-thiocarbonylimidazoyl-α-D-mannopyranoside (4). A mixture of methyl 6-*O*-benzoyl-2,3-*O*-isopropylidene-α-D-mannopyranoside[2] (8.45 g, 25 mmol, mp 110–112 °C; lit.[2] mp 111–112 °C) and *N,N'*-thiocarbonyldiimidazole (8.9 g, 50 mmol) in 1,2-dichloroethane (125 mL) was heated at gentle reflux for 4 h. The solution was concentrated in vacuo to a yellow oil that was dissolved in CH_2Cl_2 (125 mL) and washed with 1 N HCl (1 × 125 mL), 5% aqueous $NaHCO_3$ (1 × 125 mL), and water (1 × 125 mL). The organic layer was dried over anhydrous Na_2SO_4 and concentrated in vacuo to a pale yellow oil that crystallized upon addition of cold diethyl ether (2 × 15 mL). The product was collected by filtration and, after being air dried, yielded **4** as a nearly colorless solid (10.3 g, 92%): mp 129–130 °C; $[\alpha]^{18}_D$ +56.8° (*c* 1.5, $CHCl_3$); ^1H NMR ($CDCl_3$) δ 1.32 (s, 3 H), 1.58 (s, 3 H), 3.41 (s, 3 H), 4.20 (d, 1 H), 4.05–4.50 (m, 4 H), 4.99 (s, 1 H), 5.99 (dd, J = 7, 10 Hz, 1 H), 6.95–8.30 (m, 8 H).

Anal. Calcd for $C_{21}H_{24}N_2O_7S$: C, 56.23; H, 5.39; N, 6.24; S, 7.15. Found: C, 56.22; H, 5.41; N, 6.20; S, 7.11.

Methyl 6-*O*-Benzoyl-4-deoxy-2,3-*O*-isopropylidene-α-D-*lyxo*-hexopyranoside (5). A mixture of **4** (2.69 g, 6.0 mmol) in dry toluene (50 mL) was added dropwise over 30 min to a stirred solution of refluxing toluene (200 mL) and tributylstannane (2.7 g, 9.3 mmol) under N_2. After the solution was refluxed for an additional 4 h, the solvent was removed in vacuo to give a colorless oil. The crude product was dissolved in hexane (100 mL) and extracted with acetonitrile (4 × 60 mL). The combined acetonitrile extracts were washed with hexane (2 × 100 mL) and concentrated in vacuo to an oil. Recrystallization from pentane (20 mL) yielded **5** as fine colorless needles (1.67 g, 87%): mp 58–59 °C; $[\alpha]^{18}_D$ +39.3° (*c* 1.5, $CHCl_3$); ^1H NMR ($CDCl_3$) δ 1.36 (s, 3 H), 1.54 (s, 3 H), 1.65 (ddd, $J_{4a,4e}$ = 13.5, $J_{3,4a}$ = 9.3, $J_{4a,5}$ = 10.2 Hz, 1 H), 2.00 (ddd, $J_{4a,4e}$ = 13.5, $J_{3,4e}$ = 6.6, $J_{4e,5}$ = 3.0 Hz, 1 H), 3.42 (s, 3 H), 3.99 (d, 1 H) superimposed on 3.9–4.18 (m, 1 H), 4.41 (ddd, 1 H) superimposed on 4.25–4.50 (m, 2 H), 4.98 (s, 1 H), 7.25–7.65 (m, 3 H), 8.0–8.2 (m, 2 H).

Anal. Calcd for $C_{17}H_{22}O_6$: C, 63.34; H, 6.88. Found: C, 63.33; H, 6.93.

Methyl 4-Deoxy-2,3-*O*-isopropylidene-α-D-*lyxo*-hexopyranoside (6). A solution of **5** (2.58 g, 8 mmol) in 0.1 M sodium methoxide in methanol (40 mL) was allowed to stand at 20 °C for 20 h. Solid CO_2 (~1 g) was added in portions and, after 30 min, the solution was concentrated in vacuo. The residue was taken up in water and extracted with $CHCl_3$ (3 × 25 mL). The combined $CHCl_3$ extracts were dried over anhydrous Na_2SO_4 and concentrated in vacuo [70 °C (0.1 torr)] to remove methyl benzoate. The residue solidified and was recrystallized from ether/petroleum ether (1:5, 30 mL, −20 °C) to give **6** as a colorless, crystalline product (1.46 g, 83%): mp 59–60 °C; $[\alpha]^{18}_D$ +66.0° (*c* 1.5, $CHCl_3$); ^1H NMR ($CDCl_3$) δ 1.31 (s, 3 H), 1.49 (s, 3 H), 1.57 (ddd, $J_{4a,4e}$ = 13, $J_{3,4a}$ = 9.0, $J_{4a,5}$ = 9.0 Hz, 1 H), 1.87 (ddd, $J_{4a,4e}$ = 13, $J_{3,4e}$ = 6.2, $J_{4e,5}$ = 3.5 Hz, 1 H), 2.63 (br t, 1 H), 3.40 (s, 3 H), 3.5–4.0 (m, 4 H), 4.36 (ddd, 1 H), 4.91 (s, 1 H).

Anal. Calcd for $C_{10}H_{18}O_5$: C, 55.03; H, 8.31. Found: C, 55.19; H, 8.29.

4-Deoxy-*lyxo*-hexose (7). An aqueous solution (25 mL) of **6** (650 mg, 3 mmol) containing Amberlite IR 120 (H^+ form, wet weight 0.6 g) was heated at reflux for 20 h. The solution was filtered and concentrated in vacuo to a syrup that was further dried by addition and evaporation of absolute ethanol (3 × 10 mL). Trituration of the crude product with ethyl acetate induced crystallization. The solid product was filtered, washed with ether, and dried in vacuo (0.1 torr) over P_2O_5 to yield 4-deoxy-*lyxo*-hexose (416 mg, 85%): mp 54–80 °C; $[\alpha]^{18}_D$ +31.8° (3 min) → $[\alpha]^{18}_D$ +29.5° (60 min, equilibrium) (*c* 1.2, H_2O) [lit.[1] $[\alpha]^{20}_D$ +3° (*c* 0.57, H_2O)]; ^1H NMR (D_2O) δ 1.55–1.90 (m, 2 H), 3.60–4.20 (m, 5 H),

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5.27 (br s, 0.6 H, α-anomeric proton; β-anomeric proton apparently masked by HOD); ^{13}C NMR (D_2O) δ (α-anomer) 29.6, 64.7, 65.3, 68.6, 69.4, 95.2; (β-anomer) 29.0, 64.4, 69.3, 70.4, 73.2, 94.5.[6,7]

Anal. Calcd for $C_6H_{12}O_5$: C, 43.90; H, 7.37. Found: C, 43.43; H, 7.67.

The *p*-nitrobenzenesulfonylhydrazone of **7**[1] had mp 107-110 °C dec (lit.[1] mp 113-120 °C dec).

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Registry No. **3**, 63167-70-4; **4**, 73635-97-9; **5**, 73635-98-0; **6**, 73635-99-1; **7**, 18439-27-5; **7** *p*-nitrobenzenesulfonylhydrazone, 73636-00-7; *N,N'*-thiocarbonyldiimidazole, 6160-65-2.

(6) It is interesting to note that the equilibrium ratio of α- and β-anomers of **7** in aqueous solution (determined by NMR measurements) is 62:38, quite similar to the 67:33 ratio observed for D-mannose.[7]

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Reaction of Triflic Anhydride with Grignard Reagents. Oxidizing Properties of Triflic Anhydride

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As a leaving group, the trifluoromethanesulfinate ($CF_3SO_2^-$) group should possess greater lability than nonfluorinated analogues such as methanesulfinate ($CH_3SO_2^-$). This is borne out by studies of Hendrickson and coworkers[2] which show facile loss of the trifluoromethanesulfinate group in 1,2- and 1,3-eliminations as well as reductive eliminations. Additionally, our studies with α-keto triflates[3,4] have shown that base-promoted 1,2-elimination of trifluoromethanesulfinic acid (a formal oxidation process) can occur in preference to α-elimination of trifluoromethanesulfonic acid. We were therefore interested in developing methods to complement those of Hendrickson[2,5] for the preparation of trifluoromethyl sulfones (triflones) and to exploit the apparent excellent leaving-group properties of the trifluoromethanesulfinate moiety.

Hendrickson[5] has discussed some of the problems associated with the reaction of organolithium reagents with electrophilic triflating agents such as triflic anhydride. Subsequent reactions of the organolithium reagent with the "triflone" product are, in some cases, detrimental to the production of high yields. In attempting to circumvent some of these problems, we turned to Grignard reagents which we felt would be less basic, kinetically, and less nucleophilic than the corresponding organolithium reagents. Surprisingly, the reaction of phenylmagnesium bromide with triflic anhydride (-78 to 0 °C) gave only about 7% phenyl trifluoromethyl sulfone. The major product (75%) was bromobenzene (eq 1). A similar reaction occurs with phenylmagnesium iodide, giving iodobenzene (69%). A formal oxidation of Grignard reagent by triflic anhydride had occurred. This reaction appears to be quite general.

$$\underset{1}{PhMgBr} + \underset{2}{(CF_3SO_2)_2O} \rightarrow \underset{3}{PhBr} \quad (1)$$

Table I. Reaction of Grignard Reagents with Triflic Anhydride

reacn no.	RMgX	% RX	% RSO_2CF_3
1	C_6H_5MgBr	75	7
2	C_6H_5MgI	69	*a*
3	*p*-$CH_3C_6H_4MgBr$	72	5
4	*p*-$CH_3OC_6H_4MgBr$[b]	75	14
5	*p*-$CH_3OC_6H_4MgBr$[c]	57	21
6	*p*-ClC_6H_4MgBr	81	*a*
7	*m*-$CF_3C_6H_4MgBr$	78	trace[d]
8	1-naphthyl-MgBr	75	*a*
9	$C_6H_5C{\equiv}CMgBr$	63	5
10	*n*-$C_8H_{17}MgBr$	69	24
11	cyclohexyl-MgBr	78	trace[d]
12	*n*-$C_8H_{17}MgCl$	5	87
13	*n*-C_4H_9MgCl	*e*	86
14	$C_6H_5CH_2MgCl$	13	87
15	$CH_2{=}CHCH_2MgCl$	*e*	54
16	cyclohexyl-MgCl	14	16

[a] None detected. [b] Grignard reagent forms a two-phase system. This reaction was carried out by using the lower phase which was 1.69 M in Grignard reagent. [c] This reaction was carried out by using the upper phase which was 0.35 M in Grignard reagent. [d] Detected by GC-MS (<2%). [e] Not analyzed for RX.

Table I gives yields of aryl bromides produced by reaction of the corresponding arylmagnesium bromides with triflic anhydride. The yields are all good. The presence of electron-donating or electron-withdrawing substituents on the aromatic ring does not appear to drastically alter the course of the reaction. One does begin to see larger amounts (up to 21%) of the sulfone in the case of *p*-methoxyphenylmagnesium bromide. The acetylenic Grignard reagent phenylethynylmagnesium bromide gives the same type of reaction as do the aliphatic reagents *n*-octylmagnesium bromide and cyclohexylmagnesium bromide.

It is quite apparent from Table I that the reaction of aryl- and alkylmagnesium bromides is not the method of choice for the production of trifluoromethyl sulfones. However, the reaction is of interest from a mechanistic standpoint. A mechanism is suggested in eq 2-5. It is

$$2RMgBr \rightleftharpoons MgBr_2 + R_2Mg \quad (2)$$

$$MgBr_2 + (CF_3SO_2)_2O \rightarrow CF_3SO_2Br + CF_3SO_3^- \quad (3)$$

$$CF_3SO_2Br + Br^- \rightarrow Br_2 + CF_3SO_2^- \quad (4)$$

$$R_2Mg + 2Br_2 \rightarrow 2RBr + MgBr_2 \quad (5)$$

proposed that the magnesium bromide, formed from the Grignard reagent via the Schlenk equilibrium,[6] initiates the oxidation-reduction process. Nucleophilic attack of bromide on triflic anhydride could produce trifluoromethanesulfonyl bromide and triflate ion according to eq 3. Further reaction of trifluoromethanesulfonyl bromide with bromide ion could give bromine and trifluoromethanesulfinate ion, the reduced product.[7] It is suggested that the aryl (alkyl) bromides arise from reaction

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(7) This mechanism for the formation of bromine has analogy. Potassium trifluoromethanesulfinate can be prepared by the reaction of CF_3SO_2Cl with potassium iodide in acetone. Elemental iodine is also formed. See ref 2, footnote 4.

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