Fourier map was essentially featureless with no peaks greater than 0.3 e A^{-3}.

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Supplementary Material Available: Crystal data, interatomic distances, and selected bond angles (2 pages). Ordering information is given on any current masthead page.

Efficient Preparation of [(Methoxymethoxy)methyl]tributylstannane, a Convenient Hydroxymethyl Anion Equivalent

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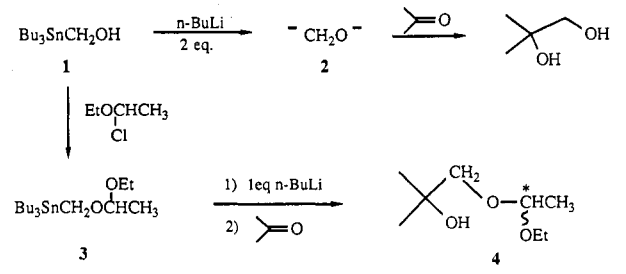
The utility of α-alkoxy organostannanes as precursors to α-alkoxy organolithiums has been demonstrated by several groups.[1] Recently Macdonald, McGarvey, and co-workers published a detailed study of the tin–lithium exchange process of this class of compounds and demonstrated the utility of tertiary α-alkoxy organostannanes for the formation of highly substituted, functionalized carbon–carbon bonds.[2] Primary α-alkoxy organostannanes[3] have been used by several investigators as hydroxymethyl anion equivalents.[4] The direct hydroxymethylation of carbonyl compounds was achieved by Seebach and Meyer,[3a] who treated (tributylstannyl)methanol (1) with 2 equiv. of *n*-BuLi to produce the dianion **2** of methanol. The dianion added to carbonyl compounds to give diols directly (Scheme I). The utility of this method, however, is limited due to the instability of the reagent and the moderate yields of addition products. Still extended the utility of **1** by protecting the free alcohol by using α-chloroethyl ethyl ether to give the stable ethoxyethyl ether **3**.[3b] Compound **3** upon treatment with 1 equiv, of *n*-BuLi gave the α-alkoxy organolithium, which added in high yield to carbonyl compounds, providing monoprotected diols **4**.

In the context of some target molecule syntheses currently under way in our laboratory, the presence of a chiral center in **3** along with its introduction into products of type **4** was disadvantageous. The related MOM derivative **5** was foreseen as fulfilling our need of an acid-sensitive protecting group, which would not introduce new diastereomers. Compound **5** has been recently prepared by McGarvey by alkylation of **1** with chloromethyl methyl ether. For the synthesis of **5** we sought an efficient and cost-effective route that avoided the use of the toxic chloromethyl methyl ether. The results of our investigation are presented here.

For the preparation of (tributylstannyl)methanol (**1**), we used the method described by Still,[3b] in which lithium diisopropylamide (LDA) is used to deprotonate tributyltin hydride. The resulting (tributylstannyl)lithium was treated with paraformaldehyde to give **1**.[5] Methoxymethylation of the crude alcohol **1** was carried out by using

Scheme I



Scheme II

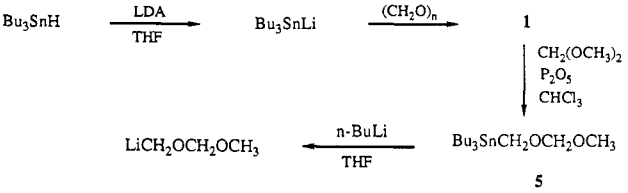
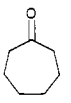
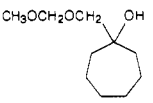
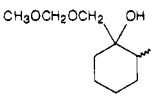
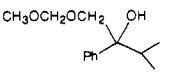
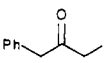
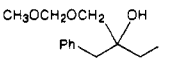
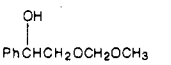
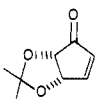
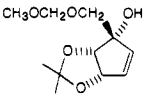
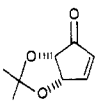
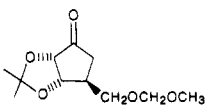
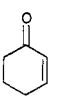
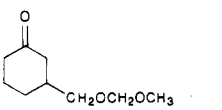
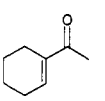
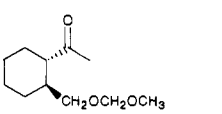


Table I. Addition of α-Alkoxy Stannane 5 to Carbonyl Compounds

entry	substrate	procedure	product	yield,[a] %
1		A		93
2		A		95
3		A		94
4		A		96
5	PhCHO	A		96
6		A		96
7		B		70
8		B		53 (82)[b]
9		B		35 (92)[b]

[a] Isolated yield of pure product. [b] Based on recovered starting material.

(1) (a) Linderman, R. J.; Godfrey, A.; Horne, K. *Tetrahedron Lett.* **1987**, *28*, 3911. (b) Linderman, R. J.; Godfrey, R. J. *Ibid.* **1986**, *27*, 3911. (c) Duchene, A.; Quintard, J. P. *J. Chem. Soc., Chem. Commun.* **1987**, 29.

(2) Sawyer, J. S.; Kucerovy, A.; Macdonald, T. L.; McGarvey, G. J. *J. Am. Chem. Soc.* **1988**, *110*, 842.

(3) (a) Seebach, D.; Meyer, N. *Angew. Chem., Int. Ed. Engl.* **1976**, *15*, 438. (b) Still, W. C. *J. Am. Chem. Soc.* **1978**, *100*, 1481.

(4) For other hydroxymethyl anion equivalents, see: (a) Corey, E. J.; Eckrich, T. M. *Tetrahedron Lett.* **1983**, *24*, 3165. (b) Imamoto, T.; Takayama, T.; Yokoyama, M. *Ibid.* **1984**, *25*, 3225. (c) Tamao, K.; Ishida, N. *Ibid.* **1984**, *25*, 4245.

(5) We explored a number of other methods for preparing tributyltin anion, all of which were found to be inferior to the LDA method; see: (a) Tamborski, C.; Ford, F. E.; Soloski, E. J. *J. Org. Chem.* **1963**, *28*, 237. (b) Lahournere, J. C.; Valade, J. *C. R. Seances Acad. Sci., Ser. C* **1970**, *C270*, 2080. (c) Corriu, R. J. P.; Guerin, C. *J. Organomet. Chem.* **1980**, *197*, C19.

0022-3263/88/1953-4131$01.50/0

a method developed by Fujita.[6] This reaction, complete in less than 20 min, involved treatment of alcohol 1 with excess dimethoxymethane in the presence of phosphorus pentoxide in chloroform to yield the methoxymethyl (MOM) ether 5. High-vacuum distillation of the crude product gave a 60% yield of 5 (97% pure)[7] based on tributyltin hydride (Scheme II). Given the acid sensitivity of the (tributylstannyl)methanol, the success of this reaction demonstrates the utility of the Fujita process. We recommend the dimethoxymethane/P_2O_5 reagent as an effective, inexpensive, and safe surrogate for chloromethyl methyl ether.

Compound 5 in THF readily transmetalates[8] with *n*-BuLi, and the resulting $MOMOCH_2Li$ adds in high yield to carbonyl compounds, providing monoprotected diols. The reagent can also be added in a conjugate fashion to enones, albeit in moderate yield, by using the methodology of Fuchs and Hutchinson[9] (Table I). Deprotection of the alcohol can be achieved in high yield by simple acid hydrolysis.

Although other hydroxymethyl anion equivalents are available, our method uses readily available, inexpensive materials for preparation of the anion equivalent and gives high yields of reaction products. The reagent can be easily prepared in 15–20-g quantities and should be useful in synthesis.

Experimental Section

Tributyltin hydride was prepared from bis(tributyltin) oxide and polymethylhydrosiloxane.[10] All alkyllithium reactions were carried out in flame-dried glassware under an argon atmosphere. Tetrahydrofuran (THF) was distilled from sodium/benzophenone ketyl immediately before use. For conjugate addition reactions, copper(I) bromide–dimethyl sulfide complex was prepared by the method of Townsend.[11]

[(Methoxymethoxy)methyl]tributylstannane (5). To a solution of diisopropylamine (9.87 g, 13.7 mL, 97.5 mmol) in THF (125 mL) at 0 °C was added dropwise *n*-BuLi (1.56 M in hexanes, 57 mL, 90 mmol). The resulting solution was stirred for 15 min at 0 °C, followed by dropwise addition of tributyltin hydride (21.8 g, 20.1 mL, 75 mmol). After 15 min at 0 °C, solid paraformaldehyde (2.7 g, 90 mmol) was added and the ice bath was removed. After the mixture was stirred for 1.5 h at ambient temperature, the clear reaction mixture was poured into petroleum ether (400 mL) and washed with water (1 × 200 mL) and brine (1 × 200 mL). The organic phase was dried over magnesium sulfate and concentrated in vacuo, to give crude (tributylstannyl)methanol (1) as a pale yellow liquid.

To a three-necked round-bottomed flask equipped with a mechanical silver were added dimethoxymethane (114 g, 133 mL, 1.5 mol), chloroform (125 mL, previously treated with P_2O_5), and phosphorus pentoxide (25 g). This mixture was stirred for 5 min, followed by addition of the crude alcohol 1 and P_2O_5 (25 g). The mixture was vigorously stirred for 15 min, at which time the reaction was complete [TLC: ethyl acetate/petroleum ether (1:20), silica gel]. The reaction mixture was carefully poured into 300 mL of saturated aqueous sodium carbonate, and the residual P_2O_5 was washed with petroleum ether (400 mL). The combined organic layers were separated, washed with brine, dried over magnesium sulfate, and concentrated in vacuo. Vacuum distillation through a 6-cm Vigreux column gave 5 as a clear liquid [bp 84–87 °C (0.05mm)] (16.4 g, 60%); ^1H NMR ($CDCl_3$) δ 0.75–1.65 (m, 27 H), 3.32 (s, 3 H), 3.75 (s, 2 H), 4.50 (s, 3 H); ^{13}C NMR ($CDCl_3$) δ 8.83, 13.59, 27.22, 29.03, 54.82, 57.54, 99.40. These data compare favorably with literature values.[2] The product is contaminated with ca. 3% of $CH_3OCH_2OCH_2OCH_2SnBu_3$.[7]

General Procedure A. Addition of 5 to Ketones and Aldehydes. To a solution of 5 (1.75 g, 4.8 mmol) in 15 mL of THF at −78 °C was added *n*-BuLi (2.5 M in hexanes, 1.84 mL, 4.6 mmol) over a period of 2 min while the temperature was maintained below −65 °C. Stirring was continued for no more than 5 min,[8] at which time the carbonyl compound was added neat via syringe. After the mixture was stirred for 15 min at −78 °C, the reaction was quenched by the addition of 20 mL of saturated aqueous ammonium chloride. The mixture was extracted with ethyl acetate, and the combined organic layers were washed consecutively with water and brine and then dried over anhydrous magnesium sulfate. The solvent was removed in vacuo to give the crude product, which was purified by flash chromatography over silica gel.

1-[(Methoxymethoxy)methyl]cycloheptanol: ^1H NMR ($CDCl_3$) δ 1.35–1.69 (m, 12 H), 2.40 (s, 1 H), 3.35 (s, 3 H), 3.35 (s, 2 H), 4.63 (s, 2 H); ^{13}C NMR ($CDCl_3$) δ 22.26, 29.86, 37.59, 55.18, 74.57, 76.10, 96.92. Anal. Calcd for $C_{10}H_{20}O_3$: C, 63.80; H, 10.71. Found: C, 63.57; H, 10.57.

1-[(Methoxymethoxy)methyl]-2-methylcyclohexanol (major diastereomer): ^1H NMR ($CDCl_3$) δ 0.905 (d, J = 6.53 Hz, 3 (H), 1.25–1.8 (m, 9 H), 1.92 (s, 1 H), 3.37 (s, 3 H), 3.45 (AB q, J = 9.58 Hz, $\Delta\nu_{AB}$ = 14.69 Hz, 2 H), 4.65 (AB q, J = 6.57, $\Delta\nu_{AB}$ = 3.72 Hz); ^{13}C NMR ($CDCl_3$) δ 15.13, 21.43, 25.34, 30.37, 34.87, 36.44, 55.27, 72.15, 74.88, 97.03. Anal. Calcd for $C_{10}H_{20}O_3$: C, 63.80; H, 10.71. Found: C, 63.56; H, 10.95.

1-(Methoxymethoxy)-3-methyl-2-phenyl-2-butanol: ^1H NMR ($CDCl_3$) δ 0.801 (d, J = 6.90 Hz, 3 H), 0.927 (d, J = 6.88 Hz, 3 H), 2.06 (m, 1 H), 3.04 (br s, 1 H), 3.21 (s, 3 H), 3.96 (AB q, J = 9.99 H, $\Delta\nu_{AB}$ = 86.3 Hz, 2 H), 4.61 (AB q, J = 6.6 Hz, $\Delta\nu_{AB}$ = 6.62 Hz, 2 H), 7.36 (m, 5 H); ^{13}C NMR ($CDCl_3$) δ 16.80, 17.39, 35.84, 55.40, 74.05, 76.08, 97.07, 126.10, 126.51, 127.65, 128.03. Anal. Calcd for $C_{13}H_{20}O_3$: C, 69.61; H, 8.99. Found: C, 69.63; H, 9.05.

2-[(Methoxymethoxy)methyl]-1-phenyl-2-butanol: ^1H NMR ($CDCl_3$) δ 0.996 (t, J = 7.39 Hz, 3 H), 1.54 (q, J = 7.38, Hz, 2 H), 2.27 (br s, 1 H), 2.85 (AB q, J = 13.81 Hz, $\Delta\nu_{AB}$ = 6.75 Hz, 2 H), 3.41 (s, 2 H), 3.43 (s, 3 H), 4.69 (s, 2 H), 7.29 (m, 5 H); ^{13}C NMR ($CDCl_3$) δ 7.72, 29.00, 42.51, 55.53, 72.85, 73.88, 97.19, 126.36, 128.13, 130.45, 133.77. Anal. Calcd for $C_{13}H_{20}O_3$: C, 69.61; H, 8.99. Found: C, 69.54; H, 9.29.

2-(Methoxymethoxy)-1-phenylethanol: ^1H NMR ($CDCl_3$) δ 3.32 (br s, 1 H), 3.38 (s, 3 H), 3.59 (m, 1 H), 3.76 (m, 1 H), 4.69 (AB q, J = 6.56 Hz, $\Delta\nu_{AB}$ = 4.66 Hz, 2 H), 4.89 (m, 1 H), 7.28–7.42 (m, 5 H); ^{13}C NMR ($CDCl_3$) δ 55.33, 72.91, 74.12, 96.86, 126.08, 127.71, 128.28, 140.32. Anal. Calcd for $C_{10}H_{14}O_3$: C, 65.91; H, 7.74. Found: C, 65.81; H, 7.55.

2,2-Dimethyl-4-[(methoxymethoxy)methyl]-3aβ,6aβ-dihydro-4*H*-cyclopenta-1,3-dioxol-4α-ol: ^1H NMR ($CDCl_3$) δ 1.40 (s, 3 H), 1.45 (s, 3 H), 3.23 (s, 1 H), 3.35 (s, 3 H), 3.56 (AB q, J = 9.93 Hz, $\Delta\nu_{AB}$ = 38.4 Hz, 2 H), 4.52 (d, J = 5.40 Hz, 1 H), 4.64 (AB q, J = 6.42 Hz, $\Delta\nu_{AB}$ = 5.34 Hz, 2 H), 5.06 (dd, J = 5.40, 5.76 Hz, 1 H), 5.77 (d, J = 5.79 Hz, 1 H), 5.93 (dd, J = 5.78, 5.76 Hz, 1 H); ^{13}C NMR ($CDCl_3$) δ 26.47, 27.62, 55.31, 71.68, 80.10, 81.48, 83.75, 96.74, 111.2, 132.88, 136.87. Anal. Calcd for $C_{11}H_{18}O_5$: C, 57.38; H, 7.88. Found: C, 57.65; H, 8.07.

General Procedure B. Conjugate Addition of 5 to Enones.[9] To a solution of $CuBr\cdot Me_2S$ (352 mg, 1.71 mmol) in 1.5 mL of diisopropyl sulfide and 1.7 mL of THF at −78 °C was added isopropylmagnesium chloride (2.0 M in diethyl ether, 20 μL) followed by stirring for 20 min. In a separate flask, a solution of 5 (568 mg, 1.56 mmol) in 7 mL of THF was treated with *n*-BuLi (2.5 M in hexanes, 0.62 mL, 1.56 mmol) as described above. The cold yellow anion solution was transferred via a cooled cannula to the copper(I) bromide solution followed by stirring at −78 °C for 15 min. To the resulting dark brown solution was added the

(6) Fuji, K.; Nakano, S.; Fujita, E. *Synthesis* **1975**, 276.

(7) The product is contaminated with up to 3% $CH_3OCH_2OCH_2OCH_2SnBu_3$. By TLC using 20:1 petroleum ether/ethyl acetate as the eluent, compound 5 displays an R_f of 0.57 and the impurity 0.48. The methylene hydrogens ($CH_3O^aCH_2O^bCH_2OCH_2SnBu_3$) a and b can be observed as singlets in the ^1H NMR spectrum at δ 4.71 and 4.68 respectively.

(8) In some cases we have made the observation that when the transmetalation mixture (5 + BuLi in THF) is stirred (15 min or more) prior to addition of the carbonyl compound, the expected addition product is contaminated with material resulting from addition of BuLi. We do not have a satisfactory explanation for this curious phenomenon, which is probably related to the formation and decomposition of ate complexes.

(9) Hutchinson, D. K.; Fuchs, P. L. *J. Am. Chem. Soc.* **1987**, *109*, 4930.

(10) Hayashi, K.; Iyoda, J.; Shiihara, I. *J. Organomet. Chem.* **1967**, *10*, 81.

(11) Theis, A. B.; Townsend, C. A. *Synth. Comm.* **1981**, *11*, 157.

enone (1.18 mmol). The mixture was stirred for 5 min, and boron trifluoride etherate (0.21 mL, 1.71 mmol) was added. The reaction mixture was stirred at −78 °C for 15 min followed by the addition of 50 mL of a 1:1 concentrated NH_4OH/saturated NH_4Cl solution. The mixture was extracted with methylene chloride, and the combined organic extracts were washed with brine and dried over anhydrous sodium sulfate. The solvent was removed in vacuo to give the crude product, which was purified by flash chromatography.

6-[(Methoxymethoxy)methyl]-2,2-dimethyl-3aβ,5,6α,6aβ-tetrahydro-4*H*-cyclopenta-1,3-dioxol-4-one: ^{1}H NMR ($CDCl_3$) δ 1.27 (s, 3 H), 1.35 (s, 3 H), 2.04 and 2.69 (ABX, J_{AB} = 18.02 Hz, J_{AX} = 9.25 Hz, J_{BX} = 1.71 Hz, 2 H), 2.53 (m, 1 H), 3.22 (s, 3 H), 3.55 (ABX, J_{AB} = 9.30 Hz, J_{AX} = 2.87 Hz, J_{BX} = 3.24 Hz, 2 H), 4.18 (d, J = 5.20 Hz, 1 H), 4.46 (AB q, J = 6.61 Hz, $\Delta\nu_{AB}$ = 11.4 Hz, 2 H), 4.60 (d, J = 5.33 Hz, 1 H); ^{13}C NMR ($CDCl_3$) δ 24.40, 26.52, 36.98, 37.24, 55.27, 68.99, 78.70, 81.32, 96.26, 111.10, 212.74. Anal. Calcd for $C_{11}H_{18}O_5$: C, 57.38; H, 7.88. Found: C, 57.22; H, 8.07.

3-[(Methoxymethoxy)methyl]cyclohexanone: ^{1}H NMR ($CDCl_3$) δ 1.30–1.64 (m, 2 H), 1.75–2.34 (m, 7 H), 3.22 (s, 3 H), 3.32 (d, J = 5.45 Hz, 2 H), 4.48 (s, 2 H); ^{13}C NMR ($CDCl_3$) δ 24.61, 27.80, 38.84, 41.05, 44.45, 54.18, 71.27, 96.18, 210.6. Anal. Calcd for $C_9H_{16}O_3$: C, 62.77; H, 9.36. Found: C, 62.92; H, 9.18.

***trans*-1-Acetyl-2-[(methoxymethoxy)methyl]cyclohexane**: ^{1}H NMR ($CDCl_3$) δ 1.20–1.88 (m, 8 H), 2.11 (s, 3 H), 2.28 (m, 1 H), 2.61 (m, 1 H), 3.27 (s, 3 H), 3.44 (d, J = 7.26 Hz, 2 H), 4.48 (s, 2 H); ^{13}C NMR ($CDCl_3$) δ 22.47, 24.04, 24.09, 27.35, 28.78, 37.45, 50.76, 55.14, 67.93, 96.48, 210.93. Anal. Calcd for $C_{11}H_{20}O_3$: C, 65.97; H, 10.06. Found: C, 66.18; H, 10.20.

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Registry No. 1, 27490-33-1; 5, 100045-83-8; Bu_3SnH, 688-73-3; $CH_2(COH_3)_2$, 109-87-5; $CH_3OCH_2OCH_2OCH_2SnBu_3$, 115384-51-5; $PhC(O)CH(CH_3)_2$, 611-70-1; $PhCH_2C(O)CH_2CH_3$, 1007-32-5; PhCHO, 100-52-7; $PhC(OH)(CH_2OCH_2OCH_3)CH(CH_3)_2$, 115384-54-8; $PhCH_2C(OH)(CH_2OCH_2OCH_3)CH_2CH_3$, 115384-55-9; $PhCH(OH)CH_2OCH_2OCH_3$, 115384-56-0; $CuBrMe_2S$, 54678-23-8; $LiCH_2OCH_2OCH_3$, 115384-62-8; cycloheptanone, 502-42-1; 2-methylcyclohexanone, 583-60-8; *cis*-3a,6a-dihydro-2,2-dimethyl-4*H*-cyclopenta-1,3-dioxol-4-one, 40269-54-3; 2-cyclohexene-1-one, 930-68-7; 1-(1-cyclohexen-1-yl)ethanone, 932-66-1; 1-[(methoxymethoxy)methyl]cycloheptanol, 115384-52-6; 1-[(methoxymethoxy)methyl]-2-methylcyclohexanol (isomer 1), 115384-53-7; 2,2-dimethyl-4-[(methoxymethoxy)methyl]-3aβ,6aβ-dihydro-4*H*-cyclopenta-1,3-dioxol-4α-ol, 115384-57-1; 6-[(methoxymethoxy)methyl]-2,2-dimethyl-3aβ,5,6α,6aβ-tetrahydro-4*H*-cyclopenta-1,3-dioxol-4-one, 115384-58-2; 3-[(methoxymethoxy)methyl]cyclohexanone, 115384-59-3; *trans*-1-acetyl-2-[(methoxymethoxy)methyl]cyclohexane, 115384-60-6; 1-[(methoxymethoxy)methyl]-2-methylcyclohexanol (isomer 2), 115384-61-7.

Regiospecific 1,4-Addition with Grignard-Derived Mixed Triorganozincate Reagents

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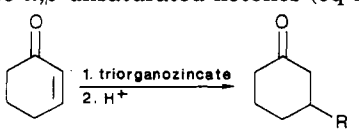
It has been previously reported that "triorganozincate" type reagents are convenient and synthetically useful reagents for transferring various organic moieties in a 1,4-fashion to α,β-unsaturated ketones (eq 1).[1-4] These



(1)

reports have involved four types of triorganozincate reagents[5]—R_3ZnLi,[1] RR'_2ZnLi,[2,3] R_3ZnMgX,[3,4] and RR'_2ZnMgX.[3] Methyl has been shown to be especially effective as the nontransferring or "dummy ligand" (R') in "mixed" (RR'_2) triorganozincates.[2,3]

We describe herein the results of our study of the 1,4-addition reactions with Grignard-derived mixed triorganozincate reagents. Although RMe_2ZnMgX type reagents were reported by Oshima and co-workers[3] during the course of our study, we are now prompted to publish our findings in view of the following differences. Our experiments were carried out at 0 °C rather than −78 °C, thus demonstrating one of the advantages of these reagents—thermal convenience. Also, our report includes enones other than 2-cyclohexene-1-one, and the yields of 1,2-addition products are given. Furthermore, the Grignard-derived mixed triorganozincates in the Oshima report were prepared by a procedure that involves two volumetric measurements (hexane/Me_2Zn solution[6] and Grignard solution) and which consequently requires prior knowledge of two concentrations. The Grignard-derived mixed triorganozincates reported herein were prepared from crystalline $ZnCl_2$·TMEDA (TMEDA = $Me_2NCH_2CH_2NMe_2$) by a procedure that involves only one volumetric measurement (the Grignard solution) and which consequently requires prior knowledge of only one concentration.

To further clarify this last point, it had been previously shown that triphenylmethane can be used to signal the end point in the formation of lithium triorganozincates (R_3ZnLi) by the addition of RLi to $ZnCl_2$ or $ZnCl_2$·TMEDA.[1] Disappointly, we had found that, when a Grignard reagent is used in place of an alkyllithium (i.e. in the preparation of R_3ZnMgX type reagents), the color change appears gradually during the addition of the 3 equiv of RMgX rather than sharply at the end of the addition of the third equivalent.[4] When preparing the Grignard-derived mixed triorganozincate reagents, we again faced this gradual color change. In order to take advantage of at least part of the convenience and accuracy of using an indicator, we arrived at the following procedure. An indicator (triphenylmethane) was used to prepare $^2/_3$ mmol of Me_3ZnLi, which was then converted to 1 mmol of Me_2Zn by adding $^1/_3$ mmol of $ZnCl_2$·TMEDA. This was followed by the addition of 1 mmol of RMgCl to give 1 mmol of the desired reagent (eq 2).

$$^2/_3ZnCl_2\cdot TMEDA \xrightarrow{2MeLi} {}^2/_3Me_3ZnLi \xrightarrow{^1/_3ZnCl_2\cdot TMEDA} Me_2Zn \xrightarrow{RMgCl} RMe_2ZnM\ (M = MgCl/Li) \quad (2)$$

(1) Isobe, M.; Kondo, S.; Nagasawa, N.; Goto, T. *Chem. Lett.* **1977**, 679-682.

(2) Watson, R. A.; Kjonaas, R. A. *Tetrahedron Lett.* **1986**, *27*, 1437-1440, and presented in part at the 19th Great Lakes Regional Meeting of the American Chemical Society, West Lafayette, IN, June 1985, No. 279.

(3) Tuckmantel, W.; Oshima, K.; Hozaki, N. *Chem. Ber.* **1986**, *119*, 1581-1593.

(4) Kjonaas, R. A.; Vawter, E. J. *J. Org. Chem.* **1986**, *51*, 3993-3996, and presented in part at the 19th Great Lakes Regional Meeting of the American Chemical Society, West Lafayette, IN, June 1985, No. 278.

(5) None of the experiments in this or any of the above referenced papers prove the existence of a discrete triorganozincate species. Formulas such as RMe_2ZnLi or $R_3ZnMgCl$ at the very least then, represent the stoichiometry involved in preparing the reagents. Spectroscopic evidence of one particular R_3ZnLi type species, however, has been reported, see: Waack, R.; Doran, M. A. *J. Am. Chem. Soc.* **1963**, *85*, 2861-2863.

(6) Solutions of Me_2Zn in ether, hexane, or any other solvent are not commercially available in the USA. Neat Me_2Zn (95%) is available from Alfa for $450/25 g. Two other companies offer higher purity material but at substantially higher prices. (Based on information given in *Chem Sources-USA*; Directories Publishing: Clemson, SC, 1987; and telephone calls to the companies listed.)

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