Evans, who recorded our mass spectra.

Registry No. 6, 87712-39-8; Ph₃SnH, 892-20-6; Ph₃SnOOCH, 47182-20-7; 4-methoxystyrene, 637-69-4; 4-nitrobenzaldehyde, 555-16-8; 4-chlorobenzaldehyde, 104-88-1; 4-methoxybenzaldehyde, 123-11-5; 3,7-dimethyl-2,6-octadienal, 5392-40-5; 3-phenyl-propionaldehyde, 104-53-0; 4-*tert*-butylcyclohexanone, 98-53-3; benzophenone, 119-61-9; 4-nitrobenzyl alcohol, 619-73-8; 4-chlorobenzyl alcohol, 873-76-7; 4-methoxybenzyl alcohol, 105-13-5; 3,7-dimethyl-2,6-octadien-1-ol, 624-15-7; 3-phenyl-1-propanol, 122-97-4; 4-*tert*-butylcyclohexanol, 98-52-2.

Transfer of Hydrogen from Alcohols. Catalysis by Compounds of Tin

James D. Wuest* and Boulos Zacharie

Département de Chimie, Université de Montréal, Montréal, Québec, H3C 3V1 Canada

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Equation 1 is a general representation of the transfers



of hydrogen that occur during Meerwein–Ponndorf–Verley reductions,¹ Cannizzaro reactions,² Grignard reductions,³ and other related processes.⁴ These precedents encouraged us to study the hypothetical reaction in equation 2, in which the transfer of hydrogen from tin formates to carbonyl compounds produces stannyl ethers and carbon dioxide. During our study of this reaction,⁵ we have made the following incidental observations.

$$\begin{array}{c} & & \\ & &$$

Although reaction 2 is slow under most conditions,⁵ we found that a wide variety of aldehydes were rapidly reduced to the corresponding alcohols in high yield at 115 °C in 1-butanol containing triphenyltin formate.⁶ These reductions did not produce measurable amounts of carbon dioxide, they failed to occur when the solvent was a tertiary alcohol, and they in fact required only catalytic amounts

(6) Ford, B. F. E.; Liengme, B. V.; Sams, J. R. J. Organomet. Chem. 1969, 19, 53-65. of triphenyltin formate. These observations indicated that the aldehydes were not being reduced by the tin formate but rather by a Meerwein–Ponndorf–Verley reaction involving 1-butanol and a catalyst containing tin.⁷ We did not attempt to detect butanal, the expected product of oxidation, but benzaldehyde was in fact formed when similar reactions were carried out in benzyl alcohol.

Further study showed that this Meerwein-Ponndorf-Verley reduction could be promoted efficiently by many (oxy)phenylstannanes, including triphenyltin acetate, triphenyltin formate, triphenyltin hydroxide, hexaphenyldistannoxane (Ph₃SnOSnPh₃), oxodiphenylstannane (Ph_2SnO) , and hydroxyoxophenylstannane $(PhSnO_2H)$.⁸ All of these compounds reacted with 1-butanol under conditions similar to those of our Meerwein-Ponndorf-Verley reductions (115 °C, 3 h, N₂) to give solutions of a substance that (1) contained no phenyl-tin bonds,⁹ (2) was converted into an insoluble oxide of tin by exposure to atmospheric moisture, and (3) was an active catalyst for the Meerwein-Ponndorf-Verley reduction. In contrast, use of triphenvltin chloride and tin tetrachloride did not lead to clean reductions. Furthermore, tetraphenyltin, oxodibutylstannane (Bu₂SnO), tricyclohexyltin hydroxide, trimethyltin formate,¹⁰ tin oxide, and simple metallic formates were not effective catalysts under the normal conditions and could be recovered unchanged from hot 1-butanol. We conclude that our Meerwein-Ponndorf-Verley reductions are preceded by a series of steps in which the (oxy)phenylstannanes are converted into catalytically active degradation products, presumably by exhaustive butanolysis of the phenyl-tin bonds.¹¹ (Oxy)alkylstannanes are ineffective precursors of this catalyst because alkyl-tin bonds are generally less easily cleaved than aryl-tin bonds.¹²

One logical product of degradation that would contain no phenyl groups, would be converted into oxides of tin by water,¹³ and that might be expected to catalyze the Meerwein–Ponndorf–Verley reaction⁷ is tin tetra-*n*-butoxide.¹³ However, treatment of representative aldehydes with catalytic or stoichiometric amounts of tin tetra-*n*butoxide¹³ under the conditions of our Meerwein–Ponndorf–Verley reductions (1-butanol, 115 °C, 3 h, N₂) produced only small amounts of the corresponding alcohols and significant amounts of the corresponding dibutyl acetals. In addition, the ¹¹⁹Sn NMR spectrum of tin tetra-*n*-butoxide (0.2 M in CDCl₃) shows a singlet near δ -612¹⁴ whereas the actual degradation product has no

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expt	alde t ko	hyde or etone	yi product	eld, ^a % p	procedu	re ^b
	1	сн30- С-сно	снзо-Снзо-снзо	H 76		
	2	сі{С	CI	^{)H} 85	A	Ĩ
	3			92		
	4	U2N-CHU	02N	79	В	
	5	ССНО	C CH2	он 83		1
	6	ССССНО	CCH2C	68		
	7	Альсни		он 67 2	- C	
	8	СН		₂ он 72		
	9	+	+он	62		
	10	02N CH3	02N CH	25		
	11	0°0		o	С	

Table I.Reductions of Representative Aldehydes and
Ketones by 1-Butanol in the
Presence of Compounds of Tin

^a Yield of isolated, chromatographically pure product. ^b (A) 115 °C for 3 h under N₂ in 1-butanol containing 2 mol of Ph₃SnOSnPh₃ per mol of aldehyde; (B) 115 °C for 4 h under N₂ in 1-butanol containing 0.17 mol of Ph₂SnO per mol of aldehyde; (C) 115 °C for 4 h under N₂ in 1butanol containing 2 mol of Ph₃SnOOCH per mol of aldehyde or ketone; (D) 115 °C for 24 h under N₂ in 1butanol containing 2 mol of Ph₃SnOOCH per mol of ketone.

well-defined absorptions betweeen δ 0 and -675. These discrepancies may arise because the two compounds are constitutionally identical but in different states of aggregation.¹⁴ However, it is more likely that the effective catalysts in our Meerwein-Ponndorf-Verley reductions are oligomeric tin alkoxides, similar to structure 1, in which bridging oxygen atoms are present.



Reductions of representative aldehydes and ketones using this catalyst are summarized in Table I, and the most noteworthy features of this procedure are described below.

a. Experiment 4 and related reactions demonstrate that only catalytic amounts of tin compounds are actually necessary, but to minimize reaction times, we have preferred to use 1 or 2 molar equiv. High solubility, commercial availability, and rapid conversion to the effective catalyst make triphenyltin hydroxide one of the most suitable (oxy)phenylstannanes. However, all of these compounds are useful and easy to handle. They are not very susceptible to hydrolysis, unlike alkoxides of aluminum or tin. Our procedure is therefore more convenient than other Meerwein-Ponndorf-Verley reductions catalyzed by tin alkoxides.⁷

b. Solvolysis of phenyl-tin bonds is not fast enough to allow convenient use of alcohols like ethanol or 2-propanol with boiling points much below that of 1-butanol. Benzyl alcohol, more easily oxidized than 1-butanol but also more difficult to remove from the products of reduction, offered no net advantage.

c. Even relatively reactive ketones are reduced significantly less rapidly than aldehydes. For example, when a mixture of 4-nitrobenzaldehyde (0.99 mmol) and 4nitroacetophenone (0.97 mmol) was subjected to the conditions of procedure A, the aldehyde was reduced to 4nitrobenzyl alcohol in 79% yield and the ketone was recovered in 69% yield. Our conditions may therefore offer useful selectivity.¹⁵ Meerwein-Ponndorf-Verley reductions under other conditions also offer some degree of selectivity,¹ of course, but our mild, nonbasic conditions seem to be particularly advantageous for the following reason: the uniformly high yields of alcohols obtained from enolizable aldehydes in experiments 6, 7, and 8 show that aldol condensations are not troublesome side reactions.

Experimental Section

All infrared (IR) spectra were recorded on a Perkin-Elmer Model 710B spectrometer. Brüker WH-90 or WH-400 spectrometers were used to obtain ¹H and ¹¹⁹Sn nuclear magnetic resonance (NMR) spectra. Chemical shifts for ¹¹⁹Sn are reported in parts per million downfield from external tetramethyltin (δ). Mass spectra were recorded at 70 eV on a V.G. Micromass 12-12 quadrupole spectrometer using electron impact (EI) or chemical ionization (CI) mass spectroscopy. Triphenyltin formate,⁶ hydroxyoxophenylstannane,⁸ trimethyltin formate,¹⁰ and tin tetra-*n*-butoxide¹³ were prepared by published procedures; all other reagents were commercial products of the highest purity obtainable.

The following general procedure is representative of the experiments summarized in Table I.

Reduction of 3-Phenylpropionaldehyde to 3-Phenyl-1propanol. A mixture of triphenyltin formate (79 mg, 0.20 mmol) and 3-phenylpropionaldehyde (13 mg, 0.097 mmol) in 1-butanol (2 mL) was warmed at 115 °C for 3 h under N₂. Volatiles were then removed by evaporation under reduced pressure, and the residue was exposed to atmospheric moisture. Soluble products were separated from precipitated oxides of tin by extraction with chloroform. Preparative thin-layer chromatography (silice gel, ethyl acetate/hexanes, 75:25) provided pure 3-phenyl-1-propanol (9.0 mg, 0.066 mmol, 68%), which was identical by NMR and IR with an authentic sample.

Acknowledgment. This work was financially supported by Research Corp., the Natural Sciences and Engineering Research Council of Canada, and le Ministère

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 α

de l'Education du Québec. We are grateful to Dr. M. T. Phan Viet, who recorded our high-field NMR spectra.

Registry No. 4-Methoxybenzaldehyde, 123-11-5; 4-chlorobenzaldehyde, 104-88-1; 4-nitrobenzaldehyde, 555-16-8; cinnamaldehyde, 104-55-2; benzenepropanal, 104-53-0; 3,4-dimethyl-2,6-octadienal, 5392-40-5; 3,7-dimethyl-6-octenal, 106-23-0; 4tert-butylcyclohexanone, 98-53-3; 4-nitroacetophenone, 100-19-6; benzophenone, 119-61-9; 4-methoxybenzenemethanol, 105-13-5; 4-chlorobenzenemethanol, 873-76-7; 4-nitrobenzenemethanol, 619-73-8; cinnamyl alcohol, 104-54-1; benzenepropanol, 122-97-4; 3,7-dimethyl-2,6-octadien-1-ol, 624-15-7; 3,7-dimethyl-6-octen-1-ol, 106-22-9; 4-tert-butylcyclohexanol, 98-52-2; α-methyl-4-nitrobenzenemethanol, 6531-13-1; 1-butanol, 71-36-3; Ph₃SnOSnPh₃, 1262-21-1; Ph₂SnO, 2273-51-0; Ph₃SnOCHO, 47182-20-7.

Preparation of α -Silylcarbanions¹

David J. Ager

Department of Chemistry, University of Toledo, Toledo, Ohio 43606

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It has already been shown that 1-(phenylthio)-1-(trimethylsilyl)alkanes (1) are masked aldehydes^{2,3} and, after reaction with lithium naphthalenide,^{4,5} gave olefins by reaction of the resultant α -silyl anion with a carbonyl compound.⁶ We now report the use of the lithium naphthalenide reaction for the preparation of substituted α -silylcarbanions, which are either difficult to prepare or are not available by other routes. The anions investigated were 1,1-bis(trimethylsilyl)-1-lithioalkanes (2), [tris(trimethylsilyl)methyl]lithium (3), 1-lithio-1-(phenylthio)-1-(trimethylsilyl)alkanes (4), and [bis(trimethylsilyl)-(phenylthio)methyl]lithium (5).

The addition of bis(trimethylsilyl)(phenylthio)methane (6) to lithium naphthalenide in THF at -78 °C gave [bis(trimethylsilyl)methyl]lithium (7),⁷ which in turn reacted with nonenolizable carbonyl compounds to give the vinylsilanes (8) in good yield (see Table I and Scheme I). With enolizable ketones, as Seebach found,⁸ the anion acted as a base and no condensation products were detected.⁹ This route has two advantages over the established method of deprotonation of bis(trimethylsilyl)methane as the synthesis of 6 is cheap and straightforward—the preparation of bis(trimethylsilyl)methane is either long or expensive—and the use of a strong base is alleviated.¹⁰⁻¹²

In a similar manner, the silanes 9 gave the 1-lithio-1,1-

Table I.	Prepa	ration	is and I	Reacti	ons of
Silylcarba	nions	with (Carbon	yl Cor	npounds

sulfide		carbonyl compd		
R1	R ²	R ³	R⁴	product yield, %
H	$SiMe_3$ (6)	Ph	Н	$(8) \ \overline{72} \ (70^8)$
Н	$SiMe_3$ (6)	Ph	Ph	$(8) 63 (65, 851^{18})$
H	SiMe ₃ (6)	-(CH	$I_{2})_{5}-$	(8) 0
Me	$SiMe_3$ (9)	Ph	H	(8) 69
Me	$SiMe_3$ (9)	Ph	Ph	(8) 57
Me	$SiMe_{3}(9)$	Н	н	(8) 71
n-Bu	SiMe ₃ (9)	Ph	н	(8) 62
n-Bu	SiMe, (9)	Ph	Ph	(8) 48
n-Bu	SiMe, (9)	н	H	(8) 73
n-Bu	SiMe, (9)	-(CH	I,),-	(8)0
Ph	SiMe, (9)	НÌ	Ή	(8) 66
SiMe,	SiMe, (10)	Ph	Н	(12) 68 (71 ⁸)
SiMe	$SiMe_{1}$ (10)	Ph	Ph	(12) 21 (25^{17})
SiMe	SiMe, (10)	Н	H	(12) 73 (70 ⁸)
SiMe	SiMe, (10)	-(CF	\mathbf{I}_{1}) -	(12)0
Н	SPh (11)	Ph	Η̈́	$(13)70(71, 1974^{20})$
H	SPh (11)	Ph	Ph	$(13)73(82.1978^{20})$
H	SPh (11)	-(CH	I.)	$(13) 61 (65, 19 68^{20})$
Me	SPh (11)	Ph	́́н	(13) 64
n-Bu	SPh (11)	Ph	н	(13) 66
n-Bu	SPh (11)	Ph	Ph	(13) 61
n-Bu	SPh (11)	Н	н	(13) 71
n-Bu	SPh (11)	<i>n</i> -Bu	Н	(13) 58
<i>n-</i> Bu	SPh (11)	-(CH	I,),-	(13) 51
SiMe,	SPh (14)	Ph	Ή	(15) 78 (76 ⁸)
SiMe	SPh (14)	Ph	Ph	(15) 49
SiMe	SPh (14)	Н	Н	(15) 80 (84 ^s)
	· · ·			

Scheme I

R LiNaph THF/-78 °C

1	= H;	$R^2 =$	SiMe ₃	(6)
1	11	D 1	, C'ÀA	(0)

$$A^{*} = aikyi; R^{*} = Siwe_{3}(9)$$

$$R^{1} = alkvl \cdot R^{2} = SPh \quad (11)$$

$$R^{1} = SiMe_{1}; R^{2} = SPh$$
 (14)



bis-(trimethylsilyl)alkanes 2, which in turn reacted with aldehydes and nonenolizable ketones to give the vinylsilanes 8 (see Table I and Scheme I). Similar results were obtained by Seebach when the anions were formed by the addition of alkyllithiums to 1,1-bis(trimethylsilyl)ethene.¹³

In an analogous manner, tris(trimethylsilyl)(phenylthio)methane (10) gave [tris(trimethyl]lithium $(3)^{14}$ when reacted with lithium naphthalenide, while 1,1-bis(phenylthio)-1-(trimethylsilyl)alkanes 11¹⁵ gave 1-lithio-1-(phenylthio)-1-(trimethylsilyl)alkanes 4. All of the anions reacted with carbonyl compounds as previously reported to give the adducts 12 or 13, respectively, in comparable yields.¹⁶⁻²⁰

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⁽¹⁾ This work was carried out at the Department of Organic Chemistry, Robert Robinson Laboratories, P.O. Box 147, Liverpool, L69 3BX, U.K.

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