yellow liquid: bp 98–99° (1.0 mm) [lit.[7] bp 103–104° (2.5 mm)]; ir (CCl_4) 2165 (C≡C), 1640 (C═), 1250 ($Si(CH_3)_3$), 865 ($Si(CH_3)_3$), 855 cm^{-1} ($Si(CH_3)_3$); nmr (CCl_4) δ 0.32 (s, 9, $Si(CH_3)_3$), 7.50 (m, 3, Ar H), 8.05 (m, 2, Ar H).

***p*-Chlorophenyl trimethylsilylethynyl ketone** was isolated as a pale yellow liquid: bp 105–106.5° (0.5 mm) [lit.[8b] bp 80–84° (10^{-3} mm)]; ir (CCl_4) 2160 (C≡C), 1645 (C═O), 1250 ($Si(CH_3)_3$), 865 ($Si(CH_3)_3$), 855 cm^{-1} ($Si(CH_3)_3$); nmr (CCl_4) δ 0.32 (s, 9, $Si(CH_3)_3$), 7.15 (d, 2, J = 9 Hz, Ar H), 7.75 (d, 2, J = 9 Hz, Ar H).

***p*-Methylphenyl trimethylsilylethynyl ketone** was isolated as a pale yellow liquid: bp 110–112° (0.75 mm) [lit.[8b] bp 74–78° (10^{-3} mm)]; ir (CCl_4) 2170 (C≡C), 1640 (C═O), 1250 ($Si(CH_3)_3$), 865 ($Si(CH_3)_3$), 855 cm^{-1} ($Si(CH_3)_3$); nmr (CCl_4) δ 0.30 (s, 9, $Si(CH_3)_3$), 2.28 (s, 3, Ar CH_3), 6.92 (d, 2, J = 8 Hz, Ar H), 7.62 (d, 2, J = 8 Hz, ArH).

Methyl trimethylsilylethynyl ketone was isolated as a colorless liquid: bp 62–64° (28 mm) [lit.[7] bp 51.5° (12 mm)]; ir (CCl_4) 2160 (C≡C), 1675 (C═O), 1255 ($Si(CH_3)_3$), 870 ($Si(CH_3)_3$), 855 cm^{-1} ($Si(CH_3)_3$); nmr (CCl_4) δ 0.25 (s, 9, $Si(CH_3)_3$), 2.25 (s, 3, $C(O)CH_3$).

***n*-Pentyl trimethylsilylethynyl ketone**[12] was isolated as a colorless liquid: bp 74–75° (1.0 mm); ir (CCl_4) 2160 (C≡C), 1670 (C═O), 1250 ($Si(CH_3)_3$), 870 ($Si(CH_3)_3$), 855 cm^{-1} ($Si(CH_3)_3$); nmr (CCl_4) δ 0.24 (s, 9, $Si(CH_3)_3$), 0.70–1.90 (cm, 9), 2.38 (t, 2, J = 6 Hz, $C(O)CH_2$).

Isopropyl trimethylsilylethynyl ketone[12] was isolated as a colorless liquid: bp 78.2–81.8° (17 mm); ir (CCl_4) 2160 (C≡C), 1670 (C═O), 1255 ($Si(CH_3)_3$), 865 ($Si(CH_3)_3$), 855 cm^{-1} ($Si(CH_3)_3$); nmr (CCl_4) δ 0.28 (s, 9, $Si(CH_3)_3$), 1.16 (d, 6, J = 7 Hz, $CH(CH_3)_2$), 2.50 (heptet, 1, J = 7 Hz, $CH(CH_3)_2$).

Acknowledgment. Acknowledgment is made to the donors of the Petroleum Research Fund, administered by the American Chemical Society, for support of this research.

Registry No.—**1**, 53210-13-2; RCOCl (R = CH_3), 75-36-5; RCOCl [R = $CH_3(CH_2)_4$], 142-61-0; RCOCl [R = $(CH_3)_2CH$], 79-30-1; RCOCl (R = C_6H_5), 98-88-4; RCOCl (R = p-$CH_3C_6H_4$), 874-60-2; RCOCl (R = p-ClC_6H_4), 122-01-0; cuprous *tert*-butoxide, 35342-67-7; trimethylsilylacetylacetylene, 1066-54-2; phenyl trimethylsilylethynyl ketone, 13829-77-1; p-chlorophenyl trimethylsilylethynyl ketone, 37166-46-4; p-methylphenyl trimethylsilylethynyl ketone, 37166-45-3; methyl trimethylsilylethynyl ketone, 5930-98-3; n-pentyl trimethylsilylethynyl ketone 53210-14-3; isopropyl trimethylsilylethynyl ketone, 53210-05-2.

References and Notes

(1) D. R. M. Walton, "Protective Groups in Organic Chemistry," J. F. W. McOmie, Ed., Plenum Press, London, 1973, pp 5–8.

(2) C. E. Castro, E. J. Gaughn, and D. C. Owsley, *J. Org. Chem.*, **31**, 4071 (1966).

(3) R. Oliver and D. R. M. Walton, *Tetrahedron Lett.*, 5209 (1972), have suggested that **1** might be an intermediate in the formation of $Me_3SI(C{\equiv}C)_2SiMe_3$ from $LiC{\equiv}CSiMe_3$ and $IC{\equiv}CSiMe_3$ in the presence of cuprous halides.

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(5) Cuprous *tert*-butoxide was not isolated as in ref 4 but was prepared *in situ* by the action of lithium *tert*-butoxide on cuprous iodide.

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(8) (a) L. Birkofer, A. Ritter, and H. Uhlenbrauck, *Chem. Ber.*, **96**, 3280 (1963); (b) D. R. M. Walton and F. Waugh, *J. Organometal. Chem.*, **37**, 45 (1972).

(9) Trimethylsilylethynyl ketones are easily desilylated with an aqueous Borax solution to give ethynyl ketones; see ref 8b for details.

(10) All boiling points are uncorrected. Ir spectra were recorded on a Perkin-Elmer Model 257 spectrophotometer. Nmr spectra were recorded on a Hitachi Perkin-Elmer Model R-20A spectrometer with tetramethylsilane as an internal standard. All column chromatography was performed on Grace Davison 950 silical gel. The acid chlorides used, except for caproyl chloride, were commercial samples that were distilled prior to use. Tetrahydrofuran was distilled from $LiAlH_4$. Cuprous iodide was obtained from Alfa Inorganics and dried in *in vacuo* at 130°. Lithium *tert*-butoxide was prepared by the action of anhydrous *tert*-butyl alcohol on 1 equiv of lithium metal suspended in anhydrous ether followed by the removal of the ether on a rotary evaporator.

(11) N. V. Komarov and O. G. Yarosh, *Zh. Obshch. Khim.*, **37**, 264 (1967), *Chem. Abstr.*, **66**, 95126 (1967).

(12) A satisfactory combustion analysis could not be obtained due to contamination with a small amount of the desilylated ketone whose presence was detected by the appearance of 3300 and 2105 cm^{-1} bands in the ir spectrum. It appears that the trimethylsilyl group is partially removed during attempted purifications.

Alkyllithium Additions to Allylic Alcohols

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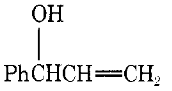
Received May 30, 1974

In some previous work, we had established that α-vinylbenzyl alcohol (**1**) can react in a variety of ways when treated with alkyllithium reagents. Treating **1** with n-butyllithium in tetrahydrofuran (THF) gave good yields of propiophenone (**2**), by way of a dianion intermediate (**3**).[2] In contrast to this result, the same starting materials in the presence of N,N,N',N'-tetramethylethylenediamine (TMEDA) and with hexane as the solvent gave the alcohol **4** (R = n-Bu) and saturated compound **5** (R = n-Bu) in a 3:1 ratio, and no ketone.[3] The alcohol, which is produced in a highly stereospecific manner, presumably arises by addition of n-butyllithium to the internal end of the double bond giving rise to intermediate **6** (R = n-Bu), which upon hydrolysis affords **4**. The saturated product **5** more than likely arises by way of addition of n-butyllithium to the terminal end of the double bond, giving rise to **7** (R = n-Bu) which eliminates Li_2O to **8** (R = n-Bu) and subsequently undergoes a second addition of n-butyllithium to the conjugated double bond.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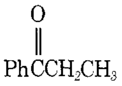
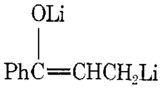
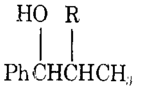
1


$Ph\overset{O}{\overset{\|}{C}}CH_2CH_3$

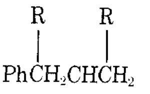
2


$Ph\overset{OLi}{\overset{|}{C}}{=}CHCH_2Li$

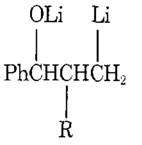
3


$Ph\overset{HO}{\overset{|}{C}}H\overset{R}{\overset{|}{C}}HCH_3$

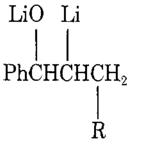
4


$PhCH_2\overset{R}{\overset{|}{C}}H\overset{R}{\overset{|}{C}}H_2$

5


$Ph\overset{OLi}{\overset{|}{C}}H\underset{\underset{R}{|}}{CH}\overset{Li}{\overset{|}{C}}H_2$

6


$Ph\overset{LiO}{\overset{|}{C}}H\overset{Li}{\overset{|}{C}}H\underset{\underset{R}{|}}{CH_2}$

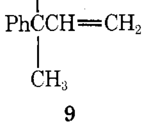
7

$PhCH{=}CHCH_2R$

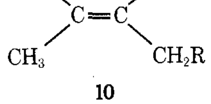
8

Treatment of α-vinylbenzyl alcohol (**1**) with *tert*-butyllithium in the presence of TMEDA and hexane gave 23% ketone (**2**), 9% *cis*-**8** (R = t-Bu), 36% *trans*-**8** (R = t-Bu), and 32% **5** (R = t-Bu).[3] Consequently, in the case of *tert*-butyllithium the only addition products that result come from addition to the terminal end of the double bond and not the internal end. These differences led us to investigate the addition of n- and *tert*-butyllithium to two other homologous alcohols.

In our previous work, we reported that 2-phenyl-3-buten-2-ol (**9**) did not show any addition or rearrangement products when treated with excess n-butyllithium. However, it has now been found (after several trials) that overnight refluxing of a solution of **9**, 3 equiv of n-butyllithium, and 1 equiv of TMEDA in the solvent hexane resulted in the production of two new compounds, together with a considerable amount of unreacted starting material remaining.


$Ph\overset{OH}{\overset{|}{C}}\underset{\underset{CH_3}{|}}{}CH{=}CH_2$

9



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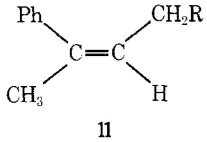

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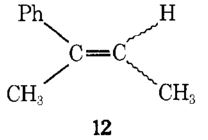


12

Table I
Spectral Data of Some Selected Substituted Styrenes

Compd	Uv (EtOH)		Nmr (CCl_4),[a] ppm				
	λ_{max}	ϵ_{max}	=CH	α-CH_3	β-allylic	γ-CH_3	Other–CH_2–
12-*cis*	247	12,800	5.75 (q of q)	1.98 (pentet)	1.80 (CH_3) (q of d)		
12-*trans*	238	9,500	5.50 (q of q)	2.00 (pentet)	1.55 (CH_3) (q of d)		
10, R = *n*-Bu	244	9,500	5.70 (t)[b]	2.01 (s)	2.1 (CH_2) (m)	0.92 (t)[c]	1.1–1.8 (m)
11, R = *n*-Bu	235	5,300	5.40 (t)[b]	2.00 (d)	<2.0 (CH_2)	0.85 (t)[c]	1.0–2.0 (m)
10, R = *t*-Bu	244	10,100	5.80 (t)	2.01 (s)	2.08 (CH_2) (d)	0.98 (s)	
11, R = *t*-Bu	233	6,000	5.50 (q of t)	2.04 (d)	1.83 (CH_2) (d)	0.83 (s)	

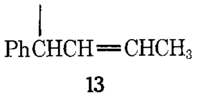
[a] All spectra integrated correctly, in accordance with the structural assignments. The aromatic signals were multiplets in the 6.8–7.5-ppm region in each case. [b] Complex. [c] Distorted.

The two new compounds were identified as *cis*- and *trans*-2-phenyl-2-octene (**10** and **11**, R = *n*-Bu), 15% and 10%, respectively.

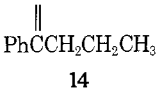
The identity of the hitherto uncharacterized olefins rests on spectra data. The mass spectra of both compounds were very similar; both display molecular ions at *m/e* 188 and base peaks at *m/e* 131, corresponding to the allylic ion which would result upon cleavage of the C-4–C-5 bond. The nmr and uv data are given in Table I. A comparison to the values known for 2-phenyl-2-butene (**12**), cis and trans, is also presented.[4,5] As can be seen from the data, the nmr signals of β-alkyl groups cis to the phenyl group are shifted upfield, while the β-vinyl protons cis to the phenyl group are shifted downfield. The uv data are consistent with the general observation that a cis β-alkyl group produces a weaker, shorter wavelength maximum than the trans β-alkyl group.[6]

The reaction of *tert*-butyllithium with alcohol **9** gave results analogous to those of the *n*-butyllithium reaction. Treatment of **9** with 2 equiv of *tert*-butyllithium and 1.5 equiv of TMEDA in hexane afforded about 70% starting alcohol, 15% of *cis*-2-phenyl-5,5-dimethyl-2-hexane (**10**, R = *t*-Bu), and 15% of *trans*-2-phenyl-5,5-dimethyl-2-hexane (**11**, R = *t*-Bu). The mass spectra of both olefin products showed molecular ions at *m/e* 188, base peaks at *m/e* 131 (allylic carbonium ion), McLafferty rearrangement ions at *m/e* 132, and an abundance of low molecular weight aliphatic ions. The nmr and uv data were consistent with the proposed structures (Table I).

The reaction of *n*-butyllithium with 1-phenyl-2-buten-1-ol (**13**) produced only small amounts of butyrophenone (**14**), starting alcohol, and possibly polymerization products using either THF or hexane–TMEDA.


$\mathrm{Ph}\overset{\mathrm{OH}}{\overset{|}{\mathrm{C}}}\mathrm{HCH{=}CHCH_3}$

13


$\mathrm{Ph}\overset{\mathrm{O}}{\overset{\|}{\mathrm{C}}}\mathrm{CH_2CH_2CH_3}$

14

In summary, the reaction of alkyllithium reagents with 2-phenyl-3-buten-2-ol (**9**) produces olefins of the type **10** and **11**, which suggests initial attack of the RLi on the external end (C-4) of the allylic system, followed by elimination of Li_2O. Although the minor products in these reactions could not be isolated, it seems reasonable to conclude that little attack occurred at C-3, in comparison to C-4. The poor yields and change in the position of attack on the allylic systems (comparing *n*-butylation of **1** and **9**) seems to indicate that these reactions are influenced considerably by steric factors. While *n*-butyllithium adds in good yield to the simple alcohol **1** in hexane–TMEDA, the additional methyl present in allylic alcohol **13** seems to retard addition reactions in favor of a weak dianion reaction, giving ketone **14**.

Experimental Section

***n*-Butylation of 2-Phenyl-3-buten-2-ol (9).** To a small three-necked, round-bottomed flask fitted with a dropping funnel, nitrogen gas inlet, drying tube, and stirrer was added 2.6 g (17.5 mmol) of 2-phenyl-3-buten-2-ol (**9**),[7] about 25 ml of hexane, and 2.0 g (17.5 mmol) of TMEDA. The solution was cooled with ice, and 30 ml (66 mmol) of a 2.2 *M* solution of *n*-butyllithium in hexane was added in a rapid dropwise fashion. The reaction mixture was allowed to warm to room temperature and stirred at reflux overnight. After quenching with water, the organic phase was separated, dried ($MgSO_4$), and evaporated under vacuum at room temperature.

Analysis by vpc (6-ft SE 30 column (¼ in). at 140°) showed seven components, several of which were quite minor. An nmr spectrum of this crude product showed methyl signals in the δ 2.0 region and vinyl protons at δ 5.4 and 5.7, indicative of products **10** and **11**. By use of preparative vpc (8-ft SE 30 column (1 in.) at 160°), three components were isolated. The first—low retention time—component (65%) was identical with the starting material **9** in its retention time and nmr and mass spectra. The second—moderate retention time—component (10%) was *trans*-2-phenyl-2-octene (**11**, R = *n*-Bu).[5] The third—long retention time—component (15%) was *cis*-2-phenyl-2-octene (**10**, R = *n*-Bu).[5]

***tert*-Butylation of 2-Phenyl-3-buten-2-ol (9).** Using a procedure similar to that above with 3.7 g (25 mmol) of **9**, 4.34 g (38 mmol) of TMEDA, 50 mmol of *n*-butyllithium, and about a total of 100 ml of hexane, the reaction mixture was refluxed overnight and worked up as before. The crude product was vacuum distilled, 75–85° (1.5 mm), and analyzed by both vpc (6-ft SE 30 column (¼ in.) at 140°) and nmr. The distilled product consisted of a mixture 70% starting alcohol **9** and 15% each of two other components of longer retention time.

The distilled product was separated by preparative vpc. The first component collected (70%) was identical in retention time and nmr and mass spectra with **9**. The second component (15%) was *trans*-2-phenyl-5,5-dimethyl-2-hexene (**11**, R = *t*-Bu).[5] The third—long retention time—component was *cis*-2-phenyl-5,5-dimethyl-2-hexene (**10**, R = *t*-Bu).[5]

Rearrangement of 1-Phenyl-2-buten-1-ol (13). In a procedure similar to above, 5.5 g (37 mmol) of **13**,[8] 4.3 g (37 mmol) of TMEDA, 80 mmol of *n*-butyllithium, and a total volume of about 100 ml of hexane were refluxed overnight. The crude product was analyzed by vpc at this point and showed only one major product—different from the starting material. The product was vacuum distilled and four fractions in the range 70° (1.5 mm)–140° (0.2 mm) were collected. The vpc traces of all the fractions were nearly the same, consisting of one major peak, which had the same retention time as butyrophenone (14),[9] and several minor peaks of intensity of 10–20%. The nmr spectra of various fractions were also similar, showing the characteristic pentet at δ 1.65, triplet at δ 1.80, and ortho aromatic protons at δ 7.85 of butyrophenone together with variable strong absorptions in the aromatic (δ 7.0–7.4), aliphatic (δ 0.8–1.4), and vinyl (δ 5–6.3) regions. The clean-looking vpc trace yet a somewhat messy nmr could possibly be explained by the existence of polymeric products which might arise during reaction, distillation, and/or chromatography. The starting alcohol **13** is fairly susceptible to dehydration and polymerization, as we found out in some of our preparations. The butyllithium could promote a base-catalyzed elimination of water and polymerization of **13**.

The reaction was repeated as before except the TMEDA was left out and THF was added in its place, so that the solvent system consisted of a mixture of hexane and THF. Analysis of the crude product by vpc (SE 30) showed, again, only one outstanding peak of retention time identical with that of authentic butyrophenone. However, the nmr of the crude product showed by overlapping comparisons to authentic samples that it was about a 1:1 mixture of alcohol **13** (which was not showing up to any great extent on the vpc possibly due to polymerization on the column) and ketone **14**, together with additional aromatic and aliphatic signals.

Registry No.—**9**, 6051-52-1; **10** (R = *n*-Bu), 53109-16-3; **10** (R = *t*-Bu), 37887-25-5; **11** (R = *n*-Bu), 53109-17-4; **11** (R = *t*-Bu), 37887-26-6; *cis*-**12**, 768-00-3; *trans*-**12**, 767-99-7; **13**, 3347-57-7; **14**, 495-40-9.

References and Notes

(1) To whom correspondence should be addressed at the Synthetics Department, Research Center, Hercules, Inc., Wilmington, Del. 19899.

(2) D. R. Dimmel and S. B. Gharpure, *J. Amer. Chem. Soc.*, **93**, 3991 (1971).

(3) D. R. Dimmel and S. Huang, *J. Org. Chem.*, **38**, 2756 (1973).

(4) Samples of these compounds were kindly supplied to us by Dr. M. A. McKinney (Marquette University). The olefins were prepared by dehydration of 2-phenyl-2-butanol according to the procedure of J. H. Rolston and K. Yates, *J. Amer. Chem. Soc.*, **91**, 1469 (1969). The olefins, present in a 3:1 ratio, were separated by preparative vpc from the terminal olefin. The major olefin is reported to be the cis isomer: J. B. Hendrickson, D. J. Cram, and G. S. Hammond, "Organic Chemistry," 3rd ed, McGraw-Hill, New York, N.Y., 1970, p 365. The major olefin possessed an ir spectrum identical with that of *cis*-2-phenyl-2-butene (Sadler, Vol. I, 1619).[5] The minor olefin possessed an ir spectrum identical with that of *trans*-2-phenyl-2-butene (Sadler, Vol. I, 1618).[5]

(5) Spectral data given in Table I.

(6) W. N. White, *et al.*, *J. Amer. Chem. Soc.*, **80**, 3271 (1958).

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(9) Purchased from Aldrich Chemical Co., Milwaukee, Wis.

Zinc Chloride Catalysis in the Reaction of Thionyl Halides with Aliphatic Alcohols

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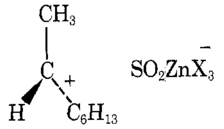
Received June 8, 1974

Since our report[2] that zinc chloride–thionyl chloride (**1**) easily converts 2,3,4,6-tetra-*O*-benzyl-α-D-glucopyranose (**2**) to 2,3,4,6-tetra-*O*-benzyl-α-D-glucopyranosyl chloride (**3**), we have extended the use of reagent **1** to aliphatic systems. Since rapid, easy, high-yield, and high-purity synthesis of **3** was a significant breakthrough for carbohydrate chemists, we have documented the effectiveness of **1** with representative alcohols. We report here (1) clear proof that zinc chloride catalyzes the reaction of thionyl chloride with alcohols (Table I), (2) comparison of zinc chloride with pyridine as a catalyst, and (3) the yield and stereochemical result of converting optically active 2-octanol (**4**) to its chloride and bromide with the appropriate thionyl halide (Table II).

In addition to **4**, the reagent **1** was allowed to react with the following alcohols: 1-butanol (**5**), 2-butanol (**6**), 2-methyl-2-propanol (**7**), and cyclohexanol (**8**). In Table I we show that reagent **1** readily converts each of these alcohols to product, under conditions where thionyl chloride alone gives little or no substitution. These data also document the synthetic utility of reagent **1** (runs 5, 8, 11, and 14). The yields of isolated products are acceptable, and no effort was made to optimize them.

The 2-chlorobutane produced from **5** (runs 4 and 5) is a primary reaction product, for 1-chlorobutane does not isomerize when treated with **1** for 2 days. This product mixture from **5** has been previously reported,[3] and led us to seek a catalyst to suppress the formation of the rearrangement product, 2-chlorobutane. Since pyridine has been widely used as a catalyst with thionyl chloride,[4] we investigated it with alcohols **5** and **6**. With **5**, pyridine was superior to zinc chloride; no rearrangement was detected by vpc (although 55% product formed in 2 days). With alcohol **6**, pyridine gave lower initial and overall conversion than was attained with reagent **1**. Attempts to combine zinc chloride and pyridine as a mixed catalyst were unsuccessful; lower conversion to product was generally observed than with either catalyst used alone.

For stereochemical studies (Table II), commercial 2-octanol (**4**) was resolved and purified according to the method of Kenyon.[5] Zinc chloride with either thionyl chloride or bromide in benzene or dioxane solution was an effective catalyst. At the concentration employed here, thionyl bromide slowly converts **4** to 2-bromooctane, but even this relatively easy reaction was greatly aided by zinc chloride. In every case where zinc chloride was used as a catalyst, the product was less optically pure than the starting **4** (*e.g.*, runs 15 and 16). This fact, along with the rearrangement product observed when reacting **5** with the reagent **1**, suggests that, at some time during the reaction, at least partial symmetry is attained by the cation. Boozer and Lewis appear to be the first to propose that ions are involved in the mechanism of transforming alcohols to alkyl chlorides.[6] We find their mechanism compatible with our data, by assuming that zinc chloride complexes at one or more stages of the reaction, makes some reaction species more ionic, and in this manner speeds the reaction. The structure **9** is a representation of a possible intermediate



9, X = Cl or Br

when **1** converts **4** to the chloride. Solvation of such an intermediate could explain the effect of dioxane on the reaction of **1** with optically active **4**. We can neither support nor reject the "ion pair" hypothesis of Sneen for solvolytic reactions.[7]

In conclusion, we have demonstrated that zinc chloride catalysis can be of value in converting alcohols to the corresponding chloride or bromide. As in the previously cited example of the conversion of **2** → **3**,[2] this can be especially important when low acidity, mild temperature reaction conditions are desired.

Experimental Section

General Procedures. All boiling points are uncorrected. All reagents were the best commercial grade available, dried and/or distilled before use, and stored appropriately so as to prevent contamination. Reaction analysis was done on the Varian 90P-3 gas chromatograph (vpc) with the Model 244 disk-chart integrator for