tetrahydropyran derivatives resulting from intramolecular electrophilic cyclization. The one example of this type which we report here presented no problem, and we were able to obtain a respectable yield of the homoallylic chloromethyl ether **1h**. These results, along with similar results from our laboratories with other homoallylic MOM ethers, are particularly interesting in view of the recent publication by Thompson et al.,[11] in which they report exclusive, highly stereoselective tetrahydropyran formation from the $TiCl_4$-mediated reaction of homoallylic MEM and ethyl vinyl ether acetals. Our procedure appears to be very useful for the preparation of chloromethyl ether derivatives of small, strained carbocyclic alcohols (entries k and l) which gave mostly formal products (i.e., $ROCH_2OR$) via reaction 1. Benzylic chloromethyl ethers are also conveniently prepared by this procedure and are generally obtained in better yield than by literature methods[2] (entries b–e). Finally, the last three entries in Table I list examples in which the procedure either failed or worked poorly. In these last examples, cleavage of the MOM ether occured with undesired regioselectivity and gave mostly or exclusively chloromethyl methyl ether.

In conclusion, the cleavage of MOM ethers with BCl_3 provides a convenient, versatile route to chloromethyl ether derivatives. The procedure complements existing methodology in that it avoids the use of protic acid and provides a route to chloromethyl ether derivatives previously unobtainable via the classical route 1.

Experimental Section

All proton nuclear magnetic resonance (NMR) spectra were recorded on a Varian EM 360 spectrometer in either chloroform-*d* or carbon tetrachloride solvent with shift values reported in δ units relative to tetramethylsilane as internal reference. All starting alcohols for the preparation of the MOM ethers were commercially available materials and were used as purchased. Tetrahydrofuran was distilled from benzophenone ketyl before use. All other solvents were reagent grade. **Caution**: Chloromethyl ethers are powerful alkylating agents and potentially carcinogenic. Due care should be exercised when working with these materials.

General Procedure for the Preparation of MOM Ethers 2. The following general procedure was used to prepare all MOM ethers.

To a stirring suspension of NaH (0.11 mol, free from oil) in 100 mL of dry THF under N_2 was added the alcohol (0.10 mol) dropwise in 20 mL of THF. The mixture was then stirred until hydrogen evolution had subsided and then heated at reflux for 2 h. After the mixture was cooled in an ice bath, there was then added chloromethyl methyl ether (0.10 mol, Aldrich) dropwise, the ice bath removed, and stirring continued at room temperature for a minimum of 1 h. The mixture was then filtered through Celite and the solvent removed either by distillation through a Vigreux column at atmospheric pressure or by rotary evaporation depending on the volatility of the product. The MOM ethers were then purified by distillation (see Table I).

General Procedure for the Preparation of Chloromethyl Ethers 1. The following general procedure was used to prepare the chloromethyl ethers **1** from their corresponding MOM ethers **2**.

To an ice-cooled, stirring mixture of the MOM ether (0.06 mol) in pentane or dichloromethane (35 mL) was added a solution of 1 M BCl_3 in hexanes (20 mL, 0.02 mol, Aldrich). After being stirred in the cold for 15 min, the mixture was then warmed to room temperature and allowed to stir for 2 h. Proton NMR analysis after this time usually indicated complete reaction. The mixture was then concentrated under reduced pressure, and the chloromethyl ether derivatives were purified by distillation (see Table I).

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Registry No. **1a**, 40556-01-2; **1b**, 104620-66-8; **1c**, 3587-60-8; **1d**, 104620-67-9; **1e**, 104620-68-0; **1f**, 104620-69-1; **1g**, 58558-40-0; **1h**, 104620-70-4; **1i**, 104620-71-5; **1j**, 104620-72-6; **1k**, 104620-73-7; **1l**, 104620-74-8; **1m**, 58567-17-2; **1n**, 55812-22-1; **1o**, 104620-75-9; **1p**, 104620-76-0; **1r**, 90331-98-9; **2a**, 24209-75-4; **2b**, 104620-55-5; **2c**, 31600-55-2; **2d**, 91764-46-4; **2e**, 104620-56-6; **2f**, 91970-13-7; **2g**, 90344-72-2; **2h**, 104620-57-7; **2i**, 104620-58-8; **2j**, 104620-59-9; **2k**, 104620-60-2; **2l**, 104620-61-3; **2m**, 42604-11-5; **2n**, 17869-83-9; **2o**, 104620-62-4; **2p**, 104620-63-5; **2q**, 824-91-9; **2r**, 104620-64-6; **2s**, 104620-65-7; *p*-$FC_6H_4CH_2OH$, 459-56-3; $PhCH(OH)CH_2CH_2CH_3$, 614-14-2; $PhCH{=}CHCH_2OH$, 104-54-1; $CH_2{=}C(CH_3)CH_2OH$, 513-42-8; $CH_3CH_2CH{=}CHCH_2CH_2OH$, 544-12-7; $CH_3CH_2CH_2CH(OH)CH{=}CH_2$, 4798-44-1; $CH_3CH_2CH_2CH(OH)C{\equiv}CH$, 105-31-7; $CH{\equiv}CC(CH_3)_2OH$, 115-19-5; $(CH_3)_3CC(CH_3)_2OH$, 594-83-2; $PhC{\equiv}CCH_2OH$, 1504-58-1; $CF_3CF_2CF_2CH_2OH$, 375-01-9; $(CH_3)_3COH$, 75-65-0; $PhCH_2OH$, 100-51-6; C_6H_5OH, 108-95-2; CH_3OCH_2Cl, 107-30-2; BCl_3, 10294-34-5; cyclobutanol, 2919-23-5; cyclooctanol, 696-71-9; α-cyclohexybenzyl alcohol, 4397-01-7; (hydroxymethyl)cyclopropane, 2516-33-8; 1-methylcyclopentanol, 1462-03-9.

Palladium-Catalyzed Cyanation of Aryl Halides by Trimethylsilyl Cyanide[1]

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Among the various methods available for the synthesis of aryl cyanides, the most convenient one is that based on the displacement of aromatic bromides or iodides by cyanide anion. It is well-known that the stoichiometric reaction of aryl halides with Cu(I)CN gives corresponding cyanides.[2] The catalytic reaction of aryl halides with KCN (or NaCN) in the presence of a palladium complex at elevated temperatures affords aryl cyanides in good to excellent yields.[3] Nickel complexes have also been found effective for cyanation of aryl halides.[4] In recent years, trimethylsilyl cyanide, Me_3SiCN (1), has been widely used in organic synthesis as a useful reagent for cyanation.[5] In this paper, we describe a new synthetic method for aryl cyanides based on a palladium-catalyzed reaction of aryl halides with **1** (eq 1).[6]

$$\text{Ar-I} \xrightarrow[\text{Pd catalyst}]{\text{Me}_3\text{SiCN}} \text{Ar-CN} \qquad (1)$$

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Table I. Effect of Solvent on Pd-Catalyzed Cyanation of Iodobenzene (2a) by Trimethylsilyl Cyanide (1)

entry	solvent	temp	time	yield,[a] %
1	Et_3N	15 °C	1 day	15
2	Et_3N	40 °C	1 h	40
			1 day	76
3	Et_3N	reflux	0.5 h	98 (88)
4	pyridine	reflux	1 day	2
5	dioxane	90 °C	1 day	2
6	DMF	90 °C	1 day	6
7	$PhCH_3$	90 °C	1 day	54
8	CH_3CN	reflux	1 day	8

[a] GLC yields based on iodobenzene. Yields in parentheses are isolated yield.

Table II. Pd-Catalyzed Cyanation of Aryl Iodide by Trimethylsilyl Cyanide (1)[a]

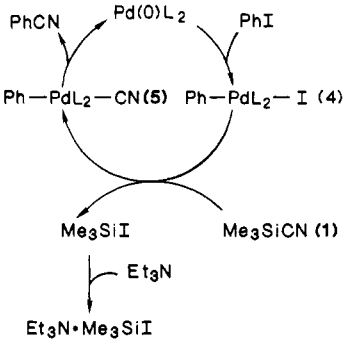
entry	iodide	cyanide	yield,[b] %
1	C_6H_5I (**2a**)	C_6H_5CN (**3a**)	88
2[c]	C_6H_5Br	**3a**	0
3[c]	C_6H_5Cl	**3a**	0
4	2-$CH_3C_6H_4I$ (**2b**)	2-$CH_3C_6H_4CN$ (**3b**)	76
5	4-$CH_3OC_6H_4I$ (**2c**)	4-$CH_3OC_6H_4CN$ (**3c**)	89 (73)[d]
6	4-ClC_6H_4I (**2d**)	4-ClC_6H_4CN (**3d**)	70
7	4-BrC_6H_4I (**2e**)	4-BrC_6H_4CN (**3e**)	56
		4-CNC_6H_4CN (**3f**)	15
8[c,e]	**2e**	**3e**	58
		3f	13
9[e]	4-IC_6H_4I (**2f**)	**3f**	53
10	4-$CH_3OOCC_6H_4I$ (**2g**)	4-$CH_3OOCC_6H_4CN$ (**3g**)	68

[a] Aryl iodide, Me_3SiCN, and $Pd(PPh_3)_4$ (1:1.5:0.02 molar ratio) in Et_3N were allowed to react under reflux for 10–30 min. [b] Yields are for isolated, purified products, based on starting halide. [c] For 1 day. [d] 50-mmol scale (see Experimental Section). [e] Me_3SiCN (5 equiv) was used.

The reaction of iodobenzene (**2a**) with **1** in the presence of $Pd(PPh_3)_4$ resulted in cyanation affording benzonitrile (**3a**). The choice of the reaction solvent is very important. The use of triethylamine as the solvent gave the best result (Table I).

The results of catalytic cyanation of various aryl halides are listed in Table II. Bromobenzene and chlorobenzene were not reactive in this reaction (entries 2 and 3). This cyanation reaction tolerated a variety of functional groups on the aromatic ring. Methyl-, chloro-, bromo-, methoxy-, and (methoxycarbonyl)phenyl iodides underwent cyanation in good yields. A byproduct of the present reaction is trimethylsilyl iodide, Me_3SiI. Although Me_3SiI is known as a powerful dealkylating reagent of ethers and esters,[5,8] such a carbon–oxygen bond cleavage did not take place at all in the present cyanation. This may due to the complexation of Me_3SiI with triethylamine leading to an insoluble solid. Although the structure of the colorless precipitates formed during the reaction was not characterized, it could well be a 1:1 complex of Et_3N and Me_3SiI. The formation of a 1:1 complex of Me_3SiI with amines such as Me_3N, pyridine, quinoline, and DABCO has been reported.[9] It was reported that the reaction of 1-chloro-3-iodobenzene with KCN in HMPA in the presence of $Pd(OAc)_2$ at 100 °C for 5.5 h afforded a mixture of 3-chlorobenzonitrile and 1,3-benzenedicarbonitrile, ratio of which was 77:23.[3b] In contrast to this result, 1-chloro-4-iodobenzene (**2d**) gave 4-chlorobenzonitrile (**3d**) as a single product in the present reaction and no 1,4-benzenedicarbonitrile (**3f**) was detected (entry 6). Dicyanation was observed to some extent in the case of 1-bromo-4-iodobenzene (**2e**) (entry 7). The dicyanation affording **3f**, however, was not complete even in the reaction using an excess amount of **1** with a prolonged reaction time (entry 8).

Scheme I. Catalytic Cycle for the Cyanation of Aryl Iodides with Me_3SiCN (1)



The mechanism of the palladium-catalyzed cyanation of aryl iodide would be similar to that of so-called cross-coupling reaction[10] and a likely mechanism is shown in Scheme I. As an alternative, **1** may react with Et_3N to afford a "soluble" cyanide ion, $Et_3NSiMe_3{}^+CN^-$, which is capable of replacing iodide in **4**.

In summary, trimethylsilyl cyanide (**1**) is effective for cyanation of aryl iodide in the presence of a palladium complex.

Experimental Section

Boiling points and melting points were uncorrected. ^1H NMR spectra were taken on a Bruker-WM 360 or a Hitachi R-24A with tetramethylsilane as the internal standard. Infrared spectra were obtained on a Hitachi 260-10 spectrometer. Mass spectra were obtained on a JMS-DX 300. GLC analyses were carried out on a Hitachi 663-50, equipped with a flame ionization detector, using a 3 m × 3 mm stainless column packed with 20 % Silicone DC-550 on 60–80 mesh Celite 545. Preparative TLC was performed with Merk Kieselgel 60 F_{254} using mixtures of hexane–EtOAc of varying composition as an eluent. Column chromatography was performed with 70–230 mesh Kieselgel 60 (Merk).

Materials. Aryl halides were commercially available and used without further purification. $Pd(PPh_3)_4$ was prepared by the procedure in the literature.[11] Triethylamine was distilled from KOH before use. Me_3SiCN was distilled from CaH_2 and stored under N_2.

General Procedures. To a solution containing aryl halide (1 mmol) and Me_3SiCN (0.2 mL, 1.5 mmol) in Et_3N (2 mL) was added $Pd(PPh_3)_4$ (23 mg, 0.02 mmol). The mixture was stirred under reflux for 30 min under nitrogen. Analysis of the product mixture by GLC indicated complete conversion to nitrile. The solution was poured into a mixture of benzene and water. The organic layer was separated and dried. The solvent was removed in vacuo. The crude product was then purified by bulb-to-bulb distillation or by preparative TLC using hexane and EtOAc as the eluent. The ^1H NMR and IR spectra of aryl nitriles were consistent with those of authentic samples.

Preparation of 4-Methoxybenzonitrile (3c) from 1-Iodo-4-methoxybenzene (2c). A mixture of 1-iodo-4-methoxybenzene

(6) Recently, we have reported the Pd-catalyzed addition of **1** to arylacetylenes.[7] In this work, we showed that **1** reacted with (4-bromophenyl)acetylene (**6**) to give a mixture of 2-(4-bromophenyl)-3-(trimethylsilyl)-(*Z*)-prop-2-enenitrile (**7**) and an unexpected product, 2-(4-cyanophenyl)-3-(trimethylsilyl)-(*Z*)-prop-2-enenitrile (**8**). The formation of the product **8** involves cyanation of the bromide **6** or **7** by **1**. This result prompted us to initiate the present work.

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(2c) (11.7 g, 50 mmol), Me_3SiCN (10 mL, 75 mmol), $Pd(PPh_3)_4$ (580 mg, 0.5 mmol), and 50 mL of Et_3N was stirred for 30 min under reflux in a 100-mL round-bottom flask under a nitrogen atmosphere. The reaction mixture was diluted with hexane and filtered. Solvent was then removed in vacuo. The residue was chromatographed (silica gel, hexane/EtOAc = 9/1) to give 4.84 g (73 %) of 4-methoxybenzonitrile (3c): mp 58–59 °C (lit.[12] mp 61–62 °C); ^1H NMR ($CDCl_3$) δ 3.80 (s, 3 H, CH_3O), 6.85 (d, J = 9 Hz, 2 H, Ph), 7.50 (d, J = 9 Hz, 2 H, Ph); IR (Nujol) 2220 (CN), 1605 (Ph) cm^{-1}; MS, m/e 133 (M^+).

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Registry No. 1, 7677-24-9; **2a**, 591-50-4; **2b**, 615-37-2; **2c**, 696-62-8; **2d**, 637-87-6; **2e**, 589-87-7; **2f**, 624-38-4; **2g**, 619-44-3; **3a**, 100-47-0; **3b**, 529-19-1; **3c**, 874-90-8; **3d**, 623-03-0; **3e**, 623-00-7; **3f**, 623-26-7; **3g**, 1129-35-7; $Pd(PPh_3)_4$, 14221-01-3.

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New Synthetic Reactions with $(Ph_3Sn)_2Zn \cdot TMEDA$ Complex. Regio- and Stereoselective Synthesis of Vinylstannanes

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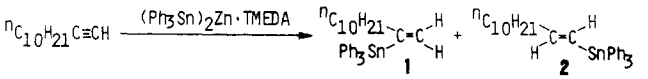
We have developed various new reactions with a reagent which is believed to have a Zn–Sn single bond.[2] This paper deals with the results of the study of the synthetic reactions with the isolable complex $(Ph_3Sn)_2Zn \cdot TMEDA$.[3] Two methods for vinylstannane synthesis with this complex have been examined: (1) addition to acetylenic bonds in the presence of transition-metal catalysts to give vinylstannanes[4] and (2) transformation of alkenyl halides and enol triflates into vinylstannanes.[5,6] The complex has behaved in the same way as the compounds that are generated from Ph_3SnLi and $ZnBr_2$ in situ in these reactions and has proved to be effective for synthetic use.

Reactions of $(Ph_3Sn)_2Zn \cdot TMEDA$ with Terminal Alkynes. Previously we have reported that the reaction of $(Bu_3Sn)_2Zn$ prepared in situ with carbon–carbon triple bonds proceeds easily in the presence of various kind of transition-metal catalysts to give vinylstannanes after aqueous workup.[2] Here we wish to report that $(Ph_3Sn)_2Zn \cdot TMEDA$ adds to acetylenic bonds in the same manner as $(Ph_3Sn)_2Zn$ derived from Ph_3SnLi and $ZnBr_2$ in situ.

Treatment of 1-dodecyne with $(Ph_3Sn)_2Zn \cdot TMEDA$ in THF in the presence of CuCN catalyst gave a mixture of 2-(triphenylstannyl)-1-dodecene (1) and the (*E*)-1-triphenylstannyl isomer (2) (83:17) in 87% combined yield (Scheme I). The regiochemistry of the reaction depends on the nature of the transition-metal catalysts. Representative results are summarized in Table I. In general, 2-stannyl-1-alkenes were obtained as major products. As shown in Scheme II, the reaction proceeded in cis fashion. The reaction of phenylacetylene with the complex in the presence of CuCN provided a mixture of 1-phenyl-1-(triphenylstannyl)ethene (3) and the 2-triphenylstannyl isomer (4). The product 3 showed ^1H NMR ($CDCl_3$, 200 MHz) signals at δ 5.64 (d, $J(H_a-H_b)$ = 1.8 Hz, $J(Sn-H_a)$ = 77 Hz, H_a) and 6.36 (d, $J(H_a-H_b)$ = 1.8 Hz, $J(Sn-H_b)$ = 165 Hz, H_b). The other isomer 4 having ^1H NMR ab-

Scheme I



Scheme II

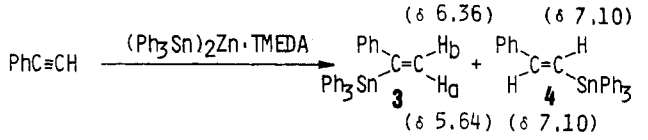
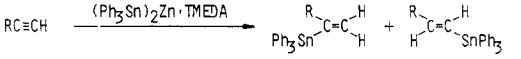
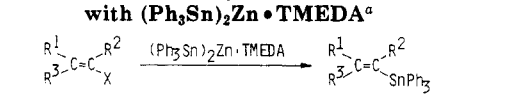


Table I. Reaction of Acetylenic Compounds with $(Ph_3Sn)_2Zn \cdot TMEDA$



acetylene R	catalyst	yield, %	ratio of I/II
n-$C_{10}H_{21}$	CuCN	87	83/17
	$PdCl_2(PPh_3)_2$	59	62/38
	$Pd(PPh_3)_4$	50	50/50
	$RhCl(PPh_3)_3$	30	40/60
$PhCH_2OCH_2CH_2$	CuCN	80	78/22
Ph	CuCN	70	60/40

Table II. Reaction of Alkenyl Halides and Enol Triflates with $(Ph_3Sn)_2Zn \cdot TMEDA$[a]



substrate				product	
R^1	R^2	R^3	X	yield, %	*E*/*Z*
n-C_6H_{13}	H	H	I	75	100/0
H	H	n-$C_{10}H_{21}$	I	67	25/75
H	n-$C_{10}H_{21}$	H	I	63	
H	n-$C_{10}H_{21}$	H	OSO_2CF_3	78	
H	n-C_5H_{11}	H	OSO_2CF_3	70	
H	CH_3	H	Br[b]	82	
H	Ph	H	OSO_2CF_3	50	
Ph	H	H	Br	65	100/0
H	H	Ph	Br	62	50/50

[a] The reactions were performed at 25 °C for alkenyl iodides and enol triflates and 60 °C for alkenyl bromides. Substrate (1.0 mmol), the complex (2.0 mmol), and $Pd(PPh_3)_4$ (0.05 mmol) were employed. [b] Bromide (3.0 mmol) and the complex (1.0 mmol) were employed.

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