Aryne Reactions of Polyhalobenzenes with Alkenyl and Alkynyl Grignard Reagents

Chi-Jen Frank Du and Harold Hart*

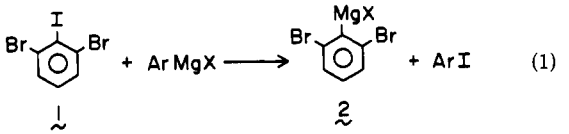
Department of Chemistry, Michigan State University, East Lansing, Michigan 48824

Received March 26, 1987

Polyhalobenzenes react with alkenyl Grignard reagents to form from one to four new aryl–alkenyl bonds, depending on the particular polyhalobenzene used, in a single operation. Three types of reactions are involved in the mechanism: organometallic exchange, followed by one or more sequences of aryne formation, and nucleophilic addition to the aryne. Similar reactions can be accomplished with alkynyl Grignard reagents by using an alkyl Grignard reagent to initiate the exchange step.

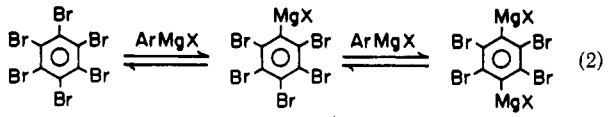
We recently described new one-flask syntheses of unsymmetric biaryls,[1] *p*-terphenyls,[1] *m*-terphenyls,[2] and 1,2,4,5-tetraarylbenzenes,[3] via the reaction of polyhalobenzenes with an excess of an aryl Grignard reagent. These reactions, which proceed via aryne intermediates, result in the formation of from one to six new carbon–carbon bonds in a single operation. We describe here how these reactions can be extended to alkenyl and alkynyl Grignard reagents.

The first step in triggering these reactions is organometallic exchange between the polyhalobenzene and the added aryl Grignard reagent. For example, the first step in the *m*-terphenyl synthesis[2] involves exchange between **1** (or a similar vicinal trihalide) and the aryl Grignard reagent to give dihalo Grignard **2** (eq 1). In the *p*-ter-



1 **2** (1)

phenyl[1] or 1,2,4,5-tetraarylbenzene[3] syntheses, two such exchanges are required to form a 1,4-di-Grignard intermediate. For example, in the latter case, the reaction with hexahalobenzenes such as **3** proceeds via di-Grignard **5** (eq 2).



3 **4** **5** (2)

These exchange steps are followed by magnesium halide elimination to form an aryne (in the case of **5**, an organometallic aryne) and regiospecific nucleophilic addition of the aryl Grignard reagent to the aryne. A second (or in the case of **5**, a third and fourth[3]) aryne is formed and captured, in tandem fashion, ultimately leading to the final product.

Two factors probably combine to facilitate the required initial Grignard exchange. First, the hybridization is the same (sp^2) in the starting aryl Grignard reagent and in the resulting halogenated aryl Grignard reagent. Second, the electron-withdrawing effect of the halogen substituents stabilizes the negative charge in the product Grignard reagent, thus shifting the equilibria in the forward direction.

These two factors should also operate favorably for the reaction of alkenyl Grignard reagents with polyhalobenzenes and, as described below, this is the case. With alkynyl Grignard reagents, however, two opposite effects are pitted against one another. On the one hand, negative charge must be transferred from an sp carbon in the alkynyl Grignard reagent to an sp^2 carbon in the desired polyhaloaryl Grignard, generally an unfavorable process. On the other hand, the halogen substituents on the product Grignard reagent might stabilize the negative charge sufficiently to overcome the unfavorable hybridization change. Consequently, the matter had to be put to an experimental test. In this event, as described below, the hybridization effect won out in the examples studied, but a trick to circumvent this factor was devised, enabling the reactions to proceed.

Results and Discussion

Alkenyl Grignard Reagents. The results of the reaction of (2-phenylvinyl)magnesium bromide (**6**) and (1,2-diphenylvinyl)magnesium bromide (**7**) with several polyhalobenzenes are summarized in Table I.

Treatment of *o*-bromoiodobenzene with nearly 3 equiv of **6** (cis–trans mixture[4]) at room temperature for 3 h gave, after aqueous quench, a 77% yield of stilbene (93% trans, 7% cis). In a similar manner, 2,6-dibromoiodobenzene and **6** (5 equiv) gave *m*-distyrylbenzene (**12**). The crystalline *E,E* isomer was isolated after column chromatography. This diene, which has an interesting and rich photochemistry,[5] was prepared previously via a double-Wittig reaction[6,7] (55% yield) or via the Siegrist reaction[8,9] (27% yield). The new route described here is comparable in efficiency and more direct.

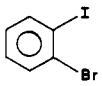
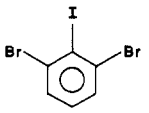
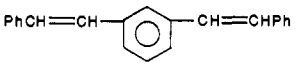
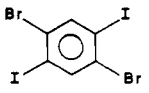
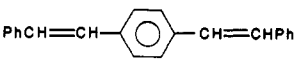
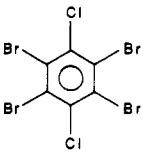
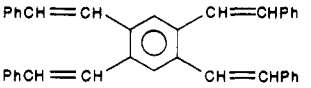
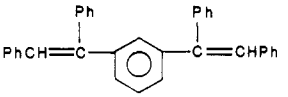
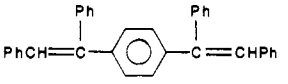
In an analogous manner, tetrahalobenzene **9** and excess **6** gave *p*-distyrylbenzene (**13**). This diene was previously prepared from terephthalaldehyde and benzylmagnesium chloride, followed by dehydration (22% yield),[10] and in better overall yield by various Wittig procedures.[7,10,11]

Of greater interest is the one-step synthesis of 1,2,4,5-tetrastyrylbenzene (**14**). This tetrene was first prepared

(1) Hart, H.; Harada, K.; Du, C.-J. F. *J. Org. Chem.* **1985**, *50*, 3104.
(2) Du, C.-J. F.; Hart, H.; Ng, K.-K. D. *J. Org. Chem.* **1986**, *51*, 3162.
(3) Harada, K.; Hart, H.; Du, C.-J. F. *J. Org. Chem.* **1985**, *50*, 5524.
(4) Aldrich Chemical Co., Catalog No. 15744-9.
(5) Zertani, R.; Meier, H. *Chem. Ber.* **1986**, *119*, 1704.
(6) Nakaya, T.; Imoto, M. *Bull. Chem. Soc. Jpn.* **1966**, *39*, 1547.
(7) Laarhoven, W. H.; Cuppen, Th. J. H. M.; Nivard, R. J. F. *Tetrahedron* **1970**, *26*, 1069.
(8) Siegrist, A. E.; Liechti, P.; Meyer, H. R.; Weber, K. *Helv. Chim. Acta* **1969**, *52*, 2521 and references cited therein.
(9) Meier, H.; Zertani, R.; Noller, K.; Oelkrug, D.; Krabichler, G. *Chem. Ber.* **1986**, *119*, 1716.
(10) Misumi, S.; Kuwana, M.; Murashima, K.; Nakagawa, M. *Bull. Chem. Soc. Jpn.* **1961**, *34*, 1833.
(11) Campbell, T. W.; McDonald, R. N. *J. Org. Chem.* **1959**, *24*, 1246.

0022-3263/87/1952-4311$01.50/0

Table I. Reaction of Alkenyl Grignard Reagents with Polyhalobenzenes

Grignard	halobenzene	product	yield, %
PhCH=CHMgBr (6)	 **8**	PhCH=CHPh	77[a]
6	 **1**	 **12**	48[b]
6	 **9**	 **13**	44[b]
6	 **10**	 **14**	49
PhCH=C(Ph)MgBr (7)	1	 **15**	64
7	9	 **16**	54

[a] Cis (7%) and trans (93%). [b] Isolated yield of *E,E* isomer; other isomers may be present.

from durene (via tetrabromination, conversion to a tetraphosphonic ester, followed by a tetra-Horner–Wadsworth–Emmons reaction with benzaldehyde) in 29% overall yield[12] and later in 2–3% yield via the Siegrist reaction.[9]

The Grignard reagent **7** from (*Z*)-1-bromo-1,2-diphenylethene[13] reacted with **1** in refluxing THF to give crystalline **15**, mp 149–150 °C, in 64% isolated yield. One isomer of this diene, mp 57 °C, was reported previously, obtained in 41% overall yield from *m*-dibenzoylbenzene and benzylmagnesium chloride, followed by dehydration.[14] Our product is probably the *Z,Z* isomer (phenyls trans), assuming that the Grignard geometry is retained in the aryne adduct.

In an analogous manner, the *Z,Z* isomer (phenyls trans) of **16**[14,15] was obtained from tetrahalobenzene **9** and excess **7**. In view of the difficulty encountered previously[15] in obtaining this isomer free of its *Z,E* and *E,E* counterparts, the method given here is the one of choice for this isomer.

Attempts to prepare the sterically hindered tetra-α-phenyl analogue of **14** (from **10** and excess **7**) failed to give a pure product.

The results described here demonstrate the construction of from one to four new aryl–ethenyl bonds from polyhalobenzenes and ethenyl Grignard reagents in a single operation. Since the actual reaction products, prior to quenching, are either mono- (from **1** and **8**) or di-Grignards (from **9** and **10**), they may be treated directly with other electrophiles than H^+, thus adding flexibility to the methodology. Extensions to alkenyl Grignard reagents that carry additional latent functionality are in progress.

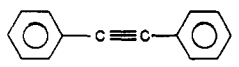
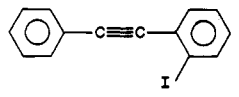
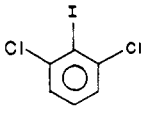
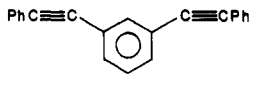
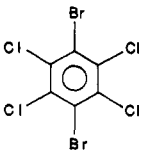
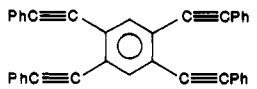
Alkynyl Grignard Reagents. The Grignard reagent used in all the studies reported here was (phenylethynyl)magnesium bromide (**17**). Treatment of any of the halobenzenes in Tables I or II with **17** at room temperature led to no reaction (recovery of the polyhalobenzene and phenylethyne after aqueous quench). Thus, the required Grignard exchange essential to aryne generation and trapping did not take place.

To circumvent this problem, ethylmagnesium bromide was used to bring about the required exchange (favorable $sp^3 \rightarrow sp^2$ hybridization change). For example, slow addition at room temperature of ethylmagnesium bromide to a mixture of *o*-bromoiodobenzene and **17** gave, after aqueous quench, a 61% yield of diphenylacetylene (**18**) (Table II). A similar reaction, but with an iodine quench, gave the previously unknown iodo derivative **19**. Clearly, this methodology provides a simple, one-step route to unsymmetric diarylacetylenes.

A similar procedure but with (2,6-dichlorophenyl)magnesium bromide (preformed from **21** and ethylmagnesium bromide at 0 °C) at reflux temperature gave 1,3-bis(phenylethynyl)benzene (**20**). Previous routes to **20** involved a multistep Wittig–Horner modification[16] or oxidative coupling of 1,3-diiodobenzene with cuprous phenylacetylide[17] and proceeded in 42–50% yield.

(12) Müller, E.; Fritz, H.-G.; Munk, K.; Straub, H. *Tetrahedron Lett.* **1969**, *10*, 5167.
(13) Avraamides, J.; Parker, A. J. *Aust. J. Chem.* **1983**, *36*, 1705. The Grignard reagent gave *trans*-stilbene (>90%) on aqueous quench.
(14) Buu-Hoi, Ng. Ph.; Loc, T. B.; Xuong, Ng. D. *J. Chem. Soc.* **1957**, 3964.
(15) Bertoniere, N. R.; Rowland, S. P.; Griffin, G. W. *J. Org. Chem.* **1971**, *36*, 2956.
(16) Zimmer, H.; Hickey, K. R.; Schumacher, R. J. *Chimia* **1974**, *28*, 656.

Table II. Aryne Trapping with (Phenylethynyl)magnesium Bromide

halobenzene	product	yield, %[b]
8	 **18**	61
8[a]	 **19**	50
 **21**	 **20**	52
 **22**	 **23**	25

[a] Iodine quench; all other entries for H^+ quench. [b] Isolated yields, not optimized.

Finally, treatment of a mixture of **17** and 1,4-dibromo-2,3,5,6-tetrachlorobenzene (**22**) with ethylmagnesium bromide gave 1,2,4,5-tetrakis(phenylethynyl)benzene (**23**). Previously, **23** was prepared in five steps and 13% overall yield from durene.[12]

The experiments summarized in Table II illustrate the feasibility and simplicity of this route to multiple aryl–ethynyl bond construction.

We believe that the reactions outlined briefly in this paper are capable of wide application, and we are extending these studies.

Experimental Section[18]

Stilbene (11). A solution of *o*-bromoiodobenzene (**8**) (1.41 g, 5 mmol) in 20 mL of dry THF was added over 1 h at room temperature to a freshly prepared solution of styrylmagnesium bromide (**6**) (from 2.7 g, 14.7 mmol of β-bromostyrene and 0.4 g of Mg turnings in 30 mL of THF). The mixture was stirred for an additional 2 h, then quenched with ice and saturated NH_4Cl. The THF was removed (rotavap), and the aqueous solution was extracted with chloroform. The organic extract was washed with sodium bicarbonate and water and dried ($MgSO_4$). After solvent removal, the residue was chromatographed over silica gel with hexane eluent to give 0.64 g of *trans*-stilbene, mp 125–126 °C (lit.[19] mp 124 °C), and 0.05 g of *cis*-stilbene, mp 3–4 °C (lit.[19] mp 5–6 °C), combined yield 77%.

***m*-Distyrylbenzene (12).** Following a similar reaction procedure as for **11**, 2,6-dibromoiodobenzene (**1**)[2] (5 mmol in 20 mL of THF) and freshly prepared **6** (25 mmol in 40 mL of THF) reacted at room temperature for 7 h. Workup and chromatography on silica gel with hexane–CH_2Cl_2 (v/v, 9:1) as the eluent gave 0.67 g (48%) of mainly (*E,E*)-**12**, mp 162–165 °C from benzene (lit.[5] mp 169 °C).

***p*-Distyrylbenzene (13).** From 1,4-dibromo-2,5-diiodobenzene (**9**)[1] (5 mmol in 30 mL of THF) and **6** (30 mmol in 50 mL of THF), reaction time 4 h at room temperature, there was obtained after the usual workup 0.61 g (44%) of (*E,E*)-**13**, mp 263–264 °C from benzene (lit.[7] mp 266 °C).

1,2,4,5-Tetrastyrylbenzene (14). From 1,4-dichloro-2,3,5,6-tetrabromobenzene (**10**)[3] (5 mmol in 30 mL of THF) and **6** (40 mmol in 50 mL of THF), reaction time 10 h at room temperature, was obtained after chromatography on silica gel with hexane–CH_2Cl_2 (v/v, 4:1) as the eluent 1.20 g (49%) of **14**, mp 268–274 °C (lit.[12] mp 273–275 °C).

1,3-Bis(1,2-diphenylvinyl)benzene (15). To a refluxing solution of **7** (prepared from 6.47 g, 25 mmol of (*Z*)-1-bromo-1,2-diphenylethene[13] and 0.72 g of Mg turnings in 40 mL of dry THF) was added over 2 h a solution of 2,6-dibromoiodobenzene (**1**) (1.81 g, 5 mmol in 30 mL of THF). After an additional 2 h, the reaction mixture was quenched with ice and dilute HCl and extracted with chloroform. The organic layer was washed with water, dried ($MgSO_4$), evaporated, and chromatographed on silica gel with hexane–CH_2Cl_2 (v/v, 9:1) as the eluent to give 1.40 g (64%) of **15**, mp 149–150 °C from benzene–ethanol: ^1H NMR δ 6.92 (s, 2 H), 7.07–7.88 (m, 24 H); mass spectrum, *m/e* (relative intensity) 435 (36), 434 (M^+, 100), 196 (39), 178 (44), 167 (33), 105 (63), 90 (32), 89 (33), 84 (50), 77 (65). Anal. Calcd for $C_{34}H_{26}$: C, 93.97; H, 6.03. Found: C, 94.02; H, 6.10.

1,4-Bis(1,2-diphenylvinyl)benzene (16). In a procedure analogous to that used for **15**, from **7** (24 mmol in 40 mL of THF) and 1,4-dibromo-2,5-diiodobenzene (**9**) (4 mmol in 30 mL of THF), reaction time 5.5 h at reflux, was obtained after workup 1.04 g (54%) of (*Z,Z*)-**16**, mp 145–145.5 °C (lit.[15] mp 145–146 °C).

Diphenylacetylene (18). To a mixture of (phenylethynyl)-magnesium bromide (**17**) (30 mmol in 30 mL of THF, prepared by maintaining a mixture of 31 mmol of ethylmagnesium bromide and 30 mmol of phenylethyne at room temperature for 3 h) and 10 mmol of *o*-bromoiodobenzene (**8**) was added over 3 h at room temperature a solution of ethylmagnesium bromide (10 mmol in 25 mL of THF). After an additional 1.5 h, the resultant mixture was quenched with ice and dilute HCl and extracted with chloroform. The organic layer was washed with water, dried ($MgSO_4$), and evaporated. Chromatography of the residue on silica gel with hexane as the eluent gave 1.10 g (61%) of **18**, mp 58–60 °C (lit.[19] mp 62.5 °C).

1-Iodo-2-(phenylethynyl)benzene (19). The procedure for **18** was followed, but, prior to aqueous workup, the mixture was treated with iodine (40 mmol). Chromatography followed by distillation gave 1.50 g (50%) of **19**: bp 160–162 °C (1 Torr); ^1H NMR δ 6.90–7.00 (m, 1 H), 7.24–7.38 (m, 4 H), 7.45–7.62 (m, 3 H), 7.85 (dd, 1 H); mass spectrum, *m/e* (relative intensity) 305 (12), 304 (84), 177 (29), 176 (100), 152 (29), 151 (51), 150 (38), 127 (20). Anal. Calcd for $C_{14}H_9I$: C, 55.15; H, 2.98. Found: C, 55.24; H, 2.85.

1,3-Bis(phenylethynyl)benzene (20). A solution of 2,6-dichloroiodobenzene (**21**)[20] (5 mmol) in 10 mL of THF was cooled to 0 °C, ethylmagnesium bromide (5.5 mmol in 10 mL of THF) was added, and the mixture was stirred for 1 h. The resulting (2,6-dichlorophenyl)magnesium bromide (shown in separate experiments by aqueous quenching to have been formed in >95% yield) was added to a refluxing solution of **17** (20 mmol in 20 mL of THF) over 3 h. Workup as usual and chromatography over silica gel with hexane–CH_2Cl_2 (v/v, 95:5) as the eluent gave 0.72 g (52%) of **20**, mp 105–108 °C (lit.[16] mp 111–113.5 °C).

1,2,4,5-Tetrakis(phenylethynyl)benzene (23). To a solution of **17** (50 mmol in 50 mL of THF) was added at room temperature a suspension of 1,4-dibromo-2,3,5,6-tetrachlorobenzene (**22**)[21] (1.87 g, 5 mmol in 20 mL of THF). After 1 h, a solution of ethylmagnesium bromide (10 mmol in 20 mL of THF) was slowly added over 2 h, and the mixture was kept at room temperature for an addition 0.5 h and then heated at reflux for 1.5 h. The usual workup and chromatography on silica gel with hexane–CH_2Cl_2 (v/v, 4:1) as the eluent gave 0.75 g (25%) of **23**, mp 198–204 °C (lit.[12] mp 204–205 °C).

Acknowledgment. We thank the National Institutes

(17) Castro, C. E.; Gaughan, E. J.; Owsley, D. C. *J. Org. Chem.* **1966**, *31*, 4071.

(18) For general procedures, see ref 2. All previously known compounds synthesized here by new methodology not only had melting points (given here) that agreed with the literature but also had ^1H NMR and mass spectra consistent with the assigned structures, omitted here to conserve space.

(19) *Dictionary of Organic Compounds*, 5th ed.; Chapman and Hall: London, 1982.

(20) Bolton, R.; Sandall, J. P. B. *J. Chem. Soc., Perkin Trans. 2* **1977**, 278.

(21) Hugel, G.; Lerer, M.; Fabre, C. *Bull. Soc. Chim. Fr.* **1954**, *21*, 836.

of Health for financial support (Grant GM 15997-17). C.-J.F.D. also thanks Dow Chemical Co. and BASF Corp. for fellowship support.

Registry No. 1, 19821-80-8; 6, 103-64-0; 7, 15022-93-2; 8, 583-55-1; 9, 63262-06-6; 10, 31604-30-5; *cis*-11, 645-49-8; *trans*-11, 103-30-0; 12, 1725-76-4; 13, 1608-41-9; 14, 25634-86-0; 15, 109764-44-5; 16, 31024-80-3; 17, 6738-06-3; 18, 501-65-5; 19, 109744-41-4; 20, 13141-36-1; 21, 19230-28-5; 22, 13074-99-2; 23, 25634-84-8; PhC≡CH, 536-74-3.

General Ether Synthesis under Mild Acid-Free Conditions. Trimethylsilyl Iodide Catalyzed Reductive Coupling of Carbonyl Compounds with Trialkylsilanes to Symmetrical Ethers and Reductive Condensation with Alkoxysilanes to Unsymmetrical Ethers[1]

Mark B. Sassaman, Kirtivan D. Kotian, G. K. Surya Prakash, and George A. Olah*

Donald P. and Katherine B. Loker Hydrocarbon Research Institute and Department of Chemistry, University of Southern California, Los Angeles, California 90089-1661

Received May 18, 1987

Facile synthesis of symmetrical ethers is achieved by either trimethylsilyl triflate or trimethylsilyl iodide catalyzed reductive coupling of carbonyl compounds (aldehydes and ketones) with trialkylsilanes. The method was also extended to the trimethylsilyl iodide catalyzed preparation of unsymmetrical ethers by reductive condensation (of carbonyl compounds) with alkoxysilanes. The scope and limitations of the reactions are discussed with emphasis on diastereoselectivity.

Introduction

The formation of carbon–oxygen single bonds is one of the oldest and most widely used functional group transformations in organic synthesis. The reaction of methyl iodide with silver oxide to prepare dimethyl ether was reported by Wurtz in 1856.[2] The Williamson ether synthesis, with its modifications, still a most widely used method, predates Wurtz's report by 6 years.[3] The attack of alkoxides on alkyl halides (Williamson's method), however, is synthetically useful only when the alkyl halide is primary. Low yields are obtained from secondary halides due to competing elimination and tertiary halides yield only elimination products.[3] Methods that involve carbocation intermediates such as the acid-catalyzed (Markovnikov) addition of alcohols to alkenes are plagued by competing rearrangements.[4] Solvomercuration, while fairly rapid, gives complicated mixtures in certain cases.[5]

In a series of papers from 1972–1975 Doyle and coworkers[6] reported acid-promoted reductive coupling of carbonyl compounds with trialkylsilanes as a route to symmetrical ethers. Their original method, however, presents the following major drawbacks: (1) Bronsted acids must be used in several-fold molar excess of the reactants, often as the solvent. Thus, the method cannot be used with acid-sensitive compounds. (2) Under these conditions, the formation of alcohol, alkene, and ester byproducts is unavoidable.

In 1979, Noyori's group reported the related trimethylsilyl triflate catalyzed reaction of acetals with trialkylsilanes.[7] This method is extremely mild and gives nearly quantitative yields of methyl ethers from the corresponding acetals. Mukaiyama et al.[8] reported a similar method from carbonyl compounds using trityl perchlorate as the catalyst. Yields ranged from 64% to 85% and circumvented most of the problems inherent in earlier methods. The use of trityl perchlorate as catalyst, however, is inconvenient and the preparation and handling of perchlorates should be avoided whenever possible.[9]

Since these latter methods overlapped with our own work involving reductive etherification of carbonyl compounds with trialkylsilanes over solid superacid catalysts,[10] we extended our studies to the reductive coupling of carbonyl compounds using trimethylsilyl triflate and trimethylsilyl iodide catalysts. In the latter case the in situ generation of trimethylsilyl iodide from hexamethyldisilane and iodine[11] was considered to provide a convenient mild way to carry out the reductive coupling of carbonyl compounds and also formation of unsymmetrical ethers. In this paper we report our studies of the trimethylsilyl iodide catalyzed preparation of ethers, its scope and limitations, as well as comparison with trimethylsilyl triflate catalyzed reactions.

The use of various alkylsilane reducing agents was also investigated and some unexpected stereochemical consequences observed.

(1) Synthetic Methods and Reactions. 130. For 129, see: Olah, G. A.; Rochin, C. *J. Org. Chem.* **1987**, *52*, 701.
(2) Wurtz, A. *Ann Chem.* **1856**, *46*, 222.
(3) March, J. *Advanced Organic Chemistry, Reactions, Mechanisms, and Structure*, 2nd ed.; McGraw-Hill Book Company: New York, 1978; pp 357–358 and references therein.
(4) March, J., ref 3, pp 699–700.
(5) Brown, H. C.; Kurek, J. T.; Rei, M.; Thompson, K. L. *J. Org. Chem.* **1984**, *49*, 2551.
(6) (a) Doyle, M. P.; DeBruyn, D. J.; Kooistra, D. A. *J. Am. Chem. Soc.* **1972**, *94*, 3659. (b) West, C. T.; Donnelly, S. J.; Kooistra, D. A.; Doyle, M. P. *J. Org. Chem.* **1973**, *38*, 2675. (c) Doyle, M. P.; DeBruyn, D. J.; Donnelly, S. J.; Kooistra, D. A.; Odubela, A. A.; West, C. T.; Zonnebelt, S. M. *J. Org. Chem.* **1974**, *39*, 2740. (d) Doyle, M. P.; West, C. T. *J. Org. Chem.* **1975**, *40*, 3821. (e) Doyle, M. P.; West, C. T. *J. Org. Chem.* **1975**, *40*, 3829. (f) Doyle, M. P.; West, C. T. *J. Org. Chem.* **1975**, *40*, 3835.
(7) Tsunoda, T.; Suzuki, M.; Noyori, R. *Tetrahedron Lett.* **1979**, 4679.
(8) Kato, J.; Iwasawa, N.; Mukaiyama, T. *Chem. Lett.* **1985**, 743.
(9) Dauben, H. J., Jr.; Honnen, L. R., Harmon, K. M. *J. Org. Chem.* **1960**, *25*, 1442.
(10) Olah, G. A.; Yamato, T.; Iyer, P. S.; Prakash, G. K. S. *J. Org. Chem.* **1986**, *51*, 2826.
(11) Olah, G. A.; Narang, S. C.; Gupta, B. G. B.; Malhotra, R. *J. Org. Chem.* **1979**, *44*, 1247.

0022-3263/87/1952-4314$01.50/0