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A Novel 1,2-Migration of Arylzincates Bearing a Leaving Group at Benzylic Position: Application to a Three-Component Coupling of *p*-Iodobenzyl Derivatives, Trialkylzincates, and Electrophiles Leading to Functionalized *p*-Substituted Benzenes

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Abstract: A three-component coupling of *p*-iodobenzyl derivatives, trialkylzincates, and electrophiles is described. Lithium trialkylzincates (R_3ZnLi) react with *p*-iodobenzyl methanesulfonate to give benzylzinc reagents p-$RC_6H_4CH_2Zn(L)$. The reaction proceeds through a mechanism involving initial iodine/zinc exchange and the 1,2-migration of the resulting arylzincates. The benzylzinc reagents, thus prepared, are subsequently used in coupling reaction with electrophiles such as aldehydes, ketones, acyl chlorides, tosyl cyanide, and chlorosilanes to give a variety of functionalized *p*-substituted benzenes. Reactions under Barbier conditions in which the corresponding benzylzinc reagents are generated in the presence of electrophiles work well for Me_3ZnLi and for magnesium zincates $R_3ZnMgBr$ derived from Grignard reagents. Generation of secondary benzylzinc reagents starting from diethyl 1-(*p*-iodophenyl)ethyl phosphate and their reaction with electrophiles are also achieved under Barbier conditions. Ketones, allyl bromides, and chlorosilanes are successfully used as electrophiles under these conditions.

Keywords: benzenes; homologation; zinc and compounds; Barbier reactions

1. Introduction

Homologation of organometallics is becoming of increasing importance in organic syntheses because subsequent bond formation of the resulting organometallics leads to a convergent construction of complex carbon frameworks by one-pot procedure. The synthetic potential has been demonstrated, for example, in recent development of useful carbon-carbon bond forming reactions using carbometalation of alkynes [1,2] and in three-component coupling approaches to a prostaglandin synthesis via conjugate addition/alkylation sequence [3-6]. Carbometalation and conjugate addition lead to a two-carbon homologation of organometallics. On the other hand, one-carbon homologation or methylene insertion is achieved by 1,2-migration of ate-type carbenoids [7-20]. Recent reports from this laboratory revealed that organozincates 1, readily generated by halogen/zinc exchange with triorganozincates (R_3ZnM', M' = Li, MgX), undergo facile 1,2-migration at low temperatures to give synthetically versatile alkenyl- [21,22], cyclopropyl- [23], and alkylzinc reagents [24] with carbon-carbon bond formation (eq 1). More recently,

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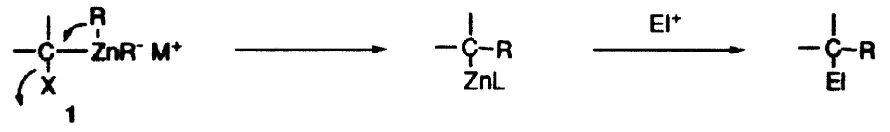
we have shown that facile 1,2-migration is also observed for alkynylzincates **2** bearing a leaving group at the γ-position with α,β-unsaturation (eq 2) [25,26]. The resulting allenic zinc reagents reacts with a variety of electrophiles regioselectively at the γ positions, affording three-component coupling products in which a nucleophile (R) and an electrophile (El) are introduced to the propargylic substrates at the 1,3-positions.



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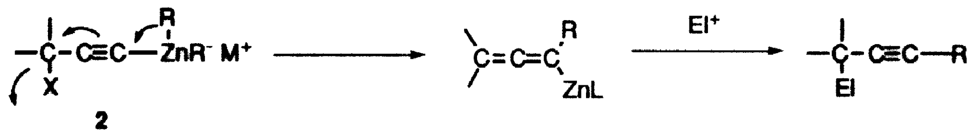

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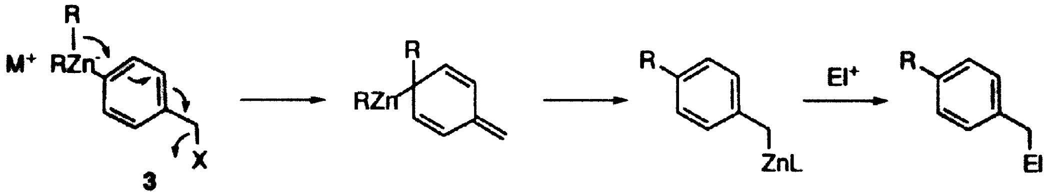

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(3)

We envisioned that a novel three-component coupling reaction in which a nucleophile and an electrophile are introduced at the remote positions of benzylic substrates could be developed through possible 1,2-migration of arylzincates **3** (eq 3). Arylzincates **3** bearing a leaving group at the benzylic position would rearrange with carbon-carbon bond formation to give a benzylzinc via a 4-methylene-2,5-cyclohexadienylzinc. Subsequent reaction of the benzylzinc with electrophiles would afford functionalized disubstituted arenes. Coexistence of the negatively charged metal atom center and the potential cationic center in **1** and **2** must be a major driving force of their facile 1,2-migration. However, for **3**, not only these two reactive centers are separated by the longer aromatic π-system but also 1,2-migration leading to the cross conjugated intermediate would suffer from a considerable loss of aromatic stabilization. Therefore, the reactivity of arylzincates **3** is also of interest in connection with the scope of 1,2-migration.

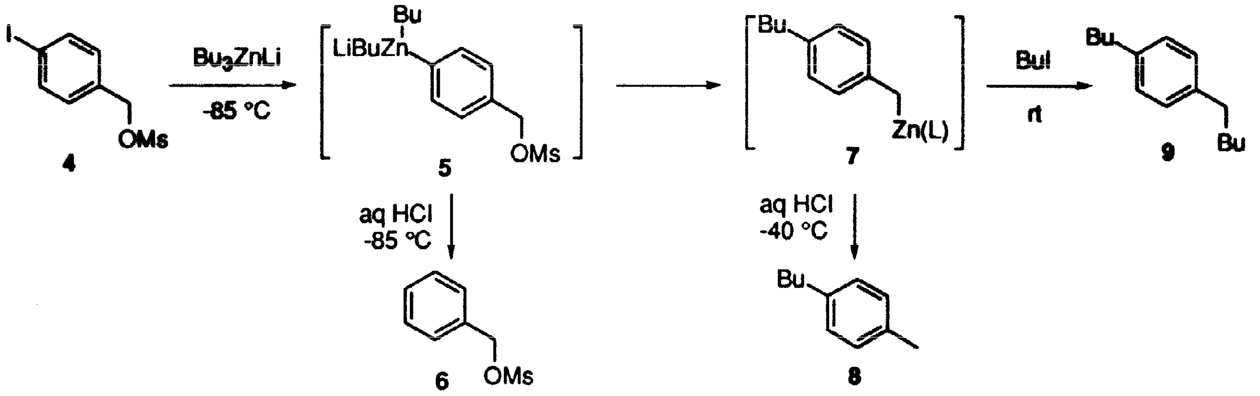
In this paper, we wish to report a three-component coupling of *p*-iodobenzyl derivatives, trialkylzincates, and electrophiles leading to *p*-disubstituted functionalized arenes via a novel 1,2-migration of arylzincates **3**. [27]

2. Results and Discussion

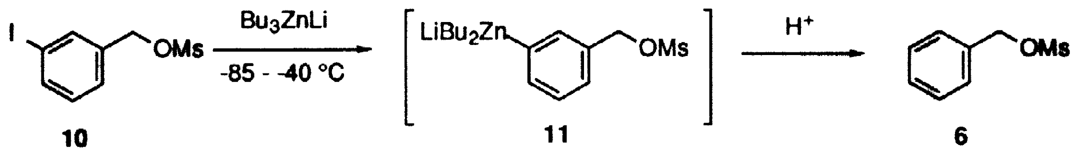
p-Iodobenzyl mesylate (**4**) was treated with Bu_3ZnLi (2.0 equiv) in THF at -85 °C for 20 min. Immediate hydrolysis of the resulting mixture by the addition of aq HCl in THF at this temperature gave benzyl mesylate (**6**) in 84% yield (Scheme 1). On the other hand, hydrolysis of the mixture after being warmed to -40 °C afforded *p*-butyltoluene (**8**) in 90% yield. In another experiment, the reaction mixture was allowed to warm to rt giving rise to the formation of *p*-butylpentylbenzene (**9**) in 57% yield. These results showed that (i) the iodine/zinc exchange reaction [28,29,21-24] proceeds smoothly at -85 °C to generate arylzincate intermediate **5** which is stable at this temperature, (ii) upon warming to -40 °C,

5 rearranges to benzylzinc **7** presumably through a mechanism involving 1,2-migration, and (iii) upon further warming to rt, **7** reacts with iodobutane, generated in situ by the initial iodine/zinc exchange, to give **9**. The efficiency of benzylzinc generation was depend on the amount of Bu_3ZnLi used: Reaction using 1.2 equiv of the reagent at -85 – - 40°C afforded *p*-butyltoluene in 61% yield. *p*-Bromobenzyl mesylate did not react with Bu_3ZnLi even at -40 °C. Reaction at 0 °C resulted in complex mixtures.

Scheme 1

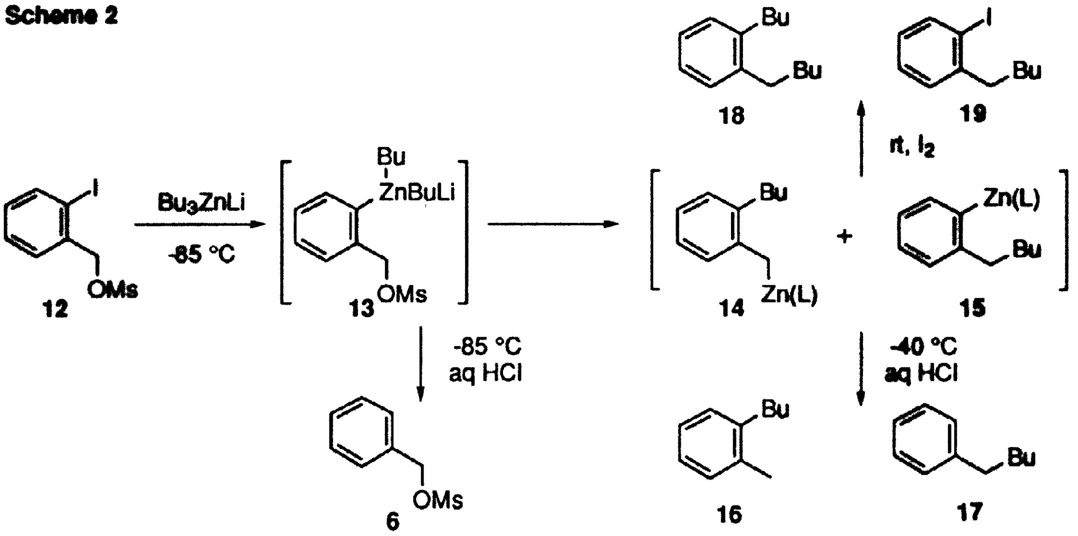


Support for the 1,2-migration mechanism was obtained from the reaction of *m*-iodobenzyl mesylate (**10**) with Bu_3ZnLi at -85 °C to -40 °C (eq 4). *m*-Substituted arylzincate **11** whose 1,2-migration would be infeasible was stable at higher temperatures and gave benzyl mesylate (71%) after hydrolysis. *o*-Substituted arylzincate **13**, on the other hand, was anticipated to undergo 1,2-migration with simultaneous liberation of the mesyloxy group to give the corresponding *o*-substituted benzylzinc reagent **14** (Scheme 2). Generation of **13** was confirmed by the formation of benzyl mesylate (63%) in the reaction of *o*-iodobenzyl mesylate (**12**) with Bu_3ZnLi (2.0 equiv) at -85 °C for 20 min. When the reaction mixture was allowed to warm to -40 °C and then hydrolyzed, *o*-butyltoluene (**16**) and pentylbenzene (**17**) were obtained in 14% and 36% yield, respectively. Treatment of arylzincate **13** at the temperatures from -85 °C to rt followed by addition of iodine afforded *o*-butylpentylbenzene (**18**) (15%) and *o*-iodopentylbenzene (**19**) (23%). The formation of **16** at -40 °C as well as the formation of iodobutane adduct **18** indicated the generation of benzylzinc **14**. However, this is not a major pathway for arylzincate **13**. Judging from the formation of **17** and **19**, *o*-pentylphenylzinc **15** was competitively produced upon warming **13** to -40 °C, probably through a mechanism involving an intramolecular transfer of the butyl ligand to the adjacent benzylic position. Alternative pathway involving intermolecular butylation of **13** with excess Bu_3ZnLi is less likely because a similar reaction using a reduced amount of the zincate (1.2 equiv) afforded **17** (30%) more selectively over **16** (5%).



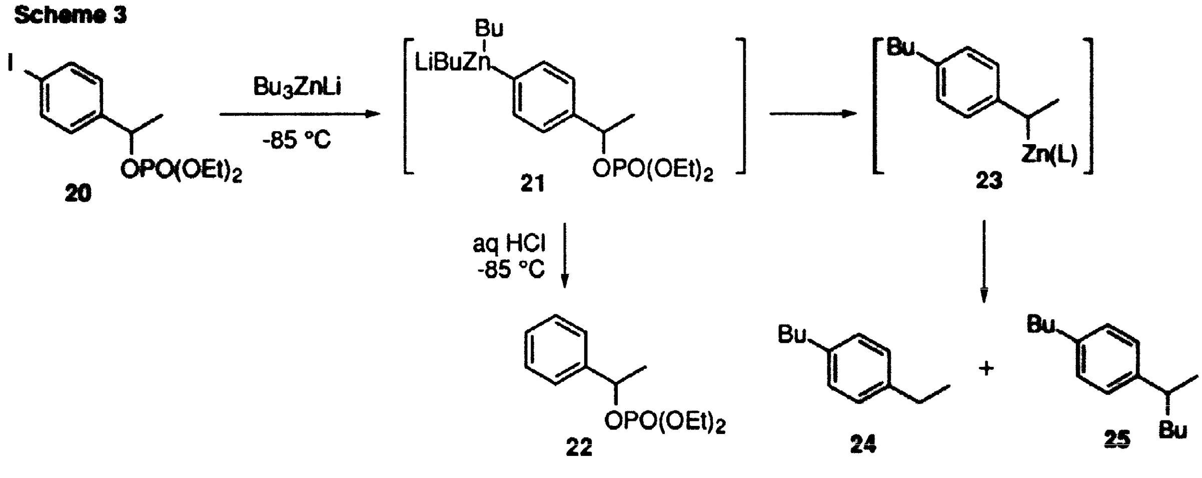
(4)

Scheme 2

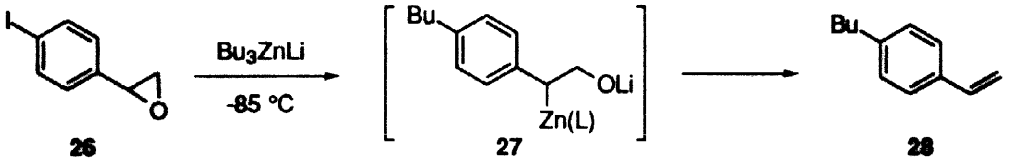


Because secondary benzyl mesylates are unstable and difficult to handle [30], the reaction of phosphate **20** was examined for the preparation of secondary benzylzinc reagents (Scheme 3). Treatment of **20** with Bu_3ZnLi (2.0 equiv) at -85 °C followed by warming up the resulting mixture to 0 °C gave dibutylation products **25** in 85% yield. The secondary benzylzinc reagent **23** is more reactive and the formation of **25** by the reaction with iodobutane was not completely retarded even at -40 °C. Thus, when a similar reaction was performed at temperatures from -85 °C to -40 °C, a mixture of phosphate **22** (51%), ethylbutylbenzene **24** (8%), and **25** (14%) was obtained. In addition, the formation of **22** as a major product under these conditions shows that 1,2-migration of arylzincate **21** bearing a less reactive phosphoryloxy group is relatively slow in comparison with **5** and **13**, derived from mesylates.

Scheme 3



The use of epoxide as a leaving group was examined in the reaction of *p*-iodostyrene oxide (**26**). Treatment of **26** with Bu_3ZnLi (2.0 equiv) at -85 °C followed by warming up the mixture to 0°C gave *p*-butylstyrene (**28**) in 39% yield. The formation of **28** is rationalized by a pathway involving elimination reaction of the benzylzinc intermediate **27**. Interestingly, the yield of **28** was improved to 47% by carrying out a similar reaction in the presence of $BF_3 \cdot Et_2O$ (2 equiv) at -85 – -45 °C.

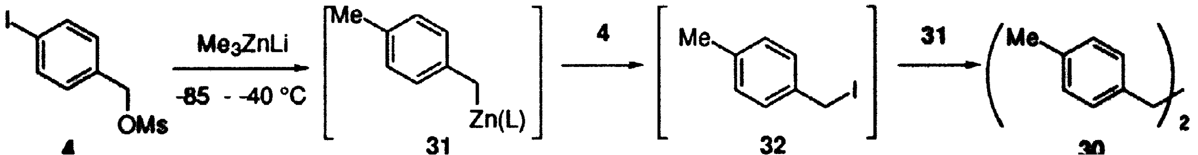


26 **27** **28**

Benzylzinc reagent **7**, prepared by treatment of mesylate **4** with Bu3ZnLi (2.0 equiv) at -85 °C followed by warming up the mixture to -40 °C, was subsequently treated with 2-phenylpropanal (1.2 equiv) at -85 °C to give alcohol **29a** in 80% yield (Table 1, entry 1). Although an excess amount of Bu3ZnLi was employed in this reaction, formation of the butylation product, 1-phenyl-3-heptanol, was not detected, indicating the higher reactivity of the benzyl moiety in zinc reagent **5**.[1] The reaction of *s*-Bu3ZnLi and *t*-Bu3ZnLi afforded the corresponding *p*-substituted benzylzinc reagents, which further reacted with isobutyraldehyde to give the corresponding adducts (entries 4 and 5). The reaction of Me3ZnLi, however, gave 4,4'-dimethylbibenzyl (**30**) as a major product but not the corresponding product (entry 6). The use of bromomagnesium zincate, prepared from BuMgBr (3 equiv) and $ZnCl_2$, resulted in lower product yield as well as byproduct formation of 4,4'-dibutylbibenzyl in 8% (entry 3). The benzylzinc reagents reacted successfully not only with aldehydes but also with ketones, acyl chlorides, tosyl cyanide, and chlorodimethylphenylsilane (entries 7-12). It should be noted that, in acylation, the formation of *tert*-alcohols derived from further reaction of the benzylzinc to the product ketones **29g,h** was not detected. It was reported that benzylzinc bromide reacts with tosyl cyanide exclusively at the ortho position to give 2-methylbenzonitrile [34]. In contrast to this, the benzylzinc reagent generated under the present conditions selectively reacted at the benzylic position to afford nitrile **29i** without the formation of such regioisomeric product.

For the successful generation of the benzylzinc reagent, it is important that halogen/metal exchange step is accomplished before subsequent 1,2-migration step. Otherwise, the resulting benzylzinc species in the form of zincate[1] would also undergo iodine/zinc exchange with the starting mesylate. A separate experiment showed that Me3ZnLi did not react with mesylate **4** at -85 °C. Owing to the lower reactivity [22], iodine/zinc exchange and subsequent 1,2-migration took place simultaneously at higher temperatures. Under these circumstances, the reaction of the resulting 4-methylbenzylzincate **31** (Zn(L) = Zn(Me)Li)[1] with **4** followed by coupling of the resulting benzyl iodide **32** with **31** would lead to the formation dimeric product **30** (Scheme 4). Formation of 4,4'-dibutylbibenzyl as a byproduct in the reaction using a BuMgCl derived zincate may also originate from its lower reactivity in the initial exchange step.

Scheme 4



4 **31** **32** **30**

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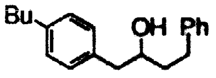
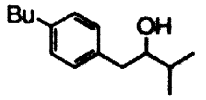
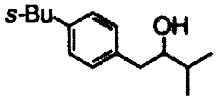
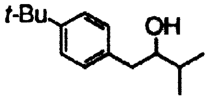
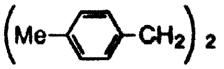
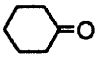
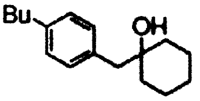
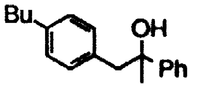
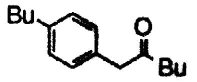
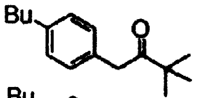
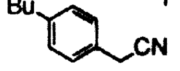
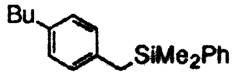
[1] Assuming a rapid ligand transfer, [31-33] the initially formed benzylzinc 7 (L = Bu) (Scheme 1) should react reversibly with Bu3ZnLi to form benzylzincates such as 7 (Zn(L) = Zn(Bu)2Li).

Table 1 Generation and Reaction of Benzylzinc Reagents [a]

entry	zincate	electrophile		product	yield (%)
1	Bu_3ZnLi	$Ph(CH_2)_2CHO$	**29a**		80
2	Bu_3ZnLi	*i*-PrCHO	**29b**		72
3[b]	$Bu_3ZnMgBr$				45
4	*s*-Bu_3ZnLi	*i*-PrCHO	**29c**		62
5	*t*-Bu_3ZnLi	*i*-PrCHO	**29d**		56
6	Me_3ZnLi	*i*-PrCHO	**30**		55
7	Bu_3ZnLi		**29e**		63
8	Bu_3ZnLi	$PhCOCH_3$	**29f**		48
9	Bu_3ZnLi	BuCOCl	**29g**		61
10	Bu_3ZnLi	*t*-BuCOCl	**29h**		48
11	Bu_3ZnLi	TsCN	**29i**		58
12	Bu_3ZnLi	$PhMe_2SiCl$	**29j**		62

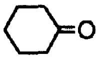
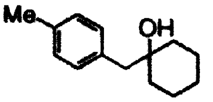
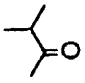
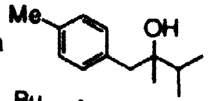
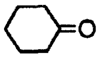
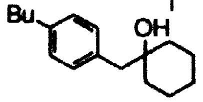
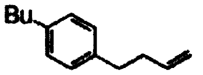
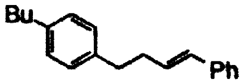
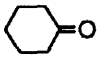
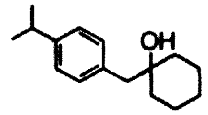
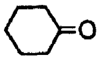
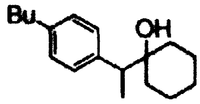
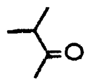
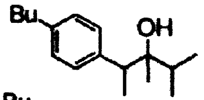
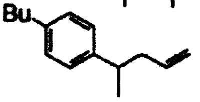
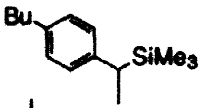
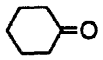
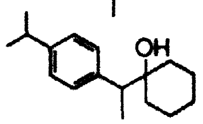
[a] Unless otherwise noted, reactions were carried out by mixing mesylate 4 and zincates (2.0 equiv) at -85 °C, warming the mixture to -40 °C, and treating with electrophiles (1.2 equiv) for 3 h at -85 °C. [b] 4,4'-dibutylbibenzyl was obtained as a byproduct in 8% yield.

We found that three-component coupling products of mesylate **4**, Me3ZnLi, and ketones were obtained in acceptable yields by carrying out the reaction under Barbier conditions (Table 2, entries 1 and 2). Thus, for example, the reaction of **4** and Me3ZnLi (2.0 equiv) in the presence of cyclohexanone (1.2 equiv) at temperatures from -85 °C to -40 °C afforded alcohol **29k** in 52% yield without formation of dimeric product **30**. Such modification of the reaction conditions was found to be also effective to the reaction using Grignard reagent-derived zincates. The yield of cyclohexanone adduct **29e** (entry 3) was comparable to that obtained in the stepwise reaction using Bu3ZnLi (Table 1, entry 7).

Allylation of the intermediate benzylzinc reagent proceeded efficiently by using 3.0 equiv of allyl bromide (entry 5). Reaction with cinnamyl bromide proceeded regioselectively to give linear adduct **29o** in high yield (entry 6). Successful results obtained under Barbier-type conditions implies a rapid reaction of benzylzinc reagents (e.g., **31**) with electrophiles which eventually retard their reaction with **4** leading to dimeric products (e.g., **30**).

Table 2

Three-Component-Coupling of 4-Iodobenzyl Esters under Barbier Conditions [a]

entry	substrate	zincate	electrophile	product	yield (%)
1	**4**	Me_3ZnLi		**29k** 	52%
2	**4**	Me_3ZnLi		**29m** 	45%
3	**4**	$Bu_3ZnMgCl$		**29e** 	68%
4	**4**		$CH_2{=}CHCH_2Br$	**29n** 	44%
5[b]	**4**				77%
6[b]	**4**		$PhCH{=}CHCH_2Br$	**29o** 	82%
7	**4**	i-$Pr_3ZnMgCl$		**29p** 	55%
8	**20**	Bu_3ZnLi		**29q** 	58%
9	**20**	$Bu_3ZnMgCl$			65%
10	**20**	Bu_3ZnLi		**29r** 	54%
11[b]	**20**		$CH_2{=}CHCH_2Br$	**29s** 	74%
12	**20**		Me_3SiCl	**29t** 	49%
13	**20**	i-$Pr_3ZnMgCl$		**29u** 	42%

[a] Unless otherwise noted, reactions were carried out by mixing **4** (or **20**), zincates (2.0 equiv), and electrophiles (1.2 equiv) at -85 °C and warming the mixture to -40 or 0 °C. [b] Three equiv. of the electrophile was used.

As noted previously, clean generation of secondary benzylzinc reagent **23** is not possible because its reaction with iodobutane is not completely retarded at temperatures where 1,2-migration of arylzincate **21** to form **23** takes place (Scheme 3). Nevertheless, the secondary zinc reagents generated from phosphate **20** and alkylzincates can be used in the three-component coupling reaction with electrophiles under Barbier conditions (entries 8-13). Both lithium and magnesium zincates gave a comparable yield of products. Ketones, allyl bromides, and chlorotrimethylsilane can be used as an electrophiles while an attempted reaction with isobutyraldehyde resulted in low yield of the product together with the formation of butylation product of the aldehyde.

3. Conclusion

In conclusion, we have described a novel three-component coupling reaction of *p*-iodobenzyl derivatives, trialkylzincates, and electrophiles leading to *p*-disubstituted functionalized arenes. For the reaction of mesylate **4** with lithium trialkylzincates other than Me_3ZnLi, a stepwise procedure in which the corresponding benzylzinc reagents are first prepared and then used works well with a variety of electrophiles involving aldehydes, ketones, acyl chlorides, tosyl cyanide, and chlorosilanes. On the other hand, for Me_3ZnLi and for bromomagnesium zincates derived from Grignard reagents, reactions under Barbier conditions where benzylzinc reagents are prepared in the presence of electrophiles effect the corresponding three-component coupling. Generation of secondary benzylzinc reagents and their reaction with electrophiles can also be achieved under Barbier conditions by using phosphate **20**. Ketones, ally bromides, and chlorosilanes are successfully used as electrophiles.

1,2-Migration of arylzincates bearing a leaving group at the remote benzylic position was found to take place in a facile manner at low temperatures although it initially leads to the formation of cross conjugated intermediates and suffers from a considerable loss of aromatic stabilization. The finding suggests that the scope of homologation by 1,2-migration would be extended to other ate complexes in which a negatively charged metal atom center and a potential cationic center are separated by an appropriate π-system.

4. Experimental Section

^1H- and ^{13}C-NMR spectra were recorded at 300 MHz and 75.6 MHz, respectively, in $CDCl_3$. All commercially available reagents were used without further purification unless otherwise noted. THF was distilled from sodium benzophenone ketyl. Triethylamine, diisopropylamine, and CH_2Cl_2 were distilled from CaH_2. Commercial anhydrous $ZnCl_2$ was dried in *vacuo* at 100 °C for 10 h over P_2O_5. All reactions were performed under argon. Reactions at -85 °C were performed with a Neslab Cryo Cool immersion cooler. Organic extracts were dried over Na_2SO_4. Flash chromatography was conducted on silica gel (Wakogel C-300).

Spectral data of following products **6** [35], **8**, **16** [36], **17** [37], **18**, **19** [38], **22** [39], and **30** [40] were in good accordance with reported values available.

4-Iodobenzyl Methanesulfonate (4)

To a solution of 4-iodobenzyl alcohol (1.73 g, 7.39 mmol) in CH_2Cl_2 (22 mL) at 0 °C were added Et_3N (1.23 mL, 8.87 mmol) and methanesulfonyl chloride (0.69 mL, 8.9 mmol). After being stirred for 30 min, the mixture was poured into 1N HCl (50 mL) and extracted twice with ether. The combined organic extracts were washed with aqueous $NaHCO_3$, dried, and concentrated *in vacuo*. Recrystallization of the reside from ethyl acetate gave 1.95 g (85% yield) of **4**: mp 92 - 93 °C; ^{1}H NMR δ 2.94 (3H, s), 5.17 (2H, s), 7.16 and 7.75 (4H, AA'BB'); IR (KBr disk) 1335, 1170, 925, 810 cm^{-1}. Anal. Calcd for $C_8H_9IO_3S$: C, 30.79; H, 2.91. Found: C, 30.79; H, 2.94.

3-Iodobenzyl Methanesulfonate (10)

The compound was prepared from 3-iodobenzyl alcohol in 97% yield by a procedure similar to that described above. **10**: mp 66 - 66.5 °C (recrystallized from ether and hexane); ^{1}H NMR δ 2.97 (3H, s), 5.16 (3H, s), 7.14 (1H, t, J = 7.8 Hz), 7.38 (1H, dd, J = 1.0 and 7.8 Hz), 7.73 (1H, br d, J = *ca.* 8 Hz), 7.76 (1H, br s); IR (KBr disk) 1335, 1170, 945, 795, 690 cm^{-1}. Anal. Calcd for $C_8H_9IO_3S$: C, 30.79; H, 2.91. Found: C, 30.76; H, 2.94.

2-Iodobenzyl Methanesulfonate (12)

The compound was prepared from 2-iodobenzyl alcohol in 93% yield by a procedure similar to that described above. **12**: mp 63 - 64 °C (recrystallized from ether and hexane); ^{1}H NMR δ 3.03 (3H, s), 5.27 (3H, s), 7.09 (1H, dt, J = 1.5 and 7.8 Hz), 7.40 (1H, dt, J = 1.2 and 7.8 Hz), 7.48 (1H, dd, J = 1.5 and 7.8 Hz), 7.89 (1H, dd, J = 1.2 and 7.8 Hz); IR (KBr disk) 1345, 1175, 840, 750 cm^{-1}. Anal. Calcd for $C_8H_9IO_3S$: C, 30.79; H, 2.91. Found: C, 30.77; H, 2.86.

General Procedure for the Preparation of Benzylzinc Reagents

To a solution of $ZnCl_2$ (2.0 mmol) in THF (10 mL) at 0 °C was added a solution of RM (6.0 mmol) (BuLi; 1.6 M in hexane; BuMgBr; 1 M in THF, MeLi; 1.4 M in ether, *s*-BuLi, 1.3 M in cyclohexane; *t*-BuLi; 1.5 M in pentane, *i*-PrMgBr, 0.67 M in THF). The mixture was stirred for 15 min at 0 °C. To the resulting solution of R_3ZnM (2.0 mmol) at -85 °C was add a THF (4 mL) solution of mesylate **4** (1.0 mmol). After being stirred for 0.5 h at this temperature, the mixture was allowed to warm to -40 °C during 1-2 h.

1-Butyl-4-methylbenzene (8) [36]

p-Butylbenzylzinc reagent was prepared by the reaction of mesylate **4** (200 mg, 0.64 mmol) and Bu_3ZnLi (1.28 mmol) in THF (8.4 mL) by a procedure similar to that described above at -85 – -40 °C. After the addition of 1N HCl (*ca.* 1 mL) at -40 °C, the mixture was poured into 1N HCl and extracted twice with hexane. The combined organic extracts were washed with aqueous $NaHCO_3$, dried, and concentrated in *vacuo*. The reside was purified by Kugelrohr distillation (160 -180 °C/12 mmHg) to give 85 mg (90% yield) of **8**.

1-Butyl-4-pentylbenzene (**9**).

p-Butylbenzylzinc reagent was prepared by the reaction of mesylate **4** (189 mg, 0.61 mmol) and Bu_3ZnLi (1.22 mmol) in THF (8.6 mL) by a procedure similar to that described above at -85 – -40 °C. After being allowed to warm to rt during 3 h, the mixture was poured into 1N HCl and extracted twice with hexane. The combined organic extracts were washed with aqueous $NaHCO_3$, dried, and concentrated in *vacuo*. The reside was purified by flash chromatography (10% benzene in hexane) to give 71 mg (57% yield) of **9**: ^1H NMR δ 0.91 (3H, t, $J = 6.9$ Hz), 0.94 (3H, t, $J = 7.0$ Hz), 1.25-1.45 (6H, m), 1.55-1.70 (4H, m), 2.58 (2H, t, $J = 7.2$ Hz), 2.59 (2H, t, $J = 7.8$ Hz), 7.10 (4H, br s); ^{13}C NMR (75.6 MHz, $CDCl_3$) d 13.88, 13.95, 22.37, 22.54, 31.27, 31.56, 33.69, 35.23, 35.53, 128.19, 128.85, 139.96, 140.01; MS, m/z (relative intensity) 204 (M^+, 62); 161 (88), 147 (100); HRMS calcd for $C_{15}H_{24}$ 204.1878, found; 204.1875.

Reaction of 3-Iodobenzyl Mesylate (**10**) *with Bu_3ZnLi at -85 – -40 °C*

To a solution of Bu_3ZnLi (1.2 mmol) in THF (10 mL) was added a solution of mesylate **10** (312 mg, 1.0 mmol) in THF (4 mL) at -85 °C. After being stirred for 15 min, the mixture was allowed to warm at -40 °C during 1 h and then quenched by the addition of 1N HCl. The mixture was poured into 1N HCl and extracted twice with ethyl acetate. The combined organic extracts were washed with aqueous $NaHCO_3$, dried, and concentrated in *vacuo*. The reside was purified by flash chromatography (10-20% ethyl acetate in hexane) to give 132 mg (71% yield) of benzyl mesylate **6** [35].

Reaction of 2-Iodobenzyl Mesylate (**12**) *with Bu_3ZnLi at -85 – -40 °C*

The reaction of **12** was carried out by a procedure similar to that described for mesylte **4**. Kugelrohr distillation (110-140 °C/30 mmHg) of the crude products gave a 1:2.6 mixture of *o*-butyltoluene (**16**) [36] and pentylbenzene (**17**) [37] in 50% combined yield.

1-Butyl-2-pentylbenzene (**18**) [38] *and 2-Iodopentylbenzene* (**19**) [38]

To a solution of Bu_3ZnLi (2.0 mmol) in THF (10 mL) was added a solution of mesylate **12** (312 mg, 1.0 mmol) in THF (4 mL) at -85 °C. After being stirred for 15 min, the mixture was allowed to warm at rt during 2.5 h. A THF (5 mL) solution of I_2 (1.27 g, 5.0 mmol) was added to the mixture at -60 °C. After being stirred for 1 h, the mixture was poured into 1N HCl and extracted twice with hexane. The combined organic extracts were washed successively with aqueous $NaHSO_3$ and aqueous $NaHCO_3$, dried, and concentrated in *vacuo*. The reside was purified by flash chromatography (hexane) to give a 1:1.6 mixture of **18** and **19** in 38% combined yield.

Diethyl 1-(4-Iodophenyl)ethyl Phosphate (**20**)

To a solution of 1-(4-iodophenyl)ethanol (4.72 g, 19.1 mmol) in THF (40 mL) at -78 °C was added a solution of LDA (21 mmol) in THF (40 mL), prepared by the reaction of diisopropylamine and BuLi. The mixture was stirred for 0.5 h. To the mixture was then added diethyl chlorophosphate (19.1 mmol, 3.30 g) at -78 °C. The mixture was allowed to warm to -20 °C during 2.5 h, poured into 1N HCl, and extracted twice with ether. The combined extracts were washed with aqueous $NaHCO_3$, dried, and concentrated in *vacuo*. The residue was purified by flash chromatography (20% ethyl acetate in hexane) to give 6.45 g (88% yield) of **20**: ^1H NMR δ 1.25 (6H, dt, J = 25.2 and 7.0 Hz), 1.59 (3H, d, J = 6.4 Hz), 3.9-4.15 (4H, m), 5.41 (1H, br quintet, J = ca 7 Hz), 7.12 and 7.68 (4H, AA'BB'); IR (liquid film); 1265, 1030, 990, 810 cm^{-1}; MS, m/z (relative intensity) 384 (M^+, 29); 230 (30), 155 (100); HRMS calcd for $C_{12}H_{18}O_4PI$ 383.9989, found; 383.9991.

1-Butyl-4-(1-methylpentyl)benzene (25)

The reaction of phosphate **20** (188 mg, 0.49 mmol) and Bu_3ZnLi (0.98 mmol) in THF (7 mL) at -85 °C - rt was performed by a procedure similar to that described for the preparation of 4-butylpentylbenzene (**9**). The crude product was purified by Kugelrohr distillation (120 °C / 0.6 mmHg) to give 91 mg (85% yield) of **25**: ^1H NMR δ 0.85 (3H, t, J = 7.1 Hz), 0.92 (3H, t, J = 7.4 Hz), 1.10-1.43 (9H, m, including d (3H, J = 7.0 Hz) at 1.21 ppm), 1.47-1.66 (2H, m), 2.57-2.68 (3H, m, including t (2H, J = 7.2 Hz) at 2.57 ppm), 7.09 (4H, br s); IR (liquid film) 835 cm^{-1}; MS, m/z (relative intensity) 218 (M^+, 15); 161 (100); HRMS calcd for $C_{16}H_{26}$ 218.2034, found; 218.2037.

Reaction of Phosphate (20) *with Bu_3ZnLi at -85 – -40 °C*

The reaction of phosphate **20** (192 mg, 0.50 mmol) and Bu_3ZnLi (1.0 mmol) in THF (7 mL) at -85 - -40 °C was carried out by a procedure similar to that described for the preparation of *p*-butyltoluene (**8**). Flash chromatography (0-20% ethyl acetate in hexane) of the crude products gave, in the order of elution, a 8:14 mixture of 1-ethyl-4-butylbenzene (**24**) and 1-butyl-4-(1-methylpentyl)benzene (**25**) (21.8 mg, 22% combined yield) and diethyl 1-phenylethyl phosphate (**22**) [39] (65.8 mg, 51% yield).

4-Butylstyrene (28)

To a solution of Bu_3ZnLi (2.0 mmol) in THF (10 mL) was added a solution of 4-iodostyrene oxide (**26**) [41] (246 mg, 1.0 mmol) in THF (4 mL) at -85 °C. After 15 min, $BF_3 \cdot OEt_2$ (0.25 mL, 2.0 mmol) was added and the mixture was allowed to warm at rt during 2.5 h, poured into 1N HCl, and extracted twice with ether. The combined organic extracts were washed with aqueous $NaHCO_3$, dried, and concentrated in *vacuo*. The reside was purified by flash chromatography (hexane) to give 75.2 mg (47% yield) of **28**.

1-(4-Butylphenyl)-4-phenyl-2-butanol (29a) *(Table 1, entry 1)*

p-Butylbenzylzinc reagent was prepared by the reaction of mesylate **4** (198 mg, 0.63 mmol) and Bu_3ZnLi (1.27 mmol) in THF (9.0 mL) by a procedure similar to that described above. To the mixture at -85 °C was added 3-phenylpropanal (103 mg, 0.77 mmol). After being stirred at this temperature for 1 h, the mixture was poured into 1N HCl and extracted twice with ethyl acetate. The combined organic extracts were washed with aqueous $NaHCO_3$, dried, and concentrated in *vacuo*. The reside was purified by flash chromatography (5% ethyl acetate in hexane) to give 142 mg (80% yield) of **29a**: ^1H NMR δ 0.94 (3H, t, J = 7.3 Hz), 1.38 (2H, quintet, J = 7.5 Hz), 1.54-1.66 (3H, m), 1.81-1.90 (2H, m), 2.57-2.92 (6H, m), 3.83 (1H, tt, J = 4.5 and 7.9 Hz), 7.13 (4H, br s), 7.18-7.33 (5H, m); IR (liquid film) 3280 (br), 1045, 730, 700 cm^{-1}; MS, m/z (relative intensity) 282 (M^+, 45); 147 (75), 85 (100); HRMS calcd for $C_{20}H_{26}O$ 282.1984, found; 232.1978. Anal. Calcd for $C_{20}H_{26}O$: C, 85.06; H, 9.28. Found: C, 84.94; H, 9.49.

The following alcohols were prepared by a procedure similar to that described above.

1-(4-Butylphenyl)-3-methyl-2-butanol (29b) *(Table 1, entries 2 and 3)*

29b: ^{1}H NMR δ 0.94 (3H, t, *J* = 7.3 Hz), 1.01, (6H, d, *J* = 6.8 Hz), 1.38 (2H, quintet, *J* = 7.5 Hz), 1.55 (1H, br s), 1.56-1.66 (2H, m), 1.76 (1H, d septet, *J* = 5.3 and 6.8 Hz), 2.57 (1H, dd, *J* = 9.4 and 13.6 Hz), 2.60 (2H, t, *J* = 7.6 Hz), 2.83 (1H, dd, *J* = 3.5 and 13.6 Hz), 3.57 (1H, m), 7.14 (4H, br s); IR (liquid film) 3420 (br), 1115, 805 cm^{-1}; MS, m/z (relative intensity) 220 (M$^+$, 10); 148 (100), 105 (90); HRMS calcd for $C_{15}H_{24}O$ 220.1827, found; 220.1833.

3-Methyl-[4-(1-methylpropyl)phenyl]-2-butanol (29c) *(Table 1, entry 4)*

29c: ^{1}H NMR δ 0.82 (3H, t, *J* = 7.3 Hz), 1.00 (6H, d, *J* = 7.1 Hz), 1.22 (3H, d, *J* = 7.0 Hz), 1.58 (3H, m), 1.75 (1H, d septet, *J* = 5.2 and 7.1 Hz), 2.56 (1H, dd, *J* = 9.6 and 13.7 Hz), 2.59 (1H, quintet, *J* = 6.9 Hz), 2.83 (1H, dd, *J* = 3.5 and 13.7 Hz), 3.57 (1H, ddd, *J* = 3.5, 5.2, and 9.6 Hz), 7.13 (4H, m); IR (liquid film) 3420 (br), 1100, 805 cm^{-1}; MS, m/z (relative intensity) 220 (M$^+$, 5); 148 (36), 119 (100); HRMS calcd for $C_{15}H_{24}O$ 220.1827, found; 220.1821.

3-Methyl-[4-(1,1-dimethylethyl)phenyl]-2-butanol (29d) *(Table 1, entry 5)*

29d: ^{1}H NMR δ 1.00 (6H, d, *J* = 6.8 Hz), 1.32 (9H, s), 1.51 (1H, br s), 1.76 (1H, br octet, *J* = *ca.* 6.5 Hz), 2.56 (1H, dd, *J* = 9.7 and 13.7 Hz), 2.83 (1H, dd, *J* = 4.1 and 13.7 Hz), 3.58 (1H, m), 7.16 and 7.34 (4H, AA'BB'); IR (liquid film) 3420 (br), 1110, 1025, 810 cm^{-1}; MS, m/z (relative intensity) 220 (M$^+$, 10); 161 (27), 133 (100); HRMS calcd for $C_{15}H_{24}O$ 220.1827, found; 220.1836.

[1-(4-Butylphenyl)methyl]cyclohexanol (29e) *(Table 1, entry 7)*

29e: ^{1}H NMR δ 0.93 (3H, t, *J* = 7.2 Hz), 1.20-1.66 (15H, m), 2.59 (2H, t, *J* = 7.6 Hz), 2.72 (2H, s), 7.12 (4H, br s); IR (liquid film) 3380 (br), 1140, 980, 835 cm^{-1}; MS, m/z (relative intensity) 246 (M$^+$, 1); 228 (5), 148 (100); HRMS calcd for $C_{17}H_{26}O$ 246.1984, found; 246.1983. Anal. Calcd for $C_{17}H_{26}O$: C, 82.47; H, 10.64. Found: C, 82.60; H, 10.73.

1-(4-Butylphenyl)-2-phenyl-2-propanol (29f) *(Table 1, entry 8)*

29f: ^{1}H NMR δ 0.92 (3H, t, *J* = 7.3 Hz), 1.34 (2H, sextet, *J* = 7.3 Hz), 1.53-1.63 (5H, m, including s (3H) at 1.56 ppm), 2.32 (1H, s), 2.57 (2H, t, *J* = 7.8 Hz), 2.99 (1H, d, *J* = 13.4 Hz), 3.11 (1H, d, *J* = 13.4 Hz), 6.92 and 7.04 (4H, AA'BB), 7.23-7.45 (5H, m); IR (liquid film) 3550 (br) ,3450 (br), 1115, 765, 700 cm^{-1}; MS, m/z (relative intensity) 268 (M$^+$, <1); 148 (96), 121 (100); HRMS calcd for $C_{19}H_{24}O$ 268.1827, found; 268.1835.

1-(4-Butylphenyl)-2-hexanone (29g) *(Table 1, entry 9)*

p-Butylbenzylzinc reagent was prepared by the reaction of mesylate **4** (297 mg, 0.95 mmol) and Bu_3ZnLi (1.90 mmol) in THF (14 mL) by a procedure similar to that described above. To the mixture at -85 °C was added pentanoyl chloride (137 mg, 1.14 mmol). After being stirred at this temperature for 2 h, the mixture was poured into 1N HCl and extracted twice with ethyl acetate. The combined organic extracts were washed with aqueous $NaHCO_3$, dried, and concentrated in *vacuo*. The reside was purified by flash chromatography (5% ethyl acetate in hexane) to give 135 mg (61% yield) of **29g**: ^1H NMR δ 0.86 (3H, t, *J* = 7.3 Hz), 0.92 (3H, t, *J* = 7.2 Hz), 1.20-1.43 (4H, m), 1.48-1.66 (4H, m), 2.44 (2H, t, *J* = 7.5 Hz), 2.59 (2H, t, *J* = 7.8 Hz), 3.64 (2H, s), 7.07-7.16 (4H, AA'BB'); IR (liquid film) 1710, 805, 735 cm^{-1}; MS, m/z (relative intensity) 232 (M^+, 45); 147 (75), 85 (100); HRMS calcd for $C_{16}H_{24}O$ 232.1827, found; 232.1819. Anal. Calcd for $C_{16}H_{24}O$: C, 82.70; H, 10.41. Found: C, 82.35; H, 10.38.

1-(4-Butylphenyl)-3,3-dimethyl-2-butanone (29h) *(Table 1, entry 10)*

The ketone was prepared by a procedure similar to that described above. **29h**: ^1H NMR δ 0.91 (3H, t, *J* = 7.3 Hz), 1.19 (9H, s), 1.34 (2H, sextet, *J* = 7.3 Hz), 1.52-1.63 (2H, m), 2.57 (2H, t, *J* = 7.6 Hz), 3.76 (2H, s), 7.05-7.14 (4H, AA'BB'); IR (liquid film) 1710, 805 cm^{-1}; MS, m/z (relative intensity) 232 (M^+, 3); 159 (5), 147 (7), 91 (100); HRMS calcd for $C_{16}H_{24}O$ 232.1827, found; 232.1828.

(4-Butylphenyl)acetonitrile (29i) *(Table 1, entry 11)*

p-Butylbenzylzinc reagent was prepared by the reaction of mesylate **4** (312 mg, 1.00 mmol) and Bu_3ZnLi (2.00 mmol) in THF (14 mL) by a procedure similar to that described above. To the mixture at -85 °C was added a THF (2.5 mL) solution of *p*-toluenesulfonyl cyanide (218 mg, 1.20 mmol). After being stirred at this temperature for 2.5 h, the mixture was poured into 1N HCl and extracted twice with ethyl acetate. The combined organic extracts were washed with aqueous $NaHCO_3$, dried, and concentrated in *vacuo*. The reside was purified by flash chromatography (5% ethyl acetate in hexane) to give 101 mg (58% yield) of **29i**: ^1H NMR δ 0.93 (3H, t, *J* = 7.3 Hz), 1.34 (2H, sextet, *J* = 7.3 Hz), 1.53-1.64 (2H, m), 2.61 (2H, t, *J* = 7.8 Hz), 3.71 (2H, s), 7.17-7.28 (4H, AA'BB'); ^{13}C NMR (75.6 MHz, $CDCl_3$) d 13.82, 22.21, 23.17, 33.45, 35.13, 117.88, 126.97, 127.74, 129.09, 142.83; IR (liquid film) 2250, 805 cm^{-1}; MS, m/z (relative intensity) 173 (M^+, 35); 130 (100), 91 (28); HRMS calcd for $C_{12}H_{15}N$ 173.1204, found; 173.1210. Anal. Calcd for $C_{12}H_{15}N$: C, 83.19; H, 8.73. Found: C, 83.19; H, 8.71.

(4-Butylphenyl)dimethylphenylsilane (29j) *(Table 1, entry 12)*

p-Butylbenzylzinc reagent was prepared by the reaction of mesylate **4** (304 mg, 0.97 mmol) and Bu_3ZnLi (1.94 mmol) in THF (14 mL) by a procedure similar to that described above. To the mixture at -85 °C was added chlorodimethylphenylsilane (198 mg, 1.16 mmol). After being stirred at this temperature for 2 h, the mixture was poured into 1N HCl and extracted twice with ethyl acetate. The combined organic extracts were washed with aqueous $NaHCO_3$, dried, and concentrated in *vacuo*. The reside was purified by flash chromatography (hexane) to give 171 mg (62% yield) of **29j**: ^1H NMR δ 0.30 (6H, s), 0.98

(3H, t, J = 7.3 Hz), 1.39 (2H, sextet, J = 7.6 Hz), 1.56-1.68 (2H, m), 2.32 (2H, s), 2.60 (2H, t, J = 7.8 Hz), 6.91 and 7.05 (4H, AA'BB'), 7.41 (3H, m), 7.51 (2H, m); IR (liquid film) 1250, 1115, 840 cm^{-1}; MS, m/z (relative intensity) 282 (M$^+$, 24); 267 (3), 135 (100); HRMS calcd for $C_{19}H_{26}Si$ 282.1804, found; 282.1808.

[1-(4-Methylphenyl)methyl]cyclohexanol (**29k**) *(Table 2, entry 1). **Typical Procedure for Three-Component Coupling under Barbier Conditions***

To a solution of Me_3ZnLi (2.1 mmol) in THF (10 mL) at -85 °C was added a THF (4 mL) solution of mesylate 4 (329 mg, 1.05 mmol). The mixture was stirred 15 min. To this at -85 °C was added cyclohexanone (0.13 mL, 1.3 mmol). The resulting mixture was allowed to warm to -40 °C during 3 h. The mixture was poured into 1N HCl and extracted twice with ethyl acetate. The combined organic extracts were washed with aqueous $NaHCO_3$, dried, and concentrated in *vacuo*. The reside was purified by silica gel flash chromatography (5% ethyl acetate in hexane) to give 111 mg (52% yield) of **29k**: ^{1}H NMR δ 1.3-1.7 (11H, m), 2.32 (3H, s), 2.70 (2H, s), 7.10 (4H, m); IR (liquid film) 3450 (br) ,975, 810 cm^{-1}; MS, m/z (relative intensity) 204 (M$^+$, 50); 161 (62), 106 (100); HRMS calcd for $C_{14}H_{20}O$ 204.1514, found; 204.1523.

The following products were prepared by a procedure similar to that described above.

2,3-Dimethyl-1-[(4-Methylphenyl)methyl]-2-butanol (**29m**) *(Table 2, entry 2)*

29m: ^{1}H NMR δ 0.98 (3H, d, J = 6.3 Hz), 0.99 (3H, d, J = 6.9 Hz), 1.03 (3H, s), 1.24 (1H, br), 1.72 (1H, sept, J = 6.5 Hz), 2.33 (3H, s), 2.67 (1H, d, J = 13.2 Hz), 2.78 (1H, d, J = 13.2 Hz), 7.12 (4H, br s); IR (liquid film) 3470 (br), 1085, 810 cm^{-1}; MS, m/z (relative intensity) 192 (M$^+$, 1); 149 (34), 106 (100); HRMS calcd for $C_{13}H_{20}O$ 192.1514, found; 192.1521.

4-(3-Butenyl)butylbenzene (**29n**) *(Table 2, entries 4 and 5)*

29n: ^{1}H NMR δ 0.93 (3H, t, J = 7.4 Hz), 1.3-1.45 (2H, m), 1.5-1.65 (2H, m), 2.3-2.45 (2H, m), 2.55-2.65 (2H, m), 2.65-2.75 (2H, m), 4.95-5.10 (2H, m), 5.87 (1H, tdd, J = 6.3, 10.5, and 17.1 Hz), 7.10 (4H, br s); IR (liquid film) 1640, 910, 825 cm^{-1}; MS, m/z (relative intensity) 188 (M$^+$, 19); 147 (100), 91 (25); HRMS calcd for $C_{14}H_{20}$ 188.1565, found; 188.1568.

p-Butyl(4-phenyl-3-butenyl)benzene (**29o**) *(Table 2 entry 6)*

29o: ^{1}H NMR δ 0.94 (3H, t, J = 7.4 Hz), 1.3-1.4 (2H, m), 1.55-1.65 (2H, m), 2.50-2.6 (4H, m), 2.75-2.8 (2H, m), 6.28 (1H, td, J = 6.9 and 15.6 Hz), 6.43 (1H, br d, J = 15.6 Hz), 7.1-7.4 (9H, m).; IR (liquid film) 965, 830, 740, 695 cm^{-1}; MS, m/z (relative intensity) 264 (M$^+$, 32); 147 (100), 117 (59); HRMS calcd for $C_{20}H_{24}$ 264.1878, found; 264.1881. Anal. Calcd for $C_{20}H_{24}$: C, 90.85; H, 9.15. Found: C, 90.55; H, 9.27.

[1-{4-(1-methylethyl)phenyl}methyl]cyclohexanol (**29p**) *(Table 2, entry 7)*

29p: ^{1}H NMR δ 1.24 (6H, d, J = 6.9 Hz), 1.27 (1H, br), 1.4-1.65 (10H, m), 2.71 (2H, s), 2.88 (1H, sept, J = 6.9 Hz), 7.1-7.2 (4H, m); IR (liquid film) 3460 (br) ,985, 850 cm^{-1};

MS, m/z (relative intensity) 232 (M+, 36); 214 (65), 134 (100), 119 (95); HRMS calcd for $C_{16}H_{24}O$ 232.1819, found; 232.1827.

[1-(4-Butylphenyl)]ethylcyclohexanol (**29q**) *(Table 2, entries 8 and 9)*

29q: : ^{1}H NMR δ 0.93 (3H, t, J = 7.3 Hz), 1.25-1.70 (18 H, m, including d (3H, J = 7.1 Hz) at 1.30 ppm), 2.58 (2H, d, J = 7.7 Hz), 2.73 (1H, q, J = 7.1 Hz), 7.12 (4H, AA'BB'); IR (liquid film) 3470 (br), 1150, 950, 840 cm^{-1}; MS, m/z (relative intensity) 260 (M+, 1); 162 (100), 119 (56); HRMS calcd for $C_{18}H_{28}O$ 260.2140, found; 260.2149. Anal. Calcd for $C_{18}H_{28}O$: C, 83.02; H, 10.84. Found: C, 83.29; H, 10.78.

2-(4-Butylphenyl)-3,4-dimethyl-3-pentanol (**29r**) *(Table 2, entry 10)*

29r: ^{1}H NMR δ 0.91 (3H, d, J = 6.9 Hz), 0.92 (3H, t, J = 6.9 Hz), 0.93 (3H, d, J = 6.9 Hz), 1.06 (3H, s), 1.28 (3H, d, J = 6.8 Hz), 1.35 (2H, m), 1.55-1.65 (4H, m), 2.58 (2H, t, J = 7.5 Hz), 2.98 (1H, q, J = 6.9 Hz), 7.10 and 7.18 (4H, AA'BB'); IR (liquid film) 3489 (br) ,1085, 835 cm^{-1}; MS, m/z (relative intensity) 248 (M+, 15); 205 (52), 162 (100); HRMS calcd for $C_{17}H_{28}O$ 248.2140, found; 248.2149.

4-Butyl(1-methyl-3-butenyl)benzene (**29s**) *(Table 2, entry 11)*

29s: ^{1}H NMR δ 0.92 (3H, t, J = 7.3 Hz), 1.23 (3H, d, J = 7.0 Hz), 1.35 (2H, septet, J = 7.3 Hz), 1.50-1.65 (2H, m), 2.19-2.43 (2H, m), 2.57 (2H, t, J = 7.7 Hz), 2.75 (1H, sextet, J = *ca.* 7 Hz), 4.92-5.03 (2H, m), 5.71 (1H, m), 7.10 (4H, br s); IR (liquid film) 1640, 990, 910, 830, 820 cm^{-1}; MS, m/z (relative intensity) 202 (M+, 8); 161 (100); HRMS calcd for $C_{15}H_{22}$ 232.1827, found; 202.1721. Anal. Calcd for $C_{15}H_{22}O$: C, 89.04; H, 10.96. Found: C, 88.79; H, 10.92.

1-(4-Butylphenyl)ethyltrimethylsilane (**29t**) *(Table 2, entry 12)*

29t: ^{1}H NMR δ -0.70 (9H, s), 0.92 (3H, t, J = 7.2 Hz), 1.3-1.4 (5H, m, including d (3H, J = 7.5 Hz) at 1.34), 1.5-1.65 (2H, m), 2.12 (1H, q, J = 7.5 Hz), 2.55 (2H, t, J = 6.6 Hz), 6.94 and 7.04 (4H, AA'BB'); IR (liquid film) 1250, 840 cm^{-1}; MS, m/z (relative intensity) 234 (M+, 33); 160 (55), 73 (100); HRMS calcd for $C_{15}H_{26}Si$ 234.1804, found; 234.1803.

1-[1-{4-(1-methylethyl)phenyl}]ethylcyclohexanol (**29u**) *(Table 2, entry 13)*

29u: ^{1}H NMR δ 1.1-1.7 (20H, m, including d (6H, J = 6.9 Hz) at 1.24 and d (3H, J = 7.2 Hz) at 1.29), 2.76 (1H, q, J = 6.9 Hz), 2.88 (1H, sept, J = 6.9 Hz), 7.15 (4H, br s); IR (liquid film) 3480 (br), 955, 840 cm^{-1}; MS, m/z (relative intensity) 246 (M+, 2); 148 (100), 133 (63); HRMS calcd for $C_{17}H_{26}O$ 246.1984, found; 246.1987.

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