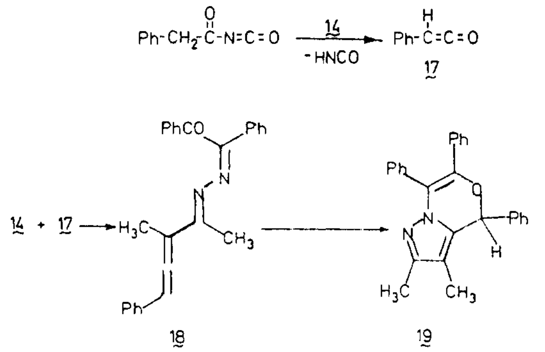
Scheme II



Preparation of 4*H*-Pyrazolo[1,5-*c*][1,3,5]oxadiazines 16. General Method. A solution of the phosphorane 14 (2.0 mmol) and acyl isocyanate (2.5 mmol) in 20 mL toluene was stirred at room temperature for 30 min and then under reflux for 2 h. The solvent was removed in vacuo, and the residue was chromatographed on a silica gel column by eluting with ethyl acetate/hexane (1/7), yielding first the 4*H*-pyrazolo[1,5-*c*][1,3,5]oxadiazine 16 and then triphenylphosphine oxide. Recrystallization of 16 from ether and/or hexane (as indicated in Table I) afforded a colorless analytically pure sample. Isolated yields, melting points, ^{1}H NMR (Table I), and ^{13}C NMR (Table II) were collected separately.

Reaction of Phosphorane 14 with Phenylacetyl Isocyanate. The reaction was undertaken in the manner described above in General Method. The chromatographed product was recrystallized from an ether/hexane mixture to yield the known 2,3-dimethyl-4,6,7-triphenyl-4*H*-pyrazolo[5,1-c][1,4]oxazine 19 (46%). The melting point, mixed melting point, and ^{1}H and ^{13}C NMR were identical with those of the previously reported sample.[2]

Acknowledgment. The partial support of the National Institute of General Medical Science (GM 27620) is gratefully acknowledged. We thank Dr. Roger Crecely and Mr. Guang Wu of the University of Delaware for obtaining the ^{1}H and ^{13}C NMR spectra reported in this work. We thank the mass spectroscopy lab of the University of Delaware for the mass spectral data. Purchase of the Bruker WM 250 was supported, in part, by a grant (GM 27616) from the National Institutes of Health.

Reaction of 2-Aryl-3-(*N*,*N*-dimethylamino)-1-propenes and Their Corresponding Quaternary Ammonium Salts with Organometallic Species and Reducing Agents

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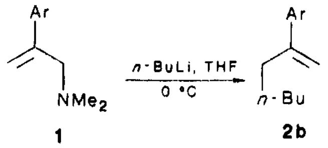
A series of 2-aryl-3-(*N*,*N*-dimethylamino)-1-propenes and their corresponding quaternary ammonium salts were reacted with a variety of organometallic species and reducing agents. The utility of such reactions for the preparation of α-substituted styrene derivatives is discussed.

We have recently reported[1] that a variety of 2-aryl-3-(*N*,*N*-dimethylamino)-1-propenes react with butyllithium and *tert*-butyllithium in THF at 0 °C to produce the corresponding 3-substituted 2-aryl-1-propenes in good yield (Scheme I).

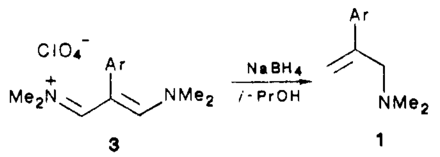
We believe this work to be interesting and useful in light of the recent studies by Richey[2] on the reaction of 1-aryl-3-(*N*,*N*-dimethylamino)-1-propenes with organolithium reagents. For example, Richey has reported that 1-(*N*,*N*-dimethylamino)-2-benzylhexane is produced as the major reaction product by the treatment of *n*-butyllithium with 1-phenyl-3-(*N*,*N*-dimethylamino)-1-propene in refluxing hexane. It has been suggested that the allylic amine group facilitates the addition of the organometallic reagent to the double bond by coordination with lithium.

Goering[3] and co-workers have also recently reported on the cross-coupling reactions of analogous allylic carboxylates with Grignard reagents under the influence of various Cu(I) salts. One example that was particularly intriguing to us was the regioselective addition of *n*-butylmagnesium bromide to the pivalate ester of 3-deuterio-2-phenyl-2-propenol to give 3-deuterio-2-phenyl-1-heptene in good yield.

Scheme I



Scheme II



Our interest in 2-aryl-3-(*N*,*N*-dimethylamino)-1-propenes stems from the fact that these substances are readily available[4] from the corresponding vinamidinium salts by sodium borohydride reduction (Scheme II).

Such systems are intriguing since the double bond has the potential of being activated to some extent by the aromatic ring. Magid[5] has extensively reviewed the nu-

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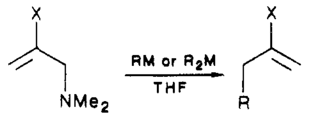
(2) Richey, H.; Heyn, A.; Erikson, W. *J. Org. Chem.* **1983**, *48*, 3822.

(3) Goering, H.; Tseng, C.; Paisley, S. *J. Org. Chem.* **1986**, *51*, 2884.

(4) Gupton, J.; Andrew, S.; Lizzi, M. *Synth. Commun.* **1982**, *12*, 361.

0022-3263/87/1952-3683$01.50/0

Table I. Reaction of 2-Aryl-3-(*N*,*N*-dimethylamino)-1-propenes with Organometallic Reagents



1 2

entry	X	R or R_2	M	% yield	bp,[a] °C (press., mm)
2a	4-MeOPh	*t*-Bu	Li	91 (61)[1]	72–76 (0.5)
2b	4-MeOPh	*n*-Bu	Li	75 (44)[1]	72–76 (0.5)
2c	4-MeOPh	Me	MgBr	[b]	
2d	4-MeOPh	Me	MgBr	[b,c]	
2e	4-MeOPh	(*n*-Bu)$_2$	CuLi	33 (22)[1]	72–76 (0.5)

[a] All compounds were purified by distillation or recrystallization and were found to be greater than 95% pure as determined by GLC analysis on a Shimadzu GC 7A gas chromatograph with a $^1/_8$ in. × 6 ft 3% SB-2401 column at 120–150 °C. The yields in parentheses represent purified products. [b] No reaction. [c] A small amount of CuI (10 mol %) was used in this reaction.

cleophilic and organometallic displacement reactions of allylic compounds and few if any substitution reactions can be found where the leaving group has the amino functionality. In many instances where allylic systems do possess poor leaving groups, transition-metal complexes must replace organolithium or Grignard reagents in order to make such substitution reactions feasible. The intent of this article will be to further define and delineate the reactions of 2-aryl-3-(*N*,*N*-dimethylamino)-1-propenes and their corresponding quaternary ammonium salts with both organometallic species and reducing agents.

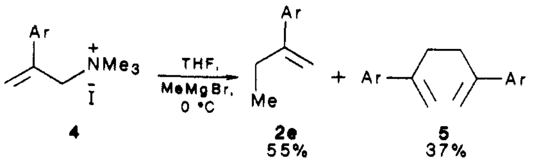
We first decided to examine the feasibility of other organometallic species reacting with the 2-aryl-3-(*N*,*N*-dimethylamino)-1-propenes (See Table I). Examples a and b in Table I represent standard runs with *n*-butylithium or *tert*-butylithium which have been previously reported.[1] Subsequent trials involving methylmagnesium bromide with and without copper(I) salts resulted in the isolation of only starting material. However, when Li(n-Bu)$_2$Cu was the reactant, substitution did occur albeit in low conversion with a substantial amount of starting material being recovered.

Since amine–metal coordination appeared to facilitate these reactions, we decided to next investigate the reaction of electrophilic reducing agents with these 2-aryl-3-(*N*,*N*-dimethylamino)-1-propenes in the hope of obtaining an intramolecular hydride transfer. When allylic amines were treated with DIBAL-H and BH_3 no reaction was observed.

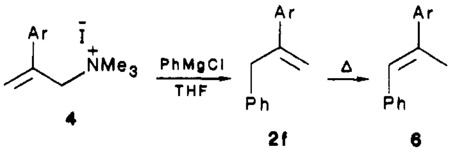
The lack of reaction of Grignard reagents and electrophilic reducing agents with the 2-aryl-3-(*N*,*N*-dimethylamino)-1-propenes was disappointing, and we then turned our attention to the preparation and reaction of the corresponding allylic quaternary ammonium salts. The quaternary salts were easily prepared in nearly quantitative yield by stirring a mixture of excess methyl iodide in THF with the requisite allylic amine and then filtering off the desired solid product. These compounds were easily characterized by their broad singlet at δ 3.05 in the ^{1}H NMR spectrum, which corresponds to the $N(CH_3)_3$ group. The downfield shift of the $CH_2N(CH_3)_3$ hydrogens to δ 4.60 after quaternization was also consistent with the anticipated structure.

The 2-aryl quaternary ammonium salts were then reacted with *tert*-butyllithium and *n*-butyllithium to give the corresponding 2-arylalkenes in good yield. These reactions proved to be more complete than for the corresponding amines since no recovered starting material was observed (see Table II). Methyllithium and phenyllithium did not react well with either the amine or the quarternary ammonium salts. However, both methylmagnesium bromide and phenylmagnesium bromide were effectve reactants. The methylmagnesium bromide reaction was intriguing since a significant amount of a coupling product was obtained (Scheme III).

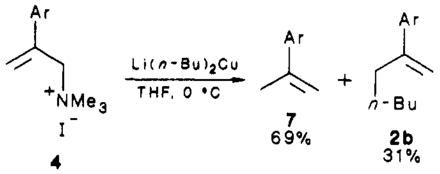
Scheme III



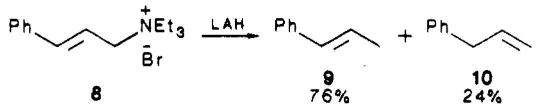
Scheme IV



Scheme V



Scheme VI



Interestingly, when the reaction was repeated (entry h, Table II) with 15 mol % CuI present, only the 2-aryl-1-butene was isolated (82% yield), and very little if any 1,5-hexadiene could be detected by TLC analyses. The reaction of phenylmagnesium chloride with the 2-aryl quaternary ammonium salts also proceeded smoothly to give a 2,3-diaryl-1-propene in good yield (Scheme IV). However, distillation of the crude material resulted in complete isomerization to the 1,2-diaryl-1-propene.

The isomerization was easily followed by the conversion of the vinylidene hydrogens to a vinyl methyl group in the ^{1}H NMR spectrum. Subsequently, the reaction of a 2-aryl quaternary ammonium salt with Li(n-Bu)$_2$Cu was studied. This reaction proved to be quite revealing in that the major product was the 2-arylpropene (Scheme V).

Since the 2-aryl quaternary ammonium salts 4 were found to react cleanly and in good yield with a variety of organometallic species, we decided to evaluate their reactivity with some metal hydride type reducing agents. This was in part suggested by the work of Nojima[6] et al. regarding the lithium aluminum hydride reduction of the isomeric system (Scheme VI).

There was no indication in this report that less reactive reducing agents than LAH would effect a reductive transformation. The results of our study are reported in Table III. A variety of 2-aryl-substituted quaternary ammonium salts were reacted with sodium borohydride

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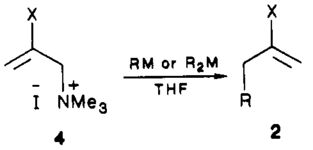
(5) Magid, R. *Tetrahedron*, **1980**, *36*, 1901.

(6) Hirabe, T.; Nojima, M.; Kusobayashi, S. *J. Org. Chem.* **1984**, *49*, 4084.

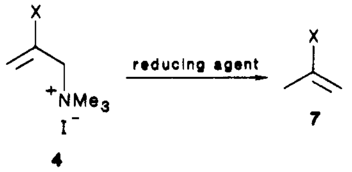
Table II. Reaction of (2-Arylprop-1-en-3-yl)trimethylammonium Iodides with Organometallic Reagents



entry	X	R or R_2	M	% yield	bp,[a] °C (press., mm)
2a	4-MeOPh	*t*-Bu	Li	92(76)	72–76[1] (0.5)
2b	4-MeOPh	*n*-Bu	Li	87 (81)	72–76[1] (0.5)
2c	4-MeOPh	Me	Li	*f*	
2d	4-MeOPh	Ph	Li	*g*	
2e	4-MeOPh	Me	MgBr	(55)[b]	53–54 (0.3)
2f	4-MeOPh	Ph	MgCl	100(84)[c]	114–115 (0.3)
2g	4-MeOPh	$(n\text{-Bu})_2$	CuLi	98[d]	
2h	4-MeOPh	Me	MgBr	82 (70)[e]	51–52 (0.3)
2i	3,4-$(MeO)_2$Ph	*n*-Bu	Li	91 (45)	85–91[1] (0.3)
2j	4-MePh	*n*-Bu	Li	100 (74)	68–69[1] (0.3)
2k	1-naphthyl	*n*-Bu	Li	100 (50)	91–96 (0.3)

[a] All compounds were purified by distillation or recrystallization and were found to be greater than 95% pure as determined by GLC analysis on a Shimadzu GC7A gas chromatograph with a $^1/_8$ in. × 6 ft 3% SB-2401 column at 120–150 °C. The yields in parentheses represent purified products. [b] This reaction mixture consisted of a 55% yield of 2-(4-methoxyphenyl)-1-butene and a 37% yield of 2,5-bis(4-methoxyphenyl)-1,5-hexadiene, which were easily separated by vacuum distillation. Please see Experimental Section for details. [c] The initially obtained product from this reaction was the anticipated 2-(4-methoxyphenyl)-3-phenyl-1-propene. Distillation of this material at reduced pressure resulted in complete isomerization to 2-(4-methoxyphenyl)-1-phenyl-1-propene. [d] This reaction mixture consisted of 69% 2-(4-methoxyphenyl)propene and 31% 2-(4-methoxyphenyl)-1-heptene[1] as determined by GLC analysis. [e] A small amount of CuI (15 mol%) was used in this reaction. [f] Gross mixture of products. [g] Starting material.

Table III. Reduction of (2-Arylprop-1-en-3-yl)trimethylammonium Iodides



entry	X	reduc agent	solv	% yield	bp,[a] °C (press., mm)
7a	4-MeOPh	$NaBH_4$	*i*-PrOH	95 (80)	42–46 (0.3)
7b	4-ClPh	$NaBH_4$	*i*-PrOH	98 (75)	43–47 (0.3)
7c	3,4-$(MeO)_2$Ph	$NaBH_4$	*i*-PrOH	92 (71)	74–75 (0.3)
7d	1-naphthyl	$NaBH_4$	*i*-PrOH	95 (79)	55–60 (0.3)
7e	4-MePh	$NaBH_4$	*i*-PrOH	61 (36)	44–45 (0.3)
7f	Ph	$NaBH_4$	*i*-PrOH	64 (25)	90–95[b] (100)
7g	4-MeOPh	L-Selectride	THF	100 (81)	45–51 (0.3)
7h	4-MeOPh	Red Al	THF	100 (90)	45–51 (0.3)
7i	4-MeOPh	$NaBH_3CN$	*i*-PrOH	[c]	

[a] All reaction products were found to be greater than 95% pure as determined by GLC analysis on a Shimadzu GC7A gas chromatograph with a $^1/_8$ in. × 6 ft 3% SB-2401 column at 120–150 °C. The yields in parentheses represent distilled products. [b] This compound was identical by NMR and IR with an authentic sample of α-methylstyrene. [c] No reaction.

in refluxing isopropyl alcohol to give the respective 2-arylpropenes in good yield. Not only did sodium borohydride work well but also L-Selectride and Red AL when THF was used as the solvent. Sodium cyanoborohydride in refluxing isopropyl alcohol did not work.

The data to date suggests that both 2-aryl-3-(*N*,*N*-dimethylamino)-1-propenes and their corresponding quaternary ammonium salts react cleanly with organolithium reagents to give 3-substituted 2-aryl-1-propenes in good yield. Although no significant mechanistic information is currently available for such reactions, an addition–elimination sequence seems plausible. In addition, Grignard reagents give analogous reaction with the quaternary ammonium salts but not with the amines. Organocuprates react in an analogous fashion with the amines but give primarily reduction products with the quaternary ammonium salts probably via an electron transfer process. The amines do not react with electrophilic reducing agents, but the quaternary ammonium salts do react with a variety of mild reducing agents to give α-methylstyrene analogues in good yield. The reduction process may be occurring by an addition–elimination sequence also.

Experimental Section[7]

The following procedures are typical of the experimental conditions used for the reaction of 2-aryl-3-(*N*,*N*-dimethylamino)-1-propenes and their corresponding quaternary ammonium salts with organometallic species and reducing agents.

Reaction of 2-(4-Methoxyphenyl)-3-(*N*,*N*-dimethylamino)-1-propene with *tert*-Butyllithium. Anhydrous THF (50 mL) and 2.0 g (0.01 mol) of 2-(*p*-methoxyphenyl)-3-(*N*,*N*-dimethylamino)-1-propene were placed in a nitrogen-flushed flask equipped with a septum inlet and magnetic stirring bar. The mixture was cooled to 0 °C with an ice bath, 17.3 mL (0.03 mol) of a solution of *tert*-butyllithium (1.8 M in pentane) was added via syringe, and the resulting mixture was stirred for 4 h. The reaction mixture was quenched with 10 mL of water, and the solvent was removed in vacuo. The residue was partitioned between water (70 mL) and chloroform (3 × 70 mL), and the combined chloroform phases were extracted with 5% aqueous hydrochloric acid (3 × 15 mL) to remove or recovery any starting material. The combined chloroform extracts were then washed with saturated $NaHCO_3$ solution, dried over anhydrous $MgSO_4$, filtered, and concentrated in vacuo. The residue[1] was subsequently distilled at reduced pressure via Kugelrohr [bp 72–76 °C (0.5 mm), 61% yield].

Preparation of [2-(4-Methoxyphenyl)prop-1-en-3-yl]trimethylammonium Iodide. 2-(4-Methoxyphenyl)-3-(*N*,*N*-dimethylamino)-1-propene (1.0 g, 0.05 mol) was dissolved in anhydrous THF (50 mL) and was placed in a 100-mL, three-necked, round-bottomed flask which had been equipped for magnetic stirring. Iodomethane (3.0 g, 0.2 mol) was added in one portion, and the resulting mixture was stirred for 4 h and filtered to yield 1.6 g (90% yield) of a white solid: mp 172–179 °C; NMR ($CDCl_3$ and CH_3SOCH_3-d_6) δ 3.05 (s, 9 H), 3.72 (s, 3 H), 4.60 (s, 2 H), 5.65 (s, 2 H), 6.80 (d, *J* = 8 Hz, 2 H), and 7.42 (d, *J* = 8 Hz, 2 H); IR (Nujol) 1600, 1520, 1250, 1180, 1030, 890, and 835 cm^{-1}.

Anal. Calcd for $C_{13}H_{20}INO$: C, 46.85; H, 6.06; N, 4.20. Found: C, 46.53; H, 6.04; N, 3.99.

[2-(4-Chlorophenyl)prop-1-en-3-yl]trimethylammonium iodide: mp 229–230 °C dec; NMR ($CDCl_3$ and CH_3SOCH_3-d_6) δ 3.00 (s, 9 H), 4.57 (s, 2 H), 5.74 (s, 1 H), 5.82 (s, 1 H), 7.27 (d, *J* = 8 Hz, 2 H), and 7.50 (d, *J* = 8 Hz, 2 H); IR (Nujol) 1490, 1085, 1010, 950, 920, 890, and 835 cm^{-1}.

[2-(3,4-Dimethoxyphenyl)prop-1-en-3-yl]trimethylammonium iodide: mp 179–180 °C; NMR ($CDCl_3$) δ 3.30 (s, 9 H), 3.80 (s, 3 H), 3.93 (s, 3 H), 4.96 (s, 2 H), 5.65 (s, 1 H), 5.83 (s, 1 H), and 6.65–7.10 (m, 3 H); IR (Nujol) 1515, 1255, 1140, 1020, 880, 845, 815, and 765 cm^{-1}.

[2-(1-Naphthyl)prop-1-en-3-yl]trimethylammonium iodide: mp 197–199 °C; NMR ($CDCl_3$) δ 3.27 (s, 9 H), 4.97 (s, 2 H), 5.78

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[7] Infrared spectra were recorded on either a Perkin-Elmer Model 1420 infrared spectrometer as thin films or Nujol mulls. NMR spectra were obtained in CCl_4, $DCCl_3$, or Me_2SO-d_6 solutions [$(CH_3)_4Si$ internal standard] at 60 MHz with a Varian EM-360 spectrometer. Some of the mass spectra were obtained on a Hitachi Perkin-Elmer RMU-6E spectrometer. All boiling points and melting points are uncorrected, and melting points were recorded on a Fisher-Johns melting point apparatus. The organolithium reagents used for this work were purchased from Aldrich Chemical.

(s, 1 H), 6.43 (s, 1 H), and 7.17–8.00 (m, 7 H); IR (Nujol) 1600, 955, 920, 890, 810, and 785 cm^{-1}.

[2-(4-Methylphenyl)prop-1-en-3-yl]trimethylammonium iodide: mp 244–245 °C dec; NMR (CDCl$_3$) δ 2.30 (s, 3 H), 3.25 (s, 9 H), 4.83 (s, 2 H), 5.69 (s, 1 H), 5.90 (s, 1 H), 7.02 (d, J = 8 Hz, 2 H), and 7.31 (d, J = 8 Hz, 2 H); IR (Nujol) 1620, 1510, 890, and 825 cm^{-1}.

(2-Phenylprop-1-en-3-yl)trimethylammonium iodide: mp 156–160 °C; NMR (CDCl$_3$) δ 3.35 (s, 9 H), 5.00 (s, 2 H), 5.85 (s, 1 H), 6.12 (s, 1 H), and 7.25–7.85 (m, 5 H); IR (Nujol) 1620, 1495, 890, 780, and 715 cm^{-1}.

Anal. Calcd for C$_{12}$H$_{15}$IN: C, 48.01; H, 5.05; N, 4.67. Found: C, 47.43; H, 6.08; N, 4.43.

Reaction of [2-(4-Methoxyphenyl)prop-1-en-3-yl]trimethylammonium Iodide with *n*-Butyllithium. A 250-mL, three-necked, round-bottomed flask was equipped with a thermometer, condenser, magnetic stirrer, and rubber septum. The apparatus was placed under a nitrogen atmosphere and 75 mL of anhydrous THF was added followed by 1.5 g (0.0045 mol) of the quaternary ammonium salt. The mixture was cooled to 0 °C and *n*-butyllithium [5.8 mL (0.009 mol) of a 1.55 M solution in hexane] was added via syringe. The resulting mixture was stirred for 2 h at 0 °C and was quenched with 10 mL of water. The solvent was removed in vacuo, and the residue was partitioned between water (70 mL) and chloroform (3 × 40 mL). The combined chloroform extracts were dried over anhydrous magnesium sulfate, filtered, and concentrated in vacuo to yield 0.80 g (87% yield) of 2-(4-methoxyphenyl)-1-heptene as a mobile liquid. This material was distilled via Kugelrohr [72–76 °C (0.3 mm)] to give 0.70 g (81% yield) of a colorless liquid.[1]

Reaction of [2-(4-Methoxyphenyl)prop-1-en-3-yl]trimethylammonium Iodide with Methylmagnesium Bromide. A 250-mL-, three-necked, round-bottomed flask was equipped with a thermometer, condenser, magnetic stirrer, and rubber septum. The apparatus was placed under a nitrogen atmosphere, and 75 mL of anhydrous THF was added along with 3.0 g (0.009 mol) of the quaternary ammoniun salt. The mixture was cooled to 0 °C, and methylmagnesium bromide [12.6 mL (0.036 mol) of a 2.85 M solution in diethyl ether] was added via syringe. The mixture was refluxed overnight, cooled to room temperature, and quenched with 10 mL of water. The solvent was removed in vacuo, and the residue was partitioned between water (70 mL) and chloroform (3 × 40 mL). The combined chloroform extracts were dried over anhydrous magnesium sulfate, filtered, and concentrated in vacuo to leave a viscous oil. This material was distilled via Kugelrohr to yield 0.8 g (55% yield) of distillate and 0.5 g (37% yield) of residue. The distillate was identified as 2-(4-methoxyphenyl)-1-butene and had the following properties: bp 53–54 °C (0.3 mm); NMR (CDCl$_3$) δ 1.10 (t, J = 7 Hz, 3 H), 2.50 (q, J = 7 Hz, 2 H), 3.80 (s, 3 H), 4.98 (s, 1 H), 5.20 (s, 1 H), 6.81 (d, J = 8 Hz, 2 H), and 7.35 (d, J = 8 Hz, 2 H); IR (thin film) 1605, 1510, 1245, 1180, 1030, 890, and 835 cm^{-1}; mass spectrum, *m/e* 162 (M$^+$).

The residue was identified as 2,5-bis(4-methoxyphenyl)-1,5-hexadiene and exhibited the following properties: mp 132 °C; NMR (CDCl$_3$) δ 2.59 (s, 4 H), 3.75 (s, 6 H), 4.93 (s, 2 H), 5.17 (s, 2 H), 6.80 (d, J = 8 Hz, 4 H), and 7.30 (d, J = 8 Hz, 4 H); IR (CHCl$_3$) 1605, 1510, 1245, 1180, 1030, 895, and 835 cm^{-1}; mass spectrum, *m/e* 294 (M$^+$).

2-(4-Methoxyphenyl)-3-phenyl-1-propene: bp 114–115 °C (0.3 mm); NMR (CDCl$_3$) δ 3.55 (s, 3 H), 3.75 (s, 2 H), 4.90 (s, 1 H), 5.37 (s, 1 H), 6.72 (d, J = 8 Hz, 2 H), and 7.00–7.70 (m, 7 H); IR (thin film) 1600, 1510, 1250, 1180, 1030, 890, 835, 740, and 700 cm^{-1}; mass spectrum, *m/e* 224 (M$^+$).

2-(4-Methoxyphenyl)-1-phenyl-1-propene: mp 95–98 °C; NMR (CDCl$_3$) δ 2.22 (s, 3 H), 3.80 (s, 3 H), 6.70–7.06 (m, 3 H), and 7.18–7.63 (m, 7 H); IR (CHCl$_3$) 1605, 1510, 1245, 1180, 1030, 830, 720, and 700 cm^{-1}; mass spectrum, *m/e* 224 (M$^+$).

Reaction of [2-(4-Methoxyphenyl)prop-1-en-3-yl]trimethylammonium Iodide with Li(*n*-Bu)$_2$Cu. A 250-mL, three-necked, round-bottomed flask was equipped with a thermometer, condenser, magnetic stirrer, and rubber septum. The apparatus was placed under a nitrogen atmosphere, and 25 mL of anhydrous THF was added along with 2.86 g (0.015 mol) of CuI. The mixture was cooled to −78 °C in an isopropyl alcohol–dry ice bath, and *n*-butyllithium (19.4 mL of a 1.55 M solution in hexane) was added via syringe. The mixture was allowed to stir for 30 min while warming to 0 °C. The quaternary ammonium salt (2.5 g, 0.0075 mol) was added in one portion and was allowed to stir for 2.5 h at 0 °C. The mixture was quenched with 10 mL of saturated aqueous ammonium chloride. The mixture was diluted with water (70 mL) and extracted with chloroform (3 × 60 mL). The combined chloroform extracts were washed with saturated, aqueous sodium bicarbonate, dried over anhydrous magnesium sulfate, filtered, and concentrated in vacuo to leave 1.2 g of a liquid. This material was analyzed by ^{1}H NMR and GLC (see notes *a* and *d*, Table II) and was found to contain 2-(4-methoxyphenyl)-1-propene (69% yield) and 2-(4-methoxyphenyl)-1-heptene (31% yield).

Reaction of [2-(4-Methoxyphenyl)prop-1-en-3-yl]trimethylammonium Iodide with Sodium Borohydride. A 250-mL, three-necked, round-bottomed flask was equipped with a thermometer, condenser, magnetic stirrer, and was placed under a nitrogen atmosphere. Into the flask were placed the quaternary ammonium salt (2.0 g, 0.006 mol), isopropyl alcohol (100 mL), and sodium borohydride (0.25 g, 0.0066 mol). The resulting mixture was refluxed overnight and cooled to room temperature. The solvent was removed in vacuo, and the residue was partitioned between water (100 mL) and chloroform (3 × 60 mL). The combined chloroform extracts were dried over anhydrous magnesium sulfate, filtered, and concentrated in vacuo to yield a liquid, which was distilled via Kugelrohr to yield 0.7 g (80% yield) of a colorless liquid: bp 42–46 °C (0.3 mm); NMR (CDCl$_3$) δ 2.10 (s, 3 H), 3.86 (s, 3 H), 4.92 (s, 1 H), 5.20 (s, 1 H), 6.78 (d, J = 8 Hz, 2 H), and 7.33 (d, J = 8 Hz, 2 H); IR (thin film) 1620, 1510, 1250, 1180, 1030, 890, and 830 cm^{-1}; mass spectrum, *m/e* 148 (M$^+$).

2-(4-Chlorophenyl)propene: bp 43–47 °C (0.3 mm); NMR (CDCl$_3$) δ 2.03 (s, 3 H), 4.93 (s, 1 H), 5.20 (s, 1 H), 7.05 (d, J = 8 Hz, 2 H), and 7.23 (d, J = 8 Hz, 2 H); IR (thin film) 1625, 1490, 1115, 1090, 1010, 895, 830, 760, and 735 cm^{-1}; mass spectrum, *m/e* 152 (M$^+$).

2-(3,4-Dimethoxyphenyl)propene: bp 75–75 °C (0.3 mm); NMR (CDCl$_3$) δ 2.12 (s, 3 H), 3.85 (s, 6 H), 5.00 (s, 1 H), 5.27 (s, 1 H), and 6.65–7.25 (m, 3 H); IR (thin film) 1600, 1510, 1460, 1250, 1180, 1140, 1110, 1025, 890, 855, and 810 cm^{-1}; mass spectrum, *m/e* 178 (M$^+$).

2-(1-Napthyl)propene: bp 55–60 °C (0.3 mm); NMR (CDCl$_3$) δ 2.15 (s, 3 H), 4.96 (s, 1 H), 5.28 (s, 1 H), and 7.10–8.02 (m, 7 H); IR (thin film) 1635, 1590, 1430, 1370, 900, 800, and 780 cm^{-1}; mass spectrum, *m/e* 168 (M$^+$).

2-(4-Methylphenyl)propene: bp 45–46 °C (0.3 mm); NMR (CDCl$_3$) δ 2.20 (s, 3 H), 2.22 (s, 3 H), 4.90 (s, 1 H), 5.22 (s, 1 H), 7.00 (d, J = 8 Hz, 2 H), and 7.26 (d, J = 8 Hz, 2 H); IR (thin film) 1625, 1510, 1450, 890, 820, and 735 cm^{-1}; mass spectrum, *m/e* 132 (M$^+$).

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