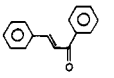
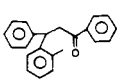
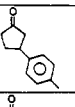
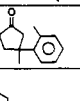
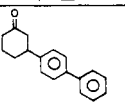
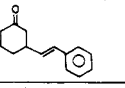
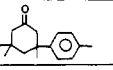
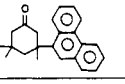
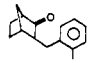
basic with 28% NH_4OH and extracted with $CHCl_3$. The extract was washed with water, dried (Na_2SO_4), and evaporated. The remaining residue was chromatographed on silica gel (10 g) by using benzene as an eluenet. Removal of the solvent gave **5**, which was recrystallized from methanol–ether. Yields and physical data are listed in Table I.

1,2,3,4-Tetrahydro-1-(α-hydroxybenzyl)-6,7-dimethoxy-2-methylisoquinoline (6). To a stirred suspension of $LiAlH_4$ (200 mg, 5.4 mmol) in THF (40 mL) was added a solution of **5d** (400 mg, 1.23 mmol) in THF (10 mL) at room temperature. After the stirring had been continued for 14 h, the mixture was decomposed with 10% NaOH. Inorganic precipitate was removed by filtration, and the solvent was evaporated. The remaining residue was extracted with $CHCl_3$. The extract was washed with water, dried (Na_2SO_4), and evaporated. The residual solid was recrystallized from methanol–ether to give **6**: 293 mg (76%); mp 155–157 °C; ^1H NMR ($CDCl_3$) δ 2.58 (3 H, s), 3.13–3.63 (4 H, m), 3.27 (3 H, s), 3.42 (1 H, d, J = 9 Hz), 3.84 (3 H, s), 4.23 (1 H, d, J = 9 Hz), 5.39 (1 H, s), 6.61 (1 H, s), 7.37 (5 H, br s); mass spectrum, m/z 314 (MH^+) (electron-impact mass spectrum did not give M^+), m/e 206 (M^+ - C_6H_5CHOH). Anal. Calcd for $C_{19}H_{23}NO_3$: C, 72.82; H, 7.40; N, 4.47. Found: C, 72.64; H, 7.46; N, 4.29.

Registry No. **2**, 695-53-4; **3a**, 5841-63-4; **3b**, 27770-23-6; **4a**, 86970-73-2; **4a** reduction product, 86970-74-3; **4b**, 86970-75-4; **4b** reduction product, 86970-76-5; **4c**, 86970-77-6; **4c** reduction product, 86970-78-7; **4d**, 86970-79-8; **4d** reduction product, 86970-80-1; **4e**, 86970-81-2; **4e** reduction product, 86970-82-3; (*R**,*R**)-**4f**, 86970-84-5; (*R**,*S**)-**4f**, 86970-83-4; **4f** reduction product, 86970-85-6; **4g**, 86970-86-7; **4g** reduction product, 86970-87-8; **5a**, 86970-88-9; **5b**, 86970-89-0; **5c**, 86970-90-3; **5d**, 86970-91-4; **5e**, 86970-92-5; **5f**, 86970-93-6; **5g**, 86970-94-7; **6**, 86970-95-8; 3,4-dimethoxybenzeneethanol, 7417-21-2; 2-thiopheneethanol, 5402-55-1; α,5-dimethyl-2-thiopheneethanol, 86970-96-9; 3-methoxybenzeneethanol, 5020-41-7.

Supplementary Material Available: A listing of spectral data of compounds **4a–g** and **5a–g** (1 page). Ordering information is given on any current masthead page.

Table I

Entry	α-enone	Ar-X	1-4 adduct	% isolated yield in THF	yield in ether	yield lit.
1				98		70[a]
2				73	57	b
3				polymers	76	
4				polymers	72	20[c]
5				92		
6				84		
7				70	94	
8				76		
9				79		

[a] via a phenyl copper reagent addition. See Ref 15; [b] 49–66% yields were obtained in similar cases from arylzinc additions. See Ref 13d; [c] See Ref 16.

Ultrasound in Organic Synthesis. 4.[1] A Simplified Preparation of Diarylzinc Reagents and Their Conjugate Addition to α-Enones

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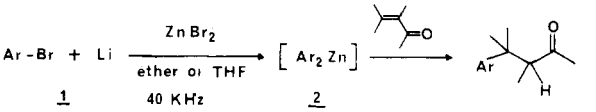
We have previously demonstrated that sonication[2] of organic halides with lithium metal in an ethereal solvent constitutes an exceptionally rapid and easy procedure for obtaining the corresponding lithio derivatives. These reagents readily react in situ with various organic (aldehydes, ketones,[3] dimethylformamide[1]) as well as inorganic (cuprous iodide[4]) species.

We now report a very simple preparation of diarylzinc derivatives by this method and their subsequent, generally high yield, reaction with conjugated enones.[5] The transmetalation reactions of organolithium and organomagnesium derivatives with zinc halides have proven to be among the most synthetically useful methods for producing organozinc reagents.[6] We have found that the preparation of diverse diarylzinc reagents **2** can be readily achieved in a one-pot process by sonication of the aryl bromides **1** in the presence of lithium wire and zinc bromide in dry ether or tetrahydrofuran (eq 1). The



(1)

reaction is usually complete within 30–45 min at 0 °C, as evidenced by the total disappearance of the metal.[7] Side reactions such as Wurtz coupling appear to be minimal under these conditions. Sonication is essential in this step;[8] much slower and less efficient reactions are observed on lowering the energy output of the sonicator and on replacing the sonication by magnetic stirring (3 h at room temperature in the case of *p*-bromotoluene).

Conjugate addition of these sonically prepared zinc reagents to various α-enones can be easily effected at room temperature in the presence of nickel acetylacetonate

(1) For the previous paper, see: Petrier, C.; Gemal, A. L.; Luche, J. L. *Tetrahedron Lett.* **1982**, *23*, 3361.

(2) For recent papers on sonochemical reactions, see: (a) Han, B. H; Boudjouk, P. *J. Org. Chem.* **1982**, *47*, 5030. (b) Kitazume, T.; Ishikawa, N. *Chem. Lett.* **1982**, 1453.

(3) Luche, J. L.; Damiano, J. C. *J. Am. Chem. Soc.* **1980**, *102*, 7926.

(4) Luche, J. L.; Petrier, C.; Gemal, A. L.; Zikra, N. *J. Org. Chem.* **1982**, *47*, 3805.

(5) The conjugate addition of reagents obtained from alkyl and vinylic bromides has not yet been optimized.

(6) See, for example: (a) Thiele, K. H.; Dimitrov, V.; Thielemann, J.; Brueser, W.; Zschunke, A. *Z. Anorg. Allg. Chem.* **1981**, *483*, 154. (b) Wakefield, B. J. "The Chemistry of Organolithium Compounds"; Pergamon Press: Oxford, **1974**; p 249.

(7) The black mixture contains a reactive organozinc species, the exact nature of which has not yet been investigated. The stoichiometry of the reaction, however, corresponds to the formation of a diarylzinc compound. The presence in the reaction medium of lithium bromide (4 equiv/Ar_2Zn) should have an effect on the reactivity. For related examples, see: Jones, P. R.; Goller, S. L.; Kauffman, W. J. *J. Org. Chem.* **1969**, *34*, 3566.

(8) The mechanism by which the sonic waves promote the reaction probably does not involve cavitational effects. These effects appear to be unlikely in solvents such as ether. See, for example: Frenkel, J. *Acta Physico Chim. URSS* **1940**, *12*, 317.

0022-3263/83/1948-3837$01.50/0

($Ni(acac)_2$). Examples of this transformation are given in Table I. These 1,4 additions proceed in high yield even with β,β-disubstituted enones.[9] This may be a consequence of the relative thermal stability of these organometallic reagents which allows the reactions to be run at room temperature. Control experiments, generally performed with *p*-bromotoluene and isophorone, demonstrate that the extent of conjugate addition is substantially decreased on using lesser amounts of the aryl bromide, and in the absence of $Ni(acac)_2$.

Organocopper compounds have been the most extensively studied reagents for effecting conjugate additions to α-enones.[10] However, their relative thermal instability necessitates low temperatures, and in some cases allylic alcohols are produced rather than the normally desired conjugate-addition adducts. This tendency is especially pronounced with β,β-disubstituted enones. Several alternative reagents have therefore been developed in order to try to overcome this problem.[10-12] Although organozinc reagents have been known for some time to add to certain enones preferentially in a conjugate fashion,[13] the number of previous studies devoted to the synthetic exploitation of this tendency is surprisingly low.[14] The ultrasonically prepared organozinc reagents readily give rise under very simple experimental conditions to β-arylation, starting from inexpensive and easily available material. The present procedure should therefore compete favorably with current methods for carrying out this type of addition (see Table I, entries 1,2,4). Preparative experiments on a 10-mmol scale give satisfactory results. Thus, reagents derived from 2-bromostyrene and *p*-bromotoluene add to cyclohexenone and isophorone, respectively, in 84% and 86% isolated yields (cf. entries 6 and 7).

The results given in this paper, although restricted to the introduction of aryl groups, clearly indicate the potential of this method. Extension and applications of this reaction are presently being studied.

Experimental Section

Ether and tetrahydrofuran were freshly distilled before use (Na, benzophenone). Lithium wire and nickel acetylacetonate were obtained from Fluka, and zinc bromide from Alfa. Sonications were run in a common ultrasound laboratory cleaner (Sonoclean, 40 KHz, 96 W/L) filled with crushed ice and water. Infrared spectra were recorded on a Perkin-Elmer Model 297 spectrometer, and a JEOL PMX-60 spectrometer was used for the NMR spectra. Mass spectra were recorded on a VG-Micromass 7070F spectrometer using chemical ionization (NH_3, isobutane).

General Procedure. The aryl bromide (2 mmol), 226 mg (1.0 mmol) of zinc bromide, and 28 mg (4.0 mmol) of lithium wire in 10 mL of anhydrous ether or tetrahydrofuran under an argon atmosphere are sonicated in a 25-mL Erlenmeyer flask equipped with a magnetic stirring bar. The mixture turns black almost immediately, and the lithium is totally consumed within 30–45 min. Sonication is then discontinued, and the black suspension is treated with stirring with 1 mmol of the enone and 3 mg (0.01 mmol) of nickel acetylacetonate dissolved in 1 mL of the reaction solvent. This addition is effected at room temperature except for entries 3 and 6. In these two cases dropwise addition of the enone–$Ni(acac)_2$ solution is carried out at 0 °C and −78 °C, respectively; then the mixture is warmed up to room temperature. The reaction time, except for entry 3 (5 min) ranges from 50 min to 4 h. (Progress of the reaction is monitored by TLC analysis of aliquots.) The mixture is poured into saturated aqueous ammonium chloride, and the product is isolated with ether in the usual manner.

3-Methyl-3-(2-methylphenyl)cyclopentanone:[16] oil; IR (neat) 3100–2850, 1740, 1600, 1490, 1460, 1400, 760, 750, 730 cm^{-1}; NMR (CCl_4) 7.2–6.9 (m, 4 H), 2.45 (s, 3 H), 2.60–2.15 (m, 6 H), 1.4 (s, 3 H) ppm.

3-(2-Methylphenyl)-1,3-diphenyl-1-propanone: mp 81–82 °C (hexane); IR (Nujol) 3070, 3050, 3020, 2950, 2850, 1680, 1600, 770, 700, 690 cm^{-1}; NMR (CCl_4) 8–6.9 (m, 14 H), 4.9 (t, 1 H), 3.6 (d, 2 H), 2.3 (s, 3 H) ppm; MS, *m/e* 318 ($[M + NH_4]^+$), 301 (M + 1), 300, 282, 181, 165, 105. Anal. Calcd for $C_{22}H_{20}O$: C, 87.96; H, 6.71. Found: C, 88.01; H, 6.67.

3-(4-Methylphenyl)cyclopentanone: oil; IR (neat) 3050, 3020, 2950, 1740, 1515, 1160, 1150, 805 cm^{-1}; NMR (CCl_4) 7.1 (s, 4 H), 3.65–3.0 (m, 1 H), 2.7–1.7 (m, 6 H), 2.35 (s, 3 H) ppm; MS, *m/e* 192 ($[M + NH_4]^+$), 175 (M + 1), 174, 118, 91. Anal. Calcd for $C_{12}H_{14}O$: C, 82.71; H, 8.10. Found: C, 82.50; H, 7.99.

3-(4-Phenylphenyl)cyclohexanone: mp 129–131 °C (hexane); IR (Nujol) 3050, 3020, 2920, 1705, 1480, 760, 700 cm^{-1}; NMR (CCl_4) 7.6–7.0 (m, 9 H), 3.2–1.5 (m, 9 H) ppm; MS, *m/e* 268 ($[M + NH_4]^+$], 251 (M + 1), 250, 193, 180, 178, 97. Anal. Calcd for $C_{18}H_{18}O$: C, 86.36; H, 7.25. Found: C, 86.18; H, 7.24.

3-(2-Phenylethenyl)cyclohexanone: oil; IR (neat) 3070, 3050, 3020, 2850, 1705, 1600, 1490, 790, 750, 700 cm^{-1}; NMR (CCl_4) 7.5–6.5 (m, 8 H), 2.5–1.0 (m, 8 H) ppm; MS, *m/e* 218 ($[M + NH_4]^+$), 201 (M + 1), 200, 183, 143, 128, 115. Anal. Calcd for $C_{14}H_{16}O$: C, 83.96; H, 8.05. Found: C, 83.83; H, 7.98.

3,3,5-Trimethyl-5-(4-methylphenyl)cyclohexanone: mp 64–65 °C (hexane); IR (Nujol) 3050, 3010, 2950, 2850, 1700, 1500, 1280, 810 cm^{-1}; NMR (CCl_4) 7.4–7.0 (q, 4 H), 3.3–1.7 (m, 6 H), 2.3 (s, 3 H) 1.3 (s, 3 H), 1.1 (s, 3 H), 0.4 (s, 3 H) ppm; MS, *m/e* 248 ($[M + NH_4]^+$), 231 (M + 1), 230, 215, 132. Anal. Calcd for $C_{16}H_{22}O$: C, 83.43; H, 9.63. Found: C, 83.23; H, 9.78.

3,3,5-Trimethyl-5-(9-phenanthryl)cyclohexanone: amorphous solid; IR (neat) 3050, 2950, 1700, 1600, 1480, 1280, 900, 890, 770, 750, 730 cm^{-1}; NMR (CCl_4) 8.9–8.3 (m, 3 H), 8.00–7.4 (m, 6 H), 3.4–2.1 (m, 6 H), 2.15 (s, 3 H), 1.7 (s, 3 H), 1.0 (s, 3 H) ppm; MS, *m/e* 334 ($[M + NH_4]^+$), 317 (M + 1), 316, 301. Anal. Calcd for $C_{23}H_{24}O$: C, 88.26; H, 7.69. Found: C, 88.30; H, 7.65.

3-[(2-Methylphenyl)methyl]bicyclo[2.2.1]heptan-2-one: oil; IR (neat) 3050, 3010, 2950, 1860, 1735, 1480, 750, 740, 720 cm^{-1}; NMR (CCl_4) 7.1 (br s, 4 H), 3.5–2.2 (m, 5 H), 2.3 (s, 3 H), 2.0–1.5 (m, 6 H) ppm; MS, *m/e* 232 ($[M + NH_4]^+$), 215 (M + 1), 214, 105. Anal. Calcd for $C_{15}H_{18}O$: C, 84.07; H, 8.47. Found: C, 84.16; H, 8.52.

Acknowledgment. We thank the Centre National de la Recherche Scientifique (ATP Chimie Fine) for financial support.

Registry No. 3-Methyl-3-(2-methylphenyl)cyclopentanone, 86921-80-4; 1-(2-methylphenyl)-1,3-diphenyl-3-propanone, 86921-81-5; 3-(4-methylphenyl)cyclopentanone, 86921-82-6; 3-(4-phenylphenyl)cyclohexanone, 86921-83-7; 3-(2-phenyl-

(9) In contrast, sonication of a mixture of the same relative amounts of isophorone, *p*-bromotoluene, lithium, and zinc bromide in dry ether in the presence of $Ni(acac)_2$ yields 34% of the 1,4 adduct and a large amount of polymer (cf. entry 7).

(10) See: Posner, G. H. "An Introduction to Syntheses Using Organocopper Reagents"; Wiley-Interscience: New York, 1980.

(11) Thermally stable hetero cuprates have recently been described; see: (a) Bertz, S. H.; Dabbagh, G. *J. Chem. Soc., Chem. Commun.* **1982**, 1030. (b) Bertz, S. H.; Dabbagh, G.; Villacorta, G. M. *J. Am. Chem. Soc.* **1982**, *104*, 5824.

(12) (a) Leyendecker, F.; Drouin, J.; Conia, J. M.; *Nouv. J. Chim.* **1978**, 271. (b) Schwartz, J.; Loots, M. J.; Kosugi, H. *J. Am. Chem. Soc.* **1980**, *102*, 1333. (c) Ashby, E. C.; Heinsohn, G.; *J. Org. Chem.* **1974**, *39*, 3297. (d) Bagnell, L; Jeffery, E. A.; Meisters, A.; Mole, T. *Aust. J. Chem.* **1975**, *28*, 801. (e) Hooz, J.; Layton, R. B. *J. Am. Chem. Soc.* **1971**, *93*, 7320. (f) Atwal, K. S.; Sahoo, S. P.; Tsai, T. Y. R.; Wiesner, K. *Heterocycles* **1982**, *19*, 641.

(13) (a) Patai, S., Ed. "Chemistry of Carbonyl Compounds"; Wiley-Interscience: New York, **1966**; p 672. (b) Kataoka, K.; Tsuruta, T. *Polym. J.* **1977**, *9*, 595. (c) Gilman, H.; Kirby, R. H. *J. Am. Chem. Soc.* **1941**, *63*, 2046. (d) Rahman, M. T.; Saha, S. L.; Hansson, A. T. *J. Organomet. Chem.* **1980**, *199*, 9. (e) Isobe, H.; Kondo, S.; Nagasawa, N.; Goto, T. *Chem. Lett.* **1977**, 679. (f) Caporusso, A. M.; Giacomelli, G.; Lardicini, L. *J. Org. Chem.* **1982**, *47*, 4640.

(14) An interesting three-component reaction involving an intermediate organozinc reagent has been published: Shono, T.; Nishiguchi, I.; Sasaki, M. *J. Am. Chem. Soc.* **1978**, *100*, 4314.

(15) Suzuki, M.; Suzuki, T.; Kawagashi, T.; Noyori, R. *Tetrahedron Lett.* **1980**, *21*, 1247.

(16) Wolff, S.; Agosta, W. C. *J. Chem. Soc., Chem. Commun.* **1972**, 226.

ethenyl)cyclohexanone, 86921-84-8; 3,3,5-trimethyl-5-(4-methylphenyl)cyclohexanone, 53577-40-5; 3,3,5-trimethyl-5-(9-phenanthryl)cyclohexanone, 86921-85-9; 3-[(2-methylphenyl)-methyl]bicyclo[2.2.1]-2-heptanone, 86921-86-0; 2-cyclopenten-1-one, 930-30-3; 3-methyl-2-cyclopenten-1-one, 2758-18-1; 2-cyclohexen-1-one, 930-68-7; 3,5,5-trimethyl-2-cyclohexen-1-one, 78-59-1; 3-methylenebicyclo[2.2.1]-2-heptanone, 5597-27-3; 9-bromophenanthrene, 573-17-1; bis(9-phenanthryl)zinc, 86921-88-2; PhCH=CHCOPh, 94-41-7; o-$BrC_6H_4CH_3$, 95-46-5; p-$BrC_6H_4CH_3$, 106-38-7; p-BrC_6H_4Ph, 92-66-0; PhCH=CHBr, 103-64-0; $ZnBr_2$, 2699-45-8; $(o\text{-}CH_3C_6H_4)_2Zn$, 7029-31-4; $(p\text{-}CH_3C_6H_4)_2Zn$, 15106-88-4; $(p\text{-}PhC_6H_4)_2Zn$, 15106-90-8; $(PhCH{=}CH)_2Zn$, 86921-87-1.

Catechol Monoether Synthesis

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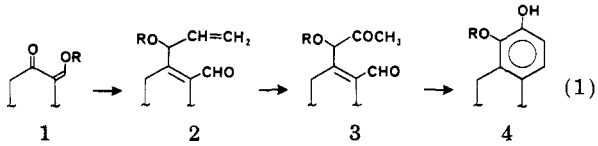
In earlier work we have demonstrated efficient methods for appending benzene rings, phenols, and pyridines onto α-methylene ketones.[1] The yields for these processes have been uniformly high, suggesting that the approach will be useful in total synthesis. The extension of this methodology to the synthesis of catechol monoethers will now be described.

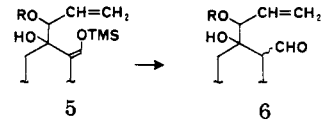
The catechol unit is a substructure of a large number of important natural products. A method for introducing this unit onto a ketone during the latter stages of a total synthesis would allow the consideration of novel strategies. A modification of our phenol synthesis suggested itself (eq 1). Addition of an alkoxyallyl nucleophile to the *O*-trimethylsilyl hydroxymethylene ketone **1** (R = $Si(CH_3)_3$) followed by hydrolysis would give enal **2**.[1] Palladium-catalyzed oxygenation[2] of the terminal alkene produces keto aldehyde **3**, which undergoes intramolecular aldol condensation and dehydration to form the catechol monoether **4**.



(1)

1 **2** **3** **4**

Potential difficulties concerning the regiochemistry of attack of the three-carbon oxygenated nucleophile and the regiochemistry of oxidation of the terminal alkene were identified at the outset. We were delighted to find that the zinc "ate" complex[3] derived from methoxyallyllithium[4] and zinc chloride attacks the carbonyl group of **1** and that the attack takes place exclusively at the α carbon of the nucleophile. Aqueous acidic hydrolysis during workup of the intermediate tertiary allylic alcohol **5** proceeded to give mixtures of enal **2** and β-hydroxy aldehyde **6** (eq 2).

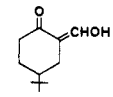
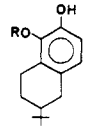
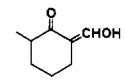
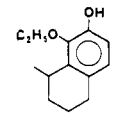
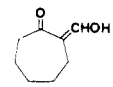
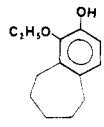
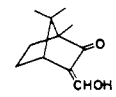
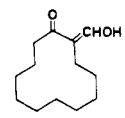
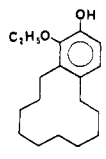
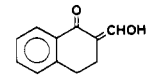
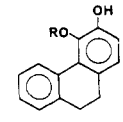
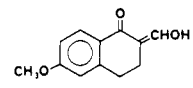
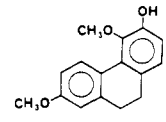
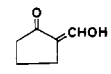
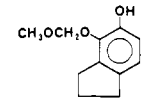


+ 2 (2)

5 **6**

Aldehyde **6** was the only product when **5** was treated with tetra-*n*-butylammonium fluoride in tetrahydrofuran. Since the palladium-catalyzed oxygenation of **6** produced a mixture of products, it was necessary to find an efficient route to **2**. Because **6** was stable to prolonged treatment with acid under a variety of conditions, it appeared that different mechanisms are responsible for the formation of **2** and **6**. Ionization of the tertiary allylic alcohol leads to **2**, whereas protonation of the trimethylsilyl enol ether leads

Table I. Catechol Monoethers

entry	starting material	catechol monoether[a]	yield[b] % (mp, °C)[c]
1		 R = CH_3, CH_2CH_3, CH_2OCH_3	78 (92) 73 (63) 61 (68-71)
2			45
3			63
4		*d*	
5			66
6		 R = CH_2CH_3, CH_2OCH_3	66 65
7			43 (91)
8			19[e]

[a] Full spectroscopic data for all products are presented in the supplementary information. [b] Overall yield from the α-hydroxymethylene ketone. [c] Melting points are uncorrected. Compounds for which no melting point has been recorded were obtained as oils. [d] Nucleophilic addition to the carbonyl group did not take place. [e] See text.

(1) (a) Tius, M. A.; Ali, S. *J. Org. Chem.* **1982**, *47*, 3163. (b) Tius, M. A.; Thurkauf, A.; Truesdell, J. W. *Tetrahedron Lett.* **1982**, *23*, 2823. (c) *Ibid.* **1982**, *23*, 2819.

(2) Tsuji, J.; Shimizu, I.; Suzuki, H.; Naito, Y. *J. Am. Chem. Soc.* **1979**, *101*, 5070. Tsuji, J. "Organic Synthesis with Palladium Compounds"; Springer-Verlag: Berlin, Heidelberg, New York, 1980.

(3) We were able to demonstrate that the "ate" complex was involved by titration of the yellow alkoxyallyllithium solutions with one-third of an equivalent of the nonhygroscopic zinc chloride—*N,N,N',N'*-tetramethylethylenediamine (TMEDA) complex to a colorless endpoint; Isobe, M.; Kondo, S.; Nagasawa, N.; Goto, T. *Chem. Lett.* **1977**, 679.

(4) Evans, D. A.; Andrews, G. C.; Buckwalter, B. *J. Am. Chem. Soc.* **1974**, *96*, 5560. Still, W. C.; Macdonald, T. L. *Ibid.* **1974**, *96*, 5561. Still, W. C. *Tetrahedron Lett.* **1976**, 2115. Still, W. C.; Macdonald, T. L. *J. Org. Chem.* **1976**, *41*, 3620. Evans, D. A.; Baillargeon, D. J.; Nelson, J. V. *J. Am. Chem. Soc.* **1978**, *100*, 2242.

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