After the solvent was removed under vacuum, the crude product was purified by flash column chromatography on silica gel to give 131 mg (82%; 72% overall from **17**) of **21a,b** as a pale yellow oil (~3:2 ratio of regioisomers): IR ($CHCl_3$) 1680 $cm^{-1}$; $^1H$ NMR ($CDCl_3$, 360 MHz) $\delta$ 0.09 (m, 2 H), 0.39 (m, 2 H), 0.85 (m, 4 H), 1.00 (m, 1 H, **21b**), 1.11 (m, 1 H, **21a**), 1.28 (d, $J$ = 7.3 Hz, 3 H, **21b**), 1.30 (d, $J$ = 7.3 Hz, 3 H, **21a**), 1.40 (m, 4 H), 1.60–1.85 (m, 8 H), 1.95 (m, 4 H), 2.52 (m, 1 H, **21b**), 2.87 (m, 1 H, **21a**), 3.58 (s, 3 H, **21a**), 3.65 (s, 3 H, **21b**), 4.09 (m, 2 H), 5.80 (m, 1 H, **21b**), 5.86 (m, 1 H, **21a**); $^{13}C$ NMR (90 MHz) $\delta$ (for **21a**) 8.6, 14.9, 17.0, 20.7, 21.0, 22.3, 25.6, 42.6, 43.9, 46.3, 51.2, 52.9, 56.8, 126.9, 139.9, 167.0; (for **21b**) 8.5, 15.1, 18.0, 21.9, 22.1, 22.3, 23.9, 42.8, 47.0, 47.1, 48.7, 52.9, 55.4, 128.3, 136.8, 167.8; HRMS ($M^+$) 279.1390 and 281.1360 calcd for $C_{16}H_{22}ClNO$, found 279.1392 and 281.1357.

**(1′*S**,2′*R**,6′*S**,7′*S**,8′*R*,9′*R**,10′*S**)-6′-Carbomethoxy-9′-chloro-8′,9′-epoxy-10′-methyl-2′-nitrospiro[cyclopropane-1,11′-bicyclo[5.3.1]undecane] (22a) and Regioisomer 22b.** To a solution of **21a,b** (110 mg, 0.4 mmol) in $CH_2Cl_2$ (20 mL) were added sequentially at 0 °C 55% mCPBA (800 mg, 2.6 mmol) and trifluoroacetic acid (103 mg, 0.9 mmol). After being stirred at rt for 18 h, the reaction mixture was quenched with aqueous saturated $Na_2CO_3$ solution. The organic layer was washed with aqueous saturated $K_2SO_3$ solution and dried ($MgSO_4$). Removal of the solvent afforded 200 mg of the concentrate. Purification of the residue by column chromatography on silica gel using 4:1 hexane–EtOAc as eluent gave 25 mg (18%) of **22a,b** as a white solid (~3:2 ratio of regioisomers). For single-crystal X-ray analysis, a 3:2 mixture of **22a,b** was separated by preparative TLC, and the major product **22a** was then recrystallized from EtOAc: mp 113–115 °C; IR ($CHCl_3$) 1750, 1560, 1445, 1360, 1250, 1200, 1160, 1020 $cm^{-1}$; $^1H$ NMR ($CDCl_3$, 360 MHz) $\delta$ 0.45 (m, 2 H), 0.62 (m, 1 H), 0.95 (m, 1 H), 1.15 (s, 1 H), 1.32 (d, $J$ = 7.2 Hz, 3 H), 1.45 (m, 1 H), 1.69 (m, 1 H), 1.76 (s, 1 H), 1.97–2.20 (m, 2 H), 2.29–2.43 (m, 2 H), 2.50 (d, $J$ = 10.8 Hz, 1 H), 2.80 (q, $J$ = 7.2 Hz, 1 H), 3.68 (s, 3 H), 3.76 (s, 1 H), 4.41 (d, $J$ = 9.8 Hz, 1 H); $^{13}C$ NMR (90 MHz) $\delta$ 6.6, 16.8, 18.9, 23.7, 28.8, 29.0, 29.3, 37.1, 42.4, 47.8, 51.0, 52.3, 64.4, 79.9, 93.6, 176.4.

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**Registry No.** **4**, 765-46-8; **9**, 144018-26-8; **10**, 144018-27-9; **11**, 144018-28-0; **12**, 144018-29-1; (±)-**13**, 144018-30-4; (±)-**14**, 144018-31-5; (±)-**15**, 144018-32-6; (±)-**16**, 144018-33-7; (±)-**17**, 144018-34-8; (±)-**18**, 144018-35-9; (±)-(*E*)-**19**, 144070-89-3; (±)-(*Z*)-**19**, 144018-36-0; (±)-**20a**, 144018-37-1; (±)-**20b**, 144018-38-2; (±)-**21a**, 144018-39-3; (±)-**21b**, 144018-40-6; (±)-**22a**, 144018-41-7; (±)-**22b**, 144018-42-8; (±)-3-chloro-2-pyrrolidinocyclohexene, 144018-25-7.

**Supplementary Material Available:** $^1H$ and $^{13}C$ NMR spectra of key intermediates (26 pages). This material is contained in many libraries on microfiche, immediately follows this article in the microfilm version of the journal, and can be ordered from the ACS; see any current masthead page for ordering information.

## A Simple Three-Component Olefin Coupling Procedure

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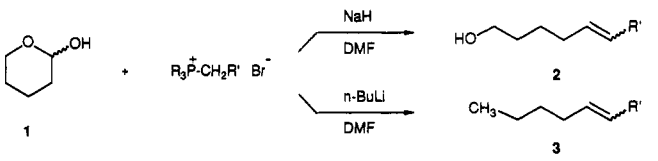
The Wittig olefination reaction remains among the most popular methods for preparing double-bond compounds from carbonyl or lactol precursors.[2] In many instances, the active phosphorus reagent can be generated in situ by deprotonation of the phosphonium salt with a strong base. During recent investigations in which unsaturated alcohols **2** were being prepared from δ-valerolactol (**1**), we observed the unusual formation of unsaturated adducts **3** in reactions for which *n*-butyllithium had been used as the base (Scheme I).

**Scheme I**




**Scheme II**

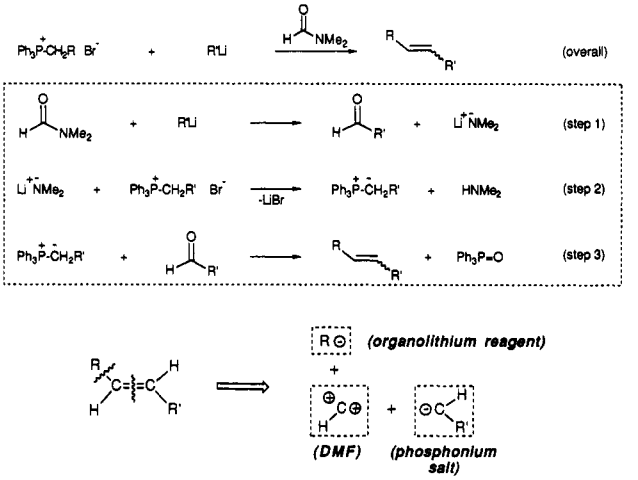






**Figure 1.**

Upon further examination, the butyl side chain of adducts **3** was found to be derived not from the starting lactol, but from *butyllithium*, with one of the new olefin centers of **3** arising from DMF. As illustrated in Figure 1, this olefination reaction can be viewed as a three-way coupling between a phosphonium salt, an organolithium reagent, and DMF. Mechanistically, the reaction presumably follows the stepwise pathway shown in Scheme II. Nucleophilic addition[3] of the organolithium reagent to DMF in step 1 generates an aldehyde[4] plus 1 equiv of dimethylamide anion. Subsequently, deprotonation of the phosphonium salt by dimethylamide anion gives the phosphorus ylide (step 2) which reacts with the aldehyde to give the olefin product (step 3). It is interesting to note that the organolithium reagent must undergo addition to DMF (step 1) more rapidly than it can deprotonate the phosphonium salt.

To study this one-pot olefin coupling procedure more fully, we surveyed several phosphorus reagents in reactions with *n*-butyllithium or phenyllithium in DMF. From the results listed in Table I, phosphonium salts which lead to stabilized ylides (entries 1–4) give the best yields. Entries 5 and 6 suggest that *phosphonate esters* can also be used in these reactions. On the other hand, reactions involving phosphonium salts of unstabilized ylides (entries 7–10) give

(1) Undergraduate research participant.

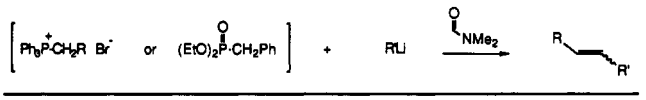
(2) Maryanoff, B. E.; Reitz, A. B. *Chem. Rev.* **1989**, *89*, 863.

(3) Organolithium reagents typically undergo formylation reactions with DMF in the absence of strong proton donors. See: Chamberlin, A. R.; Stemke, J. E.; Bond, F. T. *J. Org. Chem.* **1978**, *43*, 147. Traas, P. C.; Boelens, H. Takken, H. J. *Tetrahedron Lett.* **1976**, 2287.

(4) Variable amounts of the aldehyde are obtained in some of the reactions after aqueous workup. The aldehyde can be obtained cleanly if the reaction is carried out in the absence of the phosphonium salt and quenched with water.

0022-3263/92/1957-6667$03.00/0 

**Table I. Coupling Reactions of Phosphorus Reagents with Organolithium Reagents**



| entry | phosphorus reagent (R)[a] | organolithium reagent (R') | structure | olefin product E:Z ratio[b] | yield[c] (%) |
|---|---|---|---|---|---|
| (1) | $CO_2Et$ | $(CH_2)_3CH_3$ | $EtO_2C$, R' | 11:1 | 82% |
| (2) | $CO_2Et$ | Ph | " | >95:5 | 89% |
| (3) | Ph | $(CH_2)_3CH_3$ | Ph, R' | 1.3:1 | 94% |
| (4) | Ph | Ph | " | 1:1.2 | 95% |
| (5) | d | $(CH_2)_3CH_3$ | " | >95:5 | 84% |
| (6) | d | Ph | " | >95:5 | 95% |
| (7) | $(CH_2)_6CH_3$ | $(CH_2)_3CH_3$ | $CH_3(CH_2)_6$, R' | <5:95 | 69% |
| (8) | $(CH_2)_6CH_3$ | Ph | " | 1:4 | 69% |
| (9) | $(CH_2)_2CN$ | $(CH_2)_3CH_3$ | $NC(CH_2)_2$, R' | <5:95 | 74% |
| (10) | $(CH_2)_2CN$ | Ph | " | 1:8 | 74% |

[a] Triphenylphosphonium bromide salts used unless otherwise noted. [b] Isomer ratios determined by integration of nonoverlapping signals in the proton NMR spectrum. [c] Isolated yields after flash chromatography. [d] Diethyl benzylphosphonate.

lower yields of olefin products and are more difficult to consistently reproduce. Thusfar, this procedure appears to occur only for organolithium reagents, since attempts to employ PhMgBr or vinyl magnesium bromide in place of the organolithium reagent failed to produce the olefin adducts.

## Experimental Section

All reactions were performed under $N_2$. Glassware and syringes were pre-dried overnight in an oven at 120 °C and assembled while still hot. The phosphonium salts were prepared[5] from the appropriate alkyl bromide (0.1 mmol) and triphenylphosphine (0.1 mmol) in refluxing toluene (200 mL) and dried in an oven overnight at 120 °C. Diethyl benzylphosphonate was purchased from Aldrich Chemical Co. and used without further purification. Dimethylformamide was freshly distilled from $CaH_2$ at 1 Torr. Solvents used for extractions and chromatography were distilled. *n*-Butyllithium (1.5 M in hexane solution) and phenyllithium (1.8 M in hexane solution) were purchased from Aldrich Chemical Co. and titrated with diphenylacetic acid[6] prior to use. TLC was carried out using EM Reagents plates with fluorescence indicator ($SiO_2$-60, F-254). Flash chromatography was performed according to the method of Still[7] using J.T. Baker flash chromatography silica gel (40 μm). $^1H$ NMR spectra were recorded at 400 MHz in $CDCl_3$ with $CHCl_3$ as an internal standard. IR spectra were obtained as a thin film on NaCl plates. Mass spectra were run using electron impact or chemical ionization.

**Representative Procedure for Coupling Reactions.** To an ice-cold mixture of (carbethoxymethyl)triphenylphosphonium bromide (0.928 g, 2.16 mmol) in DMF (10 mL) was added dropwise a hexane solution of phenyllithium (1.8 M, 1.3 mL, 2.3 mmol). The reaction mixture was warmed to rt and stirred for an additional 4 h. The pale yellow solution was poured into $H_2O$ (200 mL), extracted twice with hexane (50 mL), and dried over $MgSO_4$. Evaporation of the combined organic layers and purification of the crude product mixture by flash chromatography afforded ethyl cinnamate (0.338 g, 89%) as a colorless oil. The $^1H$ NMR and IR spectra of this material matched that of a commercially available sample. The other products in Table I were prepared in a corresponding manner and characterized on the basis of their $^1H$ NMR, IR, and mass spectra. The spectra for (*E*)- and (*Z*)-stilbene were found to be identical to those of the commercial materials. Experimental data for the remaining compounds are provided below.

**Ethyl 2-heptenoate**:[8] $^1H$ NMR δ 6.96 (1 H, m), 5.81 (1 H, d, $J$ = 15.6 Hz), 4.17 (2 H, q, $J$ = 6.8 Hz), 2.20 (2 H, dt, $J$ = 8.0, 6.8 Hz), 1.43 (2 H, m), 1.33 (2 H, m), 1.27 (3 H, t, $J$ = 6.8 Hz), 0.89 (3 H, t, $J$ = 6.8 Hz); IR 2970, 2940, 2880, 1725, 1655, 1460, 1370, 1300, 1270, 1175 $cm^{-1}$; EIMS $m/z$ (relative intensity) 156 ($M^+$, 9), 127 (45), 99 (100), 55 (29); exact mass calcd for $C_9H_{16}O_2$ 156.1093, found 156.1093.

**1-Phenyl-1-hexene**:[9] $^1H$ NMR δ 7.33–7.13 (5 H, m), 6.38 (*Z* isomer, 1 H, d, $J$ = 11.6 Hz), 6.35 (*E* isomer, 1 H, d, $J$ = 16.0 Hz), 6.20 (*E* isomer, 1 H, td, $J$ = 6.7, 15.8 Hz), 5.63 (*Z* isomer, 1 H, td, $J$ = 7.2, 11.7 Hz), 2.33 (*Z* isomer, 2 H, app q, $J$ = 7.2 Hz), 2.18 (*E* isomer, 2 H, app q, $J$ = 6.7 Hz), 1.53–1.23 (4 H, m), 0.89 (*E* isomer, 3 H, t, $J$ = 7.3 Hz), 0.87 (*Z* isomer, 3 H, t, $J$ = 7.3 Hz); IR 3030, 2970, 2940, 2870 $cm^{-1}$; EIMS $m/z$ (relative intensity) 160 ($M^+$, 30), 117 (100), 115 (30), 104 (60), 91 (50), 77 (20); exact mass calcd for $C_{12}H_{16}$ 160.1252, found 160.1268.

**5-Tridecene**:[10] $^1H$ NMR δ 5.35 (2 H, app t, $J$ = 4.8 Hz), 2.02 (4 H, br), 1.9–1.0 (14 H, br), 0.85 (6 H, m); $^{13}C$ NMR (75.4 MHz) δ 130.49, 130.41, 32.20, 32.09, 29.99, 29.48, 29.42, 27.40, 27.11, 22.85, 22.52, 14.22, 14.11; IR 2940, 2910, 2840, 1440 $cm^{-1}$; EIMS $m/z$ (relative intensity) 182 ($M^+$, 10), 125 (6), 111 (17), 97 (29), 83 (37), 69 (59), 57 (72), 55 (100); exact mass calcd for $C_{13}H_{26}$ 182.2035, found 182.2031.

**1-Phenyl-1-nonene**:[11] $^1H$ NMR δ 7.4–7.1 (5 H, m), 6.40 (major isomer, 1 H, D, $J$ = 10.8 Hz), 6.37 (minor isomer, 1 H, d, $J$ = 15.6 Hz), 6.24 (minor isomer, 1 H, m), 5.66 (major isomer, 1 H, m), 2.32 (major isomer, 2 H, dt, $J$ = 8.0, 6.8 Hz), 2.20 (minor isomer, 2 H, dt, $J$ = 8.0, 6.8 Hz), 1.9–1.0 (10 H, m), 0.85 (3 H, t, $J$ = 6.8 Hz); IR 3030, 2970, 2940, 2870 $cm^{-1}$; EIMS $m/z$ (relative intensity) 202 ($M^+$, 21), 117 (94), 115 (38), 104 (100), 91 (53), 77 (13); exact mass calcd for $C_{15}H_{22}$ 202.1722, found 202.1733.

**4-Nonenenitrile**: $^1H$ NMR δ 5.56 (1 H, app td, $J$ = 7.2, 10.8 Hz), 5.36 (1 H, m), 2.38 (4 H, m), 2.06 (2 H, m), 1.34 (4 H, m), 0.90 (3 H, m); IR 3020, 2965, 2940, 2865, 2245 (m) $cm^{-1}$; EIMS $m/z$ (relative intensity) 136 (M − 1, 10), 108 (44), 97 (42), 80 (28), 67 (52), 57 (60), 55 (100), 53 (89); exact mass calcd for $C_9H_{16}N$ (M+1) 138.1284, found 138.1290.

**5-Phenyl-4-pentenenitrile**:[12] $^1H$ NMR δ 7.36 (2 H, t, $J$ = 7.8 Hz), 7.29–7.25 (3 H, m), 6.62 (major isomer, 1 H, d, $J$ = 12.0 Hz), 6.53 (minor isomer, 1 H, d, $J$ = 15.6 Hz), 6.19 (minor isomer, 1 H, td, $J$ = 6.8, 15.6 Hz), 5.67 (major isomer, 1 H, td, $J$ = 7.6, 12.0 Hz), 2.69 (major isomer, 2 H, app q, $J$ = 7.6 Hz), 2.59 (minor isomer, 2 H, app q, $J$ = 6.8 Hz), 2.52 (minor isomer, 2 H, t, $J$ = 6.8 Hz), 2.45 (major isomer, 2 H, t, $J$ = 7.6 Hz); IR 3065, 3030, 2970, 2940, 2875, 2245 (m) $cm^{-1}$; EIMS $m/z$ (relative intensity) 157 ($M^+$, 22), 117 (100), 115 (39), 91 (26); exact mass calcd for

(5) *n*-Octyltriphenylphosphonium bromide, obtained as a colorless gum, was characterized by $^1H$ NMR: (400 MHz, $CDCl_3$) δ 7.87 (6 H, dd, $J$ = 11.8, 7.8 Hz), 7.76 (3 H, dd, $J$ = 11.6, 2.4 Hz), 7.71 (6 H, dd, $J$ = 7.6, 2.4 Hz), 3.87 (2 H, m), 1.63 (4 H, m), 1.19 (8 H, m), 0.83 (3 H, t, $J$ = 7.2 Hz).

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$C_{11}H_{11}N$ 157.0892, found 157.0870.

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**Supplementary Material Available:** $^1H$ and $^{13}C$ NMR spectra for all compounds described in the Experimental Section (9 pages). This material is contained in many libraries on microfiche, immediately follows this article in the microfilm version of the journal, and can be ordered from the ACS; see any current masthead page for ordering information.

## Dehalogenation of Organic Compounds. 3. Dechlorination of Polychlorinated Biphenyls, 4-Chlorobiphenyl, and Chloro-*p*-xylene with Alkoxyborohydrides†

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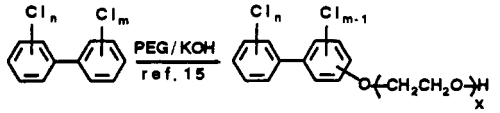
*Received May 27, 1992*

Dechlorinations of organic compounds are widely used chemical transformations for environmental decontamination.[1-7] Polychlorinated biphenyl (PCB) contamination, for example, is a serious concern at several superfund sites.[3,4] The PCB's have been employed for several decades in transformer dielectric fluids and carbonless copy paper[5] due to their thermal stability, flame retardancy, and other chemical inertness. Resistance to degradation and a tendency to accumulate in animal tissue[6] has resulted in control of PCB manufacture, use, and disposal under the Toxic Substances Control Act (TSCA).[7] Inactivation of these noxious chemicals has centered on incineration,[8] plasma incineration,[9] and other chemical methods such as wet oxidation[10] and sodium-based reduction.[11] Each of these methods has limitations. For example, incineration generates HCl while combustion produces small traces of dioxins.[12] Other chemical methods can require inert atmospheres and sensitive reagents. Thus, it is desirable to develop ways to rapidly dehalogenate organic molecules prior to their safe incineration. Hydride reducing agents ($LiAlH_4$, $NaBH_4$) are not very active in aromatic dehalogenations,[13] but reports[14] that hydrides combined with transition metal salts generate M(0) species make hydride/$M^{n+}$ combinations promising candidates for PCB dehalogenation.

### Results and Discussion

General Electric Co. pioneered dechlorination of PCB's (eq 1) using polyethylene glycol (PEG)/KOH.[15] However,




(1)

temperatures above 120 °C were routinely required, and only highly chlorinated PCB's were rapidly reduced. PCB mixtures with fewer chlorines, such as Aroclor 1016, resisted dechlorination.[15b] Aroclor 1016 gave no significant dechlorination by PEG/KOH after 14 days versus 99%

**Table I. Dechlorination[a] of Commercial Aroclors (PCB's) by PEG/KOH**

| | Aroclor (wt % Cl) | | | |
|---|---|---|---|---|
| | 1016 | 1242 | 1254 | 1260 |
| % dechlorination[b] (time in days) | 0 (14) | 27 (14) | 86 (6) | 99 (2) |
| 1[c] | 2 | 1 | | |
| 2[c] | 19 | 13 | | |
| 3[c] | 57 | 45 | 1 | |
| 4[c] | 22 | 31 | 15 | |
| 5[c] | | 10 | 53 | 12 |
| 6[c] | | | 26 | 42 |
| 7[c] | | | 4 | 38 |
| 8[c] | | | | 7 |
| 9[c] | | | | 2 |

[a] See references 6 and 15b. [b] At ambient temperature using 3000–5000 ppm by wt of Aroclor in a huge molar excess of PEG/KOH (1/1 by wt). The KOH contained 15% water by wt. [c] Number of chlorines per biphenyl unit.

**Table II. Dechlorination of Chloro-*p*-xylene**

| entry | reagents[a] (mol ratios) | time (h) | dechlorination[b] (mol %) |
|---|---|---|---|
| 1 | TEG/KOH[b] (46/1) | 168 | 42 |
| 2[c] | TEG/KOH/$NaBH_4$[b] (46/1/2) | 168 | 0 |
| 3 | $NaBH_2(OCH_2CH_2OCH_3)_2$/THF (4/47) | 168 | 28 |
| 4[d] | $NaBH_2(OCH_2CH_2OCH_3)_2/NiCl_2$/THF (4/3/47) | 3 | 81 |
| 5[e] | $NaBH_2(OCH_2CH_2OCH_3)_2/NiCl_2$/THF (4/2/47) | 2.5 | 98 |

[a] All ratios are relative to one mol of chloro-*p*-xylene. [b] Reaction temperature was 130 °C for entries 1 and 2. All other entries were run at 68 °C. [c] Phase separation of $NaBH_4$ occurred. [d] The alkoxyborohydride was added dropwise to substrate in THF/$NiCl_2$. [e] A suspension of $NiCl_2$ in THF was added incrementally to the reaction mixture using 0.20 molar equiv of $NiCl_2$ per portion.

† For the first two papers in this series see refs 1 and 2.

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