into water (2 mL), carefully neutralized with solid $NaHCO_3$, and extracted with ether. The extract was dried over anhydrous Na_2SO_4 and concentrated in vacuo to give spiro diether **7** as an oil: 290 mg (65%); bp 65–66 °C (1.0–1.5 mm) [lit.[5] bp 88–89 °C (4.5 mm)]; ^1H NMR δ 1.55 (t, CH_2CH_2O, J = 5.5 Hz, 8 H), 3.66 (t, CH_2O, J = 5.5 Hz, 8 H).

To a stirred solution of the spiro diether **7** (1.5 g, 0.96 mmol) in 48% HBr (10 mL) was added with cooling concentrated H_2SO_4 (5 mL). The mixture was warmed at 100 °C for 20 h and then worked up as described above in procedure A to afford pure tetrabromide **1**: 2.5 g (59%).

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Registry No. **1**, 5794-98-9; **3**, 29943-42-8; **4a**, 110796-52-6; **4b**, 4703-71-3; **5a**, 110796-49-1; **5b**, 110796-53-7; **5b** (methyl ester), 110796-54-8; **6**, 110796-50-4; **7**, 180-47-2; **8**, 110796-51-5; ethyl cyanoacetate, 105-56-6.

A Synthetically Useful Conversion of Benzoic Acid Derivatives to 4-Alkylphenols and 4-Alkyl-3-carbalkoxyphenols

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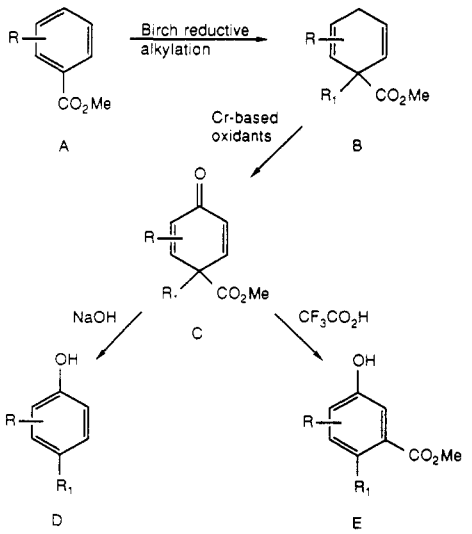
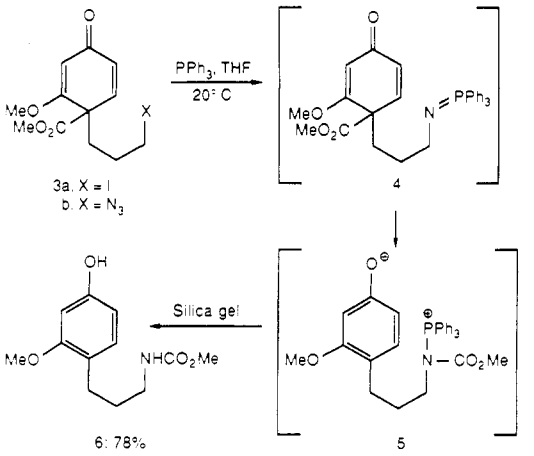
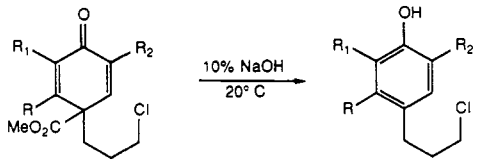
Substituted phenols play crucial roles in biosynthetic transformations, and they represent important building blocks for organic synthesis. Hence, there is a continuing need for the development of strategies for the preparation of phenolic substrates that are not readily available by conventional aromatic substitution chemistry.[2] This note describes three-step conversions of benzoic acid derivatives A to 4-alkylphenols D and 3-carbalkoxy-4-alkylphenols E. The process combines recently reported methodology used to convert benzoic acid derivatives to 2,5-cyclohexadien-1-ones[3] with familiar rearrangements of dienones to phenols.[4]



Scheme I



2,5-Cyclohexadien-1-ones **1a–d** were prepared from the corresponding benzoic ester by Birch reductive alkylation, followed by allylic oxidation.[3] Brief treatment of **1a–d** with aqueous sodium hydroxide solution at room temperature provided the 4-(3-chloropropyl)phenols **2a–d** in excellent yields.[5]



1a, R = R_1 = R_2 = H
b, R = OMe; R_1 = R_2 = H
c, R = R_2 = OMe; R_1 = H
d, R = H; R_1 = R_2 = Me

2a: 89%
2b: 87%
2c: 88%
2d: 82%

An interesting variant of this nucleophile-induced substitution process was discovered from treatment of 4-(3-azidopropyl)-2,5-cyclohexadienone **3b** with triphenylphosphine in tetrahydrofuran (THF); after chromatography on silica gel, the phenolic urethane **6** was obtained (Scheme I). This reaction presumably involves intramolecular methoxycarbonyl group transfer in the intermediate phosphine imine **4** to give zwitterion **5**, from which hydrolysis to **6** and triphenylphosphine oxide occurs during chromatography on silica gel.

Rearrangements of a series of 4,4-disubstituted 2,5-cyclohexadienones in trifluoroacetic acid to 3,4-disubstituted phenols have shown that the carbethoxy group migrates in preference to simple alkyl substituents.[6] With the literature conditions, both **1a** and **7**[7] cleanly underwent dienone–phenol rearrangements to 4-alkyl-3-carbomethoxyphenols **8a** and **8b**, respectively.

As illustrated in Scheme II, the method is particularly suited to the preparation of highly substituted 3-carbalk-

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(3) Schultz, A. G.; Lavieri, F. P.; Macielag, M.; Plummer, M. *J. Am. Chem. Soc.* **1987**, *109*, 3991.
(4) For reviews of the dienone–phenol rearrangement, see: (a) de Mayo, P. In *Elucidation of Structures by Physical and Chemical Methods*; Bentley, K. W., Ed.; Wiley-Interscience: New York, 1963; p 1106. (b) Collins, C. J.; Eastham, J. F. In *The Chemistry of the Carbonyl Group*; Patai, S., Ed.; Wiley-Interscience: New York, 1966; p 775. (c) Waring, A. J. *Adv. Alicycl. Chem.* **1966**, *1*, 207. (d) Miller, B. In *Mechanisms of Molecular Migrations*; Thyagarajan, B. S., Ed.; Wiley-Interscience: New York, 1969; Vol. 1, p 275.
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(7) The rearrangement of 4-methyl-4-carbethoxy-2,5-cyclohexadien-1-one to 4-methyl-3-carbethoxyphenol is reported in ref 6.

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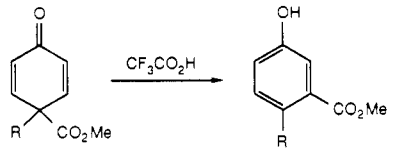


1a, R = $CH_2CH_2CH_2Cl$; 7, R = Me — 8a, R = $CH_2CH_2CH_2Cl$; b, R = Me

oxy phenols. Phenol **12** would be difficult if not impossible to prepare from readily available aromatic precursors by conventional electrophilic substitution or directed metalation procedures.

Experimental Section

See ref 3 for the preparation of 2,5-cyclohexadienones **1a–c** and general experimental procedures.

Preparation of 3-Carbomethoxy-3-(3-chloropropyl)-1,5-dimethyl-1,4-cyclohexadiene. The title compound was obtained from methyl 3,5-dimethylbenzoate by the method described in ref 3 as a colorless oil (93%): ^1H NMR ($CDCl_3$) δ 1.59 (m, 2 H), 1.72 (m, 2 H), 1.75 (s, 6 H), 2.45 (s, 2 H), 3.48 (t, J = 6.5 Hz, 2 H), 3.67 (s, 3 H), 4.40 (s, 2 H); IR ($CDCl_3$) 2940, 1715, 1430, 1245, 1195 cm^{-1}; chemical ionization mass spectrum, m/z (relative intensity) 243 ($M^+ + 1$, 100), 207 (45), 183 (57), 165 (31), 147 (27).

Preparation of 4-Carbomethoxy-4-(3-chloropropyl)-2,6-dimethyl-2,5-cyclohexadien-1-one (1d). 1d was obtained from 3-carbomethoxy-3-(3-chloropropyl)-1,5-dimethyl-1,4-cyclohexadiene by the method involving PCC described in ref 3 as a colorless oil (80%): ^1H NMR ($CDCl_3$) δ 1.6–1.71 (m, 2 H), 1.94 (s, 6 H), 2.04–2.12 (m, 2 H), 3.50 (t, J = 6.3 Hz, 2 H), 3.73 (s, 3 H), 6.75 (s, 2 H); IR (film) 1730, 1665, 1640, 1430, 1372, 1235–1215 (br) cm^{-1}; chemical ionization mass spectrum, m/z (relative intensity) 257 ($M^+ + 1$, 100), 221 (40), 177 (2.7), 163 (3.6), 137 (5.0), 135 (5.8).

Preparation of 4-(3-Chloropropyl)phenol (2a). General Method of Preparation of 4-Alkylphenols. A solution of **1a** (89 mg, 0.3 mmol) in ether (5 mL) together with 10% sodium hydroxide solution (5 mL) was rapidly stirred at 20 °C for 15 min. The organic layer was separated, and the aqueous layer was acidified to pH 2 with 10% hydrochloric acid. The aqueous mixture was washed with ether (3 × 10 mL), and the combined organic extracts were washed with water (1 × 10 mL) and brine (1 × 10 mL), and dried over anhydrous magnesium sulfate. Removal of solvent and flash chromatography (silica gel; hexane/ethyl acetate, 3:1) gave **2a** (61 mg, 89%) as a colorless oil: ^1H NMR ($CDCl_3$) δ 2.01 (m, 2 H), 2.70 (t, J = 7 Hz, 2 H), 3.49 (t, J = 7 Hz, 2 H), 4.80 (s, exchangeable with D_2O, 1 H), 6.78 (d, J = 7 Hz, 2 H), 7.06 (d, J = 8 Hz, 2 H); IR (film) 3600–3100 (br), 3020, 2960, 2940, 2860, 1610, 1595, 1510, 1440 cm^{-1}; chemical ionization mass spectrum, m/z (relative intensity) 171 ($M^+ + 1$, 100), 135 (52), 107 (53).

Anal. Calcd for $C_9H_{11}ClO$: C, 63.35; H, 6.50. Found: C, 63.27; H, 6.48.

4-(3-Chloropropyl)-3-methoxyphenol (2b): prepared from **1b** as a colorless oil in 87% yield; ^1H NMR ($CDCl_3$) δ 2.00 (m, 2 H), 2.68 (t, J = 7 Hz, 2 H), 3.52 (t, J = 7 Hz, 2 H), 3.78 (s, 3 H), 4.79 (s, exchangeable with D_2O, 1 H), 6.35 (dd, J = 8 Hz, 2 Hz, 1 H), 6.41 (d, J = 2 Hz, 1 H), 6.98 (d, J = 8 Hz, 1 H); IR (film) 3650–3100 (br), 3000, 2955, 2935, 2835, 1600, 1500, 1460, 1428, 1330 cm^{-1}; chemical ionization mass spectrum, m/z (relative intensity) 201 ($M^+ + 1$, 100), 165 (77), 137 (83).

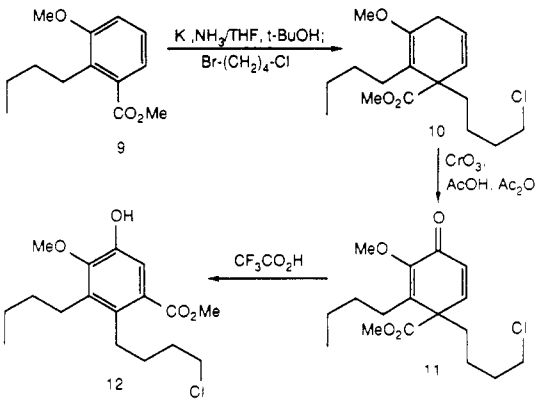
Anal. Calcd for $C_{10}H_{13}ClO_2$: C, 59.86; H, 6.53. Found: C, 59.81; H, 6.54.

4-(3-Chloropropyl)-2,5-dimethoxyphenol (2c): prepared from **1c** as a colorless oil in 88% yield; ^1H NMR ($CDCl_3$) δ 2.00 (quintet, J = 7 Hz, 2 H), 2.69 (t, J = 7 Hz, 2 H), 3.53 (t, J = 7 Hz, 2 H), 3.72 (s, 3 H), 3.81 (s, 3 H), 5.58 (s, exchangeable with D_2O, 1 H), 6.56 (s, 1 H), 6.71 (s, 1 H); IR (film) 3600–3200 (br), 3000, 2960, 2940, 2840, 1600, 1508, 1460, 1421 cm^{-1}; chemical ionization mass spectrum, m/z (relative intensity) 231 ($M^+ + 1$, 100), 195 (68), 167 (94).

Anal. Calcd for $C_{11}H_{15}ClO_3$: C, 57.27; H, 6.55. Found: C, 57.36; H, 6.50.

4-(3-Chloropropyl)-2,6-dimethylphenol (2d): prepared from **1d** as a colorless oil in 82% yield; ^1H NMR ($CDCl_3$) δ 2.02 (m, 2 H), 2.22 (s, 6 H), 2.64 (t, J = 7.6 Hz, 2 H), 3.52 (t, J = 6.5 Hz, 2 H), 4.54 (s, exchangeable with D_2O, 1 H), 6.80 (s, 2 H); IR (film) 3570–3200 (br), 2925, 1700, 1600, 1485, 1440, 1300, 1200, 1150 cm^{-1}; electron impact mass spectrum, m/z (relative intensity) 198 (M^+, 20), 135 (100), 105 (5.8), 91 (17.6), 77 (6).

Scheme II



4-Carbomethoxy-4-(3-iodopropyl)-3-methoxy-2,5-cyclohexadien-1-one (3a). 3a was prepared from **1b** by reaction with sodium iodide in refluxing acetone (24 h); flash chromatography (silica gel; ethyl acetate/hexane, 1:1) gave **3a** in 80% yield. The analytical sample was prepared by recrystallization from methylene chloride/hexane: mp 77–79 °C; ^1H NMR ($CDCl_3$) δ 1.42–1.70 (m, 2 H), 2.12 (m, 1 H), 2.38 (m, 1 H), 3.14 (t, J = 7 Hz, 2 H), 3.71 (s, 3 H), 3.79 (s, 3 H), 5.74 (d, J = 2 Hz, 1 H), 6.36 (dd, J = 10, 2 Hz, 1 H), 6.52 (d, J = 10 Hz, 1 H); IR (KBr) 3060, 2980, 2938, 1732, 1652, 1622, 1587, 1445, 1428, 1368 cm^{-1}; chemical ionization mass spectrum, m/z (relative intensity) 351 ($M^+ + 1$, 100), 223 (20).

Anal. Calcd for $C_{12}H_{15}IO_4$: C, 41.16; H, 4.32. Found: C, 41.25; H, 4.40.

4-(3-Azidopropyl)-4-carbomethoxy-3-methoxy-2,5-cyclohexadien-1-one (3b). 3b was prepared from **3a** by reaction with sodium azide in dimethylformamide at room temperature; flash chromatography (silica gel; ethyl acetate/hexane, 1:1) gave **3b** in 80% yield. The analytical sample was prepared by trituration with ether: mp 58–60 °C; ^1H NMR ($CDCl_3$) δ 1.14–1.43 (m, 2 H), 2.08 (dt, J = 13, 4 Hz, 1 H), 2.32 (dt, J = 13, 4 Hz, 1 H), 3.27 (t, J = 7 Hz, 2 H), 3.70 (s, 3 H), 3.76 (s, 3 H), 5.75 (s, 1 H), 6.36 (d, J = 10 Hz, 1 H), 6.50 (d, J = 10 Hz, 1 H); IR (film) 3065, 3020, 2945, 2870, 2100, 1738, 1655, 1628, 1597, 1450, 1431, 1370 cm^{-1}; chemical ionization mass spectrum, m/z (relative intensity) 266 ($M^+ + 1$, 70), 238 (41), 206 (48), 195 (67), 178 (64), 151 (100), 137 (81).

Anal. Calcd for $C_{12}H_{15}N_3O_4$: C, 54.33; H, 5.70. Found: C, 54.39; H, 5.63.

Conversion of 3b to Phenol 6. A solution of **3b** (37 mg, 0.14 mmol) and triphenylphosphine (37 mg, 0.14 mmol) in dry THF (2 mL) was stirred at 20 °C for 24 h. Removal of solvents followed by flash chromatography (silica gel; ethyl acetate/hexane, 3:2) gave **6** (26 mg, 78%) as a colorless oil: ^1H NMR ($CDCl_3$) δ 1.73 (m, 2 H), 2.57 (t, J = 7 Hz, 2 H), 3.16 (m, 2 H), 3.69 (br s, 3 H), 3.78 (s, 3 H), 5.27 (s, exchangeable with D_2O, 1 H), 6.38 (dd, J = 8, 2 Hz, 1 H), 6.42 (d, J = 2 Hz, 1 H), 6.95 (d, J = 8 Hz, 1 H); IR (film) 3600–3100 (br), 2960, 1690, 1610, 1595, 1530, 1508, 1465, 1430 cm^{-1}; chemical ionization mass spectrum, m/z (relative intensity) 240 ($M^+ + 1$, 44), 208 (100).

Anal. Calcd for $C_{12}H_{17}NO_4$: C, 60.24; H, 7.16. Found: C, 59.54; H, 7.46.

3-Carbomethoxy-4-(3-chloropropyl)phenol (8a). A solution of **1a** (0.11 g, 0.5 mmol) in trifluoroacetic acid (2 mL) was stirred at room temperature for 70 h. Removal of solvents followed by flash chromatography (silica gel; hexane/ethyl acetate, 3:1) gave **8a** (95 mg, 83%) as a colorless oil: ^1H NMR ($CDCl_3$) 2.02 (m, 2 H), 3.01 (t, J = 7 Hz, 2 H), 3.52 (t, J = 7 Hz, 2 H), 3.88 (s, 3 H), 5.78 (s, exchangeable with D_2O, 1 H), 6.96 (dd, J = 8, 2 Hz, 1 H), 7.15 (d, J = 8 Hz, 1 H), 7.42 (d, J = 2 Hz, 1 H); IR (film) 3650–3100 (br), 3000, 2955, 2865, 1690, 1602, 1575, 1497, 1436 cm^{-1}; chemical ionization mass spectrum, m/z (relative intensity) 229 ($M^+ + 1$, 100), 193 (31).

Anal. Calcd for $C_{11}H_{13}ClO_3$: C, 57.78; H, 5.73. Found: C, 57.81; H, 5.78.

4-Carbomethoxy-4-methyl-2,5-cyclohexadien-1-one (7). 7 was obtained from 3-carbomethoxy-3-methyl-1,4-cyclohexadiene[8] as a colorless oil in 57% yield by the method involving CrO_3 described in ref 3. Spectral data are in agreement with those reported in the literature.[5]

3-Carbomethoxy-4-methylphenol (8b). 8b was prepared from 7 as colorless crystals [mp 73–74 °C (lit.[9] mp 74–75 °C)] in 75% yield. Spectral data for 8b were not reported:[9] ^{1}H NMR ($CDCl_3$) δ 2.51 (s, 3 H), 3.89 (s, 3 H), 4.81 (s, 1 H), 6.92 (dd, J = 9 Hz, J = 3 Hz, 1 H), 7.12 (d, J = 9 Hz, 1 H), 7.40 (d, J = 3 Hz, 1 H); IR (film) 3315, 1685, 1220 cm^{-1}; chemical ionization mass spectrum, m/z (relative intensity) 167 (M^+ + 1, 100).

2-Butyl-3-carbomethoxy-3-(4-chlorobutyl)-1-methoxy-1,4-cyclohexadiene (10). A solution of 9 (0.94 g, 4.2 mmol; prepared by esterification of 2-butyl-3-methoxybenzoic acid[10]) and *tert*-butyl alcohol (0.31 g, 4.2 mmol) in dry THF (5 mL) was cooled to −78 °C. Liquid ammonia (50 mL) and then potassium (0.44 g, 11.3 mmol) were added to the stirred reaction mixture. After 0.5 h at −78 °C, 1-bromo-4-chlorobutane (0.86 g, 5 mmol) was added. The reaction mixture was stirred at −78 °C for 0.5 h and quenched with excess solid NH_4Cl. After evaporation of ammonia, the residue was partitioned between ether (60 mL) and water (10 mL). The two layers were separated, and the organic phase was washed with brine (10 mL) and dried over magnesium sulfate. Concentration under reduced pressure gave a yellow oil that was purified by flash chromatography (silica gel; hexane/ethyl acetate, 2.3:1) to afford a colorless oil (1.1 g, 82%): ^{1}H NMR ($CDCl_3$) δ 0.88 (m, 3 H), 1.18–1.40 (m, 6 H), 1.60–2.05 (m, 6 H), 2.85–2.95 (m, 2 H), 3.52 (t, J = 6.7 Hz, 2 H), 3.58 (s, 3 H), 3.65 (s, 3 H), 5.40–5.52 (m, 1 H), 5.9–6.0 (m, 1 H); IR (film) 1720, 1650 cm^{-1}; chemical ionization mass spectrum, m/z 315 (M^+ + 1).

3-Butyl-4-carbomethoxy-4-(4-chlorobutyl)-2-methoxy-2,5-cyclohexadien-1-one (11): Obtained from 10 as an oil in 34% yield by the method involving CrO_3 described in ref 3; ^{1}H NMR ($CDCl_3$) δ 0.91 (m, 3 H), 1.1–1.5 (m, 6 H), 1.65–1.9 (m, 2 H), 2.0–2.3 (m, 4 H), 3.49 (t, J = 6.5 Hz, 2 H), 3.68 (s, 3 H), 3.82 (s, 3 H), 6.40 (s, J = 9.9 Hz, 1 H), 6.65 (d, J = 9.8 Hz, 1 H); IR (film) 1730, 1660, 1640 cm^{-1}; chemical ionization mass spectrum, m/z 329 (M^+ + 1).

Anal. Calcd for $C_{17}H_{25}ClO_4$: C, 62.09; H, 7.66. Found: C, 61.88; H, 7.55.

Methyl 3-butyl-2-(4-chlorobutyl)-5-hydroxy-4-methoxybenzoate (12): obtained from 11 as a colorless oil in 60% yield; ^{1}H NMR ($CDCl_3$) δ 0.96 (t, J = 6.7 Hz, 3 H), 1.4–2.0 (m, 8 H), 2.65 (t, J = 6.7 Hz, 2 H), 2.85 (t, J = 6.7 Hz, 2 H), 3.59 (t, J = 6.6 Hz, 2 H), 3.82 (s, 3 H), 3.86 (s, 3 H), 5.68 (s, 1 H), 7.33 (s, 1 H); IR (film) 3420, 1720 cm^{-1}; chemical ionization mass spectrum, m/z 329 (M^+ + 1).

Anal. Calcd for $C_{17}H_{25}ClO_4$: C, 62.09; H, 7.66. Found: C, 61.89; H, 7.73.

Acknowledgment. This research was supported in part by the National Institute of General Medical Science (GM 26568). We thank the Sterling-Winthrop Research Institute for graduate research fellowship support for A.G.T.

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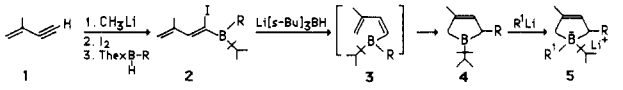
Communications

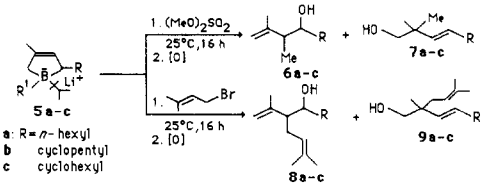
3-Borolenes. Their Regio- and Diastereoselective Conversion into Substituted Homoallylic Alcohols

Summary: The 3-borolene *ate* complexes derived from isopropenylacetylene react with dimethyl sulfate or with prenyl bromide in a regio- and diastereoselective manner to furnish substituted homoallylic alcohols. The overall reaction represents a dialkylation hydroxylation of the isopropenylacetylene precursor.

Sir: Allylic boron compounds have played an important role in the development of new methods for acyclic stereocontrol.[1] The ready accessibility of 3-borolenes (boracyclopent-3-enes) 4 from *cis*-dienylboranes 3 via reductive isomerization of alkylthexyl(1-iodo-1,3-alkadienyl)boranes 2[2] led us to explore their utility as reagents for the stereoselective preparation of substituted homoallylic alcohols. We now report that the *ate* complexes 5 derived from 3-borolenes 4 react with carbon electrophiles such as dimethyl sulfate or prenyl bromide (1-bromo-3-methyl-2-butene) in a regio- and diastereoselective manner to furnish the corresponding substituted homoallylic alcohols. Specifically, we describe our studies of borolenes derived from isopropenylacetylene (2-methyl-1-buten-3-yne, (1) whose use allows for extension of a carbon chain by one isoprene unit.



Preliminary investigations revealed that the borolenes 4[3] (R, = *n*-hexyl, cyclopentyl, cyclohexyl) did not react with dimethyl sulfate. However, their conversion into the corresponding ate complexes 5 with methyllithium enhanced their reactivities toward the alkylating agent to furnish, after oxidative workup, the methylated homoallylic alcohols 6 in 94–98% regioisomeric purities (Table I).[4] The reaction of 5 with methyl iodide was very sluggish. It



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