at −20 °C for 1 h, 2-bromophenol (81 μL, 0.66 mmol) was injected over 2 min. All the solids dissolved as soon as the addition was complete. With the cold bath left in place, the mixture was allowed to attain room temperature (over ca. 1 h), and the solvents were then evaporated. Flash chromatography of the residue over silica gel (2 × 18 cm) using 12% ether–hexane afforded crude 3, which was further purified on a Chromatotron [circular plate coated with a 2-mm-thick adsorbent (silica gel 60 PF_{254} containing gypsum)]. Successive elution with 4%, 8%, and 12% ether–hexane gave pure 3 (72 mg, 33%) as a colorless oil: FT-IR ($CHCl_3$ cast) 1720, 1475, 1183 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz) δ 1.27 (t, J = 7.0 Hz, 3 H), 1.63 (t, J = 3.8 Hz, 1 H), 1.82–2.03 (m, 3 H), 2.55–2.70 (m, 1 H), 4.13 (q, J = 7.0 Hz, 2 H), 4.55–4.65 (m, 1 H), 5.72 (br d, J = 10.2 Hz, 1 H), 6.18 (dm, J = 10.2 Hz, 1 H), 6.82–6.93 (m, 2 H), 7.20–7.30 (m, 1 H), 7.50–7.57 (m, 1 H); ^{13}C NMR ($CDCl_3$, 75.5 MHz) δ 14.26, 19.09, 20.39, 24.60, 27.09, 60.71, 71.32, 113.63, 115.77, 122.58, 126.50, 127.94, 128.36, 133.68, 154.31, 172.52; exact mass, m/z calcd for $C_{16}H_{17}{}^{79}BrO_3$ 336.0362, found 336.0350. Anal. Calcd for $C_{16}H_{17}BrO_3$: C, 56.98; H, 5.08; O, 14.23. Found: C, 56.84; H, 4.91; O, 14.28.

Ethyl (3α,4aα,9bα)-(±)-(3,4,4a,9b-Tetrahydrodibenzofuran-3-yl)acetate (4). Et_3B (1 M in hexane, 0.22 mL, 0.22 mmol) and then Bu_3SnH (0.231 mL, 0.878 mmol) were added to a stirred solution of 3 (140 mg, 0.439 mmol) in dry hexanes (18 mL). The mixture was stirred at 35 °C for 24 h. The solvent was then evaporated and the residue was diluted with ether (6 mL) and stirred with an excess of KF in water. After 1 h, the mixture was diluted with ether (20 mL) and the organic layer was washed with water (6 mL), dried ($MgSO_4$), and evaporated. Flash chromatography of the residue over silica gel (1.5 × 12 cm) using 5–10% EtOAc–hexane afforded 4 (71 mg, 67%) and an unidentified side product (12 mg). Compound 4: FT-IR ($CHCl_3$ cast) 2860, 1731, 1597, 1477, 1230 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz) δ 1.28 (t, J = 7.2 Hz, 3 H), 1.58–1.78 (m, 2 H), 2.31–2.41 [m, including a d at δ 2.35, (J = 7.2 Hz), 3 H], 2.72–2.92 (m, 1 H), 3.79 (br d, 7.2 Hz, 1 H), 4.17 (q, J = 7.2 Hz, 2 H), 5.06 (m, 1 H), 5.72 (br s, 2 H), 6.77–6.90 (m, 2 H), 7.05–7.12 (m, 2 H); ^{13}C NMR ($CDCl_3$, 75.5 MHz) δ 14.30, 26.90, 31.49, 40.25, 41.06, 60.45, 80.70, 109.94, 120.58, 124.51, 127.19, 128.28, 130.78, 130.86, 159.37, 172.12; exact mass, m/z calcd for $C_{16}H_{18}O_3$ 258.1255, found 258.1252.

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Registry No. 2, 135189-62-7; 3, 135145-37-8; 4, 135145-38-9; 5, 21449-12-7; 6, 135145-39-0; 7, 135145-40-3; 8, 85696-74-8; 9, 135145-41-4; 10, 135145-42-5; 11, 135145-43-6; 12, 135145-44-7; 13, 135145-45-8; 14, 135145-46-9; 15, 135145-47-0; 16, 135189-63-8; 17, 135145-48-1; 18, 135145-49-2; 2-bromophenol, 95-56-7.

Supplementary Material Available: ^1H NMR spectra of 2, 4, 13, 15, 16, and 18 (6 pages). Ordering information is given on any current masthead page.

$NiCl_2$(dppe)-Catalyzed Geminal Dialkylation of Dithioacetals and Trimethylation of Ortho Thioesters[1]

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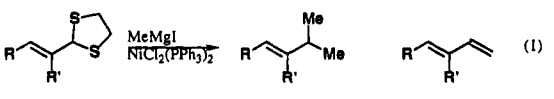
$NiCl_2$(dppe)-catalyzed cross-coupling of cinnamaldehyde dithioacetals gave the corresponding geminal dimethylation products in excellent yields. Allylic ortho thioesters afforded regioselectively the corresponding trimethylation products. The reaction may occur via an 18-electron π-allyl intermediate, which undergoes facile reductive elimination to afford the geminal dimethylation product. Benzylic dithioacetals having an ortho amino group gave 2-isopropylanilines exclusively. The reaction of benzylic dithioacetals with EtMgBr under the same conditions yielded geminal diethylation products.

We recently reported a series of new nickel-catalyzed olefination reactions of dithioacetals for the synthesis of substituted styrenes, allylsilanes, vinylsilanes, silylated butadienes, and other substituted butadienes.[3] These reactions involve a successive coupling process and a β-elimination step (eq 1). Theoretically, the two carbon–



1 2 3 (1)

sulfur bonds in dithioacetals can both be replaced by carbon–carbon bonds, hence producing *gem*-dialkyl systems. In this case, the catalytic cycle would consist of two sequential reductive elimination steps. The selectivity between geminal dialkylation versus olefination may depend on the nature of the catalyst. It is generally believed that a saturated 18-electron organometallic species would favor reductive elimination over β-elimination, and the

(1) Transition Metal Promoted Reactions. 38.

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Table I. Geminal Dimethylation of Allylic Dithioacetal

entry	substr	R	R'	catalyst[a]	% yield	ratio 2:3
1	1a	Ph	H	A	87	69:31
2	1a	Ph	H	B	93	100:0
3	1d	4-MeC_6H_4	H	B	98	100:0
4	1e	3-$MeOC_6H_4$	H	B	95	100:0
5	1f	2-naphthyl	H	B	95	100:0
6	1g	2-$MeOC_6H_4$	H	B	92	96:4
7	1b	Ph	Me	A	91	57:43
8	1b	Ph	Me	B	92	77:23
9	1c	Ph	Et	A	71	31:69
10	1c	Ph	Et	B	96	68:32

[a] A, $NiCl_2(PPh_3)_2$; B, $NiCl_2$(dppe).

reverse would be true for an unsaturated one.[4] Indeed, Kurosawa and co-workers recently found that the 18-electron (η^3-allyl-η^1-aryl)nickel(II) complexes rapidly undergo reductive elimination.[5] It is known that the reduction of carbon–sulfur bonds occurs in the $NiCl_2(PPh_3)_2$-catalyzed reactions[6] of a secondary Grignard reagent with organosulfur compounds,[7] while couplings predominante when the nickel catalyst containing bidendate ligands such as dppp ($Ph_2PCH_2CH_2CH_2PPh_2$) is employed as the catalyst.[8] In a preliminary communication, we reported that reductive elimination is a facile step in the catalytic reaction when an 18-electron (η^3-allyl)-organonickel intermediate is involved.[9] We now describe the details of this reaction.

Results and Discussion

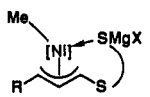
Geminal Dimethylation of Allylic Dithioacetals. Geminal dimethylation of a carbonyl group or its equivalent can be achieved by a number of methods.[10] However, these procedures are not applicable to α,β-unsaturated carbonyl compounds because a significant amount of the 1,3-dimethylation product is obtained in addition to the expected 1,1-dimethylation product.[10] We have previously shown that the sulfur moiety in allylic dithioacetals can regioselectively direct nickel-catalyzed cross-coupling with a Grignard reagent leading to an efficient synthesis of 1-silylbutadienes.[3d,f] Presumably, the coordination of sulfur to nickel causes regioselective reductive elimination leading to the formation of a carbon–carbon bond.[3d,f] Based on this conjecture and the work by Kurosawa[5] and by Felkin,[11] we felt that geminal dimethylation could be executed under the appropriate conditions. Therefore, a solution of cinnamaldehyde dithioacetal (**1a**) and 4 equiv of MeMgI in the presence of 3 mol % of $NiCl_2(PPh_3)_2$ in ether–benzene was heated under reflux for 12 h to give a mixture of geminal dimethylation product **2a** and butadiene **3a** (entry 1, Table I). These results constitute the first example of regioselective geminal dimethylation of α,β-unsaturated carbonyl equivalents.

Table II. $NiCl_2$(dppe)-Catalyzed Trimethylation of Allylic Ortho Thioesters

entry	compd	R	R'	% yield	ratio 7:8
11	6a	Ph	H	74	100:0
12	6b	2-$MeOC_6H_4$	H	67	100:0
13	6c	4-$MeOC_6H_4$	H	54	100:0
14	6d	4-MeC_6H_4	H	63	100:0
15	6e	2-naphthyl	H	59	100:0
16	6f	Ph	Me	51	72:28
17	6g	Ph	Et	64	58:42

When $NiCl_2$(dppe) was used as the catalyst, cinnamaldehyde dithioacetals **1a–d** gave **2a–d** in excellent yields as the sole products (entries 2–5). Other bidendate phosphine ligands such as dppp or dppb behaved similarly. The chemoselectivity was also improved substantially for substrates having a substituent at C-2 when $NiCl_2$(dppe) was employed as the catalyst in comparison with those using $NiCl_2(PPh_3)_2$ (entries 7–10). The results are summarized in Table I.

The reaction may occur via a similar pathway as proposed earlier,[3] proceeding via π-allyl intermediates **4** and **5**. The involvement of an 18-electron π-allyl intermediate



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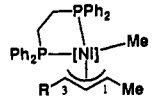

5

probably enhances the reductive elimination process that leads to the facile formation of the second carbon–carbon bond. Due to the directive effect of the concatenated thiolato anion **4**, the introduction of the first methyl group at the C-1 position is expected. The regiospecific introduction of the second methyl group may be controlled by steric factors, since C-1 in **5** is less hindered than C-3. Conjugative preferences are known in a number of regioselective nickel-catalyzed cross-coupling reactions of aryl-substituted allylic substrates,[11] and if this effect is significant, it may further enhance the regioselectivity of the geminal dimethylation reaction of allylic dithioacetals **1**.

Trimethylation of Allylic Ortho Thioesters. An ortho thioester has three carbon–sulfur bonds that could in principle undergo cross-coupling to give three new carbon–carbon bonds. When aryl-substituted allylic ortho thioesters **6** were employed, two trimethylation products (**7** and **8**) might be expected (eq 2). Compound **7** may be favored by steric factors, while **8** may be facilitated by the conjugative preference.[11]

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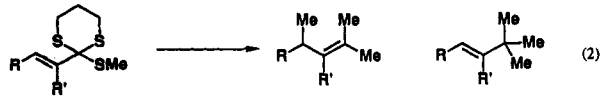
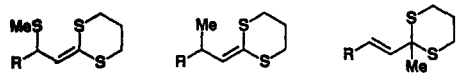


6 7 8 (2)

Treatment of **6** with 5 equiv of MeMgI in the presence of 5 mol % of $NiCl_2$(dppe) in refluxing toluene gave **7** in satisfactory yields. The results are tabulated in Table II. When there is no substituent at the C-2 position, the reaction was regiospecific, giving **7** exclusively (entries 11–15). On the other hand, upon increasing the steric congestion at C-2, the reaction became less selective and a mixture of **7** and **8** was obtained (entries 16 and 17).

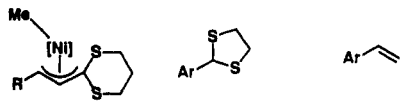
The regioselectivity of this reaction is interesting. The reaction of **6a** with MeMgI using $NiCl_2$(dppe) as the catalyst was stopped as soon as reflux temperature was achieved, and **9** and **10a** were obtained in addition to recovered **6a**. It is noteworthy that an equilibrium between



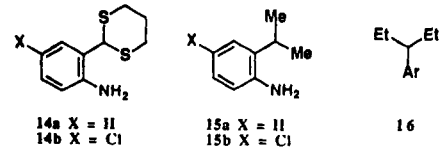
9 10a 10b

6a and **9** occurs under these conditions.[12] The isolation of **10a** indicated that the first carbon–carbon bond is formed at C-3. Such regioselectivity is somewhat striking. The directive effect of the dithiane moiety in the π-allyl intermediate **11** apparently behaves differently from that of the sulfurs in intermediate **4**. Since the environment at C-3 is less hindered than that at C-1, the coupling process would thus occur at this less crowded site. Alternatively, it has been suggested that the regiochemistry of Grignard reagent–nickel π-allyl couplings leads preferentially to the more π-acidic olefin that can more efficiently coordinate to low valent nickel.[11] Presumably, the olefin moiety in **10a** may be a better ligand for low valent nickel than the alternative styrene moiety **10b**.[11] The presence of a substituent at C-2 would destabilize **11** and the next coupling step would become less selective, yielding a mixture of **7** and **8**. Having shown that **10a** is the first intermediate in the trimethylation of allylic ortho thioesters, it becomes evident that further coupling proceeds in a similar manner as those in vinylic sulfides.[6]

Geminal Dialkylation of Benzylic Dithioacetals. The extension of the geminal dimethylation to simple benzylic dithioacetals **12** has been unsuccessful. Under the usual conditions using $NiCl_2$(dppe) as the catalyst, styrenes **13** were obtained predominantly, if not exclusively.



11 12 13



14a X = H 14b X = Cl 15a X = H 15b X = Cl 16

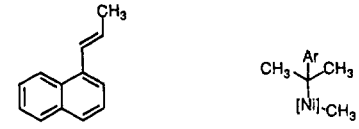
However, the presence of an *o*-amino group in dithioacetal **14** changes the situation, with 2-isopropylanilines **15** being obtained as the sole product in 60–70% yields. Since the number of the valence electrons determines the relative rates of reductive elimination versus β-elimination,[5] the nitrogen atom in these substrates may provide two "extra" electrons via coordination to the nickel atom during the reaction. Hence, the rate of reductive elimination is enhanced. The *p*-amino derivative **12** (Ar = 4-aminophenyl), as expected, afforded styrene **13** (Ar = 4-aminophenyl) in 60% yield. The *o*-hydroxy group in the phenolic analogue, on the other hand, shows no neighboring group effect, the corresponding styrene being obtained.

Table III. Nickel-Catalyzed Geminal Diethylation of Benzylic Dithioacetals

entry	substr	Ar	% yield of 16
18	**13a**	Ph	52
19	**13b**	4-MeC_6H_4	52
20	**13c**	2-thienyl	62
21	**13d**	1-naphthyl	59[a]

[a] 17 was also obtained in 14% yield.

When EtMgBr was employed in the cross-couplings with **12**, geminal diethylated products **16** were obtained. Representative results are tabulated in Table III. When a sterically hindered substrate such as **13d** was employed, β-hydride elimination from intermediate **18** becomes more competitive leading to the formation of **17** as a side product (entry 21).



17 18

It is noteworthy that geminal dialkylation other than dimethylation of carbonyl group or its equivalent cannot readily be achieved by conventional methods.[10] The observations described above is the protype of this sort of reaction.

Experimental Section

General Procedure for the Geminal Dialkylation of Dithioacetal with the Grignard Reagent. To a solution of **1** (2.0 mmol) and the nickel catalyst (0.06–0.2 mmol) in benzene (8 mL) under N_2 was added the Grignard reagent (8.0 mmol in ether solution), and the mixture was refluxed for 12 h, quenched with water, and diluted with ether (30 mL). The organic layer was washed with aqueous NaOH (10%, 10 mL) and water (20 mL × 3) before being dried anhydrous $MgSO_4$. After filtration and evaporation of the solvent, the residue was purified by chromatography on silica gel (hexane) to afford the desired product(s).

3-Methyl-1-phenyl-1-butene (2a). Following the general procedure, the reaction of **1a** (500 mg, 2.4 mmol), MeMgI (5.0 mL of a 2 M solution in ether, 10.0 mmol), and $NiCl_2$(dppe) (38.6 mg, 0.07 mmol) gave **2a**[13] (324 mg, 93%): ^{1}H NMR ($CDCl_3$, 250 MHz) δ 1.10 (d, J = 7.0 Hz, 6 H), 2.48 (m, 1 H), 6.20 (dd, J = 16.0, 6.6 Hz, 1 H), 6.35 (d, J = 16.0 Hz, 1 H), 7.12–7.40 (m, 5 H); MS m/z (rel intensity) 146 (64), 131 (100), 115 (23), 91 (66).

In a similar manner, a mixture of **1a** (300 mg, 1.4 mmol), $NiCl_2(PPh_3)_2$ (35.0 mg, 0.05 mmol), and MeMgI (4.2 mmol) in benzene (8 mL) was refluxed for 16 h to give **2a** (118 mg, 60%) and **3a**[3e] (47 mg, 27%), which exhibited identical spectroscopic properties as those of the authentic samples.

2,3-Dimethyl-1-phenyl-1-butene (2b) and 2-Methyl-1-phenylbutadiene (3b). Following the general procedure, **1b** (300 mg, 1.35 mmol) was allowed to react with MeMgI (2.5 mL 2 M solution in ether, 5.0 mmol) and $NiCl_2$(dppe) (38.6 mg, 0.07 mmol) in benzene (8 mL) to give **2b**[14] (152 mg, 71%) and **3b**[15] (42 mg, 21%). **2b**: ^{1}H NMR ($CDCl_3$, 250 MHz) δ 1.08 (d, J = 6.7 Hz,

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6 H), 1.81 (s, 3 H), 2.41 (hept, J = 6.7 Hz, 1 H), 6.06 (s, 1 H), 7.11–7.28 (m, 5 H); MS m/z (rel intensity) 160 (39), 145 (100), 129 (60), 115 (25), 91 (37). **3b**: ^{1}H NMR ($CDCl_3$, 250 MHz) δ 2.18 (s, 3 H), 5.15 (d, J = 10 Hz, 1 H), 5.26 (d, J = 18.0 Hz, 1 H), 6.53 (s, 1 H), 6.58 (dd, J = 18, 10 Hz, 1 H), 7.18–7.40 (m, 5 H); MS m/z (rel intensity) 144 (65), 129 (100), 115 (21).

In a similar manner, the reaction of **1b** (500 mg, 2.3 mmol), MeMgI (5.0 mL 2 M solution in ether, 10.0 mmol), and $NiCl_2(PPh_3)_2$ (80 mg, 0.1 mmol) in benzene gave **2b** (191 mg, 52%) and **3b** (129 mg, 39%).

3-Methyl-2-ethyl-1-phenyl-1-butene (2c) and 2-Ethyl-1-phenylbutadiene (3c). Following the general procedure, the reaction of **1c** (500 mg, 2.1 mmol), MeMgI (5.0 mL 2 M solution in ether, 10.0 mmol), and $NiCl_2$(dppe) (38.6 mg, 0.07 mmol) gave **2c**[16] (240 mg, 65%) and **3c**[17] (103 mg, 31%). **2c**: ^{1}H NMR ($CDCl_3$, 250 MHz) δ 1.05 (d, J = 6.7 Hz, 3 H), 1.13 (d, J = 6.8 Hz, 6 H), 2.27 (q, J = 6.7 Hz, 2 H), 2.43 (hept, J = 6.8 Hz, 1 H), 6.25 (s, 1 H), 7.12–7.38 (m, 5 H); MS m/z (rel intensity) 174 (38), 159 (16), 145 (100), 131 (25), 117 (43). **3c**: ^{1}H NMR ($CDCl_3$, 250 MHz) δ 1.90 (t, J = 7.5 Hz, 3 H), 2.49 (q, J = 7.5 Hz, 2 H), 5.18 (d, J = 10 Hz, 1 H), 5.35 (d, J = 18 Hz, 1 H), 6.42 (s, 1 H), 6.49 (dd, J = 18 Hz, 1 H), 7.20–7.40 (m,5 H); MS m/z (rel intensity) 188 (6), 174 (7), 159 (20), 145 (14), 129 (100), 105 (40), 91 (31).

In a similar manner, treatment of **1c** (500 mg, 2.1 mmol) with MeMgI (5.0 mL 2 M solution in ether, 10.0 mmol) and $NiCl_2(PPh_3)_2$ (80 mg, 0.1 mmol) in benzene (8 mL) afforded **2c** (82 mg, 22%) and **3c** (145 mg, 49%).

3-Methyl-1-(4-methylphenyl)-1-butene (2d). Following the general procedure, **1d** (350 mg, 1.35 mmol) was treated with MeMgI (3.0 mL 2 M solution in ether, 6.0 mmol) and $NiCl_2$(dppe) (25 mg, 0.05 mmol) in benzene to give **2d**[18] (246 mg, 98%): ^{1}H NMR ($CDCl_3$, 250 MHz) δ 1.08 (d, J = 6.7 Hz, 6 H), 2.31 (s, 3 H), 2.40–2.47 (m, 1 H), 6.12 (dd, J = 16, 6.7 Hz, 1 H), 6.30 (d, J = 16 Hz, 1 H), 7.08 (d, J = 8.2 Hz, 2 H), 7.32 (d, J = 8.2 Hz, 2 H); MS m/z (rel intensity) 160 (31), 145 (100), 128 (18), 105 (30), 91 (14).

3-Methyl-1-(2-methoxyphenyl)-1-butene (2g) and 1-(2-Methoxyphenyl)butadiene (3g). Following the general procedure, **1g** (500 mg, 2.1 mmol) was treated with MeMgI (5.0 mL 2 M solution in ether, 10.0 mmol) and $NiCl_2$(dppe) (38.6 mg, 0.07 mmol) in benzene (8 mL) to give **2g** (324 mg, 88%) and **3g**[19] (trace amount). **2g**: ^{1}H NMR ($CDCl_3$, 250 MHz) δ 1.05 (d, J = 7 Hz, 6 H), 2.50 (m, 1 H), 3.80 (s, 3 H), 6.18 (dd, J = 16, 6.8 Hz, 1 H), 6.68 (d, J = 16 Hz, 1 H), 6.81 (d, J = 8.2 Hz, 1 H), 6.90 (t, J = 8.2 Hz, 1 H), 7.17 (t, J = 8.2 Hz, 1 H), 7.41 (d, J = 8.2 Hz, 1 H); MS m/z (rel intensity) 176 (50), 161 (100), 145 (12), 121 (28); HRMS calcd for $C_{12}H_{16}O$ 176.1201, found 176.1201.

3-Methyl-1-(3-methoxyphenyl)-1-butene (2e). Following the general procedure, **1e** (270 mg, 1.1 mmol) was treated with MeMgI (3 mL 2 M solution in ether, 6.0 mmol) and $NiCl_2$(dppe) (38.6 mg, 0.07 mmol) in benzene (8 mL) to give **2e**[20] (190 mg, 95%): ^{1}H NMR ($CDCl_3$, 250 MHz) δ 1.08 (d, J = 6.8 Hz, 6 H), 2.40–2.49 (m, 1 H), 3.79 (s, 3 H), 6.18 (dd, J = 16, 6.4 Hz, 1 H), 6.31 (d, J = 16 Hz, 1 H), 6.75 (d, J = 8 Hz, 1 H), 6.88 (s, 1 H), 6.94 (d, J = 8 Hz, 1 H), 7.20 (t, J = 8 Hz, 1 H).

3-Methyl-1-(2-naphthyl)-1-butene (2f). Following the general procedure, **1f** (300 mg, 1.2 mmol) was treated with MeMgI (4 mL 2 M solution in ether, 8.0 mmol) and $NiCl_2$(dppe) (38.6 mg, 0.07 mmol) in benzene to form **2f**[21] (216 mg, 95%): ^{1}H NMR ($CDCL_3$, 250 MHz) δ 1.18 (d, J = 6.8 Hz, 6 H), 2.55–2.64 (m, 1 H), 6.21 (dd, J = 16, 6.5 Hz, 1 H), 7.07 (d, J = 16 Hz, 1 H), 7.36–7.60 (m, 4 H), 7.72 (d, J = 8 Hz, 1 H), 7.81 (d, J = 8 Hz, 1 H), 8.14 (d, J = 8 Hz, 1 H).

2-Methyl-4-phenyl-2-pentene (7a). Following the general procedure, **6a** (274 mg, 1.02 mmol) was treated with MeMgI (2 mL of a 2.5 M solution in ether, 5.0 mmol) and $NiCl_2$(dppe) (30 mg, 0.06 mmol) in refluxing benzene (10 mL) for 16 h to give **7a** (121 mg, 74%): ^{1}H NMR ($CDCl_3$, 200 MHz) δ 1.31 (d, J = 8.7 Hz, 3 H), 1.67 (d, J = 1.2 Hz, 3 H), 1.70 (s, 3 H), 3.66 (dq, J = 8.7, 6.0 Hz, 1 H), 5.62 (d, J = 6 Hz, 1 H), 7.12–7.33 (m, 5 H); ^{13}C NMR δ 17.9, 22.4, 25.8, 38.1, 125.7, 126.8, 128.3, 130.1, 130.5, 147.3; MS m/z (rel intensity) 160 (39), 145 (100), 130 (11), 117 (33), 105 (16), 91 (20), 77 (11); HRMS calcd for $C_{12}H_{16}$ 160.1252, found 160.1247.

4-(2-Methoxyphenyl)-2-methyl-2-pentene (7b). Following the general procedure, **6b** (304 mg, 1.02 mmol) was allowed to react with MeMgI (2.5 mL of a 2.5 M solution, 6.3 mmol) and $NiCl_2$(dppe) (54 mg, 0.1 mmol) in refluxing benzene (10 mL) for 16 h to give **7b** (144 mg, 67%): IR (neat) 2964, 2925, 1599, 1490, 1453, 1438, 1243, 1051, 1035, 755 cm^{-1}; ^{1}H NMR ($CDCl_3$, 200 MHz) δ 1.24 (d, J = 7 Hz, 3 H), 1.64 (s, 3 H), 1.68 (s, 3 H), 3.81 (s, 3 H), 4.06 (dq, J = 9, 7 Hz, 1 H), 5.29 (d, J = 9 Hz, 1 H), 6.58–6.94 (m, 2 H), 7.10–7.24 (m, 2 H); ^{13}C NMR δ 17.9, 21.8, 25.8, 31.3, 55.4, 110.6, 120.6, 126.5, 127.1, 129.8, 130.5, 135.8, 156.6; MS m/z (rel intensity) 190 (62), 175 (100), 160 (32), 159 (47), 145 (22), 105 (36), 91 (56), 77 (37); HRMS calcd for $C_{13}H_{18}O$ 190.1357, found 190.1353.

4-(4-Methoxyphenyl)-2-methyl-2-pentene (7c). Following the general procedure, **6c** (98 mg, 0.33 mmol) was allowed to react with MeMgI (1.0 mL of a 2.5 M solution, 2.5 mmol) and $NiCl_2$(dppe) (18 mg, 0.03 mmol) in refluxing benzene (10 mL) for 16 h to give **7c** (34 mg, 54%): IR 2964, 1611, 1510, 1452, 1247, 1080, 1042, 832 cm^{-1}; ^{1}H NMR ($CDCl_3$, 200 MHz) δ 1.27 (d, J = 7 Hz, 3 H), 1.67 (d, J = 1.3 Hz, 3 H), 1.70 (d, J = 1.3 Hz, 3 H), 3.81 (s, 3 H), 3.61 (dq, J = 9.4, 7 Hz, 1 H), 5.24 (d, J = 9.4 Hz, 1 H), 6.83 (d, J = 9 Hz, 2 H), 7.15 (d, J = 9 Hz, 2 H); ^{13}C NMR δ 17.9, 22.5, 25.8, 37.2, 55.2, 113.7, 127.7, 130.1, 130.4, 139.4, 157.6; MS m/z (rel intensity) 190 (25), 175 (100), 160 (9), 145 (6), 135 (25), 91 (11), 57 (24); HRMS calcd for $C_{13}H_{18}O$ 190.1357, found 190.1355.

2-Methyl-4-(4-methylphenyl)-2-pentene (7d). Following the general procedure, **6d** (149 mg, 0.53 mmol) was allowed to react with MeMgI (2.0 mL of a 1.5 M solution, 3.0 mmol) and $NiCl_2$(dppe) (45 mg, 0.09 mmol) in refluxing benzene (10 mL) for 16 h to give **7d** (58 mg, 63%): IR 2961, 2920, 1507, 1445, 1372, 1244, 815 cm^{-1}; ^{1}H NMR ($CDCl_3$, 200 MHz) δ 1.28 (d, J = 7 Hz, 3 H), 1.67 (d, J = 1.0 Hz, 3 H), 1.69 (d, J = 1.0 Hz, 3 H), 2.31 (s, 3 H), 3.62 (dq, J = 9.3, 7 Hz, 1 H), 5.23 (d, J = 9.3 Hz, 1 H), 7.11 (s, 4 H); ^{13}C NMR δ 17.9, 21.0, 22.5, 25.8, 37.7, 126.7, 129.0, 130.2, 130.4, 135.1, 144.3; MS m/z (rel intensity) 174 (26), 159 (100), 144 (8), 129 (11), 115 (22), 105 (8), 73 (41); HRMS calcd for $C_{13}H_{18}$ 174.1408, found 174.1393.

2-Methyl-4-(2-naphthyl)-2-pentene (7e). Following the general procedure, **6e** (160 mg, 0.51 mmol) was allowed to react with MeMgI (2.0 mL of a 1.5 M solution, 3.0 mmol) and $NiCl_2$(dppe) (39 mg, 0.07 mmol) in refluxing benzene (10 mL) for 16 h to give **7e** (62 mg, 59%): IR 2965, 2925, 1600, 1451, 1378, 1059, 858, 821, 749 cm^{-1}; ^{1}H NMR ($CDCl_3$, 200 MHz) δ 1.39 (d, J = 7 Hz, 3 H), 1.73 (s, 6 H), 3.82 (m, 1 H), 5.36 (d, J = 7 Hz, 1 H), 7.36–7.48 (m, 3 H), 7.64 (s, 1 H), 7.75–7.81 (m, 3 H); ^{13}C NMR δ 18.0, 22.4, 25.6, 38.3, 124.5, 125.1, 125.8, 126.2, 127.5, 127.6, 127.9, 130.0, 130.9, 133.7, 144.8; MS m/z (rel intensity) 210 (56), 195 (100), 180 (15), 165 (16), 153 (13); HRMS calcd for $C_{16}H_{18}$ 210.1408, found 210.1407.

2,3-Dimethyl-4-phenyl-2-pentene (7f) and 2,3,3-Trimethyl-1-phenyl-1-butene (8f). Following the general procedure, **6f** (288 mg, 1.02 mmol) was allowed to react with MeMgI (3.0 mL of a 2.0 M solution, 6.0 mmol) and $NiCl_2$(dppe) (27 mg, 0.05 mmol) in refluxing benzene (10 mL) for 16 h to give a 72:28 mixture of **7f** and **8f** (84 mg, 51%). Attempts to separate these two isomers were unsuccessful. The mixture showed the following data: ^{1}H NMR ($CDCl_3$, 200 MHz) δ 1.17 (s, 9 H, **8f**), 1.33 (d, J = 6.3 Hz, 3 H, **7f**), 1.38 (s, 3 H, **8f**), 1.77 (s, 3 H, **7f**), 1.83 (s, 3 H, **7f**), 4.14 (q, J = 6.3 Hz, 1 H, **7f**), 6.36 (s, 1 H, **8f**), 7.11–7.37 (m, 5 H); MS m/z (rel intensity) 174 (36), 159 (96), 145 (25), 131 (28), 117 (100), 105 (54), 91 (43), 77 (23); HRMS calcd for $C_{13}H_{18}$ 174.1408, found 174.1391.

3-Ethyl-2-methyl-4-phenyl-2-pentene (7g) and 2-Ethyl-3,3-dimethyl-1-phenyl-1-butene (8g). Following the general procedure, **6g** (350 mg, 1.18 mmol) was allowed to react with MeMgI (2.0 mL of a 2.5 M solution, 5.0 mmol) and $NiCl_2$(dppe) (59 mg, 0.11 mmol) in refluxing benzene (10 mL) for 16 h to give a 58:42 mixture of **7g** and **8g** (143 mg, 65%). By employing

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preparative GC (6-ft SE30 column), **8g** was obtained in pure form: ^{1}H NMR ($CDCl_3$, 200 MHz) δ 0.98 (t, J = 7.5 Hz, 3 H), 1.17 (s, 9 H), 2.30 (q, J = 7.5 Hz, 2 H), 6.36 (s, 1 H,), 7.13–7.35 (m, 5 H); ^{13}C NMR δ 15.1, 21.0, 29.7, 37.3, 122.4, 125.7, 128.6; HRMS calcd for $C_{14}H_{20}$ 188.1565, found 188.1556. **7g**: ^{1}H NMR ($CDCl_3$, 200 MHz) δ 0.70 (t, J = 7.5 Hz, 3 H), 1.36 (d, J = 7 Hz, 3 H), 1.71 (s, 3 H), 1.81 (s, 3 H,), 1.85 (q, J = 7.5 Hz, 2 H), 4.12 (q, J = 7 Hz, 1 H), 7.12–7.31 (m, 5 H); ^{13}C NMR δ 14.5, 17.5, 20.3, 20.8, 21.7, 40.3, 124.8, 125.5, 127.5, 127.9, 137.7, 145.6.

Isolation of Intermediate 10a from the Reaction of 6a with MeMgI. Following the general procedure, a mixture of **6a** (268 mg, 1.0 mmol), MeMgI (10 mL of a 0.5 M solution, 5.0 mmol), and $NiCl_2$(dppe) (33 mg, 0.06 mmol) in benzene (10 mL) was refluxed for 10 min, cooled to room temperature, and quenched with a saturated NH_4Cl solution (10 mL). The organic layer was separated and the aqueous solution was extracted with ether (50 mL). The combined organic portions were washed with 10% NaOH (2 × 20 mL) and water (2 × 20 mL), dried ($MgSO_4$), evaporated in vacuo to give a residue, which was chromatographed on silica gel (hexane) to afford **10a** (80 mg 34%), **9**[12] (62 mg, 23%), and the starting material **6a** (112 mg, 42%). **10a**: ^{1}H NMR ($CDCl_3$, 200 MHz) δ 1.34 (d, J = 7.0 Hz, 3 H), 2.08–2.20 (m, 2 H), 2.79–2.90 (m, 4 H), 4.04 (dq, J = 7.0, 9.5 Hz, 1 H), 6.05 (d, J = 9.5 Hz, 1 H), 7.10–7.30 (m, 5 H); ^{13}C NMR δ 21.3, 25.1, 29.6, 30.2, 39.3, 125.3, 126.1, 126.9, 128.4, 138.3, 145.2; MS m/z (rel intensity) 236 (84), 221 (100), 161 (38), 147 (35), 105 (12); HRMS calcd for $C_{12}H_{16}S$ 236.0693, found 236.0705.

3-Phenylpentane (16a). Following the general procedure, a benzene solution (8 mL) of **12a** (182 mg, 1.0 mmol), EtMgBr (4.0 mL of a 2.0 M solution, 8 mmol), and $NiCl_2$(dppe) 26.4 mg, 0.05 mmol) was refluxed for 16 h to give **16a**[22] (77 mg, 52%): ^{1}H NMR ($CDCl_3$, 200 MHz) δ 0.77 (t, J = 7 Hz, 6 H), 1.43–1.76 (m, 4 H), 2.21–2.30 (m, 1 H), 7.10–7.32 (m, 5 H).

3-(4-Methylphenyl)pentane (16b). Following the general procedure, a benzene solution (8 mL) of **12b** (197 mg, 1.0 mmol), EtMgBr (4.0 mL of a 2.0 M solution, 8 mmol), and $NiCl_2$(dppe) (26.4 mg, 0.05 mmol) was refluxed for 16 h to give **16b**[23] (84 mg, 52%): ^{1}H NMR ($CDCl_3$, 200 MHz) δ 0.76 (t, J = 7.4 Hz, 6 H), 1.43–1.77 (m, 4 H), 2.20–2.39 (m, 4 H including a singlet at 2.38), 7.01 (d, J = 8 Hz, 2 H), 7.10 (d, J = 8 Hz, 2 H).

3-(1-Naphthyl)pentane (16c) and 1-(1-Naphthyl)propene (17). Following the general procedure, a benzene solution (8 mL) of **12d** (232 mg, 1.0 mmol), EtMgBr (4.0 mL of a 2.0 M solution, 8 mmol), and $NiCl_2$(dppe) (26.4 mg, 0.05 mmol) was refluxed for 16 h to give a 4:1 ratio mixture of **16c** and **17**[24] (156 mg). The two compounds were separated by preparative GC. **16c**: ^{1}H NMR ($CDCl_3$, 200 MHz) δ 0.82 (t, J = 7.4 Hz, 6 H), 1.66–1.93 (m, 4 H), 3.28–3.45 (br q, J = 6 Hz, 1 H), 7.35 (d, J = 8 Hz, 1 H), 7.40–7.55 (m, 3 H), 7.67 (d, J = 8 Hz, 1 H), 7.85 (d, J = 8 Hz, 1 H), 8.13 (d, J = 8 Hz); MS m/z (rel intensity) 198 (62), 169 (100), 141 (19); HRMS calcd for $C_{15}H_{18}$ 190.1408, found 190.1405.

9,9-Diethylfluorene (20). Following the general procedure, a benzene solution (8 mL) of **19** (256 mg, 1.0 mmol), EtMgBr (4.0 mL of a 2.0 M solution, 8 mmol), and $NiCl_2$(dppe) (26.4 mg, 0.05 mmol) was refluxed for 16 h to give **20**[25] (140 mg, 69%): ^{1}H NMR ($CDCl_3$, 200 MHz) δ 0.90 (t, J = 7.4 Hz, 6 H), 1.96 (q, J = 7.4 Hz, 4 H), 7.26–7.36 (m, 4 H), 7.60–7.75 (m, 4 H).

2-Isopropylaniline (15a). Following the general procedure, the reaction of **14a**[26] (211 mg, 1.0 mmol), MeMgI (4.0 mL of a 2 M solution in ether, 8 mmol), and $NiCl_2$(dppe) (26.4 mg, 0.05 mmol) in THF (12 mL) gave **15a**[27] (93 mg, 69%): ^{1}H NMR ($CDCl_3$, 200 MHz) δ 1.25 (d, J = 6.8 Hz, 6 H), 2.88 (hept, J = 6.8 Hz, 1 H), 3.45 (bs, 2 H), 6.58–7.15 (m, 3 H).

4-Chloro-2-isopropylaniline (15b). Following the general procedure, the reaction of **14b**[26] (246 mg, 1.0 mmol), MeMgI (4.0 mL of a 2 M solution in ether 8 mmol), and $NiCl_2$(dppe) (26.4 mg, 0.05 mmol) in THF (12 mL) gave **15b** (105 mg, 62%):[28] ^{1}H NMR ($CDCl_3$, 200 MHz) δ 1.22 (d, J = 6.8 Hz, 6 H), 2.83 (hept, J = 6.8 Hz, 1 H), 3.60 (bs, 2 H), 6.56–7.07 (m, 3 H).

4-Aminostyrene (13). Following the general procedure, the reaction of **12** (Ar = 4-aminophenyl, 211 mg, 1.0 mmol), MeMgI (4.0 mL of a 2 M solution in ether, 8 mmol), and $NiCl_2$(dppe) (26.4 mg, 0.05 mmol) in THF (12 mL) gave **13** (Ar = 4-aminophenyl, 71 mg, 60%):[29] ^{1}H NMR ($CDCl_3$, 200 MHz) δ 3.69 (bs, 2 H), 5.05 (d, J = 10.9 Hz, 1 H), 5.65 (d, J = 17.5 Hz, 1 H), 6.55–6.69 (m, 3 H), 7.24 (d, J = 6.6 Hz, 2 H).

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Registry No. **1a**, 5616-58-0; **1b**, 119925-03-0; **1c**, 119925-04-1; **1d**, 119924-99-1; **1e**, 119925-00-7; **1f**, 119925-01-8; **1g**, 119925-02-9; **2a**, 1608-28-2; **2b**, 36939-20-5; **2c**, 61777-12-6; **2d**, 32094-37-4; **2e**, 119925-06-3; **2f**, 135042-74-9; **2g**, 119925-07-4; **3b**, 37580-42-0; **3c**, 119925-09-6; **3g**, 60573-56-0; **6a**, 130573-25-0; **6b**, 135042-80-7; **6c**, 135042-81-8; **6d**, 130552-14-6; **6e**, 135042-82-9; **6f**, 135042-83-0; **6g**, 135042-84-1; **7a**, 50704-01-3; **7b**, 135042-75-0; **7c**, 35029-32-4; **7d**, 135042-76-1; **7e**, 135042-77-2; **7f**, 92104-76-2; **7g**, 135042-78-3; **8f**, 29772-45-0; **8g**, 135042-79-4; **9**, 135042-85-2; **10a**, 29833-95-2; **12a**, 5616-55-7; **12b**, 23229-29-0; **12d**, 86201-62-9; **12** (Ar = 4-aminophenyl), 94838-73-0; **13**, 1520-21-4; **14a**, 53165-22-3; **14b**, 53165-23-4; **15a**, 643-28-7; **15b**, 76842-14-3; **16a**, 1196-58-3; **16b**, 22975-58-2; **16c**, 3042-56-6; **17**, 22767-77-7; **19**, 7049-31-2; **20**, 2294-79-3; $NiCl_2(PPh_3)_2$, 14264-16-5; MeMgI, 917-64-6; EtMgBr, 925-90-6; $NiCl_2$(dppe), 14647-23-5.

Supplementary Material Available: ^{1}H NMR spectra of **2g**, **7a–e**, **8g**, and **10a** (8 pages). Ordering information is given on any current masthead page.

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