Supporting Information

Regioselective Alkenylation of Aromatic Ketones with Alkenylboronates Using $\text{RuH}_2(\text{CO})(\text{PPh}_3)_3$ - Catalyst via Carbon-Hydrogen Bond Cleavage

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General Information.

Chemical shifts of ¹H NMR and ¹³C NMR signals are quoted relative to residual protiated solvent: CHCl₃ (δ = 7.26 and 77.0) or tetramethylsilane (δ = 0.00). ¹H NMR data are reported as follows: chemical shift in ppm (δ), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet, c = complex, br = broad), coupling constant (Hz), relative intensity. ¹³C NMR data are reported as follows: chemical shift in ppm (δ).

Solvent and Materials.

Acetophenone and pinacolone were dried over CaSO₄ and were distilled under nitrogen. Toluene was dried over CaH₂ and was distilled under nitrogen. RuH₂(CO)(PPh₃)₃ (**3**) was prepared by a literature method.¹ Alkenylboronates were prepared from corresponding alkenylboronic acid and 2,2-dimethyl-1,3-propandiol (2 eq.) in benzene (1.0 M) at room temperature overnight.

GC Analysis.

The conditions used for GC analysis were as follows: capillary column: CBP-10 25 m, 0.2 mm; temperature program, 70 °C (0 min)-10 °C/min-250 °C (30 min); injection temperature, 270 °C; detector temperature, 270 °C.

General Procedure

The apparatus used, which consisted of 10 mL two-necked flask equipped with a reflux condenser connected to a nitrogen line, a rubber septum, and a magnetic stirring bar, was flame-dried and cooled to room temperature under a flow of dry nitrogen. $\text{RuH}_2(\text{CO})(\text{PPh}_3)_3$ (0.02 mmol), 0.5 mL of pinacolone, the aromatic ketone (1 mmol), and the alkenylboronate (1.2 mmol) were placed in the flask. The resulting mixture was refluxed under a nitrogen atmosphere and the progress of the reaction was monitored by GC analysis. The product was isolated by column chromatography on alumina (Merck, aluminium oxide 90 active basic (0.063-0.200 mm)) and/or on silica gel (Merck, silica gel 60 (230-400 mesh ASTM)) column chromatography. Further purification of the product was performed by GPC, if necessary.

Data of Spectra

2,2-Dimethyl-1-(2-propenylphenyl)propan-1-one (7).

$R_f = 0.44$ (hexane: EtOAc = 5:1). B.p. 125 °C/1.0 mmHg; ^1H NMR (CDCl_3) δ 1.22 (s, 9H, $\text{C}(\text{CH}_3)_3$), 1.83-1.86 (m, 3H, $=\text{CCH}_3$), 6.08-6.21 (m, 1H, $=\text{CHCH}_3$), 6.25 (d, $J = 16.2$ Hz, 1H, $\text{C}=\text{CHAr}$), 7.06-7.33 (m, 3H, ArH), 7.49 (d, $J = 7.8$ Hz, 1H, ArH); ^{13}C NMR (CDCl_3) δ 18.79, 27.33, 45.17, 124.61, 125.59, 125.77, 128.33, 128.42, 128.47, 134.31, 139.49, 215.22; IR (KBr) 2966 w, 1690 s, 1481 m, 1461 w, 1446 w, 1394 w, 1365 w, 1268 w, 1185 w, 963 s, 946 w, 752 w cm^{-1} ; MS m/z (% relative intensity) 202 (M^+ , 10), 146 (12), 145 (100), 117 (65), 116 (11), 115 (47), 91 (20), 57 (12), 51 (12). Anal. Calcd for $\text{C}_{14}\text{H}_{18}\text{O}$: C, 83.12; H, 8.97%. Found: C, 82.84; H, 8.83%.

2,2-Dimethyl-1-[2-(1-methylpropenyl)phenyl]propan-1-one (*E* and *Z* isomers in a ratio of 65:35)

B.p. 130 °C/1.0 mmHg; ^1H NMR (CDCl_3) δ 1.07 (s, 9H, $\text{C}(\text{CH}_3)_3$), 1.15 (s, 9H, $\text{C}(\text{CH}_3)_3$), 1.35-1.38 (m, 3H, $=\text{CCH}_3$), 1.61-1.63 (m, 3H, $=\text{CCH}_3$), 1.88-1.90 [(m) and 1.92 (s), 3H, $=\text{CCH}_3$], 5.36 and 5.44 (q, $J = 7.1$ Hz, 1H, $=\text{CH}$), 6.97 and 7.02 [(d, $J = 8.0$ Hz) and (d, $J = 7.6$ Hz), ArH], 7.12-7.31 (m, 3H, ArH); ^{13}C NMR (CDCl_3) δ major isomer: 14.36, 17.70, 27.79, 44.88, minor isomer: 15.55, 26.33, 28.14, 44.59, the peaks of aromatic area are too complex to be identified: 123.33, 125.27, 125.71, 125.84, 127.20, 127.29, 128.25, 128.64, 129.69, 135.54, 136.62, 139.83, 141.60, 216.44; MS m/z (% relative intensity); 216.10 (M^+ , 10), 201 (15), 160 (15), 159 (70), 145 (11), 132 (10), 131 (100), 129 (15), 128 (10), 116 (15), 115 (27), 91 (44), 77 (12), 57 (24); IR (neat) 2970 s, 2927 m, 2871 w, 1689 s, 1479 m, 1365 w, 1279 w, 1194 m, 1030 w, 964 s, 764 m, 731 w, 675 m; HRMS Calcd for $\text{C}_{15}\text{H}_{20}\text{O}$: 216.1514. Found: 216.1523.

2,2-Dimethyl-1-(2-styrylphenyl) propan-1-one

M.p. 70-72 °C; $R_f = 0.42$ (hexane:EtOAc = 5:1). ^1H NMR (CDCl_3) δ 1.24 (s, 9H, $\text{C}(\text{CH}_3)_3$), 6.94 (d, $J = 16.2$ Hz, 1H, $=\text{CH}$), 7.03 (d, $J = 16.2$ Hz, 1H, $\text{CH}=\text{$), 7.14 (d, $J = 7.3$ Hz, 1H, ArH), 7.22-7.46 (m, 7H, ArH), 7.69 (d, $J = 7.3$ Hz, 1H, ArH); ^{13}C NMR (CDCl_3) δ 27.39, 45.29, 124.87, 125.58, 125.67, 126.51, 126.57, 127.86, 128.62, 128.67, 130.97, 133.70, 136.83, 140.34, 215.18; IR (KBr) 2964 w, 2868 w, 1685 s, 1496 m, 1478 w, 1461 w, 1449 w, 1363 w, 1271 w, 1195 w, 1184 w, 963 s, 945 w, 767 s, 749 w, 734 w, 694 m, 681 w, 669 w, 655 w, 531 w cm^{-1} ; MS m/z (% relative intensity) 264 (M^+ , 11), 208 (16), 207 (100), 179 (28), 178 (46), 57 (16), 51 (12). Anal. Calcd for $\text{C}_{19}\text{H}_{20}\text{O}$: C, 86.32; H, 7.63%. Found: C, 86.17; H, 7.62%.

2,2-Dimethyl-1-[2-(2-p-tolylvinyl)phenyl]propan-1-one

M.p. 73-75 °C; ^1H NMR (CDCl_3) δ 1.23 (s, 9H, $\text{C}(\text{CH}_3)_3$), 2.34 (s, 3H, ArCH_3), 6.87 (d, $J = 16.2$ Hz, 1H, $=\text{CH}$), 6.99 (d, $J = 16.2$ Hz, 1H, $\text{CH}=\text{}$), 7.07-7.38 (m, 7H, ArH), 7.67 (d, $J = 7.6$ Hz, 1H, ArH); ^{13}C NMR (CDCl_3) δ 21.26, 27.29, 45.24, 124.73, 124.92, 125.56, 126.47, 126.52, 128.73, 129.44, 130.99, 133.94, 134.16, 137.96, 140.35, 215.66; IR (KBr) 2970 m, 2932 w, 2868 w, 1687 s, 1516 m, 1476 m, 1445 w, 1392 w, 1361 w, 1268 w, 1196 w, 1183 w, 964 s, 942 w, 808 s, 776 w, 755 m, 532 m cm^{-1} ; MS m/z (% relative intensity) 278 (M^+ , 15), 222 (15), 221 (100), 178 (41), 115 (10). Anal. Calcd for $\text{C}_{20}\text{H}_{22}\text{O}$: C, 86.29; H, 7.97%. Found: C, 86.01; H, 7.96%.

1-{2-[2-(4-Methoxyphenyl)vinyl]phenyl}-2,2-dimethylpropan-1-one

M.p. 73-73 °C; ^1H NMR (CDCl_3) δ 1.24 (s, 9H, $\text{C}(\text{CH}_3)_3$), 3.82 (s, 3H, OCH_3), 6.77-7.01 (m, 4H, ArH), 7.12 (d, $J = 7.6$ Hz, 1H, ArH), 7.22 (d, $J = 7.3$ Hz, 1H, ArH), 7.32-7.40 (m, 3H, ArH), 7.67 (d, $J = 8.1$ Hz, 1H, ArH); ^{13}C NMR (CDCl_3) δ 27.40, 45.27, 55.34, 114.10, 123.56, 124.83, 125.35, 126.17, 127.76, 128.61, 129.70, 130.50, 134.00, 140.14, 159.39, 215.34; MS m/z (% relative intensity) 294 (26), 238 (18), 237 (100), 194 (12), 165 (21); IR (neat) 3028 w, 2960 w, 2835 w, 1682 s, 1604 m, 1512 s, 1468 m, 1392 w, 1362 w, 1298 w, 1254 s, 1178 m, 1109 w, 1026 m, 964 m, 945 m, 816 m, 748 m, 675 w, 536 w cm^{-1} ; Anal. Calcd for $\text{C}_{20}\text{H}_{22}\text{O}_2$: C, 81.60; H, 7.53%. Found: C, 81.31; H, 7.55%. HRMS Calcd for $\text{C}_{20}\text{H}_{22}\text{O}_2$: 294.1620. Found: 294.1628.

2,2-Dimethyl-1-{2-[2-(4-trifluoromethylphenyl)vinyl]phenyl}propan-1-one

M.p. 79-82 °C; ^1H NMR (CDCl_3) δ 1.19 (s, 9H, $\text{C}(\text{CH}_3)_3$), 6.99 (s, 2H, $\text{CH}=\text{CH}$), 7.11-7.37 (m, 3H, ArH), 7.46 (d, $J = 8.4$ Hz, 2H, ArH), 7.53 (d, $J = 8.6$ Hz, 2H, ArH), 7.65 (d, $J = 7.8$ Hz, 1H, ArH); ^{13}C NMR (CDCl_3) δ 27.26, 45.25, 124.10 (q, $J = 271.1$ Hz), 125.10, 125.66 (q, $J = 2.8$ Hz),

125.84, 126.65, 127.26, 128.26, 128.90, 129.53 (q, $J = 32.4$ Hz), 133.16, 140.37, 140.64, 215.11; IR (KBr) 2978 w, 1684 s, 1614 m, 1482 w, 1414 w, 1364 w, 1320 s, 1275 w, 1198 w, 1184 m, 1162 s, 1121 s, 1106 s, 1063 s, 1010 w, 959 s, 946 m, 873 w, 819 m, 761 m cm^{-1} ; MS m/z (% relative intensity) 332 (M^+ , 9), 276 (17), 275 (100), 227 (11), 178 (33), 57 (19). Anal.Calcd for $\text{C}_{20}\text{H}_{19}\text{F}_3\text{O}$: C, 72.28; H, 5.76%. Found: C, 71.99; H, 5.74%.

1-(2-Isopropenyl-4-methoxyphenyl)-2,2-dimethylpropan-1-one

$R_f = 0.39$ (hexane:EtOAc = 5:1). B.p. 170 °C/1.0 mmHg; ^1H NMR (CDCl_3) δ 1.19 (s, 9H, $\text{C}(\text{CH}_3)_3$), 2.10 (s, 3H, $=\text{CCH}_3$), 3.82 (s, 3H, OCH_3), 4.92 (brs, 1H, $=\text{CHH}$), 5.12 (brs, 1H, $=\text{CHH}$), 6.77-6.80 (m, 2H, ArH), 7.08 (d, $J = 6.2$ Hz, 1H, ArH). ^{13}C NMR (CDCl_3) δ 24.08, 27.99, 45.04, 55.30, 111.52, 113.04, 117.01, 127.21, 132.44, 142.09, 144.39, 159.28, 215.16; MS m/z (% relative intensity) 232 (M^+ , 4), 176 (13), 175 (100), 146 (21), 115 (17), 91 (19); IR (neat) 3322 w, 3132 w, 2952 m, 2674 w, 2540 w, 2230 w, 1918 m, 1724 m, 1677 s, 1597 s, 1564 s, 1476 s, 1462 s, 1389 m, 1358 m, 1279 s, 1216 s, 1178 s, 1080 s, 1038 s, 960 s, 936 s, 909 m, 818 m, 702 m, 644 m, 571 m, 322 w, 295 cm^{-1} ; Anal.Calcd for $\text{C}_{15}\text{H}_{20}\text{O}_2$: C, 77.55; H, 8.68%. Found: C, 77.43; H, 8.70%.

1-(4-Fluoro-2-isopropenylphenyl)-2,2-dimethylpropan-1-one

B.p. 185 °C/1.0 mmHg; ^1H NMR (CDCl_3) δ 1.18 (s, 9H, $\text{C}(\text{CH}_3)_3$), 2.10 (s, 3H, $=\text{CCH}_3$), 4.97 (brs, 1H, $=\text{CHH}$), 5.17 (brs, 1H, $=\text{CHH}$), 6.92-7.00 (m, 2H, ArH), 7.05-7.12 (m, 1H, ArH); ^{13}C NMR (CDCl_3) δ 23.77, 27.73, 45.09, 113.55 (d, $J = 21.8$ Hz), 114.28 (d, $J = 21.2$ Hz), 118.30, 127.61 (d, $J = 8.3$ Hz), 135.85 (d, $J = 3.4$ Hz), 142.50 (d, $J = 7.8$ Hz), 143.32 (d, $J = 2.2$ Hz), 162.30 (d, $J = 247.5$ Hz), 215.21; IR (neat) 3266 w, 2946 m, 1991 m, 1686 m, 1600 s, 1575 s, 1477 s, 1359 s, 1297 s, 1255 s, 1196 s, 1072 m, 1027 m, 966 s, 933 s, 911 s, 872 s, 822 s, 700 m, 569 m, 508 cm^{-1} ; MS m/z (% relative intensity) 220 (M^+ , 5), 164 (13), 163 (100), 135 (30), 133 (21), 115 (17), 109 (14), 57 (26); HRMS Calcd for $\text{C}_{14}\text{H}_{17}\text{FNaO}$: 243.11611. Found: 243.11552.

5,5-Dimethyl-2-propenyl[1,3,2]dioxaborinane (6) (*E* and *Z* isomers in a ratio of 39:61).

B.p. 60 °C/1.0 mmHg; *cis*-isomer: ^1H NMR (CDCl_3) δ 0.98 (s, 6H, $\text{C}(\text{CH}_3)_2$), 1.94 (d, $J = 7.0$ Hz, 3H, $=\text{CCH}_3$), 3.65 (s, 4H, OCH_2), 5.26 (d, $J = 14.0$ Hz, 1H, $=\text{CHH}$), 6.41 (brs, 1H, $=\text{CHH}$); ^{13}C NMR (CDCl_3) δ 18.02, 21.93, 31.79, 71.86, 147.01; *trans*-isomer: ^1H NMR (CDCl_3) δ 0.96 (s, 6H), 1.82 (d, $J = 6.8$ Hz, 3H), 3.62 (s, 4H), 5.38 (d, $J = 17.3$ Hz, 1H), 6.51-6.61 (brs, 1H); ^{13}C NMR (CDCl_3) 21.46, 21.89, 31.65, 71.98, 146.52; *cis,trans*-mixture: IR (neat) 2989 w, 2962 s, 2933 s, 2883 s, 1631 s, 1479 s, 1441 s, 1412 s, 1371 m, 1315 w, 1284 s, 1261 s, 1205 s, 1084 w, 1009 w, 748 s cm^{-1} ; MS m/z (% relative intensity) 154 (M^+ , 8), 97 (35), 96 (28), 95 (11), 69 (33), 68 (15), 56 (100), 55 (14), 54 (13); Anal. Calcd for $\text{C}_8\text{H}_{15}\text{BO}_2$: C, 62.39; H, 9.82%. Found: C, 62.26; H, 9.88%.

***Z*-5,5-Dimethyl-2-(1-methylpropenyl)[1,3,2]dioxaborinane**

B.p. 85 °C/1.0 mmHg; *cis*-isomer: ^1H NMR (CDCl_3) δ 0.99 (s, 6H, $\text{C}(\text{CH}_3)_2$), 1.72 (s, 3H, $=\text{CCH}_3$), 1.83-1.86 (m, 3H, $=\text{C}(\text{CH}_3)_3$), 3.67 (s, 4H, OCH_2), 6.06 (brs, 1H, $=\text{CH}$); ^{13}C NMR (CDCl_3) δ 16.82, 21.79, 22.46, 31.47, 71.92, 138.92.; MS m/z (% relative intensity) 168 (M^+ , 36), 167 (10), 111 (68), 110 (40), 109 (16), 95 (31), 83 (15), 82 (29), 81 (13), 71 (11), 70 (23), 69 (69), 68 (47), 67 (91), 57 (16), 56 (100), 55 (52), 54 (24), 53 (22); IR (neat) 3023 w, 2960 m, 2935 m, 2889 m, 1637 w, 1479 m, 1414 m, 1339 m, 1269 s, 1213 w, 1161 m, 1086 w, 1036 w, 993 w, 928 w, 837 w, 746 w, 708 w, 685 w, 607 w, 567 w, 418 w cm^{-1} ; Anal. Calcd for $\text{C}_9\text{H}_{17}\text{BO}_2$: C, 64.33; H, 10.20%. Found: C, 64.35; H, 10.20%; HRMS Calcd for $\text{C}_9\text{H}_{17}\text{BO}_2$: 168.1322. Found: 168.1310.

***trans*-5,5-Dimethyl-2-(2-p-tolylvinyl)[1,3,2]dioxaborinane**

M.p. 118-120 °C; ^1H NMR (CDCl_3) δ 1.01 (s, 6H, $\text{C}(\text{CH}_3)_2$), 2.34 (s, 3H, CH_3Ar), 3.70 (s, 4H, OCH_2), 6.05 (d, $J = 18.1$ Hz, 1H, $\text{CH}=\text{$), 7.13 (d, $J = 7.8$ Hz, 2H, ArH), 7.30 (d, $J = 18.1$ Hz, 1H, $=\text{CH}$), 7.38 (d, $J = 8.1$ Hz, 2H, ArH); ^{13}C NMR (CDCl_3) δ 21.29, 21.87, 31.84, 72.17, 126.93, 129.22, 135.05, 138.52, 147.05; IR (KBr) 3014 w, 2958 m, 1630 s, 1570 w, 1512 m, 1475 m, 1417 s, 1381 m, 1311 s, 1275 s, 1225 m, 1184 s, 1082 m, 1001 s, 802 s, 671 w, 633w, 492 s cm^{-1} ; MS m/z (% relative intensity) 231 (17), 230 (M^+ , 100), 229 (28), 145 (16), 144 (98), 143 (44), 118 (10), 117 (30), 116 (43), 115 (41), 91 (24), 65 (15), 56 (24), 55 (24), 53 (12); Anal. Calcd for $\text{C}_{14}\text{H}_{19}\text{BO}_2$: C, 73.07; H, 8.32%. Found: C, 72.81; H, 8.17%.

2-[2-(4-Methoxyphenyl)vinyl]-5,5-dimethyl[1,3,2]dioxaborinane

M.p. 76-80 °C; ^1H NMR (CDCl_3) δ 1.00 (s, 6H, $\text{C}(\text{CH}_3)_3$), 3.69 (s, 4H, OCH_2), 3.81 (s, 3H, OCH_3), 5.95 (d, $J = 18.1$ Hz, 1H, $\text{CH}=\text{$), 6.86 (d, $J = 7.6$ Hz, 2H, ArH), 7.29 (d, $J = 16.7$ Hz, 1H, $=\text{CH}$), 7.43 (d, $J = 7.6$ Hz, 2H, ArH); ^{13}C NMR (CDCl_3) δ 21.86, 31.84, 55.25, 72.15, 113.88, 128.32, 130.64, 146.62, 159.97; IR (neat) 3070 w, 3028 w, 2999 m, 2962 s, 2937 m, 2895 m, 2841 m, 1618 s, 1572 m, 1512 s, 1479 s, 1415 s, 1377 m, 1338 s, 1281 s, 1255 s, 1180 s, 1111 m, 1082 s, 1032 m, 1001 s, 814 s, 742 m, 719 w, 669 w, 633 m, 565 w, 509 m, 451 w, 418 w cm^{-1} ; MS m/z (% relative intensity) 247 (16), 246 (M^+ , 100), 245 (27), 160 (60), 159 (19), 145 (12), 117 (19), 65 (11), 55 (12). Anal. Calcd for $\text{C}_{14}\text{H}_{19}\text{BO}_3$: C, 68.32; H, 7.78%. Found: C, 68.29; H, 7.51%.

trans-5,5-Dimethyl-2-[2-(4-trifluoromethylphenyl)vinyl][1,3,2]dioxaborinane

M.p. 122-124 °C; ^1H NMR (CDCl_3) δ 1.01 (s, 6H, $\text{C}(\text{CH}_3)_2$), 3.71 (s, 4H, CH_2), 6.20 (d, $J = 18.4$ Hz, 1H, $=\text{CH}$), 7.33 (d, $J = 18.4$ Hz, 1H, $\text{CH}=\text{$), 7.57 (brs, 4H, ArH); ^{13}C NMR (CDCl_3) δ 21.83, 31.85, 72.23, 124.14 (q, $J = 271.1$ Hz), 125.48 (q, $J = 3.9$ Hz), 127.06, 130.09 (q, $J = 31.9$ Hz), 141.10, 145.34; IR (KBr) 3055 w, 3016 w, 2964 m, 2904 w, 2879 w, 2393 w, 1934 w, 1810 w, 1631 m, 1576 w, 1479 m, 1417 s, 1377 m,

1319 s, 1281 s, 1259 s, 1171 s, 1111 s, 1065 s, 1011 m, 991 m, 866 w, 818 m, 768 w, 737 w, 677 w, 638 w, 588 w, 499 w, 457 w cm^{-1} ; MS m/z (% relative intensity) 284 (M^+ , 14), 56 (100); Anal. Calcd for $\text{C}_{14}\text{H}_{16}\text{BF}_3\text{O}_2$: C, 59.19; H, 5.68%. Found: C, 59.02; H, 5.58%.

5,5-Dimethyl-2-(1-phenylvinyl)[1,3,2]dioxaborinane

B.p. 130 °C/1.0 mmHg; ^1H NMR (CDCl_3) δ 1.01 (s, 6H, CH_3), 3.71 (s, 4H, CH_2), 5.91 (s, 1H, $=\text{CHH}$), 6.01 (s, 1H, $=\text{CHH}$), 7.24-7.41 (m, 5H, ArH); ^{13}C NMR (CDCl_3) δ 21.94, 31.69, 72.33, 126.53, 127.43, 127.86, 129.35, 142.37; IR (neat) 3074 w, 3057 w, 3024 w, 2962 m, 2935 m, 2823 w, 2345 w, 1597 w, 1481 s, 1415 s, 1342 s, 1284 m, 1252 s, 1230 s, 1194 s, 1105 m, 1074 w, 1030 w, 1001 w, 943 w, 916 w, 816 w, 781 m, 733 m, 704 m, 644 w, 613 w, 542 w, 496 w, 455 w cm^{-1} ; MS m/z (% relative intensity) 217 (13), 216 (M^+ , 88), 215 (24), 173 (11), 131 (28), 130 (100), 129 (42), 105 (20), 104 (13), 103 (15), 102 (22), 78 (13), 77 (26), 56 (73), 55 (18), 51 (14). Anal. Calcd for $\text{C}_{13}\text{H}_{17}\text{BO}_2$: C, 72.26; H, 7.93%. Found: C, 71.97; H, 7.96%.

References

- (1) Kakiuchi, F.; Sekine, S.; Tanaka, Y.; Kamatani, A.; Sonoda, M.; Chatani, N.; Murai, S. *Bull. Chem. Soc. Jpn.* **1995**, 68, 62.

H:\B3-1NMR\fmf\fg1\su-1-28\su-

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 BF 26
 RGAIN

1H-NMR..CONDITION

DELS
COMET
DATM
OBNOC
EXMOD
OBFRO
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
FWI
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

G:*lfzf*f\NMR\1-methyl-
1H-NMR..CONDITION

SGNON
399.65 MHz
0.00 KHz
10300.00 Hz
16384
6002.40 Hz
16
2.7300 sec
0.0000 sec
6.50 usec
27.0 c
CDCL3SMEXR
1.92 ppm
0.12 Hz
12

DFILE G:\la-i\DV,\mu,8ftHf,f_1-met
 COMNT Thu Nov 16 15:44:28 2006
 DATIM 13C
 CBNUC 67.80 MHz
 EXMOD BCM
 CBFRQ 135.00 KHz
 CBSET 5200.00 Hz
 CBFIN 32768
 POINT 18315.00 Hz
 FREQU 1178
 SCANS 1.7891 sec
 ACQTM 1.2110 sec
 PD 3.90 usec
 PW1 1H
 IRNUC 21.1 c
 CTMP CDCL3
 SLVNT 77.00 ppm
 EXREF 1.20 Hz
 BF 26
 RGAIN

su-1-1-9(after HPLC 1H-NMR)

DFILE
COMMT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

270.05 MHz
112.00 KHz
5800.00 Hz
16384
5402.40 Hz
8
3.0327 sec
3.9670 sec
5.30 usec
1H
22.6 c
CDCL3
0.00 ppm
1.20 Hz
14

G:\ja-1nmr\fm\fgl\su-1-01\s
su-1-1-9(after HPLC 1H-NMR)
Wed May 22 11:33:10 2002

su-1-1-9(after HPLC 13c-NMR)

DFILE H:\05-1NMR\fmf\su-1-01\su-
 COMMENT su-1-1-9(after HPLC 13c-NMR)
 DATIM Wed May 22 11:54:00 2002
 OBNUC 13C
 EXMOD BCM

OBFRQ 67.80 MHz
 OBSET 135.00 KHz
 OBFIN 5200.00 Hz
 POINT 32768
 FREQU 18315.00 Hz
 SCANS 136
 ACQTM 1.7891 sec
 PD 1.2110 sec
 PW1 3.90 usec
 INUC 1H
 CTEMP 23.4 C
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.20 Hz
 RGAIN 26

G:\hs-1\fm\lfgl\su-1-45\Auto

DFILE
CONMT
DATIM
OBNDC
EXMOD
OBFRO
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

Fri Nov 08 09:27:43 2002

1H

NON

270.05 MHz

112.00 KHz

5800.00 Hz

16384

5405.40 Hz

8

3.0310 sec

3.9670 sec

5.30 usec

1H

19.3 c

CDCL3

1.56 ppm

1.20 Hz

18

7.688
7.660
7.384
7.347
7.317
7.262
7.243
7.236
7.208
7.158
7.130
7.105
7.026
6.966
6.912
6.853

2.340
1.560
1.225

9.00

0.96
2.54
0.93
2.53
1.77

2.82

ppm

DFILE G:\Ma-1\fmf\fgl\su-1-45\su-1
 COMNT
 DATIM Fri Nov 08 07:13:23 2002
 GENUC 13C
 EXMOD BCM
 OBFRQ 67.80 MHz
 OBSET 135.00 KHz
 OBFIN 5200.00 Hz
 POINT 32768
 FREQU 18348.60 Hz
 SCANS 8000
 ACQTM 1.7859 sec
 PD 1.2110 sec
 FW1 3.90 usec
 IRNUC 1H
 CTEMP 18.9 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.20 Hz
 RGAIN 27

D:\08-1\2-(4-methoxyphenyl)-2
 COMNT Thu Dec 07 14:05:41 2006
 DATIM 13C
 OBNUC 13C
 EXMOD BCM
 OBFRQ 67.80 MHz
 OBSET 135.00 KHz
 OBFIN 5200.00 Hz
 POINT 32768
 FREQU 18315.00 Hz
 SCANS 255
 ACQTM 1.7891 sec
 PD 1.2110 sec
 PW1 3.90 usec
 IRNUC 1H
 CTEMP 23.7 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.20 Hz
 RGAIN 27

H:\05-1NMR\fmf\fgl\au-1-47\Aut

Sat Nov 09 01:02:16 2002

DFILE
COMNT
DATIM
OBNUC 1H
EXMOD NCH
OBFRQ 270.05 MHz
OBSET 112.00 KHz
OBFIN 5600.00 Hz
POINT 16384
FREQU 5405.40 Hz
SCANS 8
AQTM 3.0310 sec
PD 3.9670 sec
PW1 5.30 usec
IRNUC 1H
CTEMP 19.5 C
SLVNT CDCL3
EXREF 1.19 ppm
BF 0.12 Hz
RGAIN 12

DFILE
COMNT
DATIM
OBNUC 1H
EXMOD NCH
OBFRQ 270.05 MHz
OBSET 112.00 KHz
OBFIN 5600.00 Hz
POINT 16384
FREQU 5405.40 Hz
SCANS 8
AQTM 3.0310 sec
PD 3.9670 sec
PW1 5.30 usec
IRNUC 1H
CTEMP 19.5 C
SLVNT CDCL3
EXREF 1.19 ppm
BF 0.12 Hz
RGAIN 12

DFILE G:\ba-i\fm\l\su-1-47\Auto
 COMNT Sat Nov 09 07:42:19 2002
 DATIM 13C
 ORNUC 13C
 EXMOD BCM
 OBFRQ 67.80 MHz
 OBSET 135.00 KHz
 OBFIN 5200.00 Hz
 POINT 32768
 FREQU 18348.60 Hz
 SCANS 8000
 ACQTM 1.7859 sec
 PD 1.2110 sec
 PW1 3.90 usec
 IRNUC 1H
 CTENP 19.0 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.20 Hz
 RGAIN 27

DFILE G:\Bs-1\ALKENYLATION\2'--(2-f
 COMNT
 DATIN Fri Feb 24 14:23:10 2006
 OBNUC 1H
 EXMOD NON
 OBFRQ 270.05 MHz
 OBSET 112.00 MHz
 OBFIN 5800.00 Hz
 POINT 16384
 FREQU 5402.40 Hz
 SCANS 8
 ACQTM 3.0327 sec
 PD 3.9670 sec
 FW1 5.30 usec
 IRNUC 1H
 CTENP 19.8 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 1.20 Hz
 RGAIN 14

DFILE G:\B-1\ALKENYLATION\2'--(2-E
 COMMT
 DATIM Fri Feb 24 14:28:46 2006
 OBNDC 13C
 EXMOD BCM
 OBFRQ 67.80 MHz
 OBSET 135.00 KHz
 OBFIN 5200.00 Hz
 POINT 32768
 FREQU 18315.00 Hz
 SCANS 100
 ACQTM 1.7891 sec
 PD 1.2110 sec
 PW1 3.90 usec
 IRNUC 1H
 CTENP 20.1 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.20 Hz
 RGAIN 26

su-3-156

DFILE G:\ba-1nmr\mf\fg,R\su-3-156
 su-3-156
 DATE Tue Jul 01 01:14:43 2003
 OBNUC 1H
 EXMOD NON
 OBFRQ 270.05 MHz
 OBSET 112.00 KHz
 OBFIN 5800.00 Hz
 POINT 16384
 FREQU 5405.40 Hz
 SCANS 8
 ACQTM 3.0310 sec
 PD 3.9670 sec
 FWH 5.30 usec
 IRNUC 1H
 CTEMP 20.7 c
 SLVNT CDCl3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 19

su-3-156

215.214

164.125
160.474
143.304
142.554
142.439
135.878
135.828
127.676
127.553
118.304
114.438
114.125
113.713
113.392

77.470
77.198
77.000
76.522
45.092
27.725
23.769

DFILE G:\ls-1NMR\ALKENYLATION\2-pz
COMNT su-3-156
DATIM Tue Jul 01 04:34:58 2003
OBNUC 13C
EXMOD BCM
OBFRQ 67.80 MHz
OBSET 135.00 KHz
OBFIN 5200.00 Hz
POINT 32768
FREQ 18348.60 Hz
SCANS 4000
ACQTM 1.7859 sec
PD 1.2110 sec
PW1 3.90 usec
IRNUC 1H
CTEMP 21.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 27

PPM
220.210.200.190.180.170.160.150.140.130.120.110.100.90.080.070.060.050.040.030.020.010.00.0-10.0

G:\ba-1\frfjf\B,tfxfyNfgf\<\\
 DFILE COMNT
 DATIM Fri Feb 24 22:00:27 2006
 OBNUC 1H
 EXMOD NOM
 OBFRQ 270.05 MHz
 OBSET 112.00 KHz
 OBFIN 5800.00 Hz
 POINT 16384
 FREQU 5402.40 Hz
 SCANS 8
 ACQTM 3.0327 sec
 PD 3.9670 sec
 PW1 5.30 usec
 IRNUC 1H
 CTEMP 20.0 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 1.20 Hz
 RGAIN 12

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBERQ
OBSET
OBEIN
POINT
FFREQ
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SSLVNT
EXREF
BF
RGAIN

$E/Z = 39/61$

1H-NMR.. CONDITION

DFILE
CONMT
DATIM
OBNUC
EXMOD
OBFRQ
OBSET
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

C:\Documents and Settings\A
1H-NMR.. CONDITION

SGNON 399.65 MHz
0.00 KHz
10300.00 Hz
16384
6002.40 Hz
16
2.7300 sec
0.0000 sec
6.50 usec
27.0 c
CDCL3SMEXR
0.00 ppm
0.12 Hz
12

13C-NMR...CONDITION

G:*Hifzf*[NMR\1-methyl
13C-NMR...CONDITION

DFILE
COMET
DATIM
OENUC
EXMOD
OBFRQ
OBSET
OBFIN
POINT
FREQU
SCANS
ACQTM
PD
PWL
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

SGBCM
100.40 MHz
0.00 KHz
10244.00 Hz
32768
25000.00 Hz
50
0.0000 sec
0.0000 sec
5.60 usec
27.0 C
CDCL3SMEXR
77.00 ppm
0.12 Hz
27

su-1-?p-tolylvinyl B

DFILE G:\ba-i\frfjfb,ifxfyfnfgf\<
 CONNT su-1-?p-tolylvinyl B
 DATIM Sun Oct 19 21:45:16 2003
 OBNUC 1H
 EXMOD NON
 OBFRQ 270.05 MHz
 OBSET 112.00 KHz
 OBFIN 5800.00 Hz
 POINT 16384
 FREQU 5405.40 Hz
 SCANS 8
 ACQTM 3.0310 sec
 PD 3.9670 sec
 FM1 5.30 usec
 IRNUC 1H
 CTMP 23.4 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 0.12 Hz
 RGAIN 18

su-1-p-tolylynyl B

DFILE G:\ba-1\frfjf\B,ifXfyfNfgf\<
 CONNT su-1-p-tolylynyl B
 DATIM Mon Oct 20 01:55:30 2003
 OBNUC 13C
 EXMOD BCN
 OBFRQ 67.80 MHz
 OBSET 135.00 KHz
 OBFIN 5200.00 Hz
 POINT 32768
 FREQU 18348.60 Hz
 SCANS 5000
 ACQTM 1.7859 sec
 PD 1.2110 sec
 PW1 3.90 usec
 IRNUC 1H
 CTEMP 23.0 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 27

p-meo-styryl-b

DEFILE G:\ba-1\frj3f.B,lfXfyfNfgf<
 COMNT p-meo-styryl-b
 DATIM Sun Mar 14 02:03:20 2004
 OBNUC 1H
 EXMOD NON
 OBSFQ 270.05 MHz
 OBSFQ 112.00 KHz
 OBSFQ 5800.00 Hz
 POINT 16384
 FREQU 5405.40 Hz
 SCANS 8
 ACQTM 3.0310 sec
 PD 3.9670 sec
 PW1 5.30 usec
 IRNUC 1H
 CTEMP 20.5 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 1.20 Hz
 RGAIN 16

p-meo-styryl-b

DFILE
 COMNT
 DATIM
 OBNUC
 EXMOD
 OBFREQ
 OBSSET
 OBSFIN
 POINT
 FREQU
 SCANS
 ACQTM
 PD
 PW1
 IRNUC
 CTMP
 SLVNT
 EXREF
 BF
 RGAIN

H:\ba-1nmr\frj\fcB,ifXfyNfgf<
 p-meo-styryl-b
 Sun Mar 14 06:13:35 2004
 13C
 BCM

67.80 MHz
 135.00 KHz
 5200.00 Hz
 32768
 18348.60 Hz
 5000
 1.7859 sec
 1.2110 sec
 3.90 usec
 1H
 20.5 C
 CDCL3
 77.00 ppm
 0.12 Hz
 27

p-tr9chloromethylphenylvinyl

G:\03-1\frjfb,ifsfyNsf\p-tr9chloromethylphenylvinyl
Thu Nov 13 00:29:44 2003

DFILE
COMNT
DATIM
OBNUC 1H
EXMOD NON
OBFRQ 270.05 MHz
OBSET 112.00 KHz
OBFIN 5800.00 Hz
POINT 16384
FREOU 5405.40 Hz
SCANS 8
ACQTM 3.0310 sec
PD 3.9670 sec
FW1 5.30 usec
IRNUC 1H
CTENP 22.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 19

p-tr9chloromethylphenylvinyl

DFILE H:\B3-1NMR\frfjfb,ifxfyNfg
 COMNT p-tr9chloromethylphenylvinyl
 DATIM Thu Nov 13 03:50:00 2003
 OBNUC 13C
 EXMOD BCM
 OBFRQ 67.80 MHz
 OBSET 135.00 KHz
 OBFIN 5200.00 Hz
 POINT 32768
 FREQU 18348.60 Hz
 SCANS 4000
 ACQTM 1.7859 sec
 PD 1.2110 sec
 PW1 3.90 usec
 IRNUC 1H
 CTEMP 22.1 C
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 27

DFILE G:\Ma-i\frfjf.B,ifXjyNfgj<
 COMNT
 DATIM Fri Feb 24 20:53:40 2006
 OBNUC 1H
 EXMOD NON
 OBFRQ 270.05 MHz
 OBSET 112.00 KHz
 OBFIN 5800.00 Hz
 POINT 16384
 FREQU 5402.40 Hz
 SCANS 8
 ACQTM 3.0327 sec
 PD 3.9670 sec
 PW1 5.30 usec
 IRNUC 1H
 CTENP 19.3 C
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 1.20 Hz
 RGAIN 17

G:\la-1\frfjf.B,ifKfyfNf9f<
 COMNT
 DATIM Thu May 29 18:29:19 2003
 OBNUC 13C
 EXMOD BCM
 OBSFQ 67.80 MHz
 OBSFQ 135.00 KHz
 OBSFQ 5200.00 Hz
 POINT 32768
 FREQU 18315.00 Hz
 SCANS 543
 ACQTM 1.7891 sec
 PD 1.2110 sec
 PW1 3.90 usec
 IIRNUC 1H
 CTMP 29.7 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 26