Pd-Catalyzed Regio- and Stereoselective Cyclization-Heck Reaction of Monoesters of 1, 2-Allenyl Phosphonic Acids with Alkenes

Fei Yu, Xiongdong Lian and Shengming Ma*

State Key Laboratory of Organometallic Chemistry, Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences, 354 Fenglin Lu, Shanghai 200032, P. R. China

Fax: (+86)21-64167510

E-mail: masm@mail.sioc.ac.cn

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1. 3-Butyl-2-ethoxy-4-chloro-5, 5-dimethyl-5H-[1, 2]-oxaphosphole 2-oxide (3a) and 3-Butyl-2-ethoxy-4-(*E*-2-cyanovinyl)-5,5-dimethyl-5H-[1, 2]-oxaphosphole 2-oxide (*E*-2a);

The reaction of monoethyl ester of (2-methylocta-2, 3-dien-4-yl) phosphonic acid **1a** (69 mg, 0.30 mmol), acrylonitrile (0.10 mL, 80 mg, 1.5 mmol), PdCl₂(PhCN)₂ (6 mg, 5 mol %), CuCl₂ (162 mg, 1.2 mmol), and K₂CO₃ (166 mg, 1.2 mmol) was stirred at 60 °C in 2 mL of DMF in air for 24 hours. Then the reaction mixture was diluted with 50 mL of ether, washed with brine (three times), and dried over Na₂SO₄. After evaporation, the residue was purification by column chromatography on silica gel to afforded 27 mg (34%) of **3a** and 12 mg (14%) of *E*-**2a**.

3a: liquid; ¹H NMR (300 M Hz, CDCl₃) δ 4.11 (dq, *J* = 6.9Hz, 9.3 Hz, 2 H), 2.42-2.18 (m, 2 H), 1.60-1.45 (m, 2 H), 1.51 (s, 3 H), 1.47 (s, 3 H), 1.38-1.24 (m, 2 H), 1.30 (t, *J* = 6.9 Hz, 3 H), 0.88 (t, *J* = 7.5 Hz, 3 H); ¹³C NMR (CDCl₃, 75.4 MHz): δ 150.6 (d, *J*_{PC} = 50.6 Hz), 124.7 (d, *J*_{PC} = 157.7 Hz), 85.4 (d, *J*_{PC} = 2.9 Hz), 63.1 (d, *J*_{PC} = 6.3 Hz), 29.4 (d, *J*_{PC} = 1.7 Hz), 26.8 (d, *J*_{PC} = 2.9 Hz), 26.2 (d, *J*_{PC} = 2.3 Hz), 25.4 (d, *J*_{PC} = 9.2 Hz), 22.4, 16.5 (d, *J*_{PC} = 5.7 Hz), 13.6; ³¹P NMR (121.5 MHz, CDCl₃) δ 32.7; IR (neat) 1630, 1464, 1368, 1272, 1151, 1042 cm⁻¹; MS (EI): *m/z* 269 (M⁺(³⁷Cl)+1, 28.14), 267 (M⁺(³⁵Cl)+1, 81.43), 240 (M⁺(³⁷Cl)-C₂H₄, 4.91), 238 (M⁺(³⁵Cl)-C₂H₄, 12.79), 226 (M⁺(³⁷Cl)-C₃H₆, 19.15), 224 (M⁺(³⁵Cl)-C₃H₆, 49.73), 43 (100); HRMS (EI): *m/z* calcd for C₁₁H₂₀O₃ClP [M⁺]: 266.08386; Found: 266.08439.

E-2a: liquid; ^1H NMR (300 MHz, CDCl_3) δ 6.99 (dd, $J = 17.1, 1.8$ Hz, 1 H), 5.58 (dd, $J = 17.1, 1.8$ Hz, 1 H), 4.18-4.01 (m, 2 H), 2.46-2.18 (m, 2 H), 1.57-1.42 (m, 8 H), 1.37-1.22 (m, 5 H), 0.85 (t, $J = 7.2$ Hz, 3 H); ^{13}C NMR (CDCl_3 , 75.4 MHz): δ 151.2 (d, $J_{\text{PC}} = 26.3$ Hz), 140.1 (d, $J_{\text{PC}} = 24.1$ Hz), 134.0 (d, $J_{\text{PC}} = 156.3$ Hz), 116.7 (d, $J_{\text{PC}} = 2.4$ Hz), 103.9 (d, $J_{\text{PC}} = 1.7$ Hz), 84.4 (d, $J_{\text{PC}} = 7.5$ Hz), 62.2 (d, $J_{\text{PC}} = 6.9$ Hz), 30.8 (d, $J_{\text{PC}} = 2.3$ Hz), 28.1 (d, $J_{\text{PC}} = 3.5$ Hz), 27.6 (d, $J_{\text{PC}} = 1.4$ Hz), 25.9 (d, $J_{\text{PC}} = 10.8$ Hz), 22.7, 16.6 (d, $J_{\text{PC}} = 5.8$ Hz), 13.7; ^{31}P NMR (121.5 MHz, CDCl_3) δ 35.5; IR (neat) 2221, 1618, 1460, 1391, 1373, 1262, 1149, 1044 cm^{-1} ; MS (EI): m/z 284 ($\text{M}^+ + \text{H}$, 12.65), 283 (M^+ , 8.98), 268 ($\text{M}^+ - \text{CH}_3$, 14.20), 43 (100); HRMS (MALDI): m/z calcd for $\text{C}_{14}\text{H}_{23}\text{NO}_3\text{P}^+ [\text{M}^+ + \text{H}]$: 284.1410; Found: 284.1417.

2. Reactions under Conditions A

(1) Synthesis of 3-Butyl-2-ethoxy-4-(2-cyanovinyl)-5, 5-dimethyl-5H-[1, 2]-oxaphosphole 2-oxides (*E*-2a and *Z*-2a);

Typical Procedure I (conditions A): A mixture of monoethyl ester of (2-methylocta-2, 3-dien-4-yl)phosphonic acid **1a** (139 mg, 0.60 mmol), acrylonitrile (0.20 mL, 161 mg, 3.0 mmol), NaI (18 mg, 0.12 mmol), CaH_2 (1 mg, 5 mol%), and $\text{PdCl}_2(\text{PhCN})_2$ (12 mg, 5 mol%) was stirred at 60 $^\circ\text{C}$ in 4 mL of DMF in air with a CaCl_2 drying tube for 24 hours. Then the reaction mixture was diluted with 50 mL of ether, washed with brine (three times), and dried over Na_2SO_4 . After evaporation, the

residue was analyzed by ^1H NMR study indicating 70% yield of **E-2a** and 4% yield of **Z-2a**. After purification by column chromatography on silica gel (petroleum ether / ethyl acetate = 3/2), 128 mg (75%) of **E-2a** was obtained. **Z-2a** was prepared by repeating this experiment for a couple of times.

Z-2a: liquid; ^1H NMR (300 MHz, CDCl_3) δ 6.76 (d, $J = 11.7$ Hz, 1 H), 5.77 (d, $J = 11.7$ Hz, 1 H), 4.20-4.08 (m, 2 H), 2.42-2.25 (m, 2 H), 1.74-1.58 (m, 2 H), 1.54 (s, 3 H), 1.46 (s, 3 H), 1.39-1.29 (m, 5 H), 0.92 (t, $J = 7.2$ Hz, 3 H); ^{13}C NMR (CDCl_3 , 75.4 MHz): δ 152.8 (d, $J_{\text{PC}} = 26.8$ Hz), 142.2 (d, $J_{\text{PC}} = 24.6$ Hz), 129.8 (d, $J_{\text{PC}} = 153.1$ Hz), 115.0, 105.7 (d, $J_{\text{PC}} = 2.6$ Hz), 84.5 (d, $J_{\text{PC}} = 8.1$ Hz), 63.1 (d, $J_{\text{PC}} = 6.8$ Hz), 29.5 (d, $J_{\text{PC}} = 2.1$ Hz), 27.7 (d, $J_{\text{PC}} = 2.9$ Hz), 26.8 (d, $J_{\text{PC}} = 2.3$ Hz), 26.7 (d, $J_{\text{PC}} = 11.5$ Hz), 22.8, 16.5 (d, $J_{\text{PC}} = 5.7$ Hz), 13.7; ^{31}P NMR (121.5 MHz, CDCl_3) δ 36.4; IR (neat) 2221, 1617, 1462, 1392, 1373, 1261, 1149, 1042 cm^{-1} ; MS (EI): m/z 284 ($\text{M}^+ + 1$, 32.47), 283 (M^+ , 8.61), 43 (100); HRMS (EI): m/z calcd for $\text{C}_{14}\text{H}_{22}\text{NO}_3\text{P}[\text{M}^+]$: 283.1337; Found: 283.1339.

(2) 3-Butyl-2-ethoxy-4-(*E*-2-methoxycarbonylvinyl)-5, 5-dimethyl-5*H*-[1, 2]-oxaphosphole 2-oxide (**E-2b**);

The reaction of monoethyl ester of (2-methylocta-2,3-dien-4-yl)phosphonic acid **1a** (69 mg, 0.30 mmol), acrylic acid methyl ester (128 mg, 1.52 mmol), CaH_2 (0.6 mg, 5 mol%), $\text{PdCl}_2(\text{PhCN})_2$ (6 mg, 5 mol %), and NaI (9 mg, 0.06 mmol) afforded 82 mg (88%) of **E-2b**: liquid; ^1H NMR (300 MHz, CDCl_3) δ 7.28 (dd, $J = 16.5, 1.2$ Hz, 1 H),

6.06 (dd, $J = 16.5, 1.2$ Hz, 1 H), 4.18-4.02 (m, 2 H), 3.77 (s, 3 H), 2.51-2.27 (m, 2 H), 1.60-1.47 (m, 8 H), 1.40-1.25 (m, 5 H), 0.88 (t, $J = 7.2$ Hz, 3 H); ^{13}C NMR (CDCl_3 , 75.4 MHz): δ 166.0, 152.5 (d, $J_{\text{PC}} = 25.3$ Hz), 133.6 (d, $J_{\text{PC}} = 23.0$ Hz), 132.0 (d, $J_{\text{PC}} = 156.0$ Hz), 124.6, 84.8 (d, $J_{\text{PC}} = 8.1$ Hz), 62.7 (d, $J_{\text{PC}} = 6.3$ Hz), 52.0, 30.6, 28.0, 27.4, 25.6 (d, $J_{\text{PC}} = 10.9$ Hz), 22.5, 16.4 (d, $J_{\text{PC}} = 5.7$ Hz), 13.6; ^{31}P NMR (121.5 MHz, CDCl_3) δ 36.6; IR (neat) 1728, 1631, 1585, 1461, 1437, 1390, 1370, 1298, 1197, 1043 cm^{-1} ; MS (EI): m/z 316 (M^+ , 4.62), 301 ($\text{M}^+ - \text{CH}_3$, 4.28), 285 ($\text{M}^+ - \text{CH}_3\text{O}$, 4.34), 257 ($\text{M}^+ - \text{CH}_3\text{COO}$, 50.86), 43 (100); HRMS (EI): m/z calcd for $\text{C}_{15}\text{H}_{25}\text{O}_5\text{P}[\text{M}^+]$: 316.1440; Found: 316.1448.

(3) 3-Butyl-2-ethoxy-4-(*E*-2-ethoxycarbonylvinyl)-5, 5-dimethyl-5*H*-[1, 2]-oxa-phosphole 2-oxide (*E*-2c)

The reaction of monoethyl ester of (2-methylocta-2,3-dien-4-yl)phosphonic acid **1a** (70 mg, 0.30 mmol), acrylic acid ethyl ester (149 mg, 1.52 mmol), CaH_2 (0.6 mg, 5 mol%), $\text{PdCl}_2(\text{PhCN})_2$ (6 mg, 5 mol %), and NaI (9 mg, 0.06 mmol) afforded 87 mg (84%) of **E-2c**: liquid; ^1H NMR (300 MHz, CDCl_3) δ 7.28 (dd, $J = 16.5, 1.5$ Hz, 1 H), 6.05 (dd, $J = 16.5, 0.9$ Hz, 1 H), 4.22 (q, $J = 7.2$ Hz, 2 H), 4.14-4.06 (m, 2 H), 2.51-2.27 (m, 2 H), 1.60-1.43 (m, 8 H), 1.39-1.24 (m, 8 H), 0.89 (t, $J = 7.5$ Hz, 3 H); ^{13}C NMR (CDCl_3 , 75.4 MHz): δ 165.6, 152.6 (d, $J_{\text{PC}} = 25.9$ Hz), 133.3 (d, $J_{\text{PC}} = 23.1$ Hz), 131.0 (d, $J_{\text{PC}} = 156.5$ Hz), 125.0, 84.9, 62.7, 61.0, 30.6, 28.1, 27.5, 25.5 (d, $J_{\text{PC}} = 10.4$ Hz), 22.5, 16.4 (d, $J_{\text{PC}} = 5.8$ Hz), 14.0, 13.6; ^{31}P NMR (121.5 MHz, CDCl_3) δ

36.6; IR (neat) 1720, 1631, 1465, 1369, 1265, 1183, 1041 cm^{-1} ; MS (EI): m/z 330 (M^+ , 4.20), 315 ($\text{M}^+ - \text{CH}_3$, 5.81), 285 ($\text{M}^+ - \text{C}_2\text{H}_5\text{O}$, 11.45), 257 ($\text{M}^+ - \text{C}_2\text{H}_5\text{COO}$, 91.83), 43 (100); HRMS (EI): m/z calcd for $\text{C}_{16}\text{H}_{27}\text{O}_5\text{P}[\text{M}^+]$: 330.1596; Found: 330.1600.

(4) 3-Butyl-2-ethoxy-5, 5-dimethyl-4-(*E*-styryl)-2, 5-dihydro-[1, 2]-oxaphosphole 2-oxide (*E*-2d)

The reaction of monoethyl ester of (2-methylocta-2,3-dien-4-yl)phosphonic acid **1a** (67 mg, 0.29 mmol), styrene (157 mg, 1.51 mmol), CaH_2 (0.6 mg, 5 mol%), $\text{PdCl}_2(\text{PhCN})_2$ (6 mg, 5 mol %), and NaI (9 mg, 0.06 mmol) afforded 50 mg (52%) of **E-2d** and 17 mg (25%) **4a**. However it is very difficult to get the pure compound by regular column chromatography on silica gel.

E-2d: liquid; ^1H NMR (300 MHz, CDCl_3) δ 7.49-7.29 (m, 5 H), 6.80 (d, $J = 16.5$ Hz, 1 H), 6.69 (d, $J = 16.5$ Hz, 1 H), 4.19-4.06 (m, 2 H), 2.59-2.37 (m, 2 H), 1.70-1.57 (m, 8 H), 1.42-1.29 (m, 5 H), 0.95 (t, $J = 7.2$ Hz, 3 H); ^{13}C NMR (CDCl_3 , 75.4 MHz): δ 155.6 (d, $J_{\text{PC}} = 24.7$ Hz), 136.0, 128.9, 128.9, 126.8, 125.3 (d, $J_{\text{PC}} = 159.4$ Hz), 118.5 (d, $J_{\text{PC}} = 23.6$ Hz), 85.3 (d, $J_{\text{PC}} = 8.7$ Hz), 62.5 (d, $J_{\text{PC}} = 6.3$ Hz), 30.8 (d, $J_{\text{PC}} = 2.3$ Hz), 28.5 (d, $J_{\text{PC}} = 2.9$ Hz), 27.8 (d, $J_{\text{PC}} = 2.3$ Hz), 25.5 (d, $J_{\text{PC}} = 11.5$ Hz), 22.8, 16.6 (d, $J_{\text{PC}} = 5.7$ Hz), 13.8; ^{31}P NMR (121.5 MHz, CDCl_3) δ 38.5; IR (neat) 1626, 1449, 1388, 1366, 1261, 1149, 1043 cm^{-1} ; MS (EI): m/z 334 (M^+ , 28.62), 319 ($\text{M}^+ - \text{CH}_3$, 3.96), 305 ($\text{M}^+ - \text{C}_2\text{H}_5$, 5.44), 292 ($\text{M}^+ - \text{C}_3\text{H}_6$, 10.09), 277 ($\text{M}^+ - \text{C}_4\text{H}_9$, 11.89),

43 (100); HRMS (EI): m/z calcd for $C_{19}H_{27}O_3P[M^+]$: 334.1698; Found: 334.1693.

(5) 3-Butyl-2-ethoxy-4-(*E*-2-methoxycarbonylvinyl)-1-oxa-2-phosphaspiro[4.4]-non-3-ene 2-oxide (*E*-2e)

The reaction of monoethyl ester of (1,1-tetramethylenehepta-1,2-dien-3-yl)phosphonic acid **1e** (77 mg, 0.30 mmol), acrylic acid methyl ester (0.14 mL, 133 mg, 1.5 mmol), CaH_2 (0.6 mg, 5 mol%), $\text{PdCl}_2(\text{PhCN})_2$ (6 mg, 5 mol %), and NaI (9 mg, 0.06 mmol) afforded 71 mg (69%) of **E-2e**: liquid; ^1H NMR (300 MHz, CDCl_3) δ 7.28 (dd, $J = 16.5, 1.5$ Hz, 1 H), 5.98 (dd, $J = 16.5, 1.2$ Hz, 1 H), 4.17-4.00 (m, 2 H), 3.75 (s, 3 H), 2.47-2.30 (m, 2 H), 2.00-1.70 (m, 8 H), 1.59-1.48 (m, 2 H), 1.39-1.24 (m, 5 H), 0.88 (t, $J = 7.2$ Hz, 3 H); ^{13}C NMR (CDCl_3 , 75.4 MHz): δ 166.1 (d, $J_{\text{PC}} = 1.6$ Hz), 149.6 (d, $J_{\text{PC}} = 25.3$ Hz), 133.8 (d, $J_{\text{PC}} = 23.0$ Hz), 133.5 (d, $J_{\text{PC}} = 156.5$ Hz), 124.4 (d, $J_{\text{PC}} = 1.1$ Hz), 95.2 (d, $J_{\text{PC}} = 7.5$ Hz), 62.7 (d, $J_{\text{PC}} = 6.2$ Hz), 52.0, 39.1 (d, $J_{\text{PC}} = 2.7$ Hz), 38.6 (d, $J_{\text{PC}} = 2.3$ Hz), 30.7 (d, $J_{\text{PC}} = 2.4$ Hz), 25.8 (d, $J_{\text{PC}} = 10.9$ Hz), 24.74, 24.70, 22.6, 16.5 (d, $J_{\text{PC}} = 6.0$ Hz), 13.6; ^{31}P NMR (121.5 MHz, CDCl_3) δ 36.9; IR (neat) 1725, 1631, 1437, 1263, 1174, 1040 cm^{-1} ; MS (EI): m/z 343 ($M^+ + 1$, 5.99), 342 (M^+ , 3.14), 285 ($M^+ - \text{C}_4\text{H}_9$, 28.36), 283 ($M^+ - \text{CH}_3\text{COO}$, 24.36), 41 (100); HRMS (EI): m/z calcd for $C_{17}H_{27}O_5P[M^+]$: 342.1596; Found: 342.1598.

(6) 3-Butyl-2-ethoxy-4-(*E*-2-methoxycarbonylvinyl)-1-oxa-2-phosphaspiro[4.5]-dec-3-ene 2-oxide (*E*-2f)

The reaction of monoethyl of (4,4-pentamethylenebuta-2,3-dien-2-yl)phosphonic acid **1f** (67 mg, 0.29 mmol), acrylic acid methyl ester (0.14 mL, 133 mg, 1.5 mmol), CaH_2 (0.6 mg, 5 mol%), $\text{PdCl}_2(\text{PhCN})_2$ (6 mg, 5 mol %), and NaI (9 mg, 0.06 mmol) afforded 72 mg (79%) of ***E*-2f**: liquid; ^1H NMR (300 MHz, CDCl_3) δ 7.16 (d, $J = 16.5$, Hz, 1 H), 6.11 (dd, $J = 16.5$, 1.2 Hz, 1 H), 4.16-4.01 (m, 2 H), 3.75 (s, 3 H), 1.96 (d, $J = 14.7$ Hz, 3 H), 1.75-1.50 (m, 10 H), 1.29 (t, $J = 7.2$ Hz, 3 H); ^{13}C NMR (CDCl_3 , 75.4 MHz): δ 165.9 (d, $J_{\text{PC}} = 1.7$ Hz), 152.9 (d, $J_{\text{PC}} = 25.3$ Hz), 134.4 (d, $J_{\text{PC}} = 23.6$ Hz), 125.8 (d, $J_{\text{PC}} = 161.1$ Hz), 125.7 (d, $J_{\text{PC}} = 2.3$ Hz), 87.4 (d, $J_{\text{PC}} = 7.5$ Hz), 62.8 (d, $J_{\text{PC}} = 6.9$ Hz), 51.9, 35.5 (d, $J_{\text{PC}} = 2.9$ Hz), 35.0 (d, $J_{\text{PC}} = 1.7$ Hz), 24.3, 21.6, 21.4, 16.5 (d, $J_{\text{PC}} = 5.2$ Hz), 10.8 (d, $J_{\text{PC}} = 12.1$ Hz); ^{31}P NMR (121.5 MHz, CDCl_3) δ 37.3; IR (neat) 1724, 1633, 1437, 1325, 1160, 1048 cm^{-1} ; MS (EI): m/z ; 315 ($\text{M}^+ + 1$, 14.20), 314 (M^+ , 16.80), 255 ($\text{M}^+ - \text{CH}_3\text{COO}$, 74.86), 41 (100); HRMS (EI): m/z calcd for $\text{C}_{15}\text{H}_{23}\text{O}_5\text{P}[\text{M}^+]$: 314.1283; Found: 314.1282.

(7) 3-Benzyl-2-ethoxy-4-(*E*-2-methoxycarbonylvinyl)-5, 5-dimethyl-5*H*-[1, 2]-oxa-phosphole 2-oxide (*E*-2g)

The reaction of monoethyl ester of (4-methyl-1-phenylpenta-2,3-dien-2-yl)-phosphonic acid **1g** (80 mg, 0.30 mmol), acrylic acid methyl ester (0.14 mL, 133 mg, 1.5 mmol), CaH_2 (0.6 mg, 5 mol%), $\text{PdCl}_2(\text{PhCN})_2$ (6 mg, 5 mol %), and NaI (9 mg, 0.06 mmol) afforded 79 mg (75%) of **E-2g**: liquid; ^1H NMR (300 MHz, CDCl_3) δ 7.37 (dd, $J = 16.5, 1.5$ Hz, 1 H), 7.30-7.15 (m, 5 H), 6.13 (dd, $J = 16.5, 1.5$ Hz, 1 H), 3.90-3.40 (m, 7 H), 1.55 (s, 3 H), 1.53 (s, 3 H), 0.94 (t, $J = 7.2$ Hz, 3 H); ^{13}C NMR (CDCl_3 , 75.4 MHz): δ 165.8, 153.3 (d, $J_{\text{PC}} = 25.3$ Hz), 136.4 (d, $J_{\text{PC}} = 2.3$ Hz), 133.5 (d, $J_{\text{PC}} = 22.5$ Hz), 130.1 (d, $J_{\text{PC}} = 158.9$ Hz), 128.6, 128.5, 126.7, 125.3 (d, $J_{\text{PC}} = 1.2$ Hz), 84.8 (d, $J_{\text{PC}} = 8.1$ Hz), 62.7 (d, $J_{\text{PC}} = 6.9$ Hz), 52.0, 31.3 (d, $J_{\text{PC}} = 11.5$ Hz), 27.9 (d, $J_{\text{PC}} = 2.9$ Hz), 27.5 (d, $J_{\text{PC}} = 2.3$ Hz), 15.9 (d, $J_{\text{PC}} = 6.9$ Hz); ^{31}P NMR (121.5 MHz, CDCl_3) δ 35.8; IR (neat) 1724, 1633, 1496, 1454, 1436, 1299, 1264, 1198, 1045 cm^{-1} ; MS (EI): m/z 350 (M^+ , 5.18), 291 ($\text{M}^+ - \text{CH}_3\text{COO}$, 100); HRMS (EI): m/z calcd for $\text{C}_{18}\text{H}_{23}\text{O}_5\text{P}[\text{M}^+]$: 350.1283; Found: 350.1285.

(8) 2-Ethoxy-5, 5-diethyl-3-hexyl-4-(E-2-methoxycarbonylvinyl)-5H-[1, 2]-oxaphosphole 2-oxide (E-2h)

The reaction of monoethyl ester of (3-ethylundeca-3,4-dien-5-yl)phosphonic acid

1h (85 mg, 0.30 mmol), acrylic acid methyl ester (0.14 mL, 133 mg, 1.5 mmol), CaH₂ (0.6 mg, 5 mol%), PdCl₂(PhCN)₂ (6 mg, 5 mol %), and NaI (9 mg, 0.06 mmol) afforded 93 mg (85%) of **E-2h**: liquid; ¹H NMR (300 MHz, CDCl₃) δ 7.22 (dd, *J* = 16.4, 1.4 Hz, 1 H), 6.05 (dd, *J* = 16.4, 1.1 Hz, 1 H), 4.22-4.10 (m, 2 H), 3.75 (s, 3 H), 2.51-2.24 (m, 2 H), 1.90-1.80 (m, 2 H), 1.78-1.61 (m, 2 H), 1.57-1.50 (m, 2 H), 1.36-1.17 (m, 9 H), 0.82-0.70 (m, 9 H); ¹³C NMR (CDCl₃, 75.4 MHz): δ 166.0 (d, *J*_{PC} = 1.7 Hz), 148.6 (d, *J*_{PC} = 25.9 Hz), 135.2 (d, *J*_{PC} = 158.9 Hz), 133.8 (d, *J*_{PC} = 23.8 Hz), 123.8 (d, *J*_{PC} = 1.2 Hz), 91.0 (d, *J*_{PC} = 6.3 Hz), 62.6 (d, *J*_{PC} = 6.6 Hz), 51.8, 31.6 (d, *J*_{PC} = 2.3 Hz), 31.5 (d, *J*_{PC} = 2.9 Hz), 31.1, 28.9, 28.6 (d, *J*_{PC} = 2.2 Hz), 25.9, 25.7, 22.2, 16.5 (d, *J*_{PC} = 5.6 Hz), 13.7, 7.3; ³¹P NMR (121.5 MHz, CDCl₃) δ 37.9; IR (neat) 1725, 1631, 1460, 1437, 1256, 1174, 1040 cm⁻¹; MS (EI): *m/z* 372 (M⁺, 0.73), 343 (M⁺-C₂H₅, 12.13), 84 (100); HRMS (EI): *m/z* calcd for C₁₉H₃₃O₅P[M⁺]: 372.2066; Found: 372.2065.

3. Reactions under Conditions B

(1) 3-Butyl-2-ethoxy-5, 5-dimethyl-4-(*E*-styryl)-2, 5-dihydro-[1, 2]-oxaphosphole 2-oxide (**E-2d**)

Typical condition II (Conditions B): The reaction of monoethyl ester of

2-methylocta- 2, 3-dien-4-yl phosphonic acid **1a** (73 mg, 0.31 mmol), styrene (157

mg, 1.51 mmol), BQ (33 mg, 0.31mmol), and PdCl₂(PhCN)₂ (6 mg, 5 mol %) in 2 mL

of DMF at 60°C for overnight. After following by the same work-up procedure as conditions A, 89 mg (85%) of **E-2d** and a trace of **4a** (less than 1%) were formed

(2) 3-Butyl-2-ethoxy-4-(E-2-methoxycarbonylpropenyl)-5, 5-dimethyl-5H-[1, 2]-oxaphosphole 2-oxide (E-2i)

The reaction of monoethyl ester of (2-methylocta-2,3-dien-4-yl)phosphonic acid **1a** (70 mg, 0.30 mmol), methyl 2-methylpropenoate (0.15 mL, 1.59 mmol), $\text{PdCl}_2(\text{PhCN})_2$ (6 mg, 5 mol %), and BQ (32 mg, 0.3 mmol) afforded 61 mg (61%) of **E-2i**: liquid; ^1H NMR (300 MHz, CDCl_3) δ 6.98 (s, 1 H), 4.19-4.07 (m, 2 H), 3.79 (s, 3 H), 2.25-1.95 (m, 2 H), 1.81 (d, $J = 1.5$ Hz, 3 H), 1.60-1.45 (m, 2 H), 1.43 (s, 3 H); 1.40 (s, 3 H), 1.38-1.20 (m, 5 H), 0.86 (t, $J = 7.2$ Hz, 3 H); ^{13}C NMR (CDCl_3 , 75.4 MHz): δ 167.1 (d, $J_{\text{PC}} = 1.2$ Hz), 155.3 (d, $J_{\text{PC}} = 26.2$ Hz), 133.9 (d, $J_{\text{PC}} = 1.4$ Hz), 130.5 (d, $J_{\text{PC}} = 22.6$ Hz), 126.3 (d, $J_{\text{PC}} = 155.9$ Hz), 85.1 (d, $J_{\text{PC}} = 8.7$ Hz), 62.6 (d, $J_{\text{PC}} = 6.9$ Hz), 52.3, 29.7 (d, $J_{\text{PC}} = 3.2$ Hz), 27.5 (d, $J_{\text{PC}} = 3.1$ Hz), 26.8 (d, $J_{\text{PC}} = 1.7$ Hz), 26.3 (d, $J_{\text{PC}} = 2.8$ Hz), 22.7, 16.6 (d, $J_{\text{PC}} = 1.4$ Hz), 14.7, 13.7; ^{31}P NMR (121.5 MHz, CDCl_3) δ 37.7; IR (neat) 1721, 1621, 1438, 1386, 1367, 1279, 1245, 1121, 1042 cm^{-1} ; MS (EI): m/z 331 ($\text{M}^+ + 1$, 44.13), 315 ($\text{M}^+ + 1 - \text{CH}_3$, 20.96), 271 ($\text{M}^+ + 1 - \text{CH}_3\text{OCO}$, 100); HRMS (EI): m/z calcd for $\text{C}_{16}\text{H}_{27}\text{O}_5\text{P}[\text{M}^+]$: 330.1596; Found: 330.1600.

(3) 3-Butyl-2-ethoxy-4-(E-2-carbamoylvinyl)-5, 5-dimethyl-5H-[1, 2]-oxa-

phosphole 2-oxide (*E*-2j);

The reaction of monoethyl ester of (2-methylocta-2,3-dien-4-yl) phosphonic acid **1a** (70 mg, 0.30 mmol), acrylamide (106 mg, 1.50 mmol), $\text{PdCl}_2(\text{PhCN})_2$ (6 mg, 5 mol %), and BQ (32 mg, 0.3 mmol) afforded 78 mg (86%) of **E-2j**: liquid; ^1H NMR (300 MHz, CDCl_3) δ 7.20 (d, $J = 15.9$ Hz, 1 H), 7.20 (bs, 1 H), 6.30 (d, $J = 15.9$ Hz, 1 H), 6.32 (bs, 1 H), 4.06-3.95 (m, 2 H), 2.41-2.24 (m, 2 H), 1.56-1.39 (m, 8 H), 1.38-1.20 (m, 2 H), 1.28 (t, $J = 7.2$ Hz, 3 H), 0.84 (t, $J = 6.9$ Hz, 3 H); ^{13}C NMR (CDCl_3 , 75.4 MHz): δ 166.8, 153.9 (d, $J_{\text{PC}} = 25.3$ Hz), 130.6 (d, $J_{\text{PC}} = 19.6$ Hz), 129.4 (d, $J_{\text{PC}} = 113.4$ Hz), 127.8, 85.3 (d, $J_{\text{PC}} = 8.7$ Hz), 62.8 (d, $J_{\text{PC}} = 6.9$ Hz), 30.5 (d, $J_{\text{PC}} = 2.3$ Hz), 28.2 (d, $J_{\text{PC}} = 3.4$ Hz), 27.4 (d, $J_{\text{PC}} = 2.3$ Hz), 25.4 (d, $J_{\text{PC}} = 10.9$ Hz), 22.5, 16.3 (d, $J_{\text{PC}} = 6.3$ Hz), 13.7; ^{31}P NMR (121.5 MHz, CDCl_3) δ 37.5; IR (neat) 3334, 3192, 1679, 1616, 1462, 1393, 1248, 1220, 1147, 1042 cm^{-1} ; MS (EI): m/z 302 ($\text{M}^+ + 1$, 44.13), 286 ($\text{M}^+ + 1 - \text{CH}_3$, 5.38), 257 ($\text{M}^+ + 1 - \text{H}_2\text{NCO}$, 100); HRMS (EI): m/z calcd for $\text{C}_{14}\text{H}_{24}\text{NO}_4\text{P}[\text{M}^+]$: 301.1443; Found: 301.1442.

(4) 2-Ethoxy-3-heptyl-4-(*E*-2-ethoxycarbonylvinyl)-5-methyl-5H-[1,2]-oxaphosphole 2-oxide (*E*-2k);

The reaction of monoethyl ester of 2, 3-undecadien-4-yl-phosphonic acid **1k** (83 mg, 0.30 mmol), acrylic acid ethyl ester (0.15 mL, 1.50 mmol), PdCl₂(PhCN)₂ (6 mg, 5 mol %), and BQ (32 mg, 0.3 mmol) afforded 71 mg (63%) of **E-2k**: liquid; ¹H NMR (300 MHz, CDCl₃) δ 7.43 (d, *J* = 16.2 Hz, 1 H), 5.90 (dd, *J* = 16.2, 4.8 Hz, 1 H), 5.13-4.86 (m, 2 H), 4.25-3.95 (m, 4 H), 2.58-2.25 (m, 2 H), 1.62-1.38 (m, 4 H), 1.38-1.05 (m, 14 H), 0.85-0.75 (m, 3 H); ³¹P NMR (121.5 MHz, CDCl₃) δ 40.3, 39.7; IR (neat) 1719, 1635, 1594, 1447, 1367, 1292, 1255, 1188, 1096, 1039 cm⁻¹; MS (EI): *m/z* 359 (M⁺+1, 22.44), 329 (M⁺+1-C₂H₅, 4.79), 285 (M⁺+1-EtOCO, 100); HRMS (EI): *m/z* calcd for C₁₈H₃₁O₅P[M⁺]: 358.1909; Found: 358.1919.

(5) 3-Butyl-2-ethoxy-4-(*E*-styryl)-5-methyl-5*H*-[1, 2]-oxaphosphole 2-oxide (*E*-2l);

The reaction of monoethyl ester of 2, 3-undecadien-4-yl-phosphonic acid **1k** (84 mg, 0.30 mmol), styrene (207 mg, 1.99 mmol), PdCl₂(PhCN)₂ (6 mg, 5 mol %), and BQ (35 mg, 0.33 mmol) afforded 73 mg (63%) of **E-2l**: liquid; ¹H NMR (300 MHz, CDCl₃) δ 7.47-7.31 (m, 5 H), 6.95 (d, *J* = 16.8 Hz, 1 H), 6.61 (dd, *J* = 16.8, 5.7 Hz, 1 H), 5.25-5.18 (m, 1 H), 4.21-3.98 (m, 2 H), 2.59-2.32 (m, 2 H), 1.76-1.50 (m, 5 H), 1.42-1.18 (m, 11 H), 0.90-0.81 (m, 3 H); ³¹P NMR (121.5 MHz, CDCl₃) δ 42.4, 41.8; IR (neat) 3058, 3038, 1628, 1591, 1496, 1449, 1377, 1252, 1162, 1096, 1042 cm⁻¹;

MS (EI): m/z 362 (M^+ , 2.23), 347 ($M^+ - CH_3$, 1.61), 263 ($M^+ - C_7H_{15}$, 9.66), 128 (100);

HRMS (EI): m/z calcd for $C_{21}H_{31}O_3P[M^+]$: 362.2011; Found: 362.2016.

(6) 3-Butyl-2-ethoxy-4-(*E*-2-ethoxycarbonylvinyl)-5*H*-[1, 2]-oxaphosphole

2-oxide (*E*-2m);

The reaction of monoethyl ester of 1,2-heptadien-3-yl phosphonic acid **1n** (54 mg, 0.27 mmol), acrylic acid ethyl ester (0.18 mL, 1.80 mmol), $PdCl_2(PhCN)_2$ (6 mg, 5 mol %), and BQ (34 mg, 0.31 mmol) afforded 37 mg (46%) of **E-2n**: liquid; 1H NMR (300 MHz, $CDCl_3$) δ 7.45 (dd, $J = 16.5, 2.4$ Hz, 1 H), 5.84 (dd, $J = 16.5, 2.1$ Hz, 1 H), 4.81-4.64 (m, 2 H), 4.19 (q, $J = 7.2$ Hz, 2 H), 4.17-4.01 (m, 2 H), 2.58- 2.25 (m, 2 H), 1.59-1.43 (m, 2 H), 1.39-1.21 (m, 8 H), 0.87 (t, $J = 7.5$ Hz, 3 H); ^{13}C NMR ($CDCl_3$, 75.4 MHz): δ 165.6(d, $J_{PC} = 2.9$ Hz), 143.6 (d, $J_{PC} = 27.6$ Hz), 133.2 (d, $J_{PC} = 159.4$ Hz), 132.7 (d, $J_{PC} = 22.5$ Hz), 122.8, 68.0 (d, $J_{PC} = 9.4$ Hz), 63.2 (d, $J_{PC} = 6.3$ Hz), 61.1, 30.8 (d, $J_{PC} = 2.3$ Hz), 25.2 (d, $J_{PC} = 11.5$ Hz), 22.6, 16.5 (d, $J_{PC} = 2.4$ Hz), 14.1, 13.6; ^{31}P NMR (121.5 MHz, $CDCl_3$) δ 43.4; IR (neat) 1717, 1637, 1600, 1465, 1367, 1298, 1259, 1188, 1151, 1026 cm^{-1} ; MS (EI): m/z 303 ($M^+ + 1$, 44.13), 287 ($M^+ + 1 - CH_3$, 6.17), 273 ($M^+ + 1 - C_4H_9$, 3.17), 229 ($M^+ + 1 - EtOCO$, 100); HRMS (EI): m/z calcd for $C_{14}H_{23}O_5P[M^+]$: 302.1283; Found: 302.1284.

(7) 3-Butyl-2-ethoxy-4-(*E*-2-carbamoyl-vinyl) - 5*H*-[1, 2]-oxaphosphole 2-oxide

(E-2n);

The reaction of monoethyl ester of 1,2-heptadien-3-yl phosphonic acid **1n** (62 mg, 0.30 mmol), acrylamide (106 mg, 1.50 mmol), $\text{PdCl}_2(\text{PhCN})_2$ (6 mg, 5 mol %), and BQ (32 mg, 0.30 mmol) afforded 40 mg (48%) of **E-2m**: liquid; ^1H NMR (300 MHz, CDCl_3) δ 7.53 (dd, $J = 15.3, 2.1$ Hz, 1 H), 5.99 (d, $J = 15.9$ Hz, 1 H), 5.92 (bs, 1H), 5.68 (bs, 1H), 4.88-4.70 (m, 2 H), 4.20-4.09 (m, 2 H), 2.61-2.38 (m, 2 H), 1.63-1.55 (m, 2 H), 1.43-1.32 (m, 5 H), 0.93 (t, $J = 7.2$ Hz, 3 H); ^{13}C NMR (CDCl_3 , 75.4 MHz): δ 166.3, 144.3 (d, $J_{\text{PC}} = 27.6$ Hz), 131.7 (d, $J_{\text{PC}} = 183.5$ Hz), 130.4 (d, $J_{\text{PC}} = 22.5$ Hz), 124.8, 68.3 (d, $J_{\text{PC}} = 9.8$ Hz), 63.2 (d, $J_{\text{PC}} = 6.9$ Hz), 30.9 (d, $J_{\text{PC}} = 2.3$ Hz), 25.2 (d, $J_{\text{PC}} = 12.1$ Hz), 22.7, 16.5 (d, $J_{\text{PC}} = 6.3$ Hz), 13.7; ^{31}P NMR (121.5 MHz, CDCl_3) δ 36.4; IR (neat) 3335, 3195, 1680, 1600, 1389, 1245, 1024 cm^{-1} ; MS (EI): m/z 274 ($\text{M}^+ + 1$, 0.83), 258 ($\text{M}^+ + 1 - \text{CH}_3$, 0.77), 229 ($\text{M}^+ + 1 - \text{H}_2\text{NCO}$, 12.62), 71 (100); HRMS (EI): m/z calcd for $\text{C}_{12}\text{H}_{20}\text{NO}_4\text{P}[\text{M}^+]$: 273.1130; Found: 273.1127.

(8) 2-Ethoxy-4-(E-2-ethoxycarbonylvinyl)-1-oxa-2-phosphaspiro[4.5]dec-3-ene

2-oxide (E-2o);

The reaction of monoethyl ester of 3,3-pentamethylenepropadienyl phosphonic acid **1o** (66 mg, 0.30 mmol), acrylamide (0.15 mL, 1.50 mmol), $\text{PdCl}_2(\text{PhCN})_2$ (6 mg,

5 mol %), and BQ (34 mg, 0.31 mmol) afforded 58 mg (61%) of **E-2o**: oil; ^1H NMR (300 MHz, CDCl_3) δ 7.14 (d, $J = 15.9$ Hz, 1 H), 6.37 (d, $J = 15.9$ Hz, 1 H), 6.19 (d, $J = 28.2$ Hz, 1 H), 4.25 (q, $J = 7.2$ Hz, 2 H), 4.19-4.08 (m, 2 H), 1.85-1.59 (m, 10 H), 1.34 (t, $J = 7.2$ Hz, 3 H), 1.31 (t, $J = 7.2$ Hz, 3 H); ^{13}C NMR (CDCl_3 , 75.4 MHz): δ 165.3, 162.4 (d, $J_{\text{PC}} = 11.5$ Hz), 134.5 (d, $J_{\text{PC}} = 24.1$ Hz), 126.4 (d, $J_{\text{PC}} = 2.3$ Hz), 113.8 (d, $J_{\text{PC}} = 167.5$ Hz), 88.0 (d, $J_{\text{PC}} = 6.3$ Hz), 63.1 (d, $J_{\text{PC}} = 6.3$ Hz), 61.2, 35.2, 34.7, 24.4, 21.6, 21.4, 16.5 (d, $J_{\text{PC}} = 5.8$ Hz), 14.2; ^{31}P NMR (121.5 MHz, CDCl_3) δ 35.6; IR (neat) 1724, 1636, 1448, 1391, 1369, 1324, 1266, 1185, 1154, 1096, 1043 cm^{-1} ; MS (EI): m/z 315 ($\text{M}^+ + 1$, 8.46), 268 ($\text{M}^+ + 1 - \text{C}_2\text{H}_5\text{O}$, 37.49), 241 ($\text{M}^+ + 1 - \text{C}_2\text{H}_5\text{OCO}$, 15.24), 158 (100); HRMS (EI): m/z calcd for $\text{C}_{15}\text{H}_{23}\text{O}_5\text{P}[\text{M}^+]$: 314.1283; Found: 314.1276.

(9) 2-Ethoxy-4-(*E*-2-carbamoylvinyl)-1-oxa-2-phospha-spiro[4.5]dec-3-ene

2-oxide (*E*-2p)

The reaction of monoethyl ester of 3,3-pentamethylenepropadienyl phosphonic acid **1o** (64 mg, 0.30 mmol), acrylic acid ethyl ester (120 mg, 1.69 mmol), $\text{PdCl}_2(\text{PhCN})_2$ (6 mg, 5 mol %), and BQ (33 mg, 0.30 mmol) afforded 29 mg (34%) of **E-2p**: solid mp: 107-110 °C (recrystallized from EtOAc: petroleum ether = 5:2); ^1H NMR (300 MHz, CDCl_3) δ 7.09 (d, $J = 15.3$ Hz, 1 H), 7.10 (bs, 1H), 6.63 (d, $J = 15.3$ Hz, 1 H), 6.13 (d, $J = 29.4$ Hz, 1 H), 5.77 (bs, 1H), 4.18-4.02 (m, 2 H), 1.80-1.57 (m,

10 H), 1.34 (t, $J = 7.2$ Hz, 3 H); ^{13}C NMR (CDCl_3 , 75.4 MHz): δ 166.0, 164.3 (d, $J_{\text{PC}} = 19.0$ Hz), 131.3 (d, $J_{\text{PC}} = 23.6$ Hz), 129.0, 111.0 (d, $J_{\text{PC}} = 168.1$ Hz), 88.6 (d, $J_{\text{PC}} = 6.9$ Hz), 63.2 (d, $J_{\text{PC}} = 6.9$ Hz), 35.3, 34.3, 24.3, 21.5, 21.4, 16.5 (d, $J_{\text{PC}} = 5.7$ Hz); ^{31}P NMR (121.5 MHz, CDCl_3) δ 36.4; IR (neat) 3333, 3191, 1679, 1617, 1448, 1391, 1278, 1243, 1151, 1042 cm^{-1} ; MS (EI): m/z 286 ($\text{M}^+ + 1$, 1.31), 268 ($\text{M}^+ + 1 - \text{H}_2\text{O}$, 8.75), 241 ($\text{M}^+ + 1 - \text{H}_2\text{NCO}$, 7.10), 142 (100); HRMS (EI): m/z calcd for $\text{C}_{13}\text{H}_{20}\text{NO}_4\text{P}[\text{M}^+]$: 285.1130; Found: 285.1135.

7.270
7.020
7.014
6.963
6.958

5.611
5.609
5.606
5.554
5.552
5.549

4.118
4.095
4.071
4.040

2.400
2.372
2.327
2.286
2.261

1.522
1.499
1.471
1.439
1.328
1.302
1.279
1.256
1.233
0.873
0.848
0.824

-0.025

yf-13-30-1

E-2a

yf-13-30-1-p

Yf-10-197-2

Z-2a

VF-10-187-2-p
Archive directory: /export/home/masm/vnmr/sys/data
Sample directory:
File: PHOSPHORUS
Pulse Sequence: szpu1

36.385

Z-2a

Yf-13-84-1

E-2b

VF-13-84-1-C

Archive directory: /export/home/masm/vnmr/sys/data
Sample directory:
File: CARBON

Pulse Sequence: szpu1

E-2b

VF-13-85-1-C

Archive directory: /export/home/masm/vmr/sys/data
Sample directory:
File: CARBON

Pulse Sequence: szpul

E-2c

1xd-1-146-C

Archive directory: /export/home/masm/vnmr/sys/data
Sample directory:
File: CARBON

Pulse Sequence: szpul

E-2d

E-2d

yf-13-91-1-p

38.538

yf-13-105-1

E-2e

E-2e

yf-13-105-1-p

36.876

7.191
7.270
7.136

6.135
6.132
6.080
6.076

4.134
4.110
4.102
4.087
4.078
4.064
4.055
4.032
3.748
3.736

1.983
1.934
1.703
1.657
1.641
1.632
1.584
1.561
1.554
1.537
1.520
1.310
1.296
1.286
1.273
1.263
1.250

Yf-13-107-1

E-2f

VF-13-107-1-C

Archive directory: /export/home/masm/vnmr/sys/data
Sample directory:
File: CARBON

Pulse Sequence: zgpg30

E-2f

E-2f

yf-13-107-1-p

37.324

yf-13-109-1

E-2g

VF-13-109-1-C

Archive directory: /export/home/masm/vmr/sys/data
Sample directory:
File: CARBON

Pulse Sequence: szpul

F-2g

2g

7.268
7.252
7.248
7.198
7.193

6.077
6.073
6.022
6.019

4.218
4.190
4.187
4.156
4.133

3.748

2.444
2.410
2.384
2.343
2.317

1.901
1.882
1.858
1.851
1.827
1.744
1.720
1.696
1.671
1.647
1.563
1.542
1.516
1.489
1.315
1.292
1.269
1.248
0.837
0.815
0.793
0.769
0.745
0.721

yf-13-110-1

E-2h

Archive directory: /export/home/nasm/vmr/sys/431
 Sample directory: 1xd-1-155depl_02Nov2006
 Pulse Sequence: DEPT

134.490
 134.176

124.159

63.095
 63.006

52.291

32.039
 32.001
 31.956
 31.591
 29.357
 29.082
 29.056
 28.329
 26.182
 22.694
 16.976
 16.907
 14.187
 7.742
 7.722

E-2h

DEPT 0 = 135°

DEPT 0 = 90°

7.256
6.975
6.763

4.176
4.152
4.145
4.129
4.125
4.121
4.102
4.097
4.078
4.073
3.788

2.196
2.165
2.144
2.121
2.097
2.089
2.066
2.022
2.000
1.810
1.805
1.563
1.549
1.536
1.526
1.517
1.493
1.485
1.471
1.443
1.425
1.403
1.349
1.326
1.315
1.302
1.289
1.263
1.239
1.222
1.216
0.898
0.883
0.859
0.833

lxd-2-56-h

1xd-2-56
 Archive directory: /export/home/masm/vnmr-sys/data
 Sample directory:
 File: CARBON
 Pulse Sequence: szpul

167.088
 155.509
 155.158

133.909
 133.939
 130.589
 130.283
 127.291
 125.223

85.220
 85.106
 77.427
 77.000
 76.580

62.658
 62.567

52.316

29.678
 29.647
 27.510
 27.464
 26.808
 26.373
 26.205
 22.717
 16.595
 16.519
 14.733
 13.667

1xd-2-56-p

- 37.698

7.226
7.202
7.173

6.323
6.270

4.048
4.024
4.018
3.995

2.404
2.381
2.350
2.320
2.309

1.515
1.482
1.475
1.436
1.347
1.323
1.300
1.276
1.252
0.858
0.843
0.820

lxd-2-80-h

1xd-2-80-c

Archive directory: /export/home/masm/vmr/sys/data
Sample directory:
File: CARBON
Pulse Sequence: szput

lxd-2-80-p

lxd-2-68-p

42.385
41.789

1xd-2-84-c
 Archive directory: /export/home/masm/vnmr/sys/data
 Sample directory:
 File: CARBON
 Pulse Sequence: szput

2m

lxd-2-87-h

1xd-2-87-c
 Archive directory: /export/home/masm/vmrys/data
 Sample directory:
 File: CARBON
 Pulse Sequence: s2pu1

7.259
7.169
7.116

6.399
6.347
6.241
6.147

4.281
4.258
4.234
4.210
4.163
4.139
4.129
4.116
4.106

1.798
1.780
1.765
1.752
1.708
1.696
1.659
1.361
1.337
1.313
1.289
1.239
1.233

lxd-2-90-h

1xd-2-90-c

Archive directory: /export/home/masm/vnmr/sys/data

Sample directory:

File: CARBON

Pulse Sequence: szpul

165.363
162.516
162.264

134.649
134.329

126.375
126.345

115.171
112.950

88.029
87.945

77.420
77.000
76.573

63.093
63.009
61.193

35.189
34.677

24.442
21.602
21.427
16.588
16.511
14.161

lx-d-2-92-h

1xd-2-92-c
 Archive directory: /export/home/mas/m/vnmrSYS/data
 Sample directory:
 File: CARBON
 Pulse Sequence: szpu1