Cycloaddition Reaction of Zirconacyclopentadienes to Quinones: Synthesis of Higher Para
Quinones

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Experimental Section:

All reactions involving organometallic compounds were carried out using standard schlenk tube under nitrogen. Tetrahydrofuran (THF) and ether was distilled from sodium and benzophenone. ^1H NMR and ^{13}C NMR spectra were recorded on 300MHz NMR spectrometer.

1,4-naphthoquinone, *p*-chloranil, *p*-benzoquinone and phenyl-*p*-benzoquinone are commercially available. All quinones should be recrystallized before usage.

A representative procedure for the preparation of 1,2,3,4-tetraethylanthracene-9,10 4aH,9aH)-dione (3a). To a solution of Cp_2ZrCl_2 (1.2 mmol, 0.352 g) in THF (5.0 mL) was added *n*-BuLi (1.6 M in hexane, 2.4 mmol) at $-78\text{ }^\circ\text{C}$ and the mixture was stirred for 1 h. After addition of 3-hexyne (2.0 mmol, 228 μL) the mixture was warmed up to room temperature and stirred for 1h. Before CuCl (2.0 mmol, 196 mg) was added, the mixture was cooled to $-30\text{ }^\circ\text{C}$ using ice-salt bath and maintained at the temperature for 10 min. Then 10 mL of THF followed by 1,4-naphthoquinone (1.2 mmol, 190 mg) was added to the suspension. The mixture was stirred at room temperature for 12 h and quenched with 3N HCl and aqueous layers were extracted with 15 mL of Et_2O for 5 times. The combined organic layers were dried over Na_2SO_4 . The solvent was evaporated and the product was purified by column chromatography with petroleum ether/ methylene dichloride =2:1 as elution to afford the target compound as a yellow liquid (268 mg, 83%). IR (neat): 1693 cm^{-1} (C=O). ^1H NMR (CDCl_3 , SiMe_4) δ 0.86-0.91 (m, 12H), 1.83-1.89 (m, 2H), 2.11-2.37 (m, 6H), 3.59 (s, 2H), 7.67-7.70 (m, 2H), 7.97-8.01 (m, 2H); ^{13}C NMR (CDCl_3 , SiMe_4) δ 14.20, 15.06, 20.86, 25.07, 53.03, 126.22, 130.57, 134.06, 135.49, 136.75, 199.70. HRMS calcd for $\text{C}_{22}\text{H}_{26}\text{O}_2$ 322.1933, found 322.1936.

1,2,3,4-Tetraethyltetracene-5,12(4aH,12aH)-dione (3b). A yellow liquid (278mg, 75%). IR (neat): 1692 cm⁻¹ (C=O). ¹H NMR (CDCl₃, SiMe₄) δ 0.87-0.97 (m, 12H), 1.89-1.96 (m, 2H), 2.19-2.40 (m, 6H), 3.66 (s, 2H), 7.62-7.67 (m, 2H), 8.00-8.03 (m, 2H), 8.54 (s, 2H); ¹³C NMR (CDCl₃, SiMe₄) δ 14.09, 14.87, 20.67, 24.89, 52.78, 127.64, 128.96, 129.84, 130.52, 131.44, 134.97, 136.60, 199.31. HRMS calcd for C₂₆H₂₈O₂, 372.2089, found 372.2092.

Compound (3d). A yellow spummy solid (351 mg, 78%). Mp 86-88 °C (decomposition). IR (neat): 1724 cm⁻¹ (C=O). ¹H NMR (CDCl₃, SiMe₄) δ 0.77-0.86 (m, 12H), 1.69-1.74 (m, 2H), 2.02 (s, 2H), 2.11-2.26 (m, 6H), 2.98 (s, 2H), 4.93 (s, 2H), 1.83-1.89 (m, 2H), 2.09-2.32 (m, 6H), 3.61 (s, 2H), 7.13-7.16 (m, 4H), 7.26-7.29 (m, 2H), 7.34-7.39 (m, 2H); ¹³C NMR (CDCl₃, SiMe₄) δ 14.08, 14.96, 20.80, 24.82, 48.69, 51.52, 52.19, 123.88, 125.09, 126.52, 126.68, 129.61, 137.15, 140.78, 141.89, 211.01. HRMS calcd for C₃₂H₃₄O₂ 450.6112, found 450.6109.

1,2,3,4-Tetrabutylanthracene-9,10(4aH,9aH)-dione (3e). IR (neat): 1724 cm⁻¹ (C=O). A yellow liquid (350 mg, 81%). ¹H NMR (CDCl₃, SiMe₄) δ 0.80-0.87 (m, 12H), 1.15-1.39 (m, 16H), 1.77-1.84 (m, 2H), 2.08-2.38 (m, 6H), 3.54(s, 2H), 7.65-7.68 (m, 2H), 7.95-7.97 (m, 2H); ¹³C NMR (CDCl₃, SiMe₄) δ 13.96, 22.81, 27.75, 31.60, 32.02, 32.42, 53.47, 126.12, 129.43, 133.94, 135.51, 136.00, 199.72. HRMS calcd for C₃₀H₄₂O₂ 434.3185, found 434.3187.

A representative procedure for the preparation of 1,2-diethyl-3,4-dipropyl anthracene-9,10(4aH,9aH)-dione (3g). A Schlenk tube under dried nitrogen was charged with Cp₂ZrCl₂ (1.2 mmol, 0.352 g) and THF (5.0 mL). The mixture was cooled to -78 °C, and then 1.6 M *n*-BuLi (2.4 mmol, 1.5 mL) was added dropwise via syringe. The reaction mixture was stirred at -78 °C for 1 h under nitrogen before ethylene gas was introduced for 1 h, and the reaction mixture was gradually warmed to room temperature. 3-Hexyne (1.0 mmol, 114 μL) was added, and the resulting mixture was stirred for 1 h at room temperature under a positive

pressure of ethylene gas. To the solution of zirconacyclopentene, 4-octyne (1.0 mmol, 142 μ L) was added. Then the solution was heated at 50 $^{\circ}$ C for 1 h and the reaction mixture changed from light yellow to red. Before CuCl (2.0 mmol, 196 mg) was added, the mixture was cooled to -30° C using ice-salt bath and maintained at the temperature for 10 min. Then 10 mL of THF followed by 1,4-naphthoquinone (1.2mmol, 190mg) was added to the suspension. The mixture was stirred at room temperature for 12 h and quenched with 3N HCl and aqueous layers were extracted with 15 mL of Et₂O for 5 times. The combined organic layers were dried over Na₂SO₄. The solvent was evaporated and the product was purified by column chromatography with petroleum ether/ methylene dichloride =2:1 as elution to afford the target compound as a yellow liquid (293 mg, 84%). IR (neat): 1693 cm⁻¹ (C=O). ¹H NMR (CDCl₃, SiMe₄) δ 0.78-0.94 (m, 12H), 1.10-1.46 (m, 4H), 1.72-1.91 (m, 2H), 2.14-2.39 (m, 6H), 3.54-3.58 (m, 2H), 7.67-7.70 (m, 2H), 7.97-8.00 (m, 2H); ¹³C NMR (CDCl₃, SiMe₄) δ 14.05, 14.11, 14.26, 15.02, 21.02, 22.54, 23.56, 25.24, 29.89, 34.20, 52.78, 53.72, 126.16, 126.22, 129.42, 130.58, 134.03, 134.10, 135.41, 135.50, 135.85, 136.99, 199.74, 199.89. HRMS calcd for C₂₄H₃₀O₂ 350.2246, found 350.2244.

1,2-Diethyl-4-propylantracene-9,10(4aH,9aH)-dione (3f). IR (neat): 1693 cm⁻¹ (C=O). A yellow liquid (263 mg, 85%). ¹H NMR (CDCl₃, SiMe₄) δ 0.82 (t, J = 7.5 Hz, 3H), 0.93 (t, J = 7.5 Hz, 3H), 0.97 (t, J = 7.5 Hz, 3H), 1.18-1.46 (m, 2H), 1.86-2.18 (m, 5H), 2.37-2.44 (m, 1H), 3.63-3.66 (m, 2H), 3.746 (s, 2H), 7.69-7.72 (m, 2H), 7.97-8.02 (m, 2H); ¹³C NMR (CDCl₃, SiMe₄) δ 13.77, 14.03, 14.19, 20.94, 24.32, 24.55, 37.05, 52.45, 53.63, 125.83, 126.25, 126.34, 129.16, 134.09, 134.22, 134.38, 135.13, 135.47, 199.14, 199.53. HRMS calcd for C₂₁H₂₄O₂ 308.1776, found 308.1740.

1,2,3,4-Tetrapropyltetracene-5,12(4aH,12aH)-dione (3i). IR (neat): 1693 cm^{-1} (C=O). A yellow liquid (368 mg, 86%). ^1H NMR (CDCl_3 , SiMe_4) δ 0.80-0.90 (m, 12H), 1.21-1.26 (m, 6H), 1.43-1.47 (m, 2H), 1.83-1.89 (m, 2H), 2.09-2.32 (m, 6H), 3.61 (s, 2H), 7.63-7.66 (m, 2H), 8.02-8.05 (m, 2H), 8.53 (s, 2H); ^{13}C NMR (CDCl_3 , SiMe_4) δ 14.02, 14.10, 22.61, 23.46, 30.09, 34.31, 53.52, 127.81, 129.16, 130.09, 131.67, 135.24, 135.90, 199.80. HRMS calcd for $\text{C}_{30}\text{H}_{36}\text{O}_2$ 428.2715, found 428.2718.

6,11-Diethyl-7,8,9,10-tetrahydrotetracene-5,12(5aH,11aH)-dione (3j). A yellow solid (215 mg, 67%). Mp 100-102 $^{\circ}\text{C}$ (decomposition). IR (neat): 1691 cm^{-1} (C=O). ^1H NMR (CDCl_3 , SiMe_4) δ 0.85 (t, $J = 7.5$ Hz, 6H), 1.52-1.59 (m, 4H), 1.83-1.90 (m, 2H), 2.26-2.38 (m, 6H), 3.62 (s, 2H), 7.67-7.71 (m, 2H), 7.98-8.01 (m, 2H); ^{13}C NMR (CDCl_3 , SiMe_4) δ 13.12, 23.51, 24.47, 26.05, 52.99, 126.18, 128.85, 131.89, 133.99, 135.46, 199.82. HRMS calcd for $\text{C}_{22}\text{H}_{24}\text{O}_2$ 320.1776, found 320.1776.

5,14-Diethyl-1,2,3,4-tetrahydropentacene-6,13(5aH,13aH)-dione (3k). A yellow solid (233 mg, 63%). Mp 87-89 $^{\circ}\text{C}$ (decomposition). IR (neat): 1692 cm^{-1} (C=O). ^1H NMR (CDCl_3 , SiMe_4) δ 0.88 (t, $J = 7.5$ Hz, 6H), 1.55-1.62 (m, 4H), 1.89-1.96 (m, 2H), 2.25-2.42 (m, 6H), 3.69 (s, 2H), 7.63-7.67 (m, 2H), 8.02-8.05 (m, 2H), 8.55 (s, 2H); ^{13}C NMR (CDCl_3 , SiMe_4) δ 13.22, 23.56, 24.49, 26.10, 52.99, 127.85, 128.99, 129.13, 130.02, 131.61, 132.03, 135.16, 199.70. HRMS calcd for $\text{C}_{26}\text{H}_{26}\text{O}_2$ 370.1933, found 370.1930.

A representative procedure for the preparation of 1,2,3,4-tetraethyl anthracene-9,10-dione (4a). To the unseparated mixture containing dihydroanthraquinone, *p*-chloranil (366 mg, 1.5 mmol) was added. The mixture was stirred at 50 $^{\circ}\text{C}$ for 6 h and quenched with 3N HCl and aqueous layers were extracted with 15mL of Et_2O for 5 times. The combined organic layers were dried over Na_2SO_4 . The solvent was evaporated and the product

was purified by flash column chromatography with petroleum ether/ methylene dichloride =2:1 as elution to afford the target compound as a red solid (256 mg, 79%). Mp 102-103 °C. IR (neat): 1668 cm⁻¹ (C=O). ¹H NMR (CDCl₃, SiMe₄) δ 1.20 (t, *J* = 7.5Hz, 6H), 1.29 (t, *J* = 7.5 Hz, 6H), 2.81 (q, *J* = 7.5Hz, 4H), 3.15 (broad, 4H), 7.62-7.65 (m, 2H), 8.02-8.05 (m, 2H); ¹³C NMR (CDCl₃, SiMe₄) δ 15.49, 15.65, 22.42, 23.62, 126.02, 132.30, 133.01, 135.10, 143.70, 148.05, 187.18. HRMS calcd for C₂₂H₂₄O₂ 320.1776, found 320.1774.

1,2,3,4-Tetraethyltetracene-5,12-dione (4b). IR (neat): 1669 cm⁻¹ (C=O). A yellow liquid (278mg, 65%). ¹H NMR (CDCl₃, SiMe₄) δ 1.23 (t, *J* = 7.5 Hz, 6H), 1.36(t, *J* = 7.5 Hz, 6H), 2.85 (q, *J* = 7.5 Hz, 4H), 3.20 (broad, 4H), 7.60-7.63 (m, 2H), 8.00-8.04 (m, 2H), 8.58 (s, 2H); ¹³C NMR (CDCl₃, SiMe₄) δ 15.51, 15.72, 22.46, 23.70, 127.61, 128.77, 129.80, 131.92, 133.43, 135.01, 143.78, 148.14, 187.32. HRMS calcd for C₂₆H₂₆O₂ 370.1933, found 370.1931

Compound (4c). A red spummy solid (305 mg, 69%). Mp 108-111 °C. IR (neat): 1652 cm⁻¹ (C=O). ¹H NMR (CDCl₃, SiMe₄) δ 1.15 (t, *J* = 7.5 Hz, 6H), 1.26 (t, *J* = 7.2 Hz, 6H), 2.76 (q, *J* = 7.2 Hz, 4H), 3.09 (q, *J* = 7.2 Hz, 4H), 5.92 (s, 2H), 7.02-7.04 (m, 4H), 7.44-7.47 (m, 4H); ¹³C NMR (CDCl₃, SiMe₄) δ 15.29, 15.46, 22.31, 23.58, 48.08, 124.35, 125.51, 130.15, 144.08, 144.27, 147.92, 153.36, 184.55. HRMS calcd for C₃₂H₃₀O₂, 446.2246, found 446.2243.

1,2-diethyl-4-propylanthracene-9,10-dione (4f). A red liquid (202 mg, 66%). IR (neat): 1668 cm⁻¹ (C=O). ¹H NMR (CDCl₃, SiMe₄) δ 1.05 (t, *J* = 7.5 Hz, 3H), 1.26(t, *J* = 7.2 Hz, 3H), 1.31 (t, *J* = 7.2 Hz, 3H), 2.77 (q, *J* = 7.5 Hz, 2H), 3.07-3.13 (m, 4H), 7.34 (s, 1H), 7.66-7.69 (m, 2H), 8.07-8.11 (m, 2H); ¹³C NMR (CDCl₃, SiMe₄) δ 14.60, 15.20, 15.56, 23.38, 24.51, 26.12, 38.14, 126.23, 126.39, 131.16, 133.21, 133.28, 133.43, 134.01, 134.47, 135.08, 137.76, 143.65, 144.21, 149.58, 185.98, 187.17. HRMS calcd for C₂₁H₂₂O₂ 306.1620, found 306.1623.

4-phenyl-1,2-dipropylanthracene-9,10-dione (4h). A red solid (265 mg, 72%). Mp 125-127 °C. IR (neat): 1671 cm^{-1} (C=O). ^1H NMR (CDCl_3 , SiMe_4) δ 1.06 (t, $J = 7.2$ Hz, 3H), 1.20 (t, $J = 7.2$ Hz, 3H), 1.66-1.77 (m, 4H), 2.75-2.80 (m, 2H), 3.17-3.22 (m, 2H), 7.28-7.49 (m, 6H), 7.63-7.74 (m, 2H), 8.00-8.03 (m, 1H), 8.17-8.20 (m, 1H); ^{13}C NMR (CDCl_3 , SiMe_4) δ 14.35, 14.98, 24.31, 24.44, 32.26, 35.41, 126.34, 126.67, 126.88, 128.04, 128.10, 131.39, 133.30, 133.40, 133.50, 133.91, 135.07, 138.20, 142.19, 143.08, 143.98, 148.03, 184.41, 186.47. HRMS calcd for $\text{C}_{26}\text{H}_{24}\text{O}_2$ 368.1776, found 368.1775.

1,2,3,4-tetrapropyltetracene-5,12-dione (4i). A yellow solid (318 mg, 85%). Mp 120-122 °C. IR (neat): 1668 cm^{-1} (C=O). ^1H NMR (CDCl_3 , SiMe_4) δ 1.09-1.18 (m, 12H), 1.49-1.59 (m, 4H), 1.62-1.72 (m, 4H), 2.70-2.75 (m, 4H), 3.10 (broad, 4H), 7.58-7.61 (m, 2H), 7.99-8.02 (m, 2H), 8.57 (s, 2H); ^{13}C NMR (CDCl_3 , SiMe_4) δ 15.06, 15.13, 24.72, 24.86, 32.23, 32.84, 127.56, 128.72, 129.75, 131.96, 133.36, 135.00, 142.47, 147.04, 187.36. HRMS calcd for $\text{C}_{30}\text{H}_{34}\text{O}_2$ 426.2559, found 426.2559.

6,11-diethyl-7,8,9,10-tetrahydrotetracene-5,12-dione (4j). A yellow solid (232 mg, 73%). Mp 106-108 °C. IR (neat): 1660 cm^{-1} (C=O). ^1H NMR (CDCl_3 , SiMe_4) δ 1.29 (t, $J = 7.2$ Hz, 6H), 1.79-1.82 (m, 4H), 2.86-2.90 (m, 4H), 3.08 (t, $J = 7.2$ Hz, 4H), 7.63-7.66 (m, 2H), 8.03-8.06 (m, 2H); ^{13}C NMR (CDCl_3 , SiMe_4) δ 14.09, 22.46, 23.13, 27.34, 125.97, 131.07, 132.91, 135.05, 143.34, 143.76, 187.04. HRMS calcd for $\text{C}_{22}\text{H}_{22}\text{O}_2$ 318.1620, found 318.1621.

5,14-diethyl-1,2,3,4-tetrahydropentacene-6,13-dione (4k). A yellow solid (333 mg, 90%). Mp 229-230 °C. IR (neat): 1666 cm^{-1} (C=O). ^1H NMR (CDCl_3 , SiMe_4) δ 1.33 (t, $J = 7.2$ Hz, 6H), 1.79-1.84 (m, 4H), 2.88-2.90 (m, 4H), 3.13 (t, $J = 7.2$ Hz, 4H), 7.57-7.60 (m, 2H), 7.97-8.00 (m, 2H), 8.55 (s, 2H); ^{13}C NMR (CDCl_3 , SiMe_4) δ 14.16, 22.46, 23.23, 27.36,

127.50, 128.62, 129.68, 131.83, 132.13, 134.90, 143.39, 143.78, 187.09, 131.07, 132.91, 135.05, 143.34, 143.76, 187.04. HRMS calcd for C₂₆H₂₄O₂ 368.1776, found 368.1774.

Compound (4l). A red spummy solid (289 mg, 65%). Mp 129-131 °C. IR (neat): 1655 cm⁻¹ (C=O). ¹H NMR (CDCl₃, SiMe₄) δ 1.22 (t, *J* = 7.2 Hz, 6H), 1.75-1.79 (m, 4H), 2.78-2.84 (m, 4H), 3.03 (q, *J* = 7.2 Hz, 4H), 5.93 (s, 2H), 7.02-7.05 (m, 4H), 7.45-7.48 (m, 4H); ¹³C NMR (CDCl₃, SiMe₄) δ 13.89, 22.50, 23.01, 27.35, 48.06, 124.34, 125.48, 129.00, 143.16, 144.21, 144.30, 153.36, 184.60. HRMS calcd for C₃₂H₂₈O₂ 444.2089, found 444.2091.

A representative procedure for the preparation of 9,10-diethyl-2-phenyl-5,6,7,8-tetrahydroanthracene-1,4-dione (4m). To a solution of Cp₂ZrCl₂ (1.2 mmol, 0.352 g) in THF (5.0 ml) was added BuLi (1.6M in hexane, 2.4 mmol) at -78 °C and the mixture was stirred for 1h. After addition of 3,9-dodecadiyne (1.0 mmol, 200 μL) the mixture was warmed up to room temperature and stirred for 1 h. Before CuCl (2.0 mmol, 196 mg) was added, the mixture was cooled to -30 °C using ice-salt bath and maintained at the temperature for 10 min. Then 10 mL of THF followed by phenyl-1,4-benzoquinone (2.0mmol, 368mg) was added to the suspension. The mixture was stirred at room temperature for 12 h and quenched with 3N HCl and aqueous layers were extracted with 15 mL of Et₂O for 5 times. The combined organic layers were dried over Na₂SO₄. The solvent was evaporated and the product was purified by column chromatography with petroleum ether/ methylene dichloride =2:1 as elution to afford the target compound as a dark brown solid. (215 mg, 63%). Mp 118-121 °C. IR (neat): 1654 cm⁻¹ (C=O). ¹H NMR (CDCl₃, SiMe₄) δ 1.23 (t, *J* = 7.5 Hz, 3H), 1.28 (t, *J* = 7.2 Hz, 3H), 1.79-1.83 (m, 4H), 1.79-1.83 (m, 4H), 2.85-2.90 (m, 4H), 3.01 (q, *J* = 7.2 Hz, 2H), 3.11 (q, *J* = 7.5 Hz, 2H), 6.88(s, 1H), 7.41-7.43 (m, 3H), 7.52-7.55 (m, 2H); ¹³C NMR (CDCl₃, SiMe₄) δ 14.10, 14.47, 22.56, 22.56, 22.62, 23.09, 27.37, 27.44, 128.56,

129.17, 129.75, 130.96, 133.79, 134.25, 142.94, 143.25, 143.33, 143.37, 148.18, 188.26, 188.92. HRMS calcd for $C_{24}H_{24}O_2$ 344.1776, found 344.1776.

9,10-diethyl-2-methyl-5,6,7,8-tetrahydroanthracene-1,4-dione (4n). A green solid (164 mg, 58%). Mp 95-96 °C. IR (neat): 1655 cm^{-1} (C=O). 1H NMR ($CDCl_3$, $SiMe_4$) δ 1.10-1.17 (m, 6H), 1.71 (b, 4H), 2.00 (s, 3H), 2.76 (b, 4H), 2.87-2.97 (m, 4H), 6.51 (s, 1H); ^{13}C NMR ($CDCl_3$, $SiMe_4$) δ 13.86, 23.92, 16.09, 22.49, 22.87, 22.27, 129.05, 129.64, 135.76, 142.73, 142.81, 143.36, 143.48, 147.20, 187.83, 188.86. HRMS calcd for $C_{19}H_{22}O_2$ 282.1620, found 282.1618.

A representative procedure for the preparation of 6,11-diphenyl-1,2,3,4-tetrahydrotetracene (5). To a solution of quinone **4j** (1.0 mmol, 0.318 g) in THF (5.0 ml) was added PhLi (2.0 M in Bu_2O , 2.0 mmol) at -78 °C and the solution was warmed up to room temperature and stirred for 3h. Then 1 mL of NH_4Cl saturated solution was added to quench the reaction for 1h. After addition of $SnCl_2 \cdot 6H_2O$ (1.0 g) and HCl (1M, 5 mL), the mixture was heated to 50 °C and stirred for 6h. The organic layer was separated and the aqueous layer were extracted with 15 mL of Et_2O for 5 times. The combined organic layers were dried over Na_2SO_4 . The solvent was evaporated and the product was purified by column chromatography with petroleum ether/ methylene dichloride =5:1 as elution to afford the target compound as a yellow solid (330 mg, 86%). Mp 187-189 °C. 1H NMR (300M, $CDCl_3$, Me_4Si) δ 0.69 (t, J = 7.2 Hz, 6H), 1.89 (b, 4H), 2.54 (q, J = 7.2 Hz, 4H), 2.92 (b, 4H), 7.28-7.34 (m, 2H), 7.54-7.60 (m, 10H), 7.72-7.77 (m, 2H); ^{13}C NMR (75M, $CDCl_3$, Me_4Si) δ 14.53, 23.18, 23.43, 28.12, 124.37, 126.51, 127.23, 128.18, 129.65, 130.79, 132.22, 134.48, 134.99, 135.72, 143.28. HRMS calcd for $C_{30}H_{24}$ 384.1878, found 384.1875.

050327-dich330H-single pulse-3Jdf
single_pulse

4a

050327-dich330C-single pulse dec-3J4f
single pulse decoupled gated NOE

4a

060619-dtch09H-single_pulse-2.jfif
single_pulse

006519 - dcd699C - single pulse dec-2,1dF
single pulse decoupled gated NOE

050327-dcd331H-single pulse-2.jdr
single_pulse

3a

050327-dichl331C-single pulse dec-2,1d1f
single pulse decoupled gated NOE

3a

4b

051222 - dht582C - single pulse dec - 2,1d1f
single pulse decoupled gated NOE

4b

059411 - chcl3&H - single pulse-2.jdr
single_pulse

3b

050411 - dichl38C - single pulse dec-2,4if
single pulse decoupled gated NOE

3b

06/511-dcd3%0H-single pulse-2.jdf
single_pulse

4i

062511 - dcd396C - single pulse dec - 2.jif
single pulse decoupled gated NOE

4i

059511 - chcl395H1-single_pulse-21d1f
single_pulse

3i

050511-chh395C-single_pulse_dec-2.jdr
single_pulse_decoupled_gated_NOE

06/29-dich417C-singl pulse dec-2,1d1f
single pulse decoupled gated NOE

3e

4k

050622-dich440C-singl pulse dec-2.jif
single pulse decoupled gated NOE

4k

060510—cdcl360Hz—single pulse-2.jdr
single_pulse

3k

050620 - dich432C - single pulse dec-2,14f
single pulse decoupled gated NOE

051206-chc1501H-single pulse-2.jdr
single_pulse

4f

051202-dh255C-singl pulse dec-2,1d1f
single pulse decoupled gated NOE

4f

051209-dict65H-single pulse-2.jdr
single pulse

051209-dich55C-singl pulse dec-2,1dF
single pulse decoupled gated NOE

3f

060116-ckck601H-single pulse-2.jdr
single_pulse

4h

060116- dich601C- single pulse dec- 2,1d1f
single pulse decoupled gated NOE

4h

051209-cha1565H-single_pulse-21d1
single_pulse

051209-dich56C-singl pulse dec-2,1d1f
single pulse decoupled gated NOE

single pulse decoupled gated NOE

050629- chcl4SH- single pulse- 2.jdr
single pulse

050622-dich437C-single pulse dec-2.jif
single pulse decoupled gated NOE

060116-ctct6t4c-singl pulse dec-2,1d1f
single pulse decoupled gated NOE

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050902-dich400H—single pulse-2.jdr
single_pulse

4m

4c

000120 - dcd609C - single pulse dec-2,3,4f
single pulse decoupled gated NOE

4c

051202-dhds3H-single pulse-2.jdr
single_pulse

3d

051202-dich25C-single pulse dec copy-2.jif
single pulse decoupled gated NOE

3d

000510- chcl3-4h- single pulse- 2Jdtf
single pulse

006510 - dich484C - single pulse dec-2,1dF
single pulse decoupled gated NOE

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