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Supporting Information

Vinylogous Nicholas Reactions in the Synthesis of Bi- and Tricyclic Cycloheptynedicobalt Complexes

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[(3,5-Dimethoxyphenyl)ethynyl]trimethylsilane (7'd)

To a mixture of Pd(PPh₃)₄ (0.6131 g, 0.531 mmol, 3 mol%) and CuI (0.1684 g, 0.884 mmol, 5 mol%) was added a solution of 1-bromo-3,5-dimethoxybenzene (3.8393 g, 17.684 mmol) in THF (12 mL), followed by trimethylsilylacetylene (5.0 mL, 35 mmol). Triethylamine (118 mL) was added and the mixture stirred for 20 h. The mixture was filtered through Celite® and subjected to a conventional extractive workup (Et₂O). Following flash chromatography (15:1 hexanes:Et₂O), compound **7'd** was isolated as a colourless solid (3.6129 g, 15.433 mmol, 87%), mp. 62-63 °C (lit., 61-65 °C¹), and which was characterized as spectroscopically identical to reported values.¹

2-(Phenylethynyl)cyclohex-1-enecarbaldehyde (9b)

Compound **9b** was synthesized from phenylacetylene (0.2773 g, 2.717 mmol) and 2-bromocyclohex-1-ene-1-carbaldehyde (**8b**)² (0.7134 g, 4.076 mmol) according to General Procedure A at a reaction temperature of 80 °C using an oil bath. The product was isolated using preparative TLC (25:1 hexanes:Et₂O) as a yellow oil (0.4687 g, 2.231 mmol, 82%). 1 H-NMR (500 MHz, CDCl₃): 10.32 (s, 1H), 7.47-7.49 (m, 2H), 7.35-7.37 (m, 3H), 2.52 (t, 2H, J = 6.1), 2.31 (t, 2H, J = 6.2), 1.66-1.75 (m, 4H); 13 C-NMR (75 MHz, CDCl₃): 193.0, 142.7, 140.1, 131.8, 129.2, 128.6, 122.4, 98.7, 86.4, 32.5, 22.2, 22.0, 21.2; IR (KBr): 2934, 2835, 2199, 1673, 1604, 1223; HRMS: m/e for C₁₅H₁₄O calculated 210.1045 (M⁺), found 210.1045.

2-[(3,4-Dimethoxyphenyl)ethynyl]cyclohex-1-enecarbaldehyde (9d)

Compound 7c³ (0.8136 g, 5.020 mmol) was subjected to Sonogashira conditions according to General Procedure A with 2-bromocyclohex-1-ene-1-carbaldehyde (8b) (1.4306 g, 7.5302

mmol). Compound **9d** was isolated as a yellow oil (1.0838 g, 4.0122 mmol, 80%) via flash chromatography (10:1 hexanes:Et₂O), and was characterized as spectroscopically identical to reported values.⁴

2-[(3,5-Dimethoxyphenyl)ethynyl]cyclohex-1-enecarbaldehyde (9f)

Compound **7'd** (0.8926 g, 3.813 mmol) was subjected to a tandem desilylation/Sonogashira reaction according to General Procedure B with 2-bromocyclohex-1-ene-1-carbaldehyde (**8b**) (1.0866 g, 5.7192 mmol). The coupled product (**9f**) was isolated as a yellow oil (0.9252 g, 3.425 mmol, 90%) following flash chromatography (10:1 hexanes:Et₂O). 1 H-NMR (500 MHz, CDCl₃): 10.30 (s, 1H), 6.60 (d, 2H, J = 2.2), 6.47 (t, 1H, J = 2.1), 3.78 (s, 6H), 2.50 (t, 2H, J = 6.1), 2.29 (t, 2H, J = 6.1), 1.64-1.73 (m, 4H); 13 C-NMR (75 MHz, CDCl₃): 192.9, 160.7, 142.9, 139.9, 123.6, 109.5, 102.6, 98.6, 85.8, 55.6, 32.4, 22.2, 22.0, 21.1; IR (KBr): 3001, 2937, 2838, 2197, 1672, 1594, 1421, 1208; HRMS: m/e for $C_{17}H_{18}O_{3}$ calculated 270.1256 (M⁺), found 270.1251.

2-[(2,5-Dimethoxyphenyl)ethynyl]cyclopent-1-enecarbaldehyde (9g)

Compound $7e^5$ (0.6523 g, 4.025 mmol) was subjected to Sonogashira chemistry according to General Procedure A with 2-bromocyclopent-1-ene-1-carbaldehyde (8a) (1.0567 g, 6.0373 mmol). Product 9g was isolated as a cream-coloured solid (0.8184 g, 3.196 mmol, 79%) following flash chromatography (10:1 hexanes:Et₂O). mp. 109-110 °C; ¹H-NMR (500 MHz, CDCl₃): 10.21 (s, 1H), 6.97 (d, 1H, J = 3.0), 6.92 (dd, 1H, J = 9.0, J = 3.1), 6.83 (d, 1H, J = 9.1), 3.85 (s, 3H), 3.78 (s, 3H), 2.82 (t, 2H, J = 7.8), 2.66 (t, 2H, J = 7.8), 2.01 (apparent pentet, 2H, J = 7.8); ¹³C-NMR (75 MHz, CDCl₃): 189.6, 155.1, 153.3, 148.1, 143.5, 117.9, 117.3, 112.1, 111.8, 97.4, 87.5, 56.5, 56.0, 38.9, 29.7, 22.4; IR (KBr): 2960, 2834, 2193, 1667, 1500, 1238; HRMS: m/e for C₁₆H₁₆O₃ calculated 256.256.1099 (M⁺), found 256.1087.

5-Iodo-1-isopropyl-2,3-dimethoxybenzene

2-(2,3-Dimethoxyphenyl)propan-2-ol ⁶ (5.5598 g, 28.332 mmol), I₂ (4.3145 g, 16.999 mmol) and (diacetoxyiodo)benzene (10.0383 g, 31.166 mmol) were ground together using a mortar and pestle for 30 min, and left for 72 h. The mixture was dissolved in a mixture of CH₂Cl₂ and Na₂S₂O₃ (aq, sat) and given a conventional extractive workup. Following Kugelrohr distillation at 0.1 Torr, the crude iodinated and dehydrated 5-iodo-1,2-dimethoxy-3-(prop-1-en-2-yl)benzene was dissolved in methanol (150 mL) along with Wilkinson's catalyst (0.5700 g, 0.6161 mmol). H₂ was bubbled through the solution, which was stirring at room temperature. Starting material

consumption was complete after 1.5 days, as assessed by 1 H-NMR spectroscopy. The solvent was removed under reduced pressure, and Kugelrohr distillation at 0.1 Torr afforded 5-Iodo-1-isopropyl-2,3-dimethoxybenzene as a pale yellow oil (7.4897 g, 24.475 mmol, 86%). 1 H-NMR (500 MHz, CDCl₃): 7.16 (d, 1H, J = 1.8), 7.04 (d, 1H, J = 1.8), 3.84 (s, 3H), 3.80 (s, 3H), 3.29 (septet, 1H, J = 7.1), 1.20 (d, 6H, J = 7.1); 13 C-NMR (125 MHz, CDCl₃): 153.4, 146.4, 144.7, 127.8, 119.1, 87.3, 61.0, 56.0, 26.7, 23.4; IR (KBr): 2962, 2870, 2004, 1568, 1479, 1291, 1218; HRMS: m/e for $C_{11}H_{15}IO_{2}$ calculated 306.0117 (M $^{+}$), found 306.0122.

$[(3\hbox{-}Isopropyl\hbox{-}4,5\hbox{-}dimethoxyphenyl) ethynyl] trimethyl silane$

[(3-Isopropyl-4,5-dimethoxyphenyl)ethynyl]trimethylsilane was synthesized from 5-iodo-1-isopropyl-2,3-dimethoxybenzene (2.2563 g, 7.3732 mmol) and trimethylsilylacetylene (2.1 mL, 15 mmol) according to General Procedure A at room temperature, with the exception that THF as solvent was substituted for DMF. The target compound was isolated by flash chromatography (10:1 hexanes:Et₂O) as a pale yellow oil (2.0005 g, 7.2441 mmol, 98%). 1 H-NMR (500 MHz, CDCl₃): 7.00 (d, 1H, J = 1.8), 6.87 (d, 1H, J = 1.8), 3.86 (s, 3H), 3.82 (s, 3H), 3.32 (septet, 1H, J = 6.9), 1.21 (d, 6H, J = 6.94), 0.27 (s, 9H); 13 C-NMR (125 MHz, CDCl₃): 152.2, 147.2, 142.5, 122.8, 118.9, 113.2, 105.5, 92.6, 61.0, 55.8, 26.8, 23.3, 0.08; IR (KBr): 2962, 2152, 1573, 1484, 1317, 1250; HRMS: m/e for C₁₆H₂₄O₂Si calculated 276.1546 (M⁺), found 276.1542.

5-Ethynyl-1-isopropyl-2,3-dimethoxybenzene (7h)

[(3-Isopropyl-4,5-dimethoxyphenyl)ethynyl]trimethylsilane (2.0005 g, 7.2441 mmol) was subjected to desilylation according to General Procedure H. The product was isolated via flash chromatography (10:1 hexanes:Et₂O) as a pale yellow oil (1.3196 g, 6.4650 mmol, 89%). 1 H-NMR (500 MHz, CDCl₃): 7.03 (d, 1H, J = 1.8), 6.89 (d, 1H, J = 1.9), 3.85 (s, 3H), 3.83 (s, 3H), 3.33 (septet, 1H, J = 6.9), 3.03 (s, 1H), 1.21 (d, 6H, J = 6.9); 13 C-NMR (125 MHz, CDCl₃): 152.3, 147.4, 142.6, 123.0, 117.4, 113.4, 84.1, 75.9, 61.0, 55.8, 26.7, 23.3; IR (KBr): 3286, 2962, 2830, 2107, 1577, 1316, 1224; HRMS: m/e for $C_{13}H_{16}O_{2}$ calculated 204.1150 (M⁺), found 204.1145.

2-[(2,5-Dimethoxyphenyl)ethynyl|cyclohex-1-enecarbaldehyde (9h)

Compound **7e** (0.7563 g, 4.666 mmol) was subjected to Sonogashira chemistry according to General Procedure A with 2-bromocyclohex-1-ene-1-carbaldehyde (**8b**) (1.3299 g, 6.9998 mmol). Product **9h** was isolated as a pale yellow solid (1.0691 g, 3.9578 mmol, 85%) following flash chromatography (10:1 hexanes:Et₂O). mp. 75-76 °C; ¹H-NMR (500 MHz, CDCl₃): 10.38

(s, 1H), 6.96 (d, 1H, J = 3.0), 6.90 (dd, 1H, J = 9.0, J = 3.0), 6.83 (d, 1H, J = 9.0), 3.85 (s, 3H), 3.78 (s, 3H), 2.54 (t, 2H, J = 5.8), 2.31 (t, 2H, J = 5.9), 1.66-1.75 (m, 4H); 13 C-NMR (75 MHz, CDCl₃): 193.7, 155.0, 153.3, 142.6, 140.2, 117.8, 116.8, 112.1, 95.1, 90.6, 56.5, 55.9, 32.3, 22.2, 22.0, 21.2; IR (KBr): 2999, 2937, 2834, 2195, 1670, 1499, 1226, 1214; HRMS: m/e for $C_{17}H_{18}O_3$ calculated 270.1256 (M), found 270.1250.

2-[(2,5-Dimethoxyphenyl)ethynyl]cyclohept-1-enecarbaldehyde (9i)

Compound **7e** (0.3400 g, 2.098 mmol) was subjected to Sonogashira chemistry according to General Procedure A with 2-bromocyclohept-1-ene-1-carbaldehyde (**8c**)² (0.6390 g, 3.147 mmol). Product **9i** was isolated as a yellow oil (0.4406 g, 1.551 mmol, 74%) following preparative TLC (10:1 hexanes:Et₂O). ¹H-NMR (500 MHz, CDCl₃): 10.34 (s, 1H), 6.94 (d, 1H, J = 3.0), 6.88 (dd, 1H, J = 8.9, J = 3.1), 6.80 (d, 1H, J = 9.1), 3.83 (s, 3H), 3.76 (s, 3H), 2.69-2.72 (m, 2H), 2.52-2.54 (m, 2H), 1.81 (apparent pentet, 2H, J = 5.9), 1.68 (apparent pentet, 2H, J = 5.7), 1.46 (apparent pentet, 2H, J = 6.0); ¹³C-NMR (125 MHz, CDCl₃): 193.0, 154.9, 153.2, 148.2, 145.9, 117.6, 116.9, 112.0, 111.9, 96.8, 92.0, 56.4, 55.8, 37.4, 32.3, 25.8, 24.3; IR (KBr): 2999, 2922, 2852, 2188, 1667, 1499, 1221; HRMS: m/e for $C_{18}H_{20}O_3$ calculated 284.1412 (M⁺), found 284.1412.

2-[(2,3,4-Trimethoxyphenyl)ethynyl]cyclopent-1-enecarbaldehyde (9j)

Compound $7\mathbf{f}^7$ (0.9223 g, 4.802 mmol) was subjected to Sonogashira coupling with 2-bromocyclopent-1-ene-1-carbaldehyde (**8a**) (1.2606 g, 7.2025 mmol) according to General Procedure A. Flash chromatography (2:1 hexanes:Et₂O) afforded the product (**9j**) as a pale yellow solid (1.1881 g, 4.1524 mmol, 86%). mp. 74-76 °C; ¹H-NMR (500 MHz, CDCl₃): 10.19 (s, 1H), 7.17 (d, 1H, J = 9.0), 6.66 (d, 1H, J = 9.0), 3.98 (s, 3H), 3.89 (s, 3H), 3.88 (s, 3H), 2.81 (t, 2H, J = 7.9), 2.66 (t, 2H, J = 7.9), 2.01 (apparent pentet, 2H, J = 7.9); ¹³C-NMR (75 MHz, CDCl₃): 189.2, 155.6, 155.3, 147.3, 143.8, 142.4, 128.5, 109.4, 107.6, 97.6, 86.4, 61.5, 61.2, 56.2, 39.0, 29.7, 22.3; IR (KBr): 2942, 2841, 2191, 1668, 1496, 1295; HRMS: m/e for C₁₇H₁₈O₄ calculated 286.1205 (M⁺), found 286.1214.

2-[(2,3,4-Trimethoxyphenyl)ethynyl]cyclohex-1-enecarbaldehyde (9k)

Compound **7f** (1.2647 g, 6.5843 mmol) was subjected to Sonogashira conditions with 2-bromocyclohex-1-ene-1-carbaldehyde (**8b**) (1.8764 g, 9.8764 mmol) according to General Procedure A. Coupled product **9k** was isolated via flash chromatography (2:1 hexanes:Et₂O) for the last purification step. The product was obtained as a yellow solid (1.6237 g, 5.4099 mmol,

82%). mp. 119-120 °C; 1 H-NMR (300 MHz, CDCl₃): 10.32 (s, 1H), 7.12 (d, 1H, J = 8.7), 6.63 (d, 1H, J = 8.7), 3.95 (s, 3H), 3.86 (s, 3H), 3.85 (s, 3H), 2.51 (m, 2H), 2.28 (m, 2H), 1.62-1.74 (m, 4H); 13 C-NMR (75 MHz, CDCl₃): 193.3, 155.2, 155.1, 142.3, 142.0, 140.5, 128.2, 109.6, 107.6, 95.3, 89.3, 61.5, 61.2, 56.2, 32.4, 22.2, 22.0, 21.2; IR (KBr): 2937, 2190, 1668, 1494, 1276; HRMS: m/e for $C_{18}H_{20}O_{4}$ calculated 300.1362 (M⁺), found 300.1355.

2-[(3,4,5-Trimethoxyphenyl)ethynyl]cyclopent-1-enecarbaldehyde (91)

Compound $7g^8$ (0.2514 g, 1.309 mmol) was subjected to General Procedure A with 2-bromocyclopent-1-ene-1-carbaldehyde (8a) (0.3436 g, 1.963 mmol). Preparative TLC (2:1 hexanes:Et₂O) afforded the product as a yellow solid (0.3109 g, 1.087 mmol, 83%). mp. 131-133 °C; ¹H-NMR (500 MHz, CDCl₃): 10.15 (s, 1H), 6.71 (s, 2H), 3.85 (s, 9H), 2.79 (t, 2H, J = 7.8), 2.64 (t, 2H, J = 7.8), 1.99 (apparent pentet, 2H, J = 7.9); ¹³C-NMR (125 MHz, CDCl₃): 189.0, 153.3, 148.0, 143.3, 139.9, 117.0, 109.2, 101.0, 82.6, 61.1, 56.3, 39.0, 29.7, 22.3; IR (KBr): 2941, 2834, 2192, 1661, 1239; HRMS: m/e for $C_{17}H_{18}O_4$ calculated 286.1205 (M⁺), found 286.1206.

2-[(3,4,5-Trimethoxyphenyl)ethynyl]cyclohex-1-enecarbaldehyde (9m)

Compound **7g** (0.3386 g, 1.763 mmol) was subjected to General Procedure A with 2-bromocyclohex-1-ene-1-carbaldehyde (**8b**) (0.5024 g, 2.644 mmol). Preparative TLC (2:1 hexanes:Et₂O) was used to isolate **9m** as a cream-coloured solid (0.4510 g, 1.503 mmol, 85%). mp. 121-123 °C; ¹H-NMR (500 MHz, CDCl₃): 10.31 (s, 1H), 6.70 (s, 2H), 3.87 (s, 9H), 2.52 (t, 2H, J = 5.9), 2.31 (t, 2H, J = 5.9), 1.67-1.74 (m, 4H); ¹³C-NMR (75 MHz, CDCl₃): 193.0, 153.3, 142.6, 140.2, 139.6, 117.4, 109.0, 98.8, 85.6, 61.1, 56.3, 32.5, 22.2, 22.0, 21.2; IR (KBr): 2933, 2195, 1664, 1238; HRMS: m/e for $C_{18}H_{20}O_4$ calculated 300.1362 (M⁺), found 300.1361.

2-[(3,4,5-Trimethoxyphenyl)ethynyl]cyclohept-1-enecarbaldehyde (9n)

Compound **7g** (0.2422 g, 1.261 mmol) was subjected to General Procedure A with 2-bromocyclohept-1-ene-1-carbaldehyde (**8c**) (0.3841 g, 1.891 mmol). Preparative TLC (2:1 hexanes:Et₂O) isolated the product as a cream-coloured solid (0.3408 g, 1.085 mmol, 86%). mp. 115-116 °C; 1 H-NMR (500 MHz, CDCl₃): 10.29 (s, 1H), 6.70 (s, 2H), 3.87 (s, 9H), 2.69-2.71 (m, 2H), 2.53-2.55 (m, 2H), 1.83 (apparent pentet, 2H, J = 5.2), 1.68 (apparent pentet, 2H, J = 5.3), 1.47 (apparent pentet, 2H, J = 5.3); 13 C-NMR (75 MHz, CDCl₃): 192.4, 153.3, 148.3, 145.9, 139.8, 117.4, 109.0, 100.6, 87.0, 68.1, 61.1, 56.3, 37.6, 32.3, 25.9, 24.4; IR (KBr): 2924, 2850, 2185, 1668, 1575, 1503, 1238; HRMS: m/e for C₁₉H₂₂O₄ calculated 314.1518 (M⁺), found

314.1526.

2-[(3-Isopropyl-4,5-dimethoxyphenyl)ethynyl]cyclohex-1-enecarbaldehyde (90)

Compound **7h** (1.3196 g, 6.4650 mmol) was subjected to Sonogashira conditions according to General Procedure A with 2-bromocyclohex-1-ene-1-carbaldehyde (**8b**) (1.8424 g, 9.6975 mmol). Compound **9o** was isolated via flash chromatography (10:1 hexanes:Et₂O) as a yellow oil (1.7456 g, 5.5918 mmol, 86%). 1 H-NMR (500 MHz, CDCl₃): 10.31 (s, 1H), 6.97 (d, 1H, J = 1.7), 6.83 (d, 1H, J = 1.7), 3.84 (s, 3H), 3.82 (s, 3H), 3.31 (septet, 1H, J = 7.0), 2.50 (t, 2H, J = 6.1), 2.28 (t, 2H, J = 6.1), 1.63-1.72 (m, 4H), 1.19 (d, 6H, J = 7.1); 13 C-NMR (125 MHz, CDCl₃): 193.0, 152.4, 147.7, 142.8, 142.2, 140.3, 122.6, 117.6, 112.8, 99.2, 85.2, 61.0, 55.8, 32.4, 26.8, 23.3, 22.1, 21.9, 21.1; IR (KBr): 2936, 2868, 2192, 1673, 1484, 1323, 1226; HRMS: m/e for $C_{20}H_{24}O_{3}$ calculated 312.1725 (M⁺), found 312.1727.

2-(Thiophen-3-ylethynyl)cyclohex-1-enecarbaldehyde (9p)

3-(Trimethylsilylethynyl)thiophene⁹ (**7'i**) (0.3651 g, 2.028 mmol) was subjected to General Procedure B with 2-bromocyclohex-1-ene-1-carbaldehyde (**8b**) (0.5779 g, 3.042 mmol). The reaction flask was placed in an oil bath set to 75 °C for the overnight (20 h) portion of the reaction. The product (**9p**) (0.3511 g, 1.625 mmol, 80%) was isolated via preparative TLC (15:1 hexanes:Et₂O) as a yellow oil. ¹H-NMR (500 MHz, CDCl₃): 10.29 (s, 1H), 7.53 (d, 1H, J = 2.1), 7.32 (dd, 1H, J = 5.0, J = 3.1), 7.15 (d, 1H, J = 5.4), 2.51 (t, 2H, J = 6.1), 2.31 (t, 2H, J = 6.2), 1.66-1.75 (m, 4H); ¹³C-NMR (75 MHz, CDCl₃): 193.0, 142.6, 140.1, 129.8, 129.7, 125.9, 121.5, 93.8, 86.1, 32.4, 22.2, 22.0, 21.2; IR (KBr): 3320, 3106, 2936, 2861, 2834, 2201, 1668, 1596; HRMS: m/e for $C_{13}H_{12}OS$ calculated 216.0609 (M⁺), found 216.0616.

[2-(Phenylethynyl)cyclohex-1-enyl]methyl acetate (10b)

Compound **9b** (0.4687 g, 2.231 mmol) was subjected to reduction and acetylation according to General Procedure C. Product **10b** was isolated via preparative TLC (15:1 hexanes:Et₂O) as a yellow oil (0.4889 g, 1.924 mmol, 86%). ¹H-NMR (500 MHz, CDCl₃): 7.43-7.45 (m, 2H), 7.29-7.33 (m, 3H), 4.90 (s, 2H), 2.31 (m, 2H), 2.18 (m, 2H), 2.10 (s, 3H), 1.66-1.71 (m, 4H); ¹³C-NMR (75 MHz, CDCl₃): 171.3, 139.1, 131.6, 128.4, 128.2, 123.5, 120.1, 93.2, 88.2, 66.7, 30.3, 27.2, 22.3, 22.1, 21.1; IR (KBr): 3058, 2934, 2861, 1740, 1228; HRMS: m/e for C₁₇H₁₈O₂ calculated 254.1307 (M⁺), found 254.1302.

[2-((3,4-Dimethoxyphenyl)ethynyl)cyclohex-1-enyl|methyl acetate (10d)

Compound 9d (1.0838 g, 4.0122 mmol) was subjected to General Procedure C. The product 10d

was isolated via flash chromatography (5:1 hexanes:Et₂O), as a pale yellow oil (1.1207 g, 3.5674 mmol, 89%). 1 H-NMR (500 MHz, CDCl₃): 7.03 (dd, 1H, J = 8.2, J = 1.9), 6.93 (d, 1H, J = 1.9), 6.79 (d, 1H, J = 8.3), 4.89 (s, 2H), 3.88 (s, 6H), 2.29 (m, 2H), 2.16 (m, 2H), 2.08 (s, 3H), 1.63-1.70 (m, 4H); 13 C-NMR (125 MHz, CDCl₃): 171.2, 149.3, 148.6, 138.4, 124.7, 120.1, 115.7, 114.1, 111.0, 93.2, 86.7, 66.6, 56.0, 55.9, 30.3, 27.0, 22.2, 22.0, 21.0; IR (KBr): 2934, 2837, 1737, 1514, 1247; HRMS: m/e for $C_{19}H_{22}O_4$ calculated 314.1518 (M⁺), found 314.1513.

[2-((3,5-Dimethoxyphenyl)ethynyl)cyclopent-1-enyl|methyl acetate (10e)

Compound **9e** (1.0474 g, 4.0896 mmol) was subjected to reduction and acetylation according to General Procedure C. Product **10e** was isolated as a yellow oil (1.0852 g, 3.6157 mmol, 88%) following flash chromatography (5:1 hexanes:Et₂O). ¹H-NMR (300 MHz, CDCl₃): 6.58 (d, 2H, J = 2.3), 6.41 (t, 1H, J = 2.3), 4.86 (s, 2H), 3.76 (s, 6H), 2.60 (t, 2H, J = 7.5), 2.49 (t, 2H, J = 7.5), 2.07 (s, 3H), 1.93 (apparent pentet, 2H, J = 7.57); ¹³C-NMR (75 MHz, CDCl₃): 170.9, 160.6, 145.0, 124.6, 122.7, 109.2, 101.8, 94.9, 84.2, 61.9, 55.4, 37.0, 34.2, 22.4, 20.8; IR (KBr): 3002, 2842, 2202, 1741, 1595, 1420, 1231; HRMS: m/e for $C_{18}H_{20}O_4$ calculated 300.1362 (M⁺), found 300.1357.

[2-((3,5-Dimethoxyphenyl)ethynyl)cyclohex-1-enyl|methyl acetate (10f)

Compound **9f** (0.9252 g, 3.425 mmol) was subjected to reduction and acetylation according to General Procedure C. Product **10f** was isolated as a yellow oil (0.9761 g, 3.107 mmol, 91%) following flash chromatography (5:1 hexanes:Et₂O). ¹H-NMR (500 MHz, CDCl₃): 6.58 (d, 2H, J = 2.3), 6.42 (t, 1H, J = 2.3), 4.88 (s, 2H), 3.78 (s, 6H), 2.30 (m, 2H), 2.16 (m, 2H), 2.09 (s, 3H), 1.64-1.70 (m, 4H); ¹³C-NMR (75 MHz, CDCl₃): 171.2, 160.6, 139.4, 124.8, 119.9, 109.2, 101.7, 93.2, 87.8, 66.6, 55.5, 30.2, 27.1, 22.2, 22.0, 21.0; IR (KBr): 3001, 2936, 2840, 2201, 1739, 1590, 1420, 1233; HRMS: m/e for $C_{19}H_{22}O_4$ calculated 314.1518 (M⁺), found 314.1519.

[2-((2,5-Dimethoxyphenyl)ethynyl)cyclopent-1-enyl|methyl acetate (10g)

Compound **9g** (0.8184 g, 3.196 mmol) was subjected to reduction and acetylation according to General Procedure C. Product **10g** was isolated as a colourless solid (0.8595 g, 2.864 mmol, 90%) following flash chromatography (7:1 hexanes:Et₂O). mp. 63-65 °C; ¹H-NMR (500 MHz, CDCl₃): 6.95 (d, 1H, J = 2.7), 6.84 (dd, 1H, J = 8.9, J = 2.8), 6.80 (d, 1H, J = 9.0), 4.92 (s, 2H), 3.84 (s, 3H), 3.77 (s, 3H), 2.64 (t, 2H, J = 7.8), 2.51 (t, 2H, J = 8.0), 2.09 (s, 3H), 1.96 (apparent pentet, 2H, J = 7.8); ¹³C-NMR (75 MHz, CDCl₃): 171.3, 154.5, 153.3, 145.0, 123.2, 117.8, 115.8, 113.1, 112.1, 91.2, 88.9, 62.2, 56.5, 55.9, 37.0, 34.2, 22.6, 21.0; IR (KBr): 3002, 2960,

2834, 1746, 1504, 1228; HRMS: m/e for $C_{18}H_{20}O_4$ calculated 300.1362 (M⁺), found 300.1340.

[2-(2,5-Dimethoxyphenyl)ethynyl)cyclohex-1-enyl|methyl acetate (10h)

Compound **9h** (0.6059 g, 2.243 mmol) was subjected to reduction and acetylation according to General Procedure C. Product **10h** was isolated as a yellow solid (0.6362 g, 2.025 mmol, 90%) following preparative TLC (7:1 hexanes:Et₂O). mp. 54-56 °C; ¹H-NMR (500 MHz, CDCl₃): 6.94 (d, 1H, J = 2.9), 6.82 (dd, 1H, J = 9.0, J = 2.9), 6.79 (d, 1H, J = 9.0), 4.95 (s, 2H), 3.84 (s, 3H), 3.77 (s, 3H), 2.33 (m, 2H), 2.17 (m, 2H), 2.09 (s, 3H), 1.64-1.70 (m, 4H); ¹³C-NMR (125 MHz, CDCl₃): 171.2, 154.2, 153.2, 139.1, 120.1, 117.6, 115.5, 113.2, 112.0, 92.4, 89.2, 66.8, 56.4, 55.8, 30.0, 27.0, 22.2, 22.0, 21.0; IR (KBr): 2935, 2835, 1737, 1500, 1234; HRMS: m/e for $C_{19}H_{22}O_4$ calculated 314.1518 (M⁺), found 314.1526.

[2-((2,5-Dimethoxyphenyl)ethynyl)cyclohept-1-enyl|methyl acetate (10i)

Compound **9i** (0.4406 g, 1.551 mmol) was subjected to reduction and acetylation according to General Procedure C. Product **10i** was isolated as a pale yellow oil (0.4327 g, 1.318 mmol, 85%) following preparative TLC (7:1 hexanes:Et₂O). ¹H-NMR (500 MHz, CDCl₃): 6.92 (d, 1H, J = 2.7), 6.80 (dd, 1H, J = 8.5, J = 2.5), 6.77 (d, 1H, J = 9.0), 4.96 (s, 2H), 3.82 (s, 3H), 3.75 (s, 3H), 2.49-2.51 (m, 2H), 2.30-2.32 (m, 2H), 2.08 (s, 3H), 1.78 (apparent pentet, 2H, J = 5.8), 1.61 (apparent pentet, 2H, J = 5.4), 1.52 (apparent pentet, 2H, J = 5.5); ¹³C-NMR (125 MHz, CDCl₃): 171.2, 154.1, 153.2, 145.2, 126.0, 117.4, 115.4, 113.3, 111.8, 94.0, 90.0, 68.1, 56.3, 55.8, 34.7, 32.4, 31.3, 26.2, 26.1, 21.1; IR (KBr): 2919, 2850, 1739, 1498, 1228; HRMS: m/e for C₂₀H₂₄O₄ calculated 328.1675 (M⁺), found 328.1683.

[2-((2,3,4-Trimethoxyphenyl)ethynyl)cyclopent-1-enyl]methyl acetate (10j)

Compound **9j** (1.1881 g, 4.1524 mmol) was treated according to General Procedure C. Flash chromatography (2:1 hexanes:Et₂O) afforded **10j** as a pale yellow oil (1.2573 g, 3.8083 mmol, 92%). 1 H-NMR (500 MHz, CDCl₃): 7.10 (d, 1H, J = 8.9), 6.60 (d, 1H, J = 8.9), 4.88 (s, 2H), 3.95 (s, 3H), 3.85 (s, 6H), 2.60 (t, 2H, J = 7.8), 2.49 (t, 2H, J = 7.9), 2.07 (s, 3H), 1.94 (apparent pentet, 2H, J = 7.8); 13 C-NMR (75 MHz, CDCl₃): 171.1, 154.7, 154.4, 144.1, 143.0, 128.0, 123.5, 110.6, 107.4, 91.0, 87.4, 62.1, 61.3, 61.2, 56.2, 37.0, 34.1, 22.5, 21.0; IR (KBr): 2940, 2841, 2199, 1739, 1490, 1226; HRMS: m/e for $C_{19}H_{22}O_{5}$ calculated 330.1467 (M⁺), found 330.1468.

[2-((2,3,4-Trimethoxyphenyl)ethynyl)cyclohex-1-enyl|methyl acetate (10k)

Compound 9k (0.8787 g, 2.928 mmol) was reacted according to General Procedure C. Product

10k was isolated as a yellow oil (0.8956 g, 2.600 mmol, 89%) via flash chromatography (2:1 hexanes:Et₂O). 1 H-NMR (500 MHz, CDCl₃): 7.10 (d, 1H, J = 8.8), 6.61 (d, 1H, J = 8.9), 4.93 (s, 2H), 3.96 (s, 3H), 3.87 (s, 6H), 2.32 (m, 2H), 2.17 (m, 2H), 2.09 (s, 3H), 1.65-1.70 (m, 4H); 13 C-NMR (75 MHz, CDCl₃): 171.3, 154.7, 154.3, 142.3, 138.4, 127.9, 120.4, 110.8, 107.4, 91.0, 89.2, 66.8, 61.3, 61.2, 56.2, 30.3, 27.1, 22.3, 22.1, 21.1; IR (KBr): 2940, 2839, 2196, 1746, 1494, 1234; HRMS: m/e for $C_{20}H_{24}O_{5}$ calculated 344.0624 (M⁺), found 344.0627.

1-[2-((2,3,4-Trimethoxyphenyl)ethynyl)cyclohex-1-enyl]ethyl acetate (10kk)

Compound **9k** (0.7450 g, 2.482 mmol) was subjected to General Procedure C, where DIBAL-H was substituted with MeLi (1.6 M in Et₂O, 3.1 mL, 5.0 mmol). The product (**10kk**) was isolated as a yellow/orange oil (0.7888 g, 2.202 mmol, 89%) following flash chromatography (2:1 hexanes:Et₂O). 1 H-NMR (500 MHz, CDCl₃): 7.12 (d, 1H, J = 8.6), 6.60 (d, 1H, J = 8.7), 6.13 (q, 1H, J = 6.5), 3.98 (s, 3H), 3.86 (s, 3H), 3.85 (s, 3H), 2.27 (m, 2H), 2.15 (m, 2H), 2.04 (s, 3H), 1.62-1.69 (m, 4H), 1.36 (d, 3H, J = 6.6); 13 C-NMR (75 MHz, CDCl₃): 170.2, 154.6, 154.2, 143.2, 142.3, 128.0, 117.0, 111.0, 107.4, 91.0, 89.7, 72.7, 61.4, 61.2, 56.2, 30.1, 23.4, 22.4, 22.1, 21.4, 18.8; IR (KBr): 2935, 2839, 2194, 1737, 1593, 1494, 1243; HRMS: m/e for C₂₁H₂₆O₅ calculated 358.1780 (M⁺), found 358.1777.

[2-((3,4,5-Trimethoxyphenyl)ethynyl)cyclopent-1-enyl]methyl acetate (10l)

Compound **91** (0.3099 g, 1.083 mmol) was subjected to General Procedure C. The product compound (**101**) was isolated as a yellow oil (0.3093 g, 0.9368 mmol, 89%) via preparative TLC (2:1 hexanes:Et₂O). ¹H-NMR (500 MHz, CDCl₃): 6.64 (s, 2H), 4.84 (s, 2H), 3.82 (s, 6H), 3.81 (s, 3H), 2.58 (t, 2H, J = 7.7), 2.47 (t, 2H, J = 7.8), 2.05 (s, 3H), 1.92 (apparent pentet, 2H, J = 7.7); ¹³C-NMR (75 MHz, CDCl₃): 171.0, 153.1, 144.7, 138.8, 122.8, 118.8, 108.0, 95.0, 83.8, 62.0, 61.0, 56.2, 37.1, 34.2, 22.4, 20.9; IR (KBr): 2940, 1741, 1503, 1234; HRMS: m/e for $C_{19}H_{22}O_5$ calculated 330.1467 (M⁺), found 330.1464.

[2-((3,4,5-Trimethoxyphenyl)ethynyl)cyclohex-1-enyl|methyl acetate (10m)

Compound **9m** (0.1994 g, 0.6644 mmol) was subjected to General Procedure C. The product compound (**10m**) was isolated as a yellow oil (0.2003 g, 0.5820 mmol, 88%) using preparative TLC (2:1 hexanes:Et₂O). 1 H-NMR (500 MHz, CDCl₃): 6.66 (s, 2H), 4.89 (s, 2H), 3.85 (s, 6H), 3.84 (s, 3H), 2.30 (m, 2H), 2.17 (m, 2H), 2.09 (s, 3H), 1.64-1.70 (m, 4H); 13 C-NMR (75 MHz, CDCl₃): 171.2, 153.2, 139.2, 138.8, 120.0, 118.6, 108.8, 93.3, 87.3, 66.7, 61.1, 56.3, 30.3, 27.2, 22.3, 22.1, 21.1; IR (KBr): 2938, 1737, 1576, 1504, 1237; HRMS: m/e for C₂₀H₂₄O₅ calculated

344.1624 (M⁺), found 344.1631.

Phenyl[2-((3,4,5-trimethoxyphenyl)ethynyl)cyclohex-1-enyl]methyl acetate (10mm)

Compound **9m** (0.2516 g, 0.8383 mmol) was subjected to General Procedure C, where DIBAL-H was substituted with PhMgBr (1.0 M in THF, 1.7mL, 1.7 mmol). The product **10mm** was isolated as a yellow oil (0.2826 g, 0.6725 mmol, 80%) following preparative TLC (2:1 hexanes:Et₂O). ¹H-NMR (500 MHz, CDCl₃): 7.43 (apparent d, 2H, J = 7.2), 7.37 (apparent t, 2H, J = 7.6), 7.29-7.32 (m, 1H), 7.27 (s, 1H), 6.77 (s, 2H), 3.90 (s, 6H), 3.89 (s, 3H), 2.33-2.41 (m, 2H), 2.22 (s, 3H), 1.92-1.98 (m, 2H), 1.55-1.73 (m, 4H); ¹³C-NMR (75 MHz, CDCl₃): 169.8, 153.2, 142.6, 139.5, 138.8, 128.4, 127.5, 125.7, 119.1, 118.7, 108.8, 93.5, 88.2, 76.3, 61.1, 56.3, 30.2, 23.8, 22.2, 22.0, 21.3; IR (KBr): 3062, 3004, 2939, 2839, 2197, 1731, 1574, 1505, 1411, 1234; HRMS: m/e for $C_{26}H_{28}O_{5}$ calculated 420.1937 (M⁺), found 420.1950.

[2-((3,4,5-Trimethoxyphenyl)ethynyl)cyclohept-1-enyl]methyl acetate (10n)

Compound **9n** (0.3408 g, 1.085 mmol) was subjected to General Procedure C. The product compound (**10n**) was isolated as a yellow oil (0.3498 g, 0.9766 mmol, 90%) via preparative TLC (2:1 hexanes:Et₂O). ¹H-NMR (500 MHz, CDCl₃): 6.66 (s, 2H), 4.90 (s, 2H), 3.86 (s, 6H), 3.85 (s, 3H), 2.48-2.50 (m, 2H), 2.31-2.33 (m, 2H), 2.10 (s, 3H), 1.79 (apparent pentet, 2H, J = 5.8), 1.61 (apparent pentet, 2H, J = 5.4), 1.53 (apparent pentet, 2H, J = 5.5); ¹³C-NMR (75 MHz, CDCl₃): 171.3, 153.4, 145.1, 138.8, 125.9, 118.7, 108.7, 94.0, 88.8, 68.0, 61.1, 56.3, 34.9, 32.4, 31.4, 26.3, 26.1, 21.1; IR (KBr): 2959, 2929, 2858, 1730, 1576, 1464, 1275; HRMS: m/e for $C_{21}H_{26}O_{5}$ calculated 358.1780 (M⁺), found 358.1776.

[2-((3-Isopropyl-4,5-dimethoxyphenyl)ethynyl)cyclohex-1-enyl|methyl acetate (100)

Compound **90** (1.7456 g, 5.5918 mmol) was subjected to General Procedure C. Product **100** was purified via flash chromatography (5:1 hexanes:Et₂O) as a pale yellow oil (1.7960 g, 5.0421 mmol, 90%). ¹H-NMR (500 MHz, CDCl₃): 6.93 (d, 1H, J = 1.5), 6.82 (d, 1H, J = 1.5), 4.89 (s, 2H), 3.84 (s, 3H), 3.81 (s, 3H), 3.30 (septet, 1H, J = 7.0), 2.30 (m, 2H), 2.16 (s, 3H), 2.08 (m, 2H), 1.64-1.70 (m, 4H), 1.20 (d, 6H, J = 7.0); ¹³C-NMR (125 MHz, CDCl₃): 171.2, 152.3, 146.8, 142.5, 138.6, 122.1, 120.0, 118.8, 112.7, 93.4, 86.9, 66.6, 61.0, 55.8, 30.3, 27.0, 26.8, 23.4, 22.2, 22.0, 21.0; IR (KBr): 2935, 2869, 2836, 2198, 1739, 1573, 1484, 1341, 1273; HR m/e for $C_{22}H_{28}O_4$ calculated 356.1988 (M⁺), found 356.1990.

[2-(Thiophen-3-ylethynyl)cyclohex-1-enyl]methyl acetate (10p)

Compound 9p (0.3511 g, 1.625 mmol) was subjected to reduction and acetylation according to

General Procedure C. The product (**10p**) was isolated as a yellow oil (0.3957 g, 1.521 mmol, 94%) via preparative TLC (10:1 hexanes:Et₂O). 1 H-NMR (500 MHz, CDCl₃): 7.42 (d, 1H, J = 2.2), 7.27 (dd, 1H, J = 4.8, J = 2.9), 7.11 (d, 1H, J = 5.0), 4.88 (s, 2H), 2.30 (m, 2H), 2.17 (m, 2H), 2.10 (s, 3H), 1.65-1.68 (m, 4H); 13 C-NMR (75 MHz, CDCl₃): 171.3, 139.0, 129.9, 128.3, 125.4, 122.5, 120.0, 88.2, 87.7, 66.7, 30.2, 27.1, 22.2, 22.1, 21.1; IR (KBr): 3108, 2934, 2860, 2205, 1738, 1233; HR-MS: m/e for C₁₅H₁₆O₂S calculated 260.0871 (M⁺), found 260.0876.

Hexacarbonyl[μ-η⁴(2-(phenylethynyl)cyclohex-1-enyl|methyl acetate)|dicobalt (1b)

Compound **10b** (0.4889 g, 1.924 mmol) was subjected to complexation procedures according to General Procedure D. Product **1b** was isolated as a dark brown solid (0.9272 g, 1.717 mmol, 89%) following flash chromatography (15:1 hexanes:Et₂O). 1 H-NMR (500 MHz, CDCl₃): 7.41 (apparent d, 2H, J = 7.1), 7.30-7.36 (m, 3H), 4.53 (s, 2H), 2.40 (t, 2H, J = 6.1), 2.14 (t, 2H, J = 6.2), 1.95 (s, 3H), 1.72-1.80 (m, 4H); 13 C-NMR (75 MHz, CDCl₃): 199.6, 170.8, 138.7, 133.4, 132.1, 129.3, 128.8, 127.8, 93.7, 91.7, 65.3, 33.3, 28.5, 23.4, 22.2, 20.9; IR (KBr): 2935, 2861, 2088, 2047, 2016, 1743, 1233; HRMS: m/e for $C_{23}H_{18}Co_{2}O_{8}$ calculated 483.9767 (M-2CO⁺), found 483.9759.

Hexacarbonyl[μ - η^4 (2-((3,4-dimethoxyphenyl)ethynyl)cyclohex-1-enyl)methyl acetate)|dicobalt (1d)

Compound **10d** (1.1207 g, 3.5674 mmol) was complexed according to General Procedure D. After washing the column of silica with 100% hexanes to remove excess, uncomplexed $Co_2(CO)_8$, the product **1d** was eluted using 5:1 hexanes: Et_2O , and isolated as a dark brown solid (2.0001 g, 3.3336 mmol, 93%). ¹H-NMR (500 MHz, CDCl₃): 7.04 (dd, 1H, J = 8.3, J = 2.0), 6.92 (d, 1H, J = 2.0), 6.84 (d, 1H, J = 8.4), 4.61 (s, 2H), 3.92 (s, 3H), 3.89 (s, 3H), 2.40 (t, 2H, J = 6.2), 2.14 (t, 2H, J = 6.0), 1.98 (s, 3H), 1.72-1.81 (m, 4H); ¹³C-NMR (75 MHz, CDCl₃): 199.6, 170.8, 149.0, 148.9, 133.4, 132.0, 130.7, 122.1, 112.5, 111.4, 94.0, 91.4, 65.3, 56.0, 55.9, 33.4, 28.3, 23.4, 22.2, 20.7; IR (KBr): 2935, 2834, 2086, 2045, 2014, 1742, 1509, 1228; HRMS: m/e for $C_{25}H_{22}Co_2O_{10}$ calculated 571.9928 (M-CO⁺), found 571.9925.

$Hexacarbonyl[\mu-\eta^4(2-((3,5-dimethoxyphenyl)ethynyl)cyclohex-1-enyl)methyl acetate)]$ dicobalt (1f)

Compound **10f** (0.9761 g, 3.107 mmol) was subjected to complexation according to General Procedure D. The complexed compound (**1f**) was isolated via flash chromatography (5:1 hexanes:Et₂O) following removal of excess, uncomplexed Co₂(CO)₈ with 100% hexanes. The

product was isolated as a dark brown solid (1.7135 g, 2.8559 mmol, 92%). 1 H-NMR (500 MHz, CDCl₃): 6.57 (d, 2H, J = 2.2), 6.41 (t, 1H, J = 2.2), 4.58 (s, 2H), 3.81 (s, 6H), 2.39 (t, 2H, J = 5.9), 2.13 (t, 2H, J = 5.9), 1.98 (s, 3H), 1.70-1.80 (m, 4H); 13 C-NMR (75 MHz, CDCl₃): 199.7, 170.8, 160.8, 140.8, 133.6, 131.9, 107.8, 99.6, 93.8, 91.6, 65.2, 55.4, 33.3, 28.5, 23.4, 22.2, 20.7; IR (KBr): 2937, 2836, 2089, 2012, 1740, 1590, 1421, 1234; HRMS: m/e for $C_{25}H_{22}Co_{2}O_{10}$ calculated 543.9979 (M-2CO $^{+}$), found 543.9975.

Hexacarbonyl[μ - η^4 (2-((2,5-dimethoxyphenyl)ethynyl)cyclopent-1-enyl)methyl acetate)]dicobalt (1g)

Compound **10g** (0.8595 g, 2.864 mmol) was subjected to complexation according to General Procedure D. The complexed compound **1g** was isolated using flash chromatography (7:1 hexanes:Et₂O) following removal of excess, uncomplexed $Co_2(CO)_8$ with 100% hexanes. The product was isolated as a dark brown solid (1.5256 g, 2.6035 mmol, 91%). ¹H-NMR (500 MHz, CDCl₃): 7.05 (d, 1H, J = 3.0), 6.87 (dd, 1H, J = 8.8, J = 3.1), 6.77 (d, 1H, J = 9.0), 4.59 (s, 2H), 3.80 (s, 3H), 3.74 (s, 3H), 2.76 (t, 2H, J = 7.8), 2.54 (t, 2H, J = 7.8), 2.0 (apparent pentet, 2H, J = 7.9), 1.98 (s, 3H); ¹³C-NMR (75 MHz, CDCl₃): 199.8, 170.9, 153.6, 150.6, 137.7, 137.0, 127.3, 117.3, 113.7, 110.5, 89.0, 88.0, 61.2, 55.8, 54.6, 39.8, 36.2, 22.1, 20.8; IR (KBr): 2959, 2835, 2087, 2047, 2014, 1746, 1494, 1223; HRMS: m/e for $C_{24}H_{20}Co_2O_{10}$ calculated 529.9822 (M-2CO⁺), found 529.9818.

Hexacarbonyl[μ - η^4 (2-(2,5-dimethoxyphenyl)ethynyl)cyclohex-1-enyl)methyl acetate)] dicobalt (1h)

Compound **10h** (0.6362 g, 2.025 mmol) was subjected to complexation according to General Procedure D. The complexed compound (**1h**) was eluted via flash chromatography (7:1 hexanes:Et₂O) following removal of excess, uncomplexed $Co_2(CO)_8$ with 100% hexanes. The product was isolated as a dark brown solid (1.0050 g, 1.6750 mmol, 83%). ¹H-NMR (500 MHz, CDCl₃): 7.03 (d, 1H, J = 2.9), 6.85 (dd, 1H, J = 8.8, J = 3.0), 6.74 (d, 1H, J = 8.9), 4.50 (s, 2H), 3.80 (s, 3H), 3.72 (s, 3H), 2.37 (m, 2H), 2.11 (m, 2H), 1.95 (s, 3H), 1.70-1.75 (m, 4H); ¹³C-NMR (75 MHz, CDCl₃): 199.9, 171.0, 153.6, 150.3, 133.2, 132.5, 127.7, 117.4, 113.6, 110.4, 95.0, 89.9, 65.1, 55.8, 54.6, 33.1, 28.3, 23.5, 22.4, 20.9; IR (KBr): 2938, 2834, 2086, 2049, 2016, 1740, 1490, 1228; HR-MS: m/e for $C_{25}H_{22}Co_2O_{10}$ calculated 543.9979 (M-2CO⁺), found 543.9979.

$Hexacarbonyl[\mu-\eta^4(2\text{-}((2,5\text{-}dimethoxyphenyl)ethynyl)cyclohept-1-enyl)methyl$

acetate)|dicobalt (1i)

Compound **10i** (0.4327 g, 1.318 mmol) was subjected to complexation according to General Procedure D. The complexed compound (**1i**) was isolated via flash chromatography (5:1 hexanes:Et₂O) following removal of excess, uncomplexed $Co_2(CO)_8$ with 100% hexanes. The product was isolated as a dark brown-green solid (0.6867 g, 1.118 mmol, 85%). ¹H-NMR (500 MHz, CDCl₃): 7.01 (d, 1H, J = 3.0), 6.86 (dd, 1H, J = 8.9, J = 3.1), 6.75 (d, 1H, J = 8.9), 4.52 (s, 2H), 3.81 (s, 3H), 3.74 (s, 3H), 2.60-2.62 (m, 2H), 2.30-2.32 (m, 2H), 1.95 (s, 3H), 1.82 (apparent pentet, 2H, J = 5.8), 1.54-1.63 (m, 4H); ¹³C-NMR (75 MHz, CDCl₃): 199.9, 171.0, 153.6, 150.2, 139.2, 138.8, 127.8, 117.3, 113.6, 110.3, 96.0, 91.1, 65.7, 55.8, 54.6, 37.6, 32.7, 32.5, 26.7, 26.4, 21.0; IR (KBr): 2926, 2852, 2085, 2058, 2013, 1742, 1486, 1222; HRMS: m/e for $C_{26}H_{24}Co_2O_{10}$ calculated 558.0106 (M-2CO⁺), found 558.0117.

Hexacarbonyl[μ - η^4 (2-((2,3,4-trimethoxyphenyl)ethynyl)cyclopent-1-enyl)methyl acetate)]dicobalt (1j)

Compound **10j** (1.0012 g, 3.0326 mmol) was subjected to complexation according to General Procedure D. After washing excess, uncomplexed $Co_2(CO)_8$ off a column of silica, the product (**1j**) was eluted using 2:1 hexanes:Et₂O as a dark brown solid (1.6176 g, 2.6260 mmol, 87%). ¹H-NMR (500 MHz, CDCl₃): 7.16 (d, 1H, J = 8.8), 6.64 (d, 1H, J = 8.9), 4.65 (s, 2H), 3.96 (s, 3H), 3.90 (s, 3H), 3.85 (s, 3H), 2.76 (t, 2H, J = 7.8), 2.56 (t, 2H, J = 7.8), 2.00 (apparent pentet, 2H, J = 7.8), 1.99 (s, 3H); ¹³C-NMR (75 MHz, CDCl₃): 199.9, 171.0, 154.4, 151.0, 141.1, 138.0, 136.5, 126.6, 122.9, 106.6, 89.7, 87.5, 61.3, 60.9, 60.2, 56.1, 40.2, 36.2, 22.1, 20.8; IR (KBr): 2944, 2844, 2086, 2045, 2014, 1742, 1486, 1231; HRMS: m/e for $C_{25}H_{22}Co_2O_{11}$ calculated 448.0131 (M-6CO⁺), found 448.0139.

Hexacarbonyl[μ - η^4 (2-((2,3,4-trimethoxyphenyl)ethynyl)cyclohex-1-enyl)methyl acetate)]dicobalt (1k)

Compound **10k** (0.8956 g, 2.600 mmol) was subjected to complexation according to General Procedure D. The complexed product (**1k**) was isolated as a dark green solid (1.5445 g, 2.4516 mmol, 94%) following flash chromatography (2:1 hexanes:Et₂O). ¹H-NMR (500 MHz, CDCl₃): 7.14 (d, 1H, J = 9.0), 6.64 (d, 1H, J = 9.0), 4.53 (s, 2H), 3.95 (s, 3H), 3.90 (s, 3H), 3.84 (s, 3H), 2.39 (t, 2H, J = 6.0), 2.13 (t, 2H, J = 6.1), 1.94 (s, 3H), 1.71-1.77 (m, 4H); ¹³C-NMR (75 MHz, CDCl₃): 200.1, 170.9, 154.4, 150.5, 140.8, 132.8, 132.4, 126.6, 123.0, 106.4, 94.4, 90.3, 65.3, 60.9, 60.0, 56.0, 33.3, 28.3, 23.5, 22.4, 20.8; IR (KBr): 2940, 2838, 2084, 2044, 2012, 1742,

1486, 1229; HRMS: m/e for C₂₆H₂₄Co₂O₁₁ calculated 462.0288 (M-6CO⁺), found 462.0298.

$Hexacarbonyl \\ [\mu-\eta^4-(1-[2-((2,3,4-trimethoxyphenyl)ethynyl)cyclohex-1-enyl]ethyl\\ acetate)] \\ dicobalt \\ l\ (1kk)$

Compound **10kk** (0.7888 g, 2.202 mmol) was subjected to complexation according to General Procedure D. Compound **1kk** (1.2896 g, 2.0024 mmol, 91%) was isolated as a dark green solid following flash chromatography (2:1 hexanes:Et₂O). ¹H-NMR (500 MHz, CDCl₃): 7.11 (d, 1H, J = 8.7), 6.64 (d, 1H, J = 8.7), 6.14 (q, 1H, J = 6.3), 3.96 (s, 3H), 3.90 (s, 3H), 3.83 (s, 3H), 2.15-2.33 (m, 4H), 1.93 (s, 3H), 1.60-1.75 (m, 4H), 1.26 (d, 3H, J = 6.3); ¹³C-NMR (75 MHz, CDCl₃): 200.1, 170.0, 154.2, 150.2, 140.9, 137.4, 130.2, 126.6, 123.6, 106.3, 93.2, 92.8, 70.6, 60.9, 60.0, 56.0, 33.0, 24.5, 23.6, 22.4, 21.3, 18.3; IR (KBr): 2938, 2839, 2084, 2046, 2016, 1737, 1485, 1241; HRMS: m/e for $C_{27}H_{26}Co_2O_{11}$ calculated 588.0241 (M-2CO⁺), found 588.0226.

Hexacarbonyl[μ - η^4 -(2-((3,4,5-trimethoxyphenyl)ethynyl)cyclopent-1-enyl)methyl acetate)]dicobalt (11)

Compound **10I** (0.3093 g, 0.9368 mmol) was complexed using General Procedure D to afford product **1I** (0.5156 g, 0.8370 mmol, 89%) as a dark brown solid following flash chromatography (1:1 hexanes:Et₂O). 1 H-NMR (500 MHz, CDCl₃): 6.71 (s, 2H), 4.72 (s, 2H), 3.90 (s, 3H), 3.88 (s, 6H), 2.81 (t, 2H, J = 7.8), 2.56 (t, 2H, J = 7.9), 2.04 (apparent pentet, 2H, J = 7.8), 2.02 (s, 3H); 13 C-NMR (75 MHz, CDCl₃): 199.5, 170.7, 153.3, 138.0, 137.7, 136.9, 133.7, 106.5, 93.4, 84.4, 61.2, 61.0, 56.2, 39.9, 36.2, 21.8, 20.7; IR (KBr): 2940, 2088, 2050, 2020, 1743, 1576, 1230; HRMS: m/e for $C_{25}H_{22}Co_2O_{11}$ calculated 559.9928 (M-2CO $^+$), found 559.9924.

$Hexacarbonyl[\mu-\eta^4-(2-((3,4,5-trimethoxyphenyl)ethynyl)cyclohex-1-enyl)methyl acetate)] dicobalt~(1m)$

Compound **10m** (0.2003 g, 0.5820 mmol) was complexed using General Procedure D to afford product **1m** (0.3162 g, 0.5019 mmol, 86%) as a dark brown solid following flash chromatography (1:1 hexanes:Et₂O). ¹H-NMR (500 MHz, CDCl₃): 6.65 (s, 2H), 4.62 (s, 2H), 3.89 (s, 3H), 3.87 (s, 6H), 2.39 (t, 2H, J = 6.1), 2.13 (t, 2H, J = 6.1), 1.98 (s, 3H), 1.72-1.81 (m, 4H); ¹³C-NMR (75 MHz, CDCl₃): 199.4, 170.8, 153.2, 137.9, 134.0, 133.6, 131.8, 106.7, 94.1, 91.5, 65.3, 61.0, 56.2, 33.4, 28.3, 23.4, 22.2, 20.8; IR (KBr): 2938, 2087, 2048, 2020, 1742, 1232; HRMS: m/e for $C_{26}H_{24}Co_2O_{11}$ calculated 574.0084 (M-2CO⁺), found 574.0071.

Hexacarbonyl[μ - η^4 -(phenyl[2-((3,4,5-trimethoxyphenyl)ethynyl)cyclohex-1-enyl]methyl acetate)] dicobalt (1mm)

Compound **10mm** (0.2826 g, 0.6725 mmol) was complexed using General Procedure D to afford product **1mm** (0.4118 g, 0.5833 mmol, 87%) as a dark brown solid following flash chromatography (1:1 hexanes:Et₂O). ¹H-NMR (500 MHz, CDCl₃): 7.18-7.20 (m, 3H), 6.94 (s, 1H), 6.87-6.89 (m, 2H), 6.36 (s, 2H), 3.85 (s, 3H), 3.70 (s, 6H), 2.48-2.50 (m, 2H), 2.09 (s, 3H), 2.00-2.05 (m, 2H), 1.60-1.88 (m, 4H); ¹³C-NMR (75 MHz, CDCl₃): 199.7, 169.8, 152.4, 137.9, 137.0, 136.3, 135.0, 131.3, 128.2, 127.6, 126.7, 107.2, 98.0, 92.1, 75.2, 60.9, 55.8, 33.0, 26.1, 23.4, 22.4, 21.1; IR (KBr): 3001, 2938, 2860, 2835, 2091, 2047, 2034, 1738, 1578, 1407, 1235; HRMS: m/e for C₃₂H₂₈Co₂O₁₁ calculated 538.0601 (M-6CO⁺), found 538.0601.

Hexacarbonyl[μ - η^4 -(2-((3,4,5-trimethoxyphenyl)ethynyl)cyclohept-1-enyl)methyl acetate)|dicobalt (1n)

Compound **10n** (0.3498 g, 0.9766 mmol) was complexed using General Procedure D to afford product **1n** (0.5788 g, 0.8987 mmol, 92%) as a dark green solid following flash chromatography (1:1 hexanes:Et₂O). ¹H-NMR (500 MHz, CDCl₃): 6.64 (s, 2H), 4.69 (s, 2H), 3.89 (s, 3H), 3.86 (s, 6H), 2.62-2.64 (m, 2H), 2.33-2.35 (m, 2H), 1.99 (s, 3H), 1.83 (apparent pentet, 2H, J = 6.0), 1.62 (apparent pentet, 2H, J = 5.4), 1.55 (apparent pentet, 2H, J = 5.42); ¹³C-NMR (75 MHz, CDCl₃): 199.4, 170.9, 153.2, 139.5, 138.9, 137.9, 134.2, 106.6, 95.8, 91.8, 65.7, 61.0, 56.2, 37.5, 32.9, 32.3, 27.1, 26.2, 20.7; IR (KBr): 2928, 2853, 2087, 2049, 1742, 1577, 1501, 1408, 1231; HRMS: m/e for C₂₇H₂₆Co₂O₁₁ calculated 588.0241 (M-2CO⁺), found 588.0234.

Hexacarbonyl[μ - η^4 -(2-((3-isopropyl-4,5-dimethoxyphenyl)ethynyl)cyclohex-1-enyl)methyl acetate)] dicobalt (10)

Compound **10o** (1.7960 g, 5.0421 mmol) was complexed according to General Procedure D. After washing the column of silica with 100% hexanes to remove excess, uncomplexed $Co_2(CO)_8$, the product (**1o**) was eluted using 5:1 hexanes:Et₂O, and isolated as a dark brown solid (2.8905 g, 4.5021 mmol, 89%). ¹H-NMR (500 MHz, CDCl₃): 6.92 (d, 1H, J = 1.8), 6.80 (d, 1H, J = 1.8), 4.66 (s, 2H), 3.86 (s, 3H), 3.86 (s, 3H), 3.34 (septet, 1H, J = 7.1), 2.40 (t, 2H, J = 6.0), 2.13 (t, 2H, J = 6.2), 1.98 (s, 3H), 1.70-1.80 (m, 4H), 1.22 (d, 6H, J = 7.1); ¹³C-NMR (75 MHz, CDCl₃); 199.7, 170.8, 152.5, 146.1, 142.7, 133.8, 133.5, 131.8, 119.9, 110.8, 94.5, 91.3, 65.2, 61.0, 55.8, 33.4, 28.3, 26.9, 23.5, 23.4, 22.2, 20.8; IR (KBr): 2962, 2936, 2869, 2834, 2086, 2048, 2019, 1743, 1464, 1308, 1230; HRMS: m/e for $C_{28}H_{28}Co_2O_{10}$ calculated 558.0499 (M-3CO⁺), found 558.0486.

[2-(Thiophen-3-ylethynyl)cyclohex-1-enyl]methyl acetate dicobalt hexacarbonyl (1p)

Compound **10p** (0.3957 g, 1.521 mmol) was subjected to complexation procedures according to General Procedure D. The product (**1p**) was isolated as a dark brown solid (0.7527 g, 1.379 mmol, 91%) via flash chromatography (10:1 hexanes:Et₂O). ¹H-NMR (500 MHz, CDCl₃): 7.32-7.34 (m, 2H), 7.06 (dd, 1H, J = 4.6, J = 1.7), 4.57 (s, 2H), 2.38 (t, 2H, J = 6.0), 2.14 (t, 2H, J = 6.1), 1.99 (s, 3H), 1.69-1.80 (m, 4H); ¹³C-NMR (75 MHz, CDCl₃): 199.5, 170.8, 139.1, 133.6, 132.0, 128.5, 126.4, 123.7, 91.2, 87.3, 65.3, 33.2, 28.6, 23.3, 22.2, 20.9; IR (KBr): 2936, 2862, 2088, 2049, 2020, 1742, 1231; HRMS: m/e for $C_{21}H_{16}Co_{2}O_{8}S$ calculated 461.9382 (M-3CO⁺), found 461.9398.

Hexacarbonyl[μ - η^4 -((10,11- η :10,11- η)-2,3,4,5-tetrahydro-7,8-dimethoxy-1*H*-dibenzo[a,d]cycloheptene)] dicobalt (2d) and Hexacarbonyl[μ - η^4 -((10,11- η :10,11- η)-2,3,4,5-tetrahydro-6,7-dimethoxy-1*H*-dibenzo[a,d]cycloheptene)] dicobalt (2d')

Compound **1d** (1.0023 g, 1.6705 mmol) was reacted according to General Procedure E, using BF₃-OEt₂ (635 μ L, 5.01 mmol) as the Lewis acid. The product was obtained as a pair of regioisomers, with **2d** (0.7280 g, 1.348 mmol, 81%) as the major product, and **2d'** (0.0823 g, 0.152 mmol, 9%) as the minor product. Both were isolated as maroon solids. The major product, **2d**, eluted as the first band upon purification via flash chromatography (10:1 hexanes:Et₂O). ¹H-NMR (500 MHz, CDCl₃): 7.14 (s, 1H), 6.64 (s, 1H), 3.92 (s, 6H), 3.20 (s, 2H), 2.37 (t, 2H, J = 6.2), 2.29 (t, 2H, J = 6.0), 1.68-1.79 (m, 4H); ¹³C-NMR (125 MHz, CDCl₃): 200.1, 149.2, 148.4, 136.3, 130.5, 129.7, 114.6, 112.3, 95.1, 90.5, 56.0, 42.6, 33.8, 30.5, 23.1, 22.7; IR (KBr): 2935, 2084, 2043, 2012, 1505, 1265; HRMS: m/e for C₂₃H₁₈Co₂O₈ calculated 511.9716 (M-CO⁺), found 511.9711.

Compound **2d'** eluted as the second band in the chromatography purification sequence. The two products had a combined yield of 90%, and a ratio of 8.8:1 para attack:ortho attack (i.e., major:minor (**2d:2d'**)). 1 H-NMR (500 MHz, CDCl₃): 7.39 (d, 1H, J = 8.6), 6.87 (d, 1H, J = 8.7), 3.90 (s, 3H), 3.84 (s, 3H), 3.34 (s, 2H), 2.31-2.36 (m, 4H), 1.67-1.79 (m, 4H); 13 C-NMR (75 MHz, CDCl₃): 200.2, 153.4, 145.7, 136.3, 131.2, 131.1, 130.9, 128.2, 110.9, 95.2, 90.4, 61.4, 55.9, 33.8, 33.2, 30.6, 23.1, 22.8; IR (KBr): 2962, 2917, 2849, 2085, 2048, 2017, 1463, 1283; HRMS: m/e for $C_{23}H_{18}Co_{2}O_{8}$ calculated 539.9666 (M⁺), found 539.9672.

 $Hexacarbonyl [\mu-\eta^4-(9,10-didehydro-5,8-dimethoxy-1,2,3,4-tetrahydrobenzo[f] azulene)] dicobalt~(2g)$

Compound 1g (0.0502 g, 0.0857 mmol) was reacted according to General Procedure E using

BF₃-OEt₂ (32 μL, 0.26 mmol) and at -40 °C. Starting material consumption was complete after 2 h, as assessed by TLC analysis. The cyclized product **2g** was isolated by flash chromatography (15:1 hexanes:Et₂O) as a maroon solid (0.0028 g, 0.0053 mmol, 6%). ¹H-NMR (500 MHz, CDCl₃): 6.90 (½ABq, 1H, J = 9.0), 6.74 (½ABq, 1H, J = 9.0), 3.86 (s, 3H), 3.80 (s, 3H), 3.58 (s, 2H), 2.71 (t, 2H, J = 7.5), 2.52 (t, 2H, J = 7.7), 2.04 (apparent pentet, 2H, J = 7.6); IR (KBr): 2919, 2850, 2086, 2048, 2024, 1650, 1464, 1263; HRMS: m/e for $C_{22}H_{16}Co_2O_8$ calculated 469.9611 (M-2CO⁺), found 469.9628.

Hexacarbonyl[μ - η ⁴-((11,12- η :11,12- η)-11,12-didehydro-5,6,7,8,9,10-hexahydro-1,4-dimethoxybenzo[b]heptalene)] dicobalt (2i)

Compound **1i** (0.2234 g, 0.3638 mmol) was reacted according to General Procedure E using BF₃-OEt₂ (138 μ L, 1.09 mmol). Starting material consumption was complete after 1 h, as assessed by TLC analysis. The cyclized product (**2i**) was isolated by flash chromatography (15:1 hexanes:Et₂O) as a maroon solid (0.1687 g, 0.3045 mmol, 85%). ¹H-NMR (500 MHz, CDCl₃): 6.92 (d, 1H, J = 9.0), 6.74 (d, 1H, J = 9.0), 3.86 (s, 3H), 3.82 (s, 3H), 3.40 (s, 2H), 2.52-2.56 (m, 4H), 1.78 (apparent pentet, 2H, J = 6.0), 1.56-1.67 (m, 4H); ¹³C-NMR (75 MHz, CDCl₃): 200.6, 153.8, 150.2, 141.7, 136.8, 127.1, 126.7, 112.3, 108.5, 97.7, 85.5, 56.8, 54.6, 38.6, 35.6, 34.7, 31.4, 26.2; IR (KBr): 2965, 2919, 2849, 2085, 2051, 2029, 1466, 1261; HRMS: m/e for $C_{24}H_{20}Co_{2}O_{8}$ calculated 553.9822 (M⁺), found 553.9802.

Hexacarbonyl[μ - η^4 -(9,10-dehydro1,2,3,4-tetrahydro-6,7,8-trimethoxybenzo[f]azulene)]dicobalt (2j)

Compound **1j** (1.2170 g, 1.9757 mmol) was subjected to General Procedure E, using SnCl₄ (69 μ L, 5.9 mmol). Starting material consumption was complete in 1 h, as monitored by TLC. Flash chromatography on neutralized silica (20:1 hexanes:Et₂O) afforded the product as a maroon solid (0.0903 g, 0.162 mmol, 8%). ¹H-NMR (500 MHz, CDCl₃): 6.42 (s, 1H), 4.05 (s, 3H), 3.89 (s, 3H), 3.84 (s, 3H), 3.41 (s, 2H), 2.71 (t, 2H, J = 7.8), 2.54 (t, 2H, J = 7.8), 2.05 (apparent pentet, 2H, J = 7.7); ¹³C-NMR (75 MHz, CDCl₃): 200.3, 154.7, 153.9, 141.1, 139.8, 136.2, 131.3, 123.3, 108.6, 89.6, 84.5, 60.8, 60.2, 56.0, 38.8, 38.0, 35.5, 22.6; IR (ATR): 2931, 2850, 2083, 2025, 2009, 1993, 1588, 1487, 1319, 1120; HRMS: m/e for C₂₃H₁₈Co₂O₉ calculated 499.9716 (M-2CO⁺), found 499.9699.

Hexacarbonyl[μ - η^4 -(10,11-didehydro-2,3,4,5-tetrahydro-7,8,9-trimethoxy-1*H*-dibenzo[a,d]cycloheptene)]dicobalt (2k)

Compound **1k** (0.8100 g, 1.286 mmol) was subjected to General Procedure E, using SnCl₄ (90 μ L, 7.7 mmol). Starting material consumption was complete in 1 h, as assessed by TLC analysis. The cyclized product **2k** (0.2865 g, 0.5026 mmol, 39%) was isolated as a maroon solid following flash chromatography using neutralized silica (20:1 hexanes:Et₂O). ¹H-NMR (500 MHz, CDCl₃): 6.48 (s, 1H), 4.04 (s, 3H), 3.90 (s, 3H), 3.85 (s, 3H), 3.18 (s, 2H), 2.35 (t, 2H, J = 6.3), 2.28 (t, 2H, J = 6.2), 1.67-1.78 (m, 4H); ¹³C-NMR (75 MHz, CDCl₃): 200.5, 154.3, 153.9, 141.2, 135.3, 133.1, 131.3, 123.2, 108.1, 96.7, 84.8, 60.8, 60.2, 56.0, 43.4, 33.6, 30.6, 23.1, 22.8; IR (ATR): 2932, 2855, 2086, 2044, 1992, 1586, 1484, 1326; HRMS: m/e for C₂₄H₂₀Co₂O₉ calculated 513.9873 (M-2CO⁺), found 513.9852.

Hexacarbonyl[μ - η^4 -(10,11-didehydro-2,3,4,5-tetrahydro-7,8,9-trimethoxy-5-methyl-1*H*-dibenzo[a,d]cycloheptene)]dicobalt (2kk)

Compound **1kk** (0.8530 g, 1.324 mmol) was subjected to General Procedure E, using SnCl₄ (46 μ L, 4.0 mmol). After 1 h reaction time, starting material consumption was complete. The cyclized product (**2kk**) was isolated as a maroon solid (0.3018 g, 0.5168 mmol, 39%) following flash chromatography (20:1 hexanes:Et₂O). ¹H-NMR (500 MHz, CDCl₃): 6.44 (s, 1H), 4.07 (s, 3H), 3.90 (s, 3H), 3.86 (s, 3H), 3.25 (q, 1H, J = 7.7), 2.16-2.46 (m, 4H), 1.75 (m, 4H), 1.26-1.3 (m, 3H); ¹³C-NMR (75 MHz, CDCl₃): 200.6, 154.5, 154.3, 140.7, 139.1, 137.7, 129.3, 121.9, 106.5, 95.3, 83.8, 60.8, 60.1, 56.0, 47.4, 31.0, 23.2, 22.8, 19.5; IR (ATR): 2930, 2859, 2081, 2038, 1993, 1586, 1486, 1319, 1259; HRMS: m/e for C₂₅H₂₂Co₂O₉ calculated 555.9979 (M-CO⁺), found 555.9996.

Hexacarbonyl[μ - η^4 -(9,10-didehydro-1,2,3,4-tetrahydro-5,6,7-trimethoxybenz[f]azulene)] dicobalt (2l)

Compound **11** (0.4123 g, 0.6693 mmol) was cyclized according to General Procedure E with BF₃-OEt₂ (254 μ L, 2.01 mmol) as Lewis acid. Starting material consumption was complete within 30 minutes, as assessed by TLC analysis. The cyclized product **21** (0.3159 g, 0.5682 mmol, 85%) was isolated via flash chromatography (5:1 hexanes:Et₂O) as a dark maroon solid. ¹H-NMR (500 MHz, CDCl₃): 6.99 (s, 1H), 3.92 (s, 3H), 3.89 (s, 3H), 3.84 (s, 3H), 3.48 (s, 2H), 2.71 (t, 2H, J = 7.5), 2.56 (t, 2H, J = 7.4), 2.06 (apparent pentet, 2H, J = 7.6); ¹³C-NMR (75 MHz, CDCl₃): 199.9, 152.4, 150.9, 143.0, 141.6, 135.3, 133.5, 121.6, 122.3, 91.0, 88.0, 61.3, 60.9, 56.0, 39.3, 35.5, 27.5, 22.7; IR (KBr): 2938, 2086, 2048, 2018, 1118; HRMS: m/e for C₂₃H₁₈Co₂O₉ calculated 527.9666 (M-CO⁺), found 527.9654.

Hexacarbonyl[μ - η^4 -(10,11-didehydro-2,3,4,5-tetrahydro-6,7,8-trimethoxy-1*H*-dibenzo[a,d]cycloheptene)] dicobalt (2m)

Compound **1m** (0.3033 g, 0.4814 mmol) was cyclized according to General Procedure E with BF₃-OEt₂ (183 μ L, 1.44 mmol) as Lewis acid. Starting material consumption was complete within 30 minutes, as assessed by TLC analysis. The cyclized product (**2m**) (0.2368 g, 0.4154 mmol, 86%) was isolated via flash chromatography (5:1 hexanes:Et₂O) as a dark maroon solid. ¹H-NMR (500 MHz, CDCl₃): 6.98 (s, 1H), 3.92 (s, 3H), 3.89 (s, 3H), 3.88 (s, 3H), 3.25 (s, 2H), 2.32-2.36 (m, 4H), 1.69-1.78 (m, 4H); ¹³C-NMR (75 MHz, CDCl₃): 200.1, 152.5, 150.5, 143.0, 137.1, 133.8, 130.9, 123.4, 111.1, 95.3, 90.5, 61.7, 60.9, 56.1, 33.8, 32.9, 30.5, 23.1, 22.8; IR (KBr): 2936, 2085, 2045, 2016, 1591, 1127; HRMS: m/e for C₂₄H₂₀Co₂O₉ calculated 541.9822 (M-CO⁺), found 541.9821.

Hexacarbonyl[μ - η^4 -(1,2,3-trimethoxy-5-(-2-(6-(phenylmethylene)-1-cyclohexen-1-yl)ethynyl)benzene)]dicobalt (11c) and Hexacarbonyl[μ - η^4 -(10,11,-didehydro-2,3,4,5-tetrahydro-6,7,8-trimethoxy-5-phenyl-1*H*-dibenzo[a,d]cycloheptene)]dicobalt (2mm)

Compound **1mm** (0.1300 g, 0.1841 mmol) was reacted according to General Procedure E using BF₃-OEt₂ (70 μ L, 0.55 mmol). Starting material consumption was complete after 1 h, as assessed by TLC analysis. The cyclized product (**2mm**) was separated from its elimination isomer (**11c**) by flash chromatography (10:1 hexanes:Et₂O). The elimination product came off the column as the second band, and was isolated as a green solid (0.0546 g, 0.0845 mmol, 46%). ¹H-NMR (500 MHz, CDCl₃): 7.26 (apparent t, 2H, J = 7.7), 7.16 (apparent t, 1H, J = 7.3), 7.03 (d, 2H, J = 7.9), 6.82 (s, 2H), 6.67 (t, 1H, J = 4.6), 6.49 (s, 1H), 3.91 (s, 3H), 3.81 (s, 6H), 2.77 (t, 2H, J = 6.4), 2.42 (apparent q, 2H, J = 5.7), 1.81 (apparent pentet, 2H, J = 6.3); ¹³C-NMR (75 MHz, CDCl₃): 199.9, 153.2, 137.9, 137.7, 137.3, 136.2, 134.0, 133.8, 129.2, 128.2, 127.9, 126.6, 107.6, 95.8, 93.8, 61.0, 56.1, 27.6, 27.4, 22.7; IR (KBr): 3000, 2937, 2835, 2083, 2046, 2032, 1574, 1498, 1409, 1322, 1232; HRMS: m/e for C₃₀H₂₄Co₂O₉ calculated 562.0237 (M-3CO⁺), found 562.0231.

The cyclized product came off the column first, and was isolated as a maroon solid (0.0406 g, 0.0628 mmol, 34%). The combined yield was 80%, and a ratio of cyclized:elimination of 1:1.3 was determined. 1 H-NMR (500 MHz, CDCl₃): 7.05-7.15 (m, 4H), 6.90 (apparent d, 2H, J = 7.6), 5.32 (s, 1H), 3.98 (s, 3H), 3.92 (s, 3H), 3.87 (s, 3H), 2.37-2.68 (m, 4H), 1.71-1.94 (m, 4H); 13 C-NMR (75 MHz, CDCl₃): 199.8, 152.8, 151.6, 142.9, 141.0, 138.4, 132.8, 131.5, 128.5, 126.9,

126.4, 125.1, 112.3, 91.7, 87.7, 61.8, 60.9, 55.8, 47.6, 35.9, 31.5, 23.5, 22.9; IR (KBr): 2928, 2858, 2084, 2027, 2015, 1638, 1448, 1242; HRMS: m/e for C₃₀H₂₄Co₂O₉ calculated 562.0237 (M-3CO⁺), found 562.0240.

Alternatively, **1mm** (0.2081 g , 0.2947mol) was reacted according to General Procedure E using $SnCl_4$ (103 μ L, 0.884 mmol). Starting material consumption was complete after 1 h, as assessed by TLC analysis. Cyclized product **2mm** (0.1509 g, 79% yield) was isolated following flash chromatography (10:1 hexanes: Et_2O).

Hexacarbonyl[μ - η^4 -(11,12-didehydro-5,6,7,8,9,10-hexahydro-2,3,4-trimethoxybenzo[b]heptalene)]dicobalt (2n)

Compound **1n** (0.4989 g, 0.7747 mmol) was cyclized according to General Procedure E with BF₃-OEt₂ (294 μ L, 2.32 mmol) as Lewis acid. Starting material consumption was complete within 30 minutes, as assessed by TLC analysis. The cyclized product (**2n**) (0.3804 g, 0.6514 mmol, 84%) was isolated via flash chromatography (5:1 hexanes:Et₂O) as a maroon solid. ¹H-NMR (500 MHz, CDCl₃): 6.97 (s, 1H), 3.92 (s, 3H), 3.89 (s, 3H), 3.87 (s, 3H), 3.32 (s, 2H), 2.54-2.58 (m, 4H), 1.78 (apparent pentet, 2H, J = 5.8), 1.59-1.67 (m, 4H); ¹³C-NMR (75 MHz, CDCl₃): 200.2, 152.5, 150.3, 142.9, 136.4, 133.6, 123.4, 110.8, 97.2, 91.0, 61.8, 61.0, 56.1, 39.1, 35.5, 35.0, 31.7, 26.3; IR (KBr): 2918, 2850, 2085, 2046, 2016, 1590, 1480, 1328; HRMS: m/e for C₂₅H₂₂Co₂O₉ calculated 528.0029 (M-2CO⁺), found 528.0030.

Hexacarbonyl[μ - η^4 -(10,11-didehydro-2,3,4,5-tetrahydro-6-isopropyl-7,8-dimethoxy-1*H*-dibenzo[a,d]cycloheptene)]dicobalt (20) and Hexacarbonyl[μ - η^4 -(10,11-didehydro-2,3,4,5-tetrahydro-8-isopropyl-6,7-dimethoxy-1*H*-dibenzo[a,d]cycloheptene)]dicobalt (20')

Compound **1o** (0.2042 g, 0.3180 mmol) was reacted according to General Procedure E, using SnCl₄ (111 μ L, 0.954 mmol). Starting material consumption was complete after 1 h. The product was obtained as a pair of regioisomers, **2o** (0.1361 g, 0.2338 mmol, 74%) and **2o'** (0.0098 g, 0.0168 mmol, 5%). The two regioisomers were separable by flash chromatography on neutralized silica (25:1 hexanes:Et₂O). The major product, **2o**, eluted as the first band, and was isolated as a maroon solid. ¹H-NMR (500 MHz, CDCl₃): 7.09 (s, 1H), 3.88 (s, 6H), 3.47-3.53 (m, 1H), 3.15 (s, 2H), 2.33-2.37 (m, 4H), 1.69-1.80 (m, 4H), 1.40 (d, 6H, J = 7.2); NOE (500 MHz, CDCl₃): Irradiation at δ 3.15 resonance gave enhancement of the δ 1.40 resonance; ¹³C-NMR (75 MHz, CDCl₃): 200.3, 151.9, 149.0, 138.8, 138.0, 133.7, 131.2, 128.1, 113.9, 95.7, 92.1, 60.8, 55.7, 36.7, 33.0, 30.2, 29.0, 23.1, 22.8, 20.3; IR (ATR): 2955, 2931, 2871, 2083,

2042, 2006, 1586, 1459, 1307, 1241; HRMS: m/e for $C_{26}H_{24}Co_2O_8$ calculated 526.0237 (M- $2CO^+$), found 526.0241.

Product compound **20'** came off the column as the second band, and was isolated as a maroon solid. The combined yield was 79%, with a ratio of 13.9:1 major:minor (**20:20'**). ¹H-NMR (500 MHz, CDCl₃): 7.28 (s, 1H), 3.90 (s, 3H), 3.85 (s, 3H), 3.30 (septet, 1H, J = 7.2), 3.28 (s, 2H), 2.30-2.36 (m, 4H), 1.67-1.79 (m, 4H), 1.24 (d, 6H, J = 7.0); ¹³C-NMR (125 MHz, CDCl₃): 200.1, 151.0, 149.5, 141.5, 136.6, 133.8, 131.0, 128.3, 125.2, 95.0, 90.6, 61.0, 60.6, 33.7, 33.0, 30.5, 26.9, 23.4, 23.0, 22.7; IR (ATR): 2961, 2920, 2849, 2084, 2043, 2010, 1407, 1306, 1226; HRMS: m/e for $C_{26}H_{24}Co_2O_8$ calculated 554.0186 (M-CO⁺), found 554.0197.

$Hexacarbonyl[\mu-\eta^4-((6-Methylenecyclohex-1-enyl)ethynyl)benzene]dicobalt (11b)$

Compound **1b** (0.1068 g, 0.1978 mmol) was subjected to General Procedure E using BF₃-OEt₂ (75 μ L, 0.59 mmol). Starting material consumption was complete within 2 h, as determined by TLC analysis. Product **11b** was isolated as a dark brown-green solid (0.0494 g, 0.103 mmol, 52%) following flash chromatography with 100% hexanes. ¹H-NMR (500 MHz, CDCl₃): 7.48 (apparent d, 2H, J = 7.1), 7.28-7.36 (m, 3H), 6.51 (t, 1H, J = 4.2), 4.85 (s, 1H), 4.73 (s, 1H), 2.48 (t, 2H, J = 6.4), 2.35 (apparent q, 2H, J = 5.7), 1.83 (apparent pentet, 2H, J = 6.3); ¹³C-NMR (75 MHz, CDCl₃): 199.9, 140.5, 138.5, 136.8, 135.2, 130.2, 128.7, 127.8, 112.9, 94.9, 93.6, 32.9, 29.8, 27.7, 22.9; IR (KBr): 2940, 2828, 2087, 2047, 2015, 1633; HRMS: m/e for C₂₁H₁₄Co₂O₆ calculated 479.9454 (M⁺), found 479.9465.

2-[(Trimethylsilyl)ethynyl]cyclohex-1-enecarbaldehyde (12a)

2-Bromocyclohex-1-ene-1-carbaldehyde (**8b**) (2.5314 g, 13.466 mmol) was subjected to General Procedure A with trimethylsilylacetylene (2.6453 g, 26.932 mmol), and with THF in place of DMF. Compound **12a** was isolated as a yellow oil (2.3527 g, 11.415 mmol, 85%) via flash chromatography (20:1 hexanes:Et₂O). The material was spectroscopically identical to reported values.¹⁰

2-[(Trimethylsilyl)ethynyl]cyclohept-1-enecarbaldehyde (12b)

2-Bromocyclohept-1-ene-1-carbaldehyde (**8c**) (0.5512 g, 2.729 mmol) was subjected to General Procedure A with trimethylsilylacetylene (0.5360 g, 5.457 mmol), and with THF in place of DMF. Compound **12b** was isolated as a yellow oil (0.5393 g, 2.450 mmol, 90%) via preparative TLC (20:1 hexanes:Et₂O). 1 H-NMR (500 MHz, CDCl₃): 10.13 (s, 1H), 2.53-2.55 (m, 2H), 2.42-2.44 (m, 2H), 1.74 (apparent pentet, 2H, J = 5.8), 1.58 (apparent pentet, 2H, J = 5.6), 1.38

(apparent pentent, 2H, J = 5.6), 0.17 (s, 9H); 13 C-NMR (125 MHz, CDCl₃): 192.4, 148.3, 145.4, 106.3, 102.7, 37.2, 32.2, 25.6, 25.5, 24.1, -0.27; IR (KBr): 2958, 2925, 2853, 2133, 1675, 1449, 1251; HRMS: m/e for $C_{13}H_{20}OSi$ calculated 220.1283 (M⁺), found 220.1274.

[2-((Trimethylsilyl)ethynyl)cyclohept-1-enyl|methyl acetate (13b)

Compound **12b** (0.5340 g, 2.426 mmol) was subjected to General Procedure C. The product was isolated as a yellow oil (0.6001 g, 2.272 mmol, 94%) via radial chromatography (10:1 hexanes:Et₂O). ¹H-NMR (500 MHz, C_6D_6): 4.99 (s, 2H), 2.28-2.30 (m, 2H), 2.06-2.09 (m, 2H), 1.65 (s, 3H), 1.45 (apparent pentet, 2H, J = 5.9), 1.26-1.35 (m, 4H), 0.16 (s, 9H); ¹³C-NMR (125 MHz, C_6D_6): 169.6, 146.5, 125.6, 105.7, 98.3, 67.3, 34.4, 32.1, 30.9, 25.9, 25.8, 20.0, -0.27; IR (ATR): 2922, 2851, 2136, 1740, 1375, 1226; HRMS: m/e for $C_{15}H_{24}O_2Si$ calculated 264.1546 (M⁺), found 264.1547.

(2-Ethynylcyclohept-1-enyl)methyl acetate (14b)

Compound **13b** (0.6001 g, 2.272 mmol) was desilylated according to General Procedure F except that the reaction was kept at 0 °C over 1.5 h. Compound **14b** was isolated as a yellow oil (0.3601 g, 1.874 mmol, 82%) following radial chromatogrpahy (10:1 hexanes:Et₂O). ¹H-NMR (500 MHz, C_6D_6): 4.95 (s, 2H), 2.94 (s, 1H), 2.23-2.26 (m, 2H), 2.04-2.06 (m, 2H), 1.66 (s, 3H), 1.44 (apparent pentet, 2H, J = 5.8), 1.25-1.34 (m, 4H); ¹³C-NMR (125 MHz, C_6D_6): 169.8, 146.7, 124.7, 83.7, 82.0, 67.2, 34.4, 32.0, 30.8, 25.8, 25.7, 20.0; IR (ATR): 3282, 2922, 2850, 1736, 1376, 1227; HRMS: m/e for $C_{12}H_{16}O_2$ calculated 192.1150 (M⁺), found 192.1142.

[2-(3-((Trimethylsilyl)methyl)but-3-en-1-ynyl)cyclohept-1-enyl[methyl acetate (15b)

Compound **14b** (0.3601 g, 1.874 mmol) was subjected to Sonogashira conditions according to General Procedure A with 2-bromo-3-(trimethylsilyl)-1-propene (0.6118 g, 3.186 mmol). The coupled compound (**15b**) was isolated as a yellow oil (0.5127 g, 1.685 mmol, 90%) via preparative TLC (10:1 hexanes:Et₂O). ¹H-NMR (500 MHz, C_6D_6): 5.28 (d, 1H, J = 2.2), 4.96 (s, 2H), 4.92-4.93 (m, 1H), 2.31-2.33 (m, 2H), 2.10-2.12 (m, 2H), 1.68 (s, 3H), 1.66 (d, 2H, J = 1.0), 1.50 (apparent pentet, 2H, J = 5.8), 1.38 (apparent pentet, 2H, J = 5.6), 1.32 (apparent pentet, 2H, J = 5.7), 0.08 (s, 9H); ¹³C-NMR (75 MHz, C_6D_6): 169.7, 144.8, 129.2, 125.9, 118.4, 96.4, 89.0, 67.5, 34.7, 32.2, 31.1, 28.1, 26.0, 26.0, 20.2, -1.8; IR (ATR): 2921, 2850, 1739, 1374, 1246; HRMS: m/e for $C_{18}H_{28}O_2Si$ calculated 304.1859 (M⁺), found 304.1872.

 $Hexacarbonyl \\ [\mu-\eta^4-(2-(3-((trimethylsilyl)methyl)but-3-en-1-ynyl)cyclohept-1-enyl)methyl\\ acetate)] \\ dicobalt~(3b)$

Compound **15b** (0.5127 g, 1.685 mmol) was complexed using General Procedure G to afford complexed product **3b** (0.9250 g, 1.568 mmol, 93%) as a dark green solid, which eluted off a flash chromatographic column of neutralized silica using 10:1 hexanes:Et₂O after all the excess, uncomplexed $Co_2(CO)_8$ had been washed off with 100% hexanes. ¹H-NMR (500 MHz, C_6D_6): 5.40 (s, 1H), 5.15 (s, 1H), 4.94 (s, 2H), 2.50-2.52 (m, 2H), 2.14-2.16 (m, 2H), 1.84 (s, 2H), 1.71 (s, 3H), 1.49-1.55 (m, 4H), 1.36 (apparent pentet, 2H, J = 5.4), 0.09 (s, 9H); ¹³C-NMR (75 MHz, C_6D_6): 200.0, 169.8, 144.3, 139.3, 138.5, 115.9, 101.6, 94.4, 65.5, 37.5, 32.6, 32.1, 27.0, 26.9, 25.9, 20.1, -1.1; IR (KBr): 2926, 2854, 2087, 2049, 2020, 1743, 1229; HRMS: m/e for $C_{24}H_{28}Co_2O_8Si$ calculated 422.0523 (M⁺-6CO), found 422.0512.

$Hexacarbonyl[\mu-\eta^4-(9,10-dehydro-1,2,3,4,5,6,7,8-octahydro-8-methyleneheptalene)] dicobalt \eqno(4b)$

Compound **3b** (0.6755 g, 1.145 mmol) was treated according to General Procedure H. Starting material consumption was complete within 20 minutes, as assessed by TLC. The cyclized product (**4b**) was isolated as a maroon solid (0.4346 g, 0.9490 mmol, 83%), using 100% hexanes for flash chromatography on neutralized silica. 1 H-NMR (500 MHz, $C_{6}D_{6}$): 5.60 (s, 1H), 5.21 (s, 1H), 2.45-2.47 (m, 2H), 2.30-2.32 (m, 2H), 2.15-2.18 (m, 2H), 1.99-2.01 (m, 2H), 1.45-1.58 (m, 4H), 1.28 (apparent pentet, 2H, J = 5.7); 13 C-NMR (75 MHz, $C_{6}D_{6}$): 200.3, 147.6, 147.0, 133.9, 118.4, 95.4, 90.0, 39.2, 38.4, 34.7, 33.8, 32.2, 26.5, 26.1; IR (KBr): 2924, 2851, 2086, 2046, 2016, 1598, 1432, 1213; HRMS: m/e for $C_{19}H_{16}Co_{2}O_{6}$ calculated 457.9611 (M⁺), found 457.9631.

2-[(3-Methoxyphenyl)ethynyl] benzaldehyde (18a)

3-Iodoanisole (1.5271 g, 6.5275 mmol) and 2-ethynylbenzaldehyde¹¹ (0.5659 g, 4.352 mmol), were subjected to reaction via General Procedure A. Flash chromatography (25:1 hexanes:Et₂O) afforded the product **18a** as a yellow oil (0.8583 g, 3.646 mmol, 84%), which was characterized as spectroscopically identical to reported values.¹²

2-[(3,5-Dimethoxyphenyl)ethynyl]benzaldehyde (18b)

Compound 2-ethynylbenzaldehyde (0.6020 g, 4.629 mmol) was subjected to General Procedure A along with 1-bromo-3,5-dimethoxybenzene (1.4997 g, 6.9439 mmol), with the modification that Pd(PPh₃)₂Cl₂ (0.0975 g, 0.139 mmol, 3 mol%) was used as catalyst instead of Pd(PPh₃)₄, and the reaction flask was placed in an oil bath set to a temperature of 60 °C instead of room temperature for overnight (20 h). The product **18b** was isolated using flash chromatography

(10:1 hexanes:Et₂O) as a yellow solid (0.9732 g, 3.657 mmol, 79%), mp. 75-77 °C (lit. mp. 76-77 °C¹³), and which was characterized as spectroscopically identical to reported values.¹³

2-[(3,5-Dimethoxyphenyl)ethynyl]benzyl acetate (19b)

Compound **19b** was synthesized according to General Procedure C from **18b** (0.9732 g, 3.657 mmol). The product was isolated as a pale yellow oil (0.9656 g, 3.114 mmol, 85%) via flash chromatography (7:1 hexanes:Et₂O). ¹H-NMR (300 MHz, CDCl₃): 7.55-7.58 (m, 1H), 7.40-7.43 (m, 1H), 7.28-7.37 (m, 2H), 6.71 (dd, 2H, J = 2.4, J = 0.5), 6.48 (t, 1H, J = 2.3), 5.36 (s, 2H), 3.79 (s, 6H), 2.12 (s, 3H); ¹³C-NMR (75 MHz, CDCl₃): 170.8, 160.7, 137.6, 132.3, 128.6, 128.5, 128.2, 124.3, 122.6, 109.4, 102.1, 94.5, 86.2, 64.8, 55.4, 21.0; IR (ATR): 2953, 2836, 1742, 1585, 1355, 1233; HRMS: m/e for $C_{19}H_{18}O_4$ calculated 310.1205 (M⁺), found 310.1205.

Hexacarbonyl[μ - η^4 -(2-(3,5-dimethoxyphenyl)ethynyl)benzyl acetate)]dicobalt (5b)

Compound **19b** (0.9656 g, 3.114 mmol) was complexed using General Procedure D to afford product **5b** (1.7463 g, 2.9302 mmol, 94%) as a dark brown solid. The product was eluted from a column of silica using 7:1 hexanes:Et₂O. 1 H-NMR (500 MHz, CDCl₃): 7.67 (dd, 1H, J = 7.3, J = 1.85), 7.42 (dd, 1H, J = 7.6, J = 1.7), 7.33-7.40 (m, 2H), 6.63 (d, 2H, J = 2.2), 6.47 (t, 1H, J = 2.3), 5.16 (s, 2H), 3.80 (s, 6H), 2.05 (s, 3H); 13 C-NMR (75 MHz, CDCl₃): 1992.1, 170.5, 160.9, 140.7, 136.0, 134.6, 132.4, 129.6, 128.8, 128.4, 107.6, 100.0, 95.3, 89.0, 63.6, 55.4, 20.8; IR (ATR): 2940, 2839, 2085, 2032, 2000, 1737, 1586, 1421, 1241; HRMS: m/e for C₂₅H₁₈Co₂O₁₀ calculated 483.9767 (M-4CO⁺), found 483.9752.

3-[(3,5-Dimethoxyphenyl)ethynyl]furan-2-carbaldehyde (18d)

[(3,5-Dimethoxyphenyl)ethynyl]trimethylsilane (**7'd**) (0.9212 g, 3.935 mmol) was subjected to tandem desilylation/Sonogashira chemistry according to General Procedure B with 3-bromo-2-formylfuran (1.1977 g, 6.8861 mmol) and Pd(PPh₃)₂Cl₂ (0.0828 g, 0.118 mmol, 3 mol%) as the catalyst, with the exception that the mixture was warmed to rt. The product (**18d**) was isolated with flash chromatography (7:1 hexanes:Et₂O) as a light yellow solid (0.8263 g, 3.227 mmol, 82%). mp. 88-88.5 °C; 1 H-NMR (500 MHz, CDCl₃): 9.85 (d, 1H, J = 0.8), 7.64 (dd, 1H, J = 1.8, J = 0.8), 6.68 (d, 2H, J = 2.3), 6.66 (d, 1H, J = 1.8), 6.50 (t, 1H, J = 2.3), 3.79 (s, 6H); 13 C-NMR (125 MHz, CDCl₃): 176.1, 160.6, 152.8, 147.6, 123.1, 119.4, 115.2, 109.5, 102.7, 97.4, 77.8, 55.5; IR (ATR): 2940, 2832, 2219, 1669, 1586, 1424, 1208; HRMS: m/e for C₁₅H₁₂O₄ calculated 256.0736 (M⁺), found 256.0731.

[3-((3,5-Dimethoxyphenyl)ethynyl)furan-2-yl|methyl acetate (19d)

Compound **18d** (0.8263 g, 3.227 mmol) was treated according to General Procedure C. The product (**19d**) was isolated using flash chromatography (5:1 hexanes:Et₂O) as a colourless solid (0.8552 g, 2.850 mmol, 88%). mp. 58.5-60 °C; 1 H-NMR (500 MHz, CDCl₃): 7.39 (d, 1H, J = 1.9), 6.66 (d, 2H, J = 2.3), 6.50 (d, 1H, J = 1.8), 6.46 (t, 1H, J = 2.2), 5.20 (s, 2H), 3.78 (s, 6H), 2.10 (s, 3H); 13 C-NMR (125 MHz, CDCl₃): 170.5, 160.6, 152.2, 143.1, 124.1, 113.1, 109.2, 108.0, 101.9, 93.4, 79.3, 56.6, 55.4, 20.8; IR (KBr): 3125, 2941, 2840, 2218, 1745, 1590, 1156; HRMS: m/e for C₁₇H₁₆O₅ calculated 300.0998 (M⁺), found 300.0998.

Hexacarbonyl[μ - η^4 -(3-((3,5-dimethoxyphenyl)ethynyl)furan-2-yl)methyl acetate)]dicobalt (5d)

Compound **19d** (0.8552 g, 2.850 mmol) was complexed according to General Procedure D. The complexed product **5d** was isolated using flash chromatography (5:1 hexanes:Et₂O) after washing the column with 100% hexanes to remove any excess, uncomplexed $Co_2(CO)_8$. The product (1.5958 g, 2.7235 mmol, 96%) was a dark brown solid in appearance. ¹H-NMR (500 MHz, CDCl₃): 7.46 (d, 1H, J = 2.0), 6.68 (d, 2H, J = 2.2), 6.56 (d, 1H, J = 1.9), 6.46 (t, 1H, J = 2.3), 5.08 (s, 2H), 3.81 (s, 6H), 2.04 (s, 3H); ¹³C-NMR (125 MHz, CDCl₃): 198.9, 170.5, 160.9, 146.8, 143.4, 140.0, 122.7, 112.9, 107.4, 100.0, 92.9, 79.3, 56.6, 55.4, 20.5; IR (KBr): 2972, 2941, 2089, 2049, 2028, 2008, 1994, 1743, 1587, 1225; HRMS: m/e for $C_{23}H_{16}Co_2O_{11}$ calculated 529.9458 (M⁺), found 529.9470.

Hexacarbonyl[μ - η^4 -(10,11-dehydro-2,4-dimethoxy-5*H*-dibenzo[*a,d*]cycloheptene)]dicobalt (6b)

Compound **6b** was synthesized according to General Procedure E from starting material **5b** (0.2052 g, 0.3443 mmol), using SnCl₄ (121 μ L, 1.03 mmol). The reaction was allowed warm to rt and starting material consumption was complete in 15 h, as monitored by TLC. The product was recovered as a dark maroon solid (0.0938 g, 0.175 mmol, 51%) using flash chromatography (10:1 hexanes:Et₂O). ¹H-NMR (500 MHz, CDCl₃): 7.68-7.71 (m, 1H), 7.31-7.38 (m, 3H), 6.87 (d, 1H, J = 2.5), 6.54 (d, 1H, J = 2.5), 3.94 (s, 2H), 3.90 (s, 3H), 3.86 (s, 3H); ¹³C-NMR (75 MHz, CDCl₃): 199.6, 159.6, 157.2, 139.4, 138.1, 137.6, 132.0, 129.8, 128.6, 127.5, 118.4, 108.3, 99.1, 91.6, 90.9, 56.1, 55.4, 31.8; IR (ATR): 2938, 2840, 2090, 2052, 1996, 1572, 1138; HRMS: m/e for C₂₃H₁₄Co₂O₈ calculated 479.9454 (M-2CO⁺), found 479.9455.

Hexacarbonyl[μ - η^4 -(4,5-dehydro-7,9-dimethoxy-10*H*-benzo[5,6]cyclohepta[1,2-*b*] furan)]dicobalt (6d)

Compound **5d** (0.1582 g, 0.2700 mmol) was treated according to General Procedure E, using SnCl₄ (95 μ L, 0.81 mmol). Starting material consumption was complete in 2 h, as monitored by TLC. The product **6d** was isolated using flash chromatography (neutralized silica, 15:1 hexanes:Et₂O) as a maroon solid (0.0232 g, 0.0441 mmol, 17%). ¹H-NMR (500 MHz, CDCl₃): 7.38 (d, 1H, J = 1.4), 6.86 (d, 1H, J = 2.2), 6.60 (d, 1H, J = 1.4), 6.50 (d, 1H, J = 2.2), 4.14 (s, 2H), 3.87 (s, 3H), 3.85 (s, 3H); ¹³C-NMR (125 MHz, CDCl₃): 199.3, 159.5, 157.3, 150.1, 142.5, 139.3, 118.0, 114.5, 111.9, 109.9, 98.9, 91.6, 81.5, 55.9, 55.3, 25.4; IR (ATR): 2928, 2836, 2086, 2016, 1995, 1561, 1318, 1141; HRMS: m/e for C₂₁H₁₂Co₂O₉ calculated 469.9247 (M-2CO⁺), found 469.9245.

2,3,4,4a,5,10,11,11a-Octahydro-7,8-dimethoxy-1*H*-dibenzo[*a,d*]cycloheptene (20)

To a stirred solution of compound **2d** (0.5101 g, 0.9446 mmol), dissolved in degassed 1,2-dichloroethane (14.4 mL), was added bis(trimethylsilyl)acetylene (429 μ L , 1.89 mmol) and triethylsilane (754 μ L, 4.72 mmol). The reaction was placed in an oil bath set at 65 °C, and allowed to stir for 6 h under a nitrogen atmosphere. The oil bath was removed, and the solution allowed to cool to room temperature, at which point, trifluoroacetic acid (3.6 mL) was added. After stirring for an additional 12 h, the mixture was dissolved in Et₂O (75 mL) and extracted with dH₂O (3 x 75 mL). The organic fraction was dried over MgSO₄, filtered, and the solvent removed under reduced pressure. Preparative TLC (15:1 hexanes:Et₂O) afforded compound **20** as a colourless solid of inseparable diastereomers (1:1) (0.1980 g, 0.7610 mmol, 81%). ¹H-NMR (500 MHz, CDCl₃): 6.66 (s, 1H), 6.65 (s, 1H), 6.64 (s, 1H), 6.60 (s, 1H), 3.86 (s, 9H), 3.85 (s, 3H), 2.86 (apparent t, 1H, J = 13.1), 2.76 (dd, 1H, J = 10.4, J = 14.0), 2.69 (m, 1H), 2.61 (dd, 1H, J = 14.0, J = 6.7), 2.32 (d, 1H, J = 14.0), 0.89-1.96 (m, 27H); ¹³C-NMR (125 MHz, CDCl₃): 146.6, 146.5, 146.5, 146.4, 135.4, 135.1, 134.3, 113.9, 113.1, 112.6, 112.5, 56.1, 56.0, 55.9, 48.5, 44.0, 43.8, 38.1, 36.4, 35.9, 35.4, 35.0, 26.8, 26.4; IR (KBr): 2919, 2851, 1516, 1449, 1271; HRMS: m/e for C₁₇H₂₄O₂ calculated 260.1776 (M⁺), found 260.1775.

6,6-Dimethyl-7,8-dihydro-4H-benzo[d][1,3]dioxin-5(6H)-one (23)

A stirred solution of diisopropylamine (2.7 mL, 19 mmol) in THF (27.1 mL) was cooled to -78 °C. ⁿBuLi (2.5 M in hexanes, 6.8 mL 17 mmol) was added dropwise, and the solution allowed to stir for 30 minutes to generate LDA. Following the 0.5 h, dioxinone **24**, ¹⁴ (2.0906 g, 13.570 mmol) dissolved in THF (13.6 mL), was added to the reaction flask dropwise over 5 minutes. This solution was allowed to stir for 1 h at -78 °C. MeI (3.8522 g, 27.140 mmol), dissolved in

THF (7.7 mL), was added to the reaction following the hour, and stirring continued at -78 °C for 1 h. The reaction was warmed to -30 °C over 30 minutes, and stirred at this temperature for a further hour. It was then warmed to 0 °C and stirred for 3 h. At this point, NH₄Cl (aq, sat) was added, and subjected to a conventional extractive workup (Et₂O). This provided the monomethylated product in a sufficiently pure state for further use as a yellow oil (1.8475 g, 10.992 mmol, 81%). This product was verified by ¹H- and ¹³C-NMR spectroscopy, and found to be identical to reported values. ¹⁴

This monomethylated compound, without further purification, was then resubjected to the identical procedure to generate the *gem*-dimethyl product (**23**) (1.9012 g, 10.441 mmol, 95%), which was isolated as a viscous pale yellow oil following flash chromatography (2:1 hexanes:Et₂O). 1 H-NMR (500 MHz, CDCl₃): 5.11 (s, 2H), 4.39 (t, 2H, J = 1.9), 2.40-2.44 (m, 2H), 1.81 (t, 2H, J = 6.4), 1.10 (s, 6H); 13 C-NMR (125 MHz, CDCl₃): 201.2, 168.2, 109.7, 91.3, 63.2, 40.1, 34.3, 24.8, 24.4; IR (ATR): 2961, 2928, 2865, 1630, 1392, 1236; HRMS: m/e for C_{10} H₁₄O₃ calculated 182.0943 (M⁺), found 182.0944.

2-(Hydroxymethyl)-4,4-dimethyl-3-[(trimethylsilyl)ethynyl]cyclohex-2-enone (25)

Compound 25 was synthesized according to methods adapted from Majetich and Grove, 14 and Brummond and Gao. 15 In a round bottom flask, (trimethylsilyl)acetylene (1.7652 g, 17.972 mmol) was dissolved in THF (30 mL). The reaction flask was cooled to -78 °C, at which point, ⁿBuLi (2.5 M in hexanes, 5.4 mL, 13 mmol) was added dropwise into the stirred solution, and allowed to stir for 30 minutes. After the 30 minutes, the reaction mixture was allowed to warm to 0 °C, at which point 23 (1.6363 g, 8.9860 mmol), dissolved in THF (9.0 mL), was added dropwise into the reaction flask, and the solution allowed to stir for 1 h. The reaction was allowed to warm to room temperature and proceed for another 6 h. NH₄Cl (ag, sat) was added, and the mixture subjected to a conventional extractive workup (Et₂O). The viscous yellow oil was then dissolved in THF (38.0 mL), and 3 M HCl (1.3 mL) was added dropwise into the flask. This reaction was allowed to stir for 1 h at room temperature, after which NaHCO₃ (aq, sat) was added. Following a conventional extractive workup (Et₂O), flash chromatography (1:1 hexanes:Et₂O) afforded **25** (1.8928 g, 7.5670 mmol, 84%) as a pale vellow oil. ¹H-NMR (500 MHz, CDCl₃): 4.44 (d, 2H, J = 6.7), 3.04 (t, 1H, J = 6.8), 2.44 (t, 2H, J = 6.9), 1.82 (t, 2H, J = 6.9) 6.9), 1.21 (s, 6H), 0.18 (s, 9H); ¹³C-NMR (125 MHz, CDCl₃): 199.7, 148.1, 139.2, 113.0, 99.8, 60.6, 35.8, 35.4, 34.2, 27.4, -0.41; IR (KBr): 3474, 2963, 2930, 2902, 2869, 2137, 1664, 1581,

1356, 1251; HRMS: m/e for $C_{14}H_{22}O_2Si$ calculated 250.1389 (M⁺), found 250.1387.

3-Ethynyl-2-(hydroxymethyl)-4,4-dimethylcyclohex-2-enone (26)

Compound **25** (1.8928 g, 7.5670 mmol) was desilylated according to General Procedure F except that the reaction was complete within 30 minutes at 0 °C. The desilylated product (**26**) was isolated as a colourless solid (1.1875 g, 6.67 mmol, 88%) following flash chromatography (1:1 hexanes:Et₂O). mp. 83-84.5 °C; 1 H-NMR (500 MHz, CDCl₃): 4.39 (s, 2H), 3.82 (s, 1H), 3.04 (s, 1H), 2.43 (t, 2H, J = 6.9), 1.81 (t, 2H, J = 6.9), 1.19 (s, 6H); 13 C-NMR (75 MHz, CDCl₃): 199.9, 147.4, 140.4, 93.6, 73.9, 60.0, 35.9, 35.4, 34.2, 27.3; IR (ATR): 3395, 3201, 2956, 2930, 2895, 2867, 2080, 1644, 1574, 1360, 1196; HRMS: m/e for C₁₁H₁₄O₂ calculated 178.0994 (M⁺), found 178.0994.

2-(Hydroxymethyl)-3-[(3-isopropyl-4-methoxyphenyl)ethynyl]-4,4-dimethylcyclohex-2-enone (28)

To a mixture of Pd(PPh₃)₄ (0.1008 g, 0.08723 mmol, 5 mol%) and CuI (0.0266 g, 0.140 mmol, 8 mol%) was added a solution of 27^{16} (0.7225 g, 2.618 mmol) dissolved in dry DMF (2.9 mL), followed by a solution of 26 (0.3108 g, 1.745 mmol) in dry DMF (2.9 mL). Diisopropylamine (11.6 mL), which had been degassed for 1.5 h, was then added to the reaction. The reaction mixture was allowed to stir for 48 h under a nitrogen atmosphere and at room temperature. The mixture was then filtered through Celite®, and subjected to a conventional extractive workup (Et₂O). Preparative TLC (1:1 hexanes:Et₂O) afforded 28 as a viscous yellow oil (0.3983 g, 1.221 mmol, 70%). ¹H-NMR (500 MHz, CDCl₃): 7.30-7.34 (m, 2H), 6.80 (d, 1H, J = 8.4), 4.61 (d, 2H, J = 6.7), 3.84 (s, 3H), 3.28 (septet, 1H, J = 6.9), 3.16 (t, 1H, J = 6.8), 2.52 (t, 2H, J = 6.8), 1.91 (t, 2H, J = 6.8), 1.34 (s, 6H), 1.20 (d, 6H, J = 6.9); ¹³C-NMR (125 MHz, CDCl₃): 199.7, 158.2, 149.3, 137.6, 131.1, 129.8, 113.9, 110.4, 107.8, 84.1, 60.9, 55.4, 36.0, 35.8, 34.3, 27.7, 26.7, 22.4; IR (ATR): 3453, 2961, 2929, 2869, 2839, 2183, 1648, 1493, 1245; HRMS: m/e for C₂₁H₂₆O₃ calculated 326.1882 (M⁺), found 326.1882.

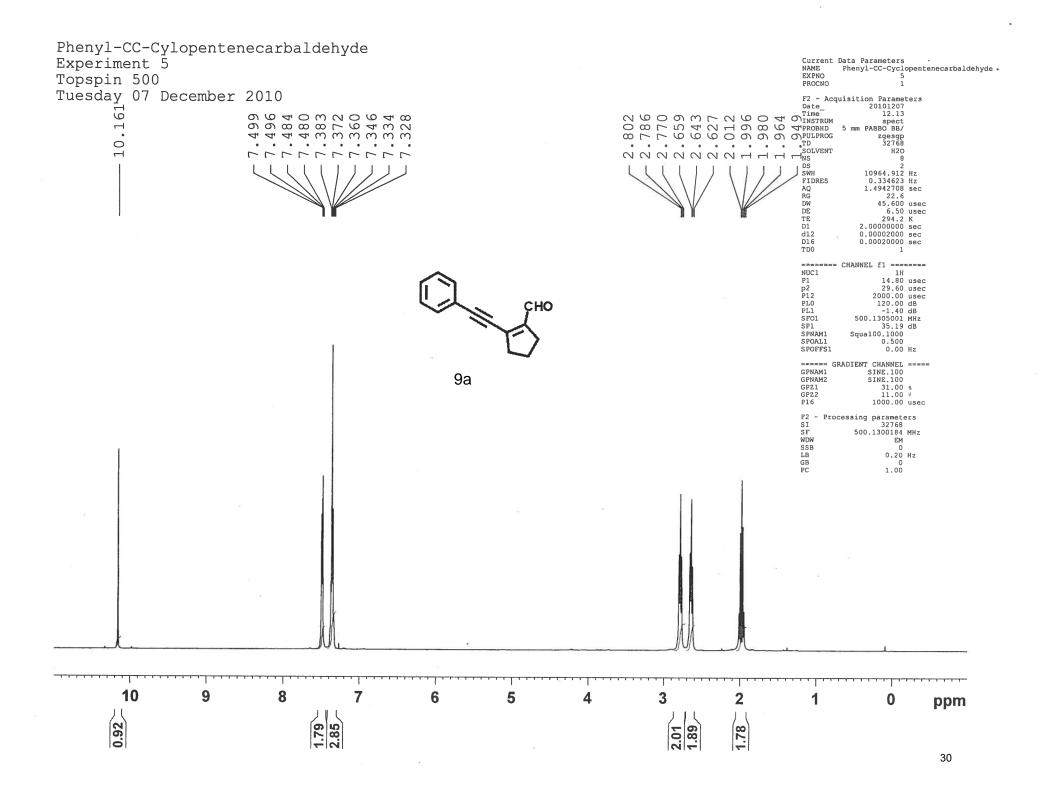
[2-((3-Isopropyl-4-methoxyphenyl)ethynyl)-3, 3-dimethyl-6-oxocyclohex-1-enyl] methyl acetate (29)

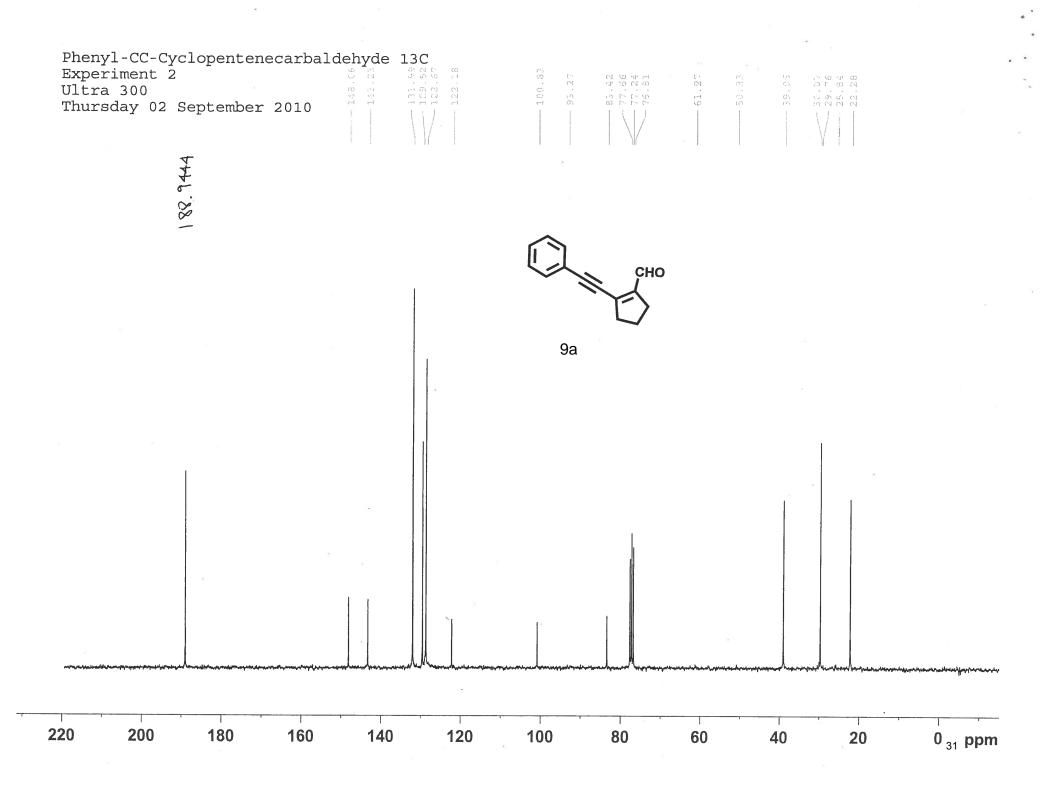
Compound **28** (0.3983 g, 1.221 mmol) was dissolved in dry THF (14.1 mL), and the solution was cooled to a temperature of -78 °C. Pyridine (3.0 mL, 37 mmol) was added to the reaction, followed by acetic anhydride (5.8 mL, 61 mmol) and DMAP (0.7459 g, 6.105 mmol). The reaction was allowed to warm to room temperature under a nitrogen atmosphere over the course

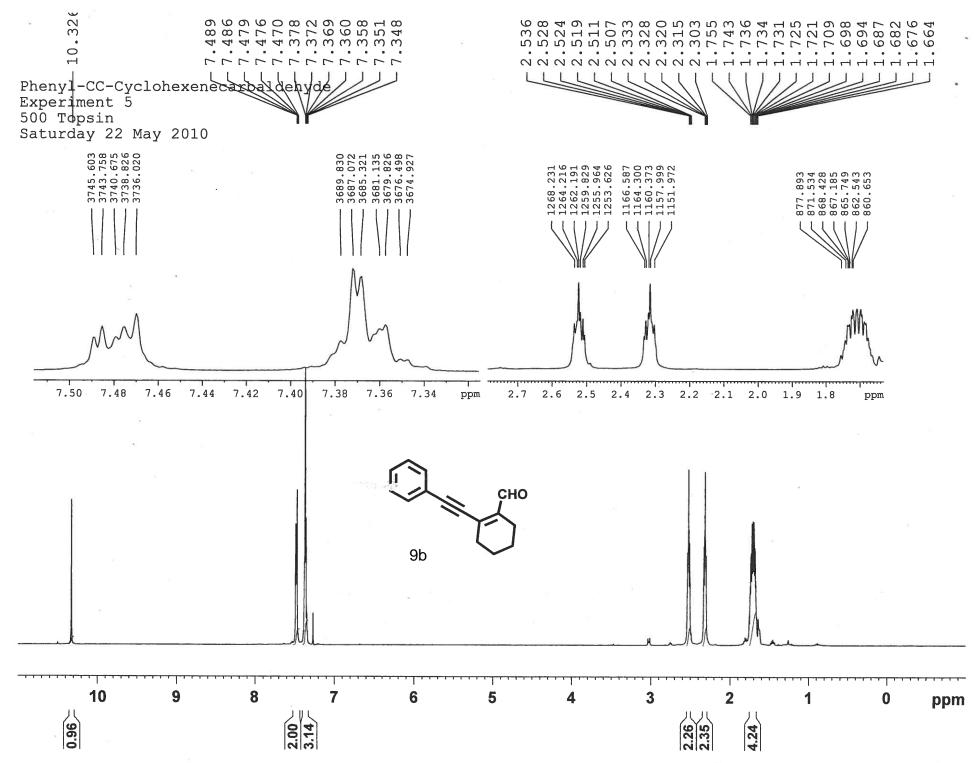
of 4 h, at which point TLC analysis showed complete starting material consumption. Following the addition NH₄Cl (aq, sat), the mixture was subjected to a conventional extractive workup (Et₂O). Preparative TLC (1:1 hexanes:Et₂O) afforded compound **29** as an off-white solid (0.4240 g, 1.152 mmol, 94%). mp. 123.5-124.5 °C; 1 H-NMR (500 MHz, CDCl₃): 7.30-7.32 (m, 2H), 6.78 (d, 1H, J = 8.3), 5.00 (s, 2H), 3.80 (s, 3H), 3.25 (septet, 1H, J = 6.9), 2.51 (t, 2H, J = 6.8), 1.99 (s, 3H), 1.90 (t, 2H, J = 6.8), 1.33 (s, 6H), 1.18 (d, 6H, J = 7.0); 13 C-NMR (75 MHz, CDCl₃): 196.3, 170.8, 158.4, 153.1, 137.6, 134.0, 131.4, 130.1, 113.8, 110.4, 108.7, 84.3, 59.7, 55.5, 36.1, 36.0, 34.1, 27.8, 26.7, 22.4, 21.0; IR (ATR): 2956, 2938, 2868, 2181, 1725, 1668, 1248; HRMS: m/e for C₂₃H₂₈O₄ calculated 368.1988 (M⁺), found 368.1998.

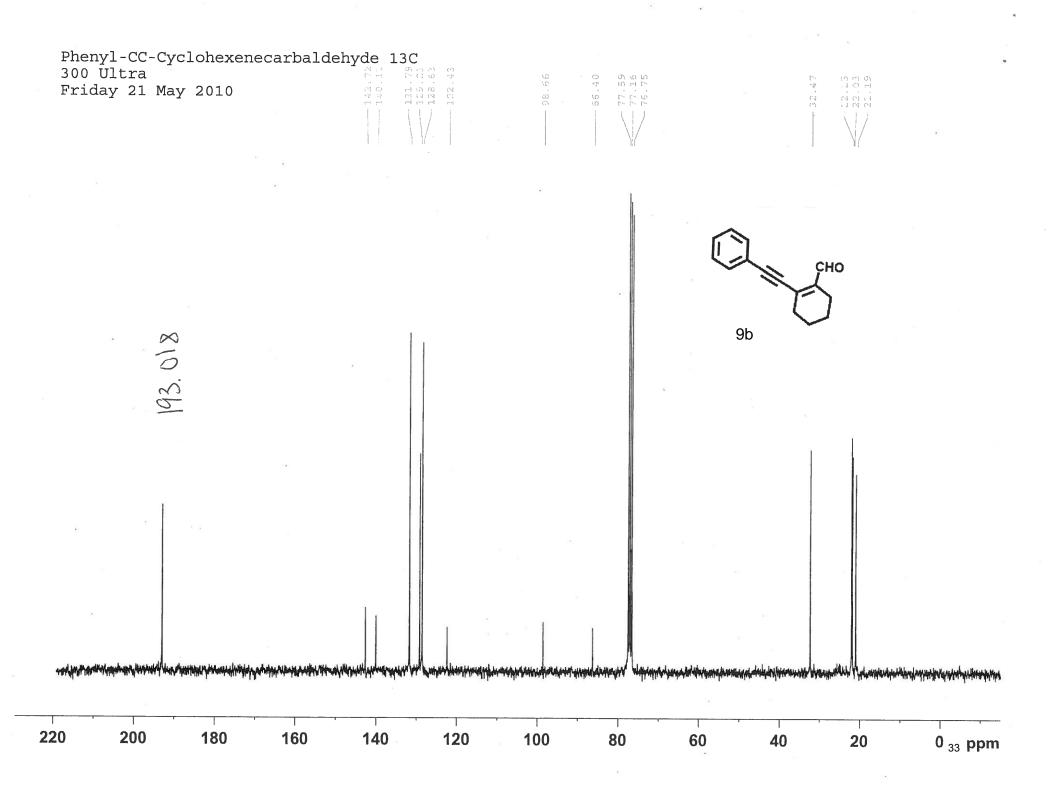
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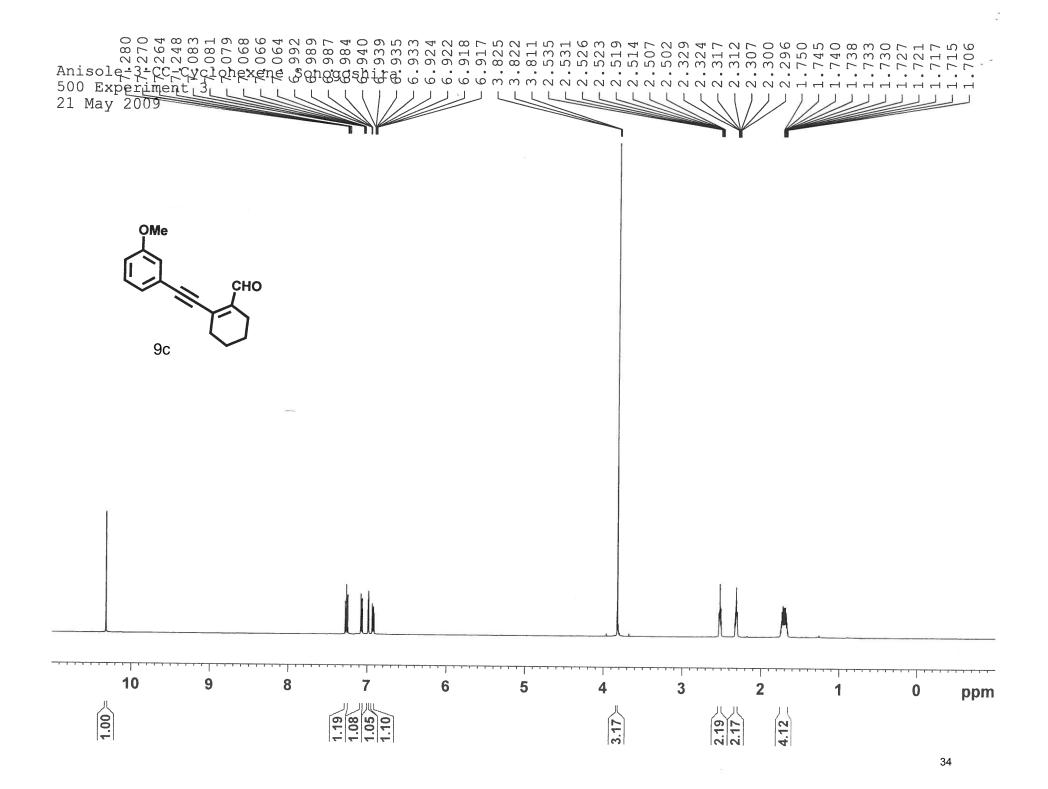
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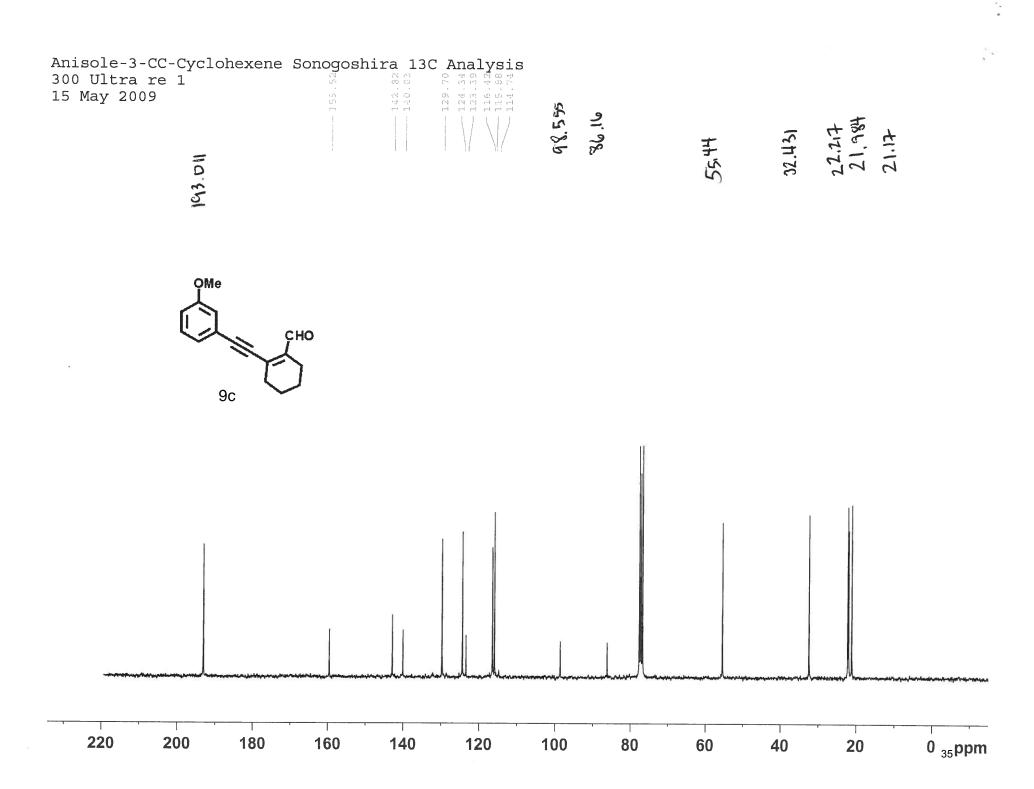


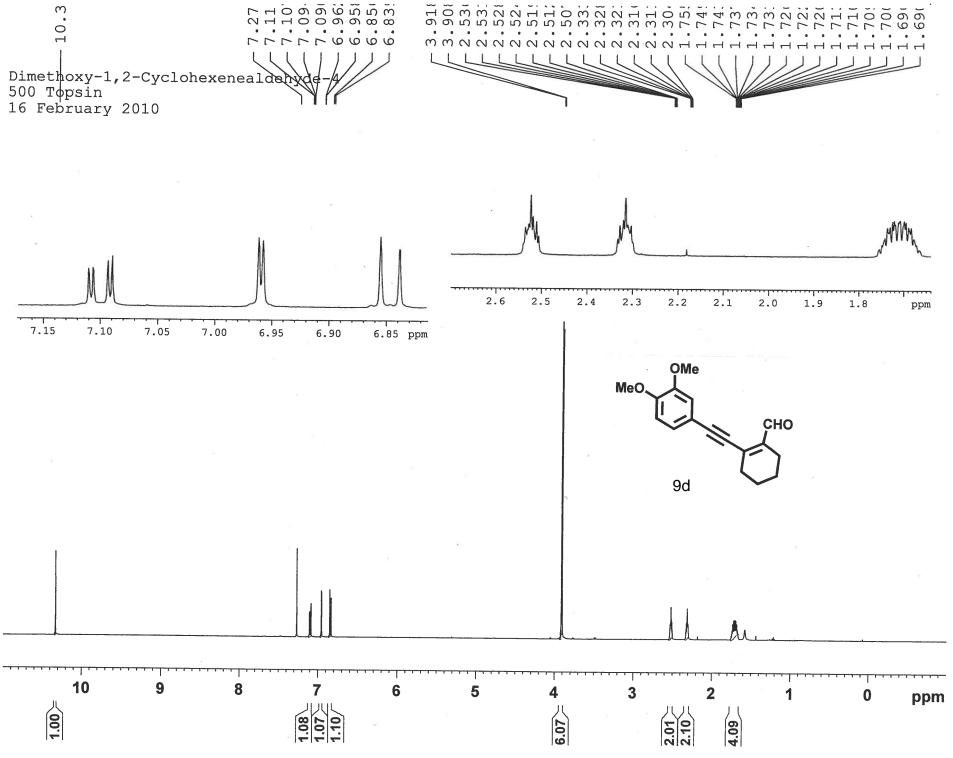


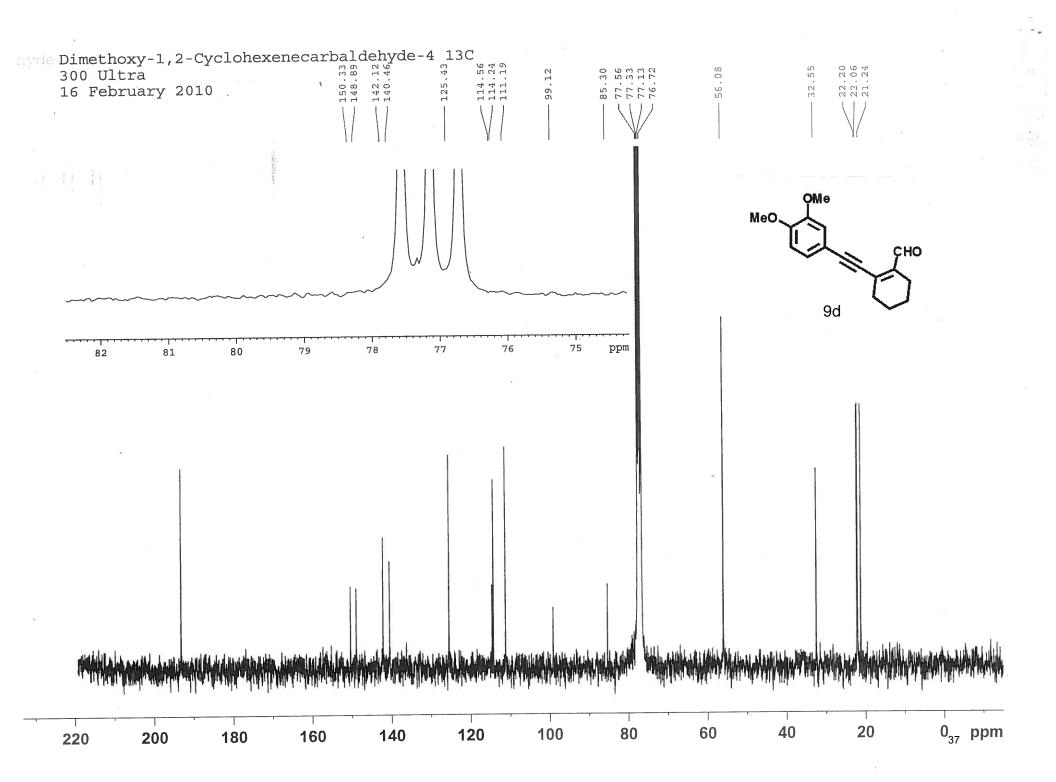


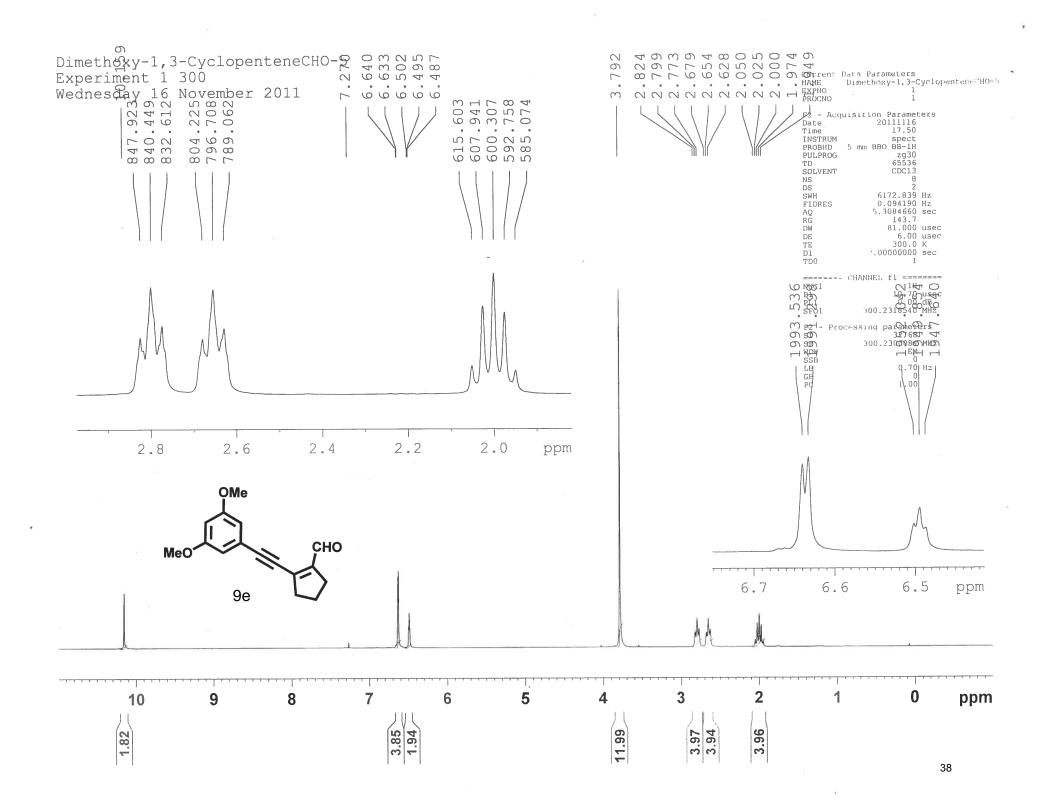


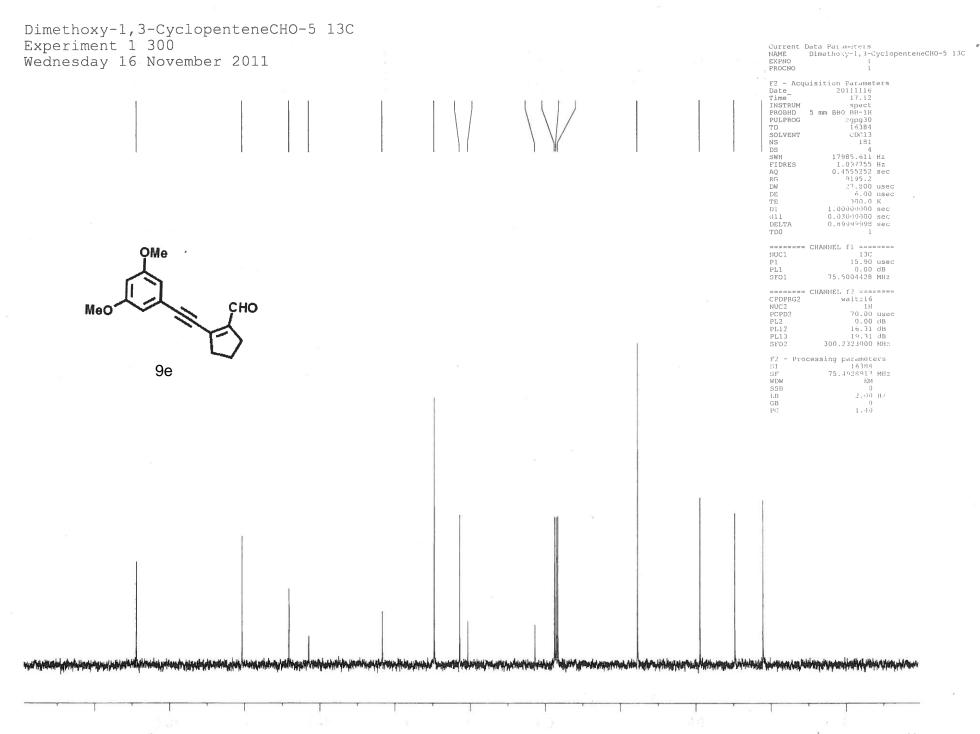


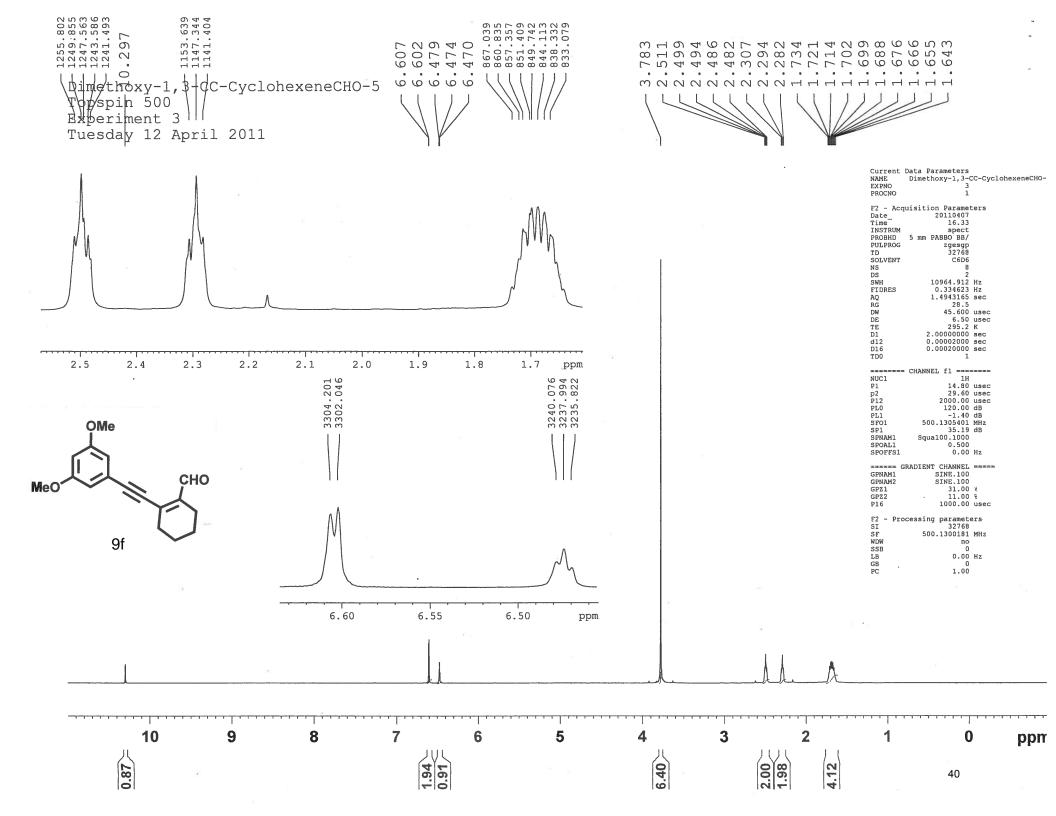




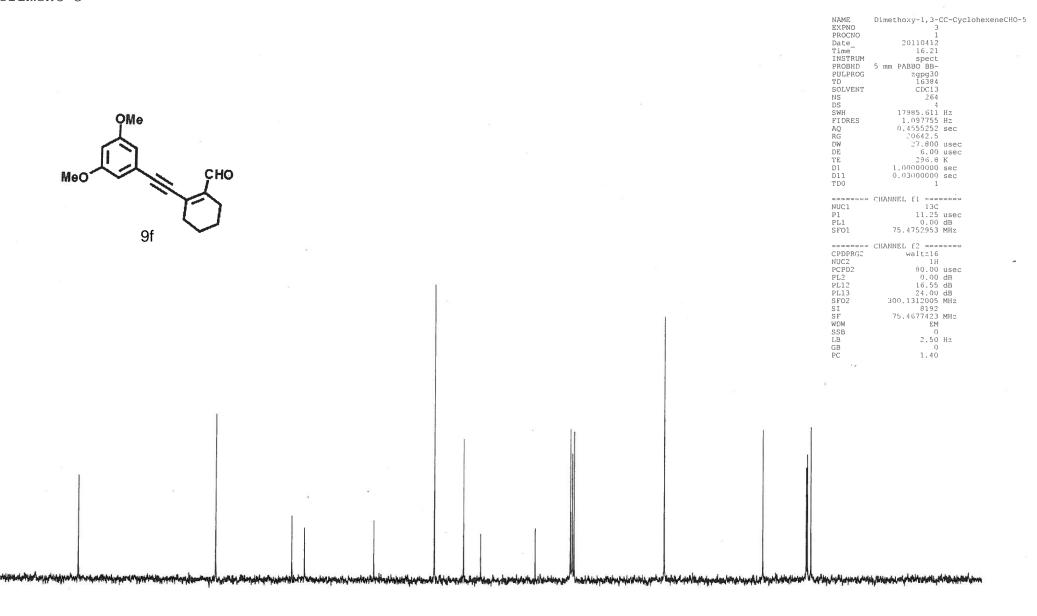




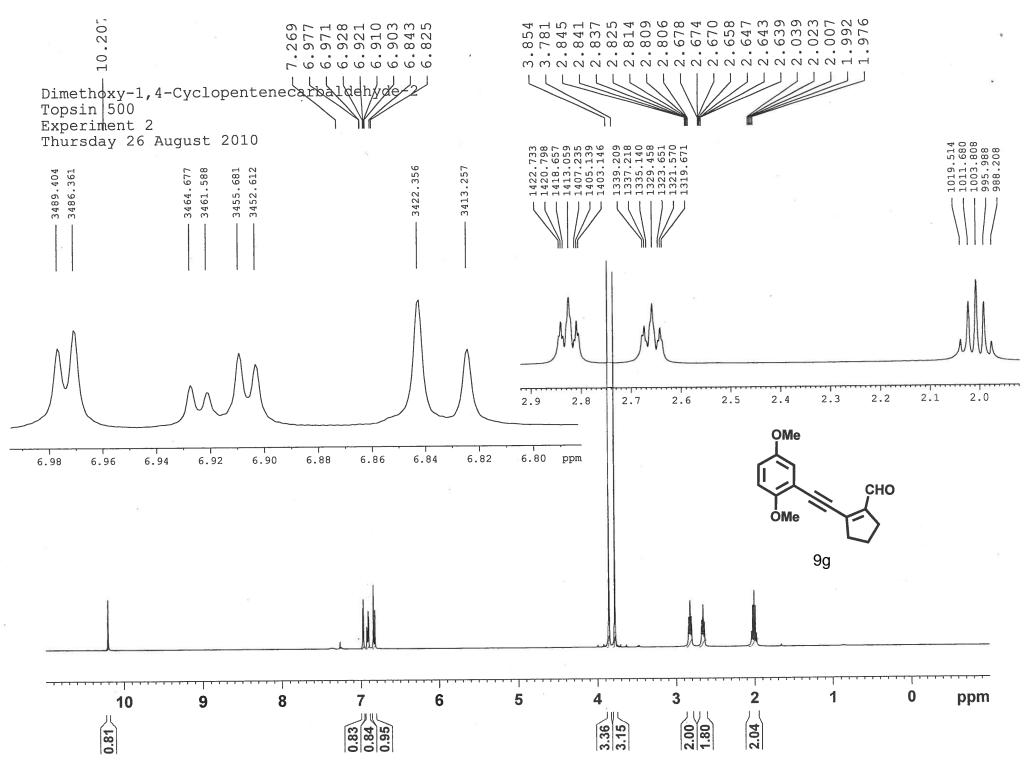


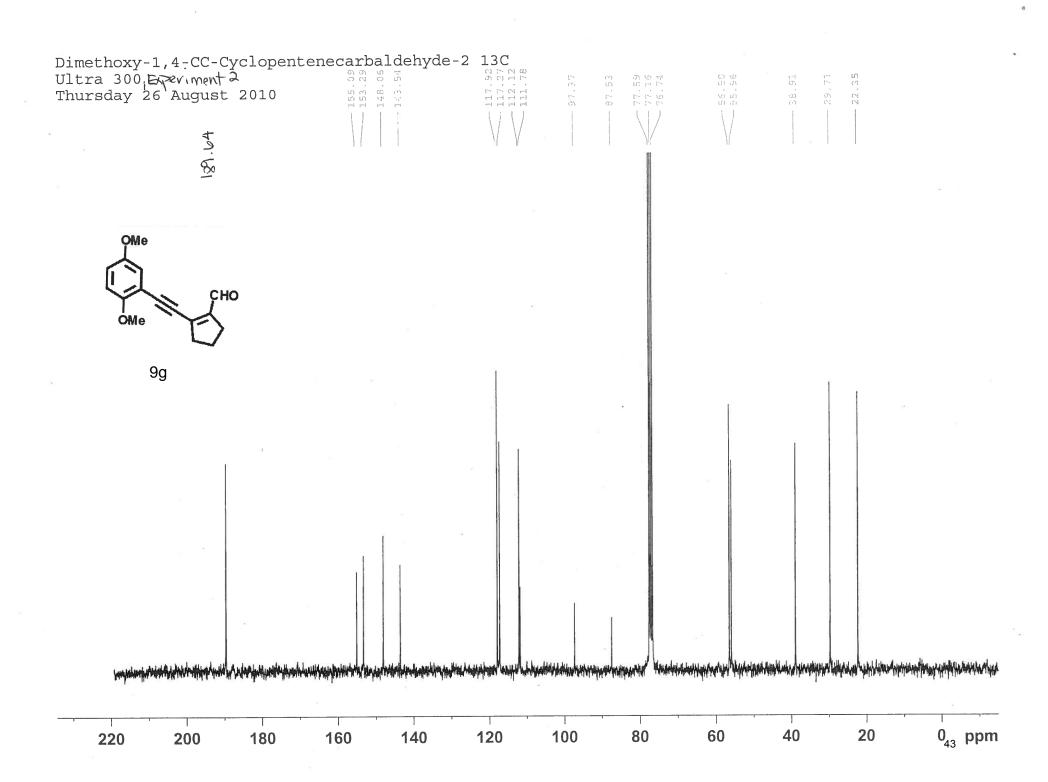


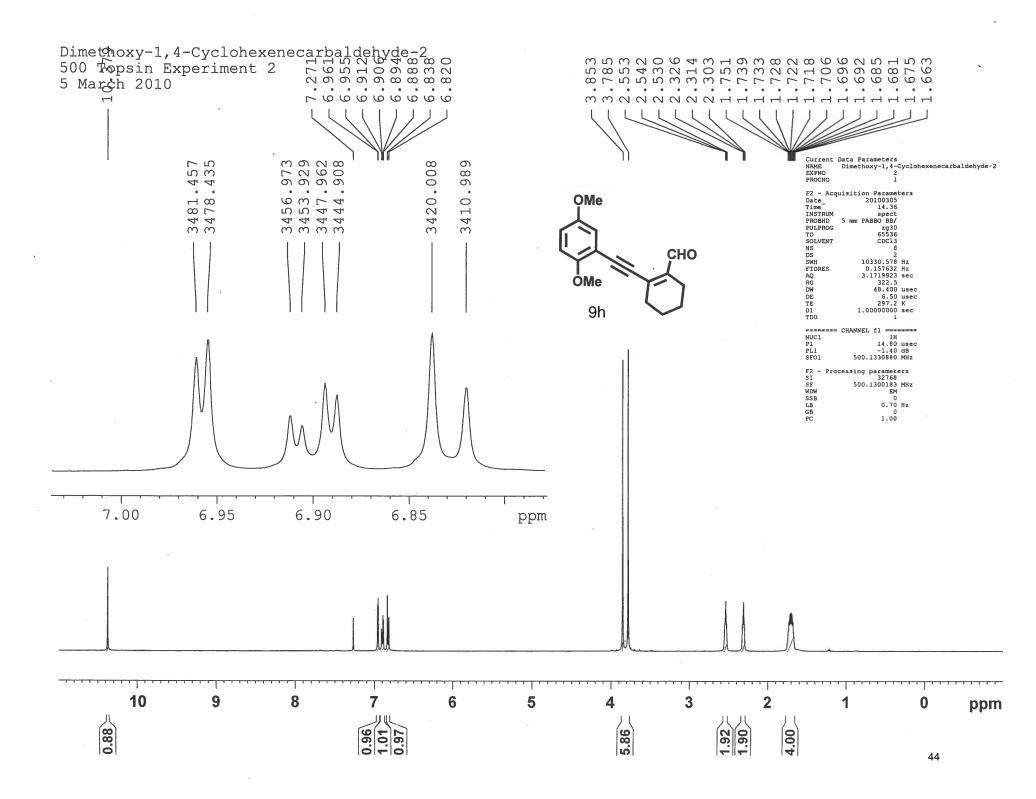
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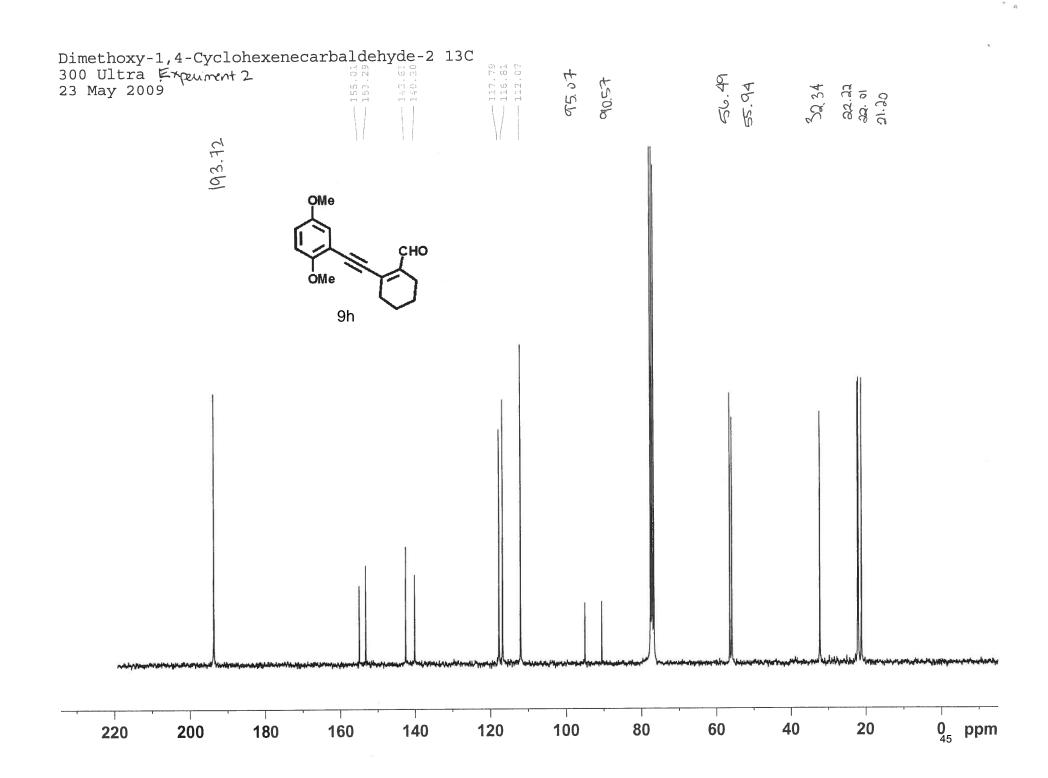


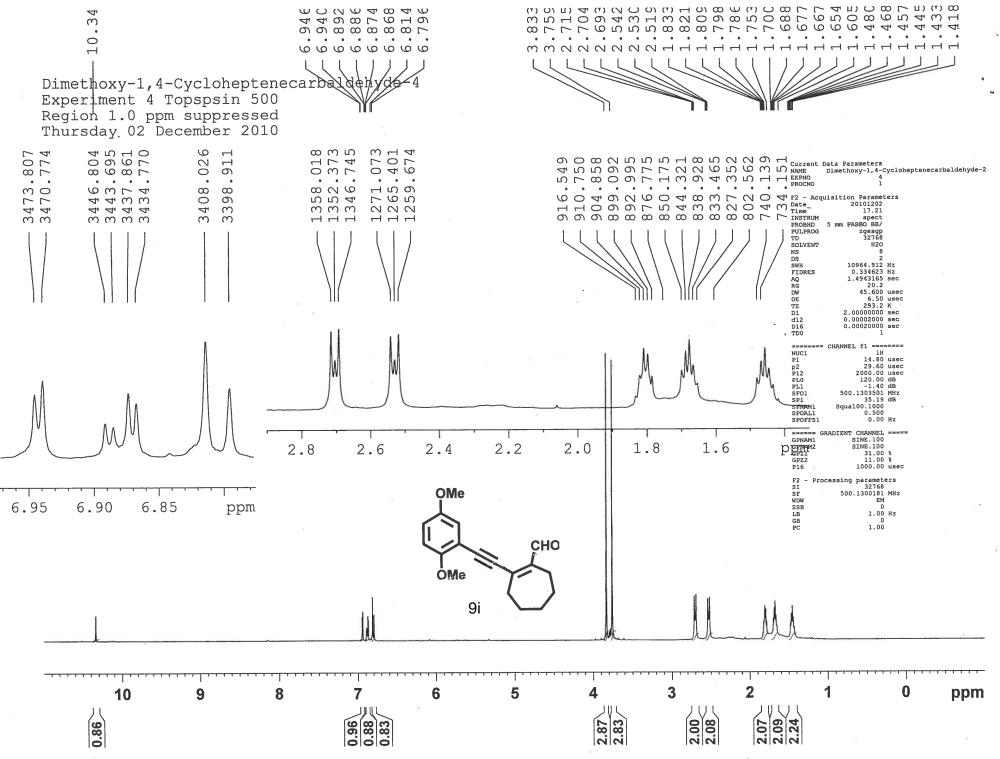
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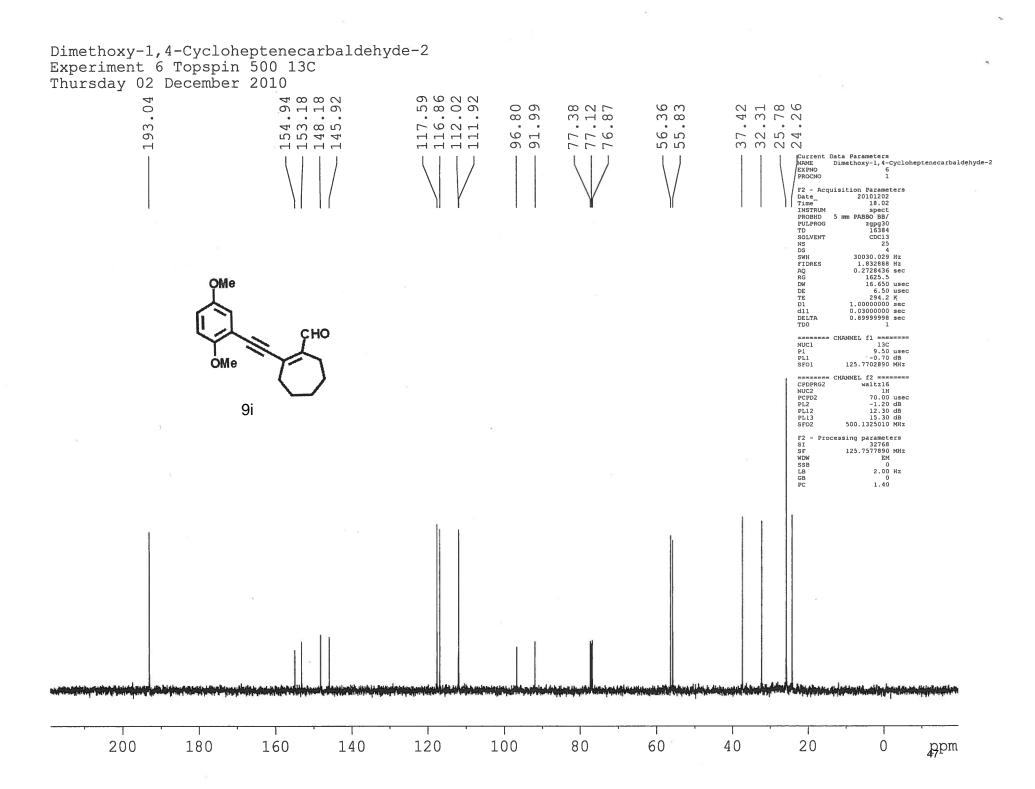


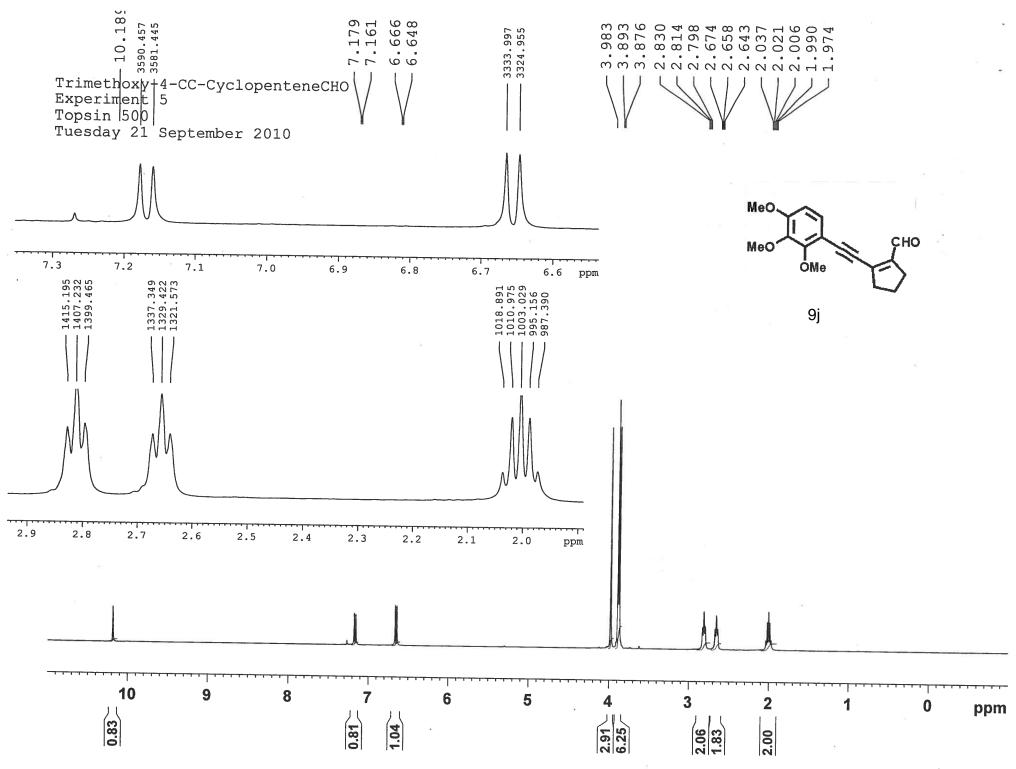


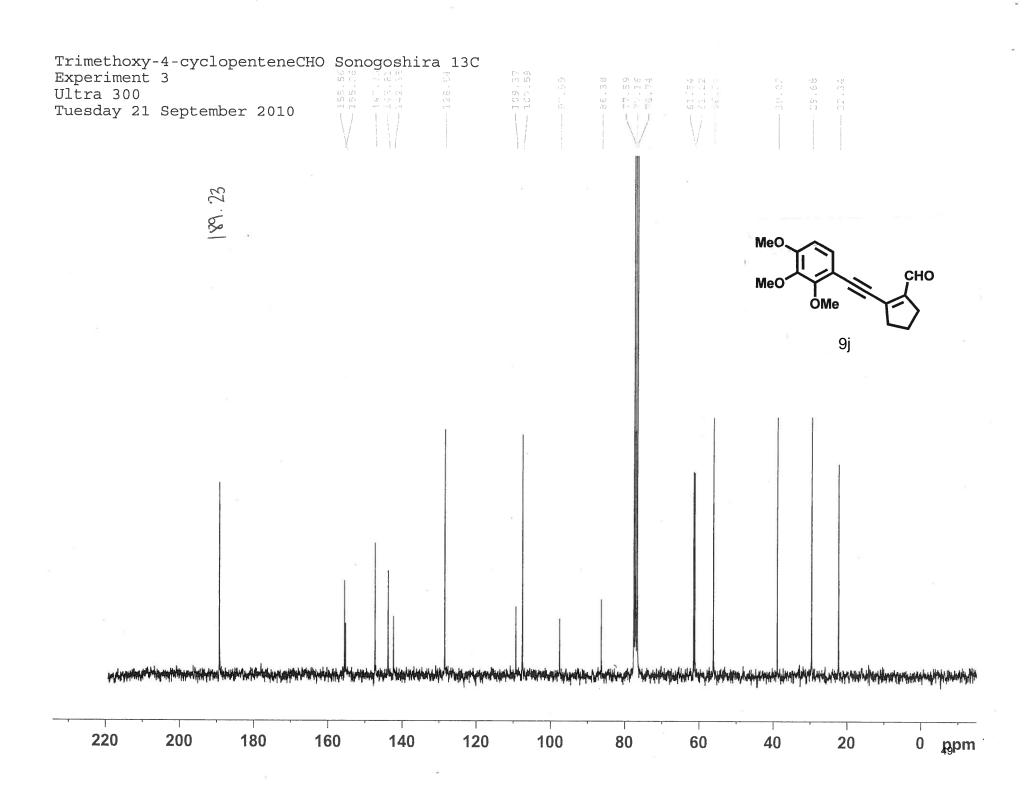


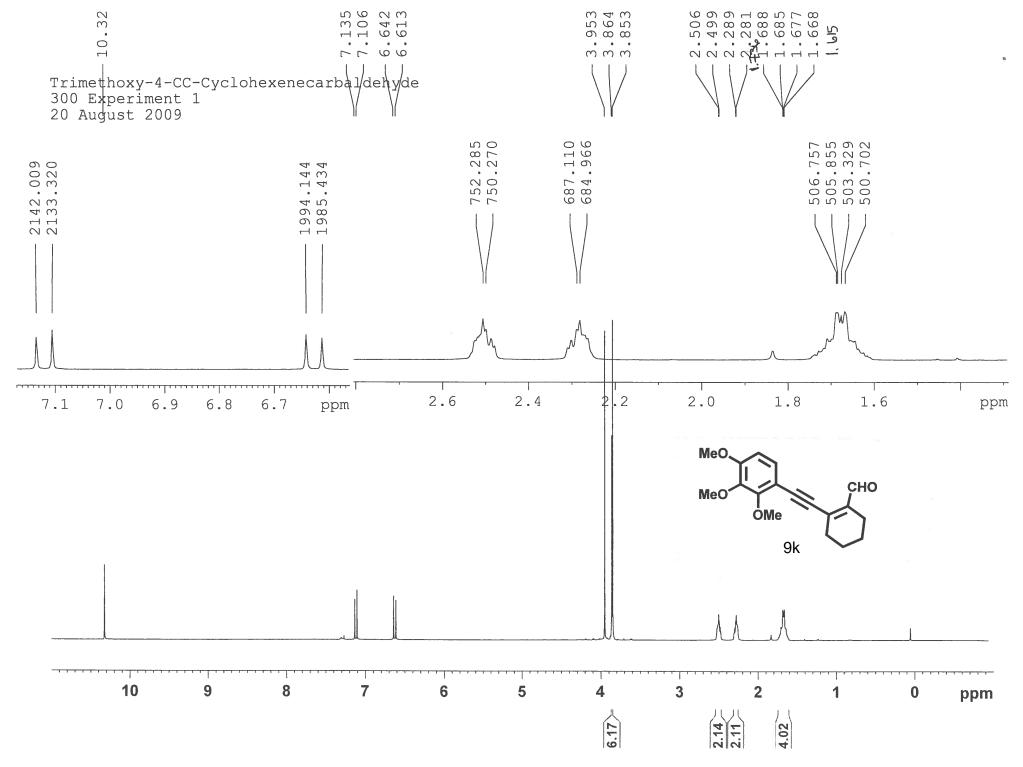


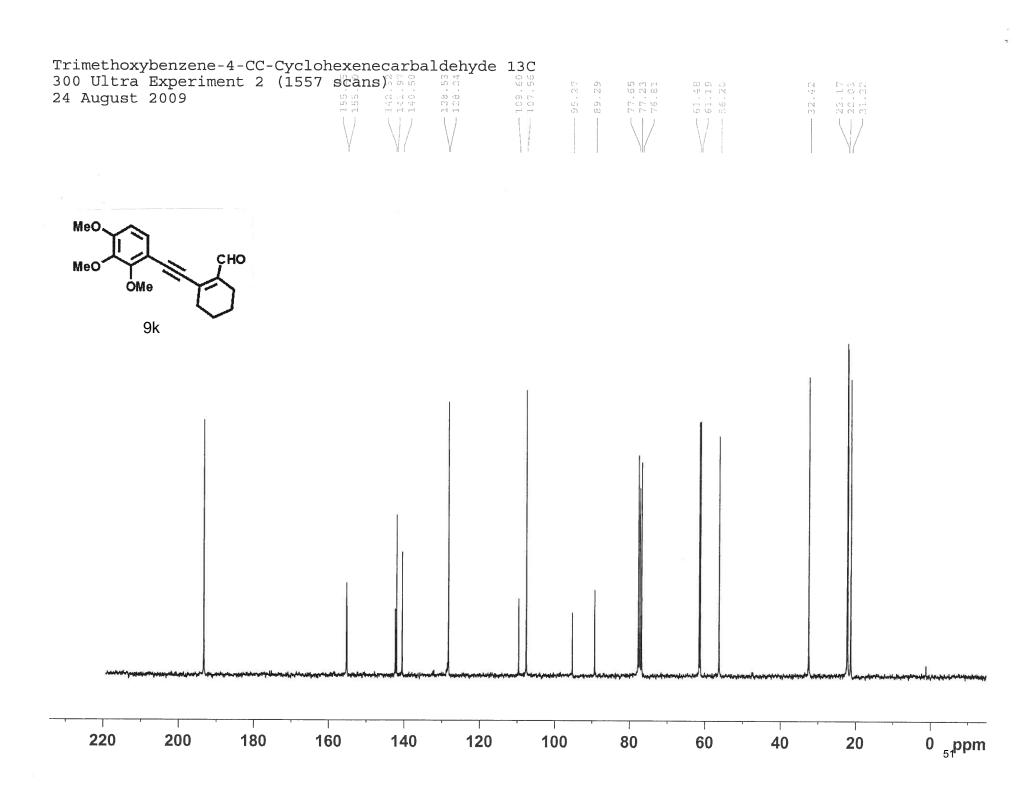


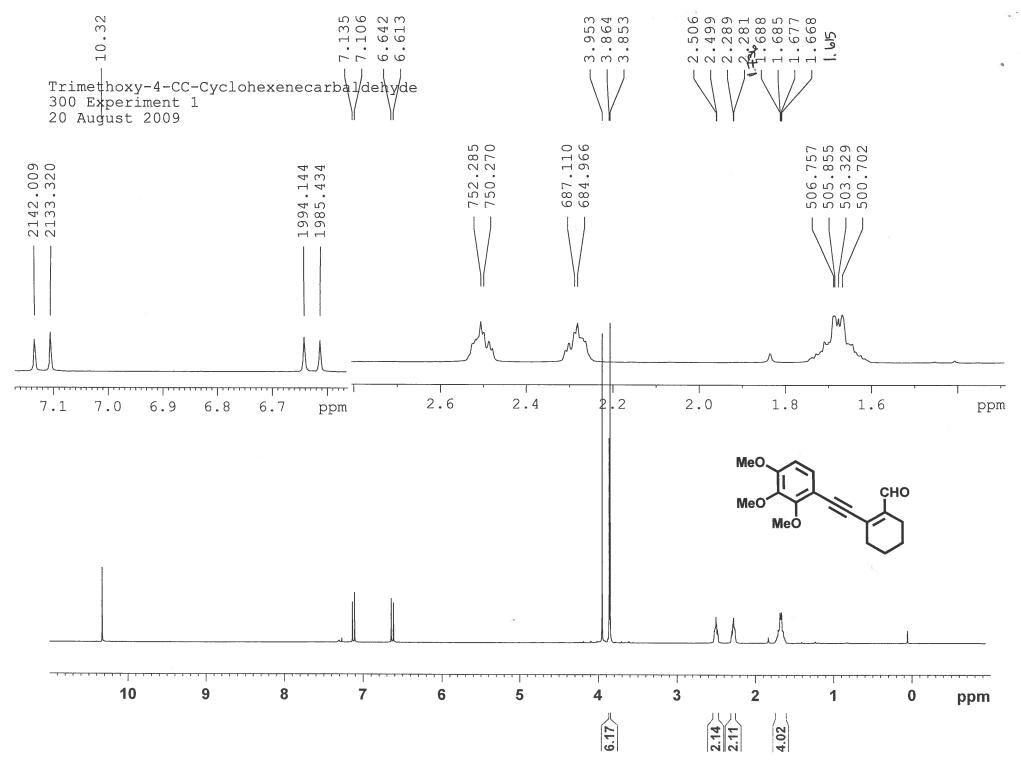


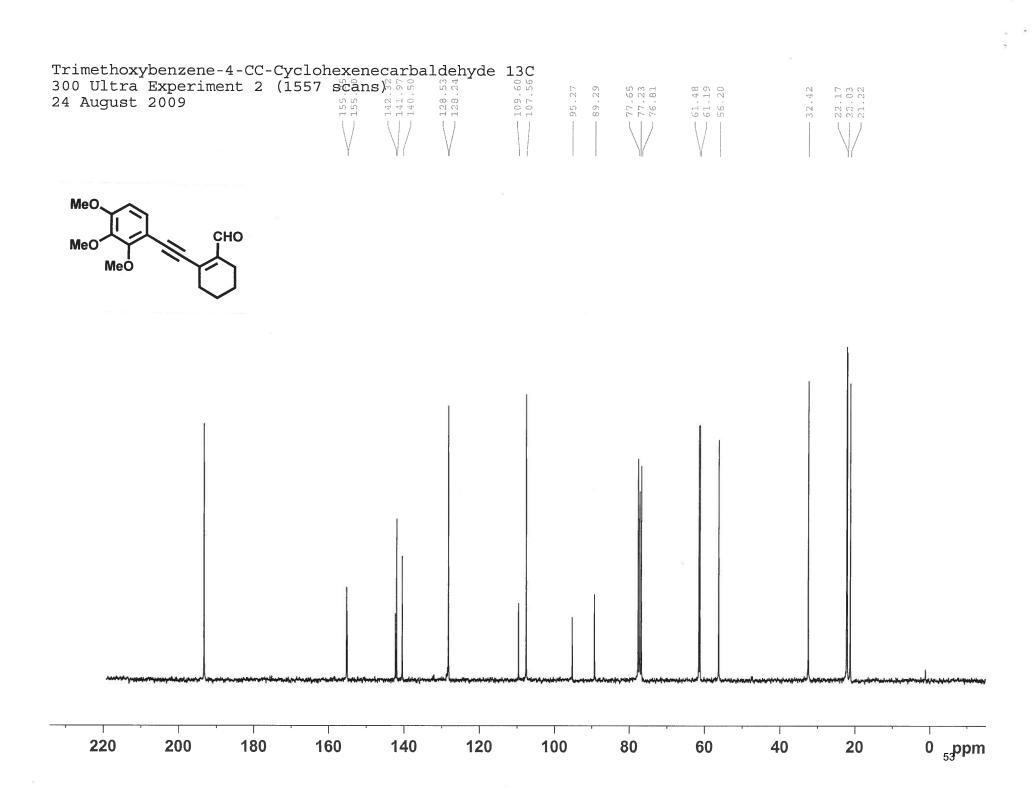


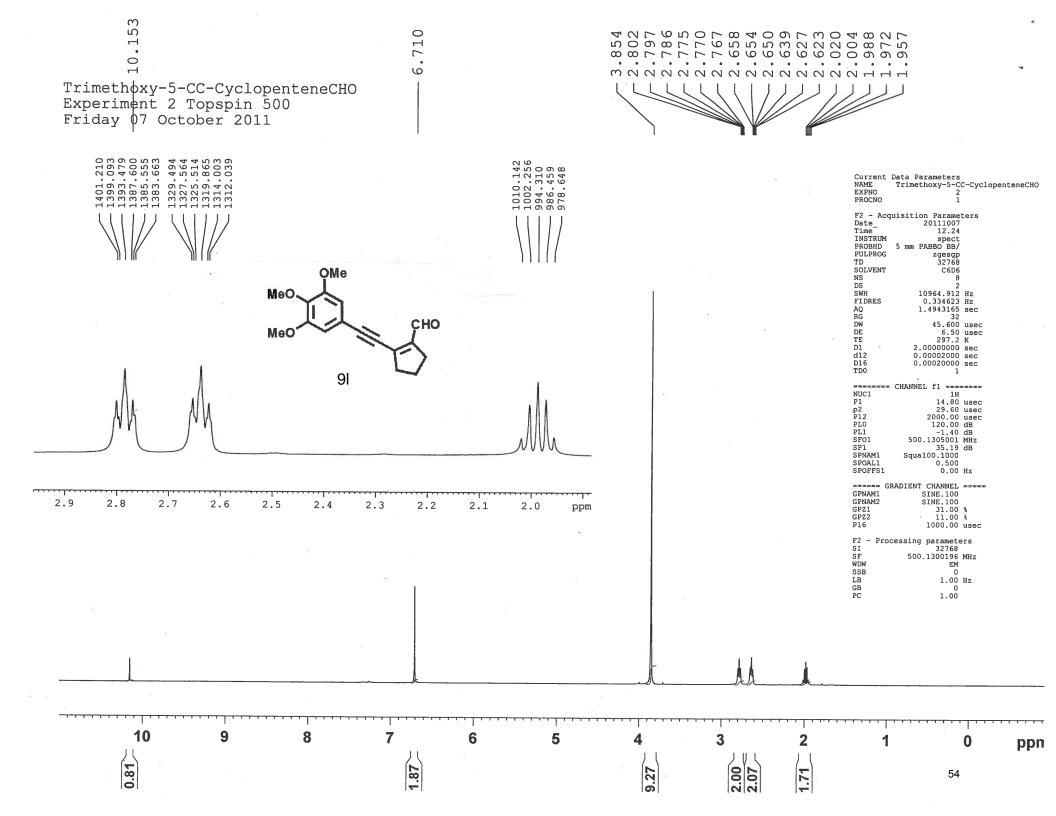


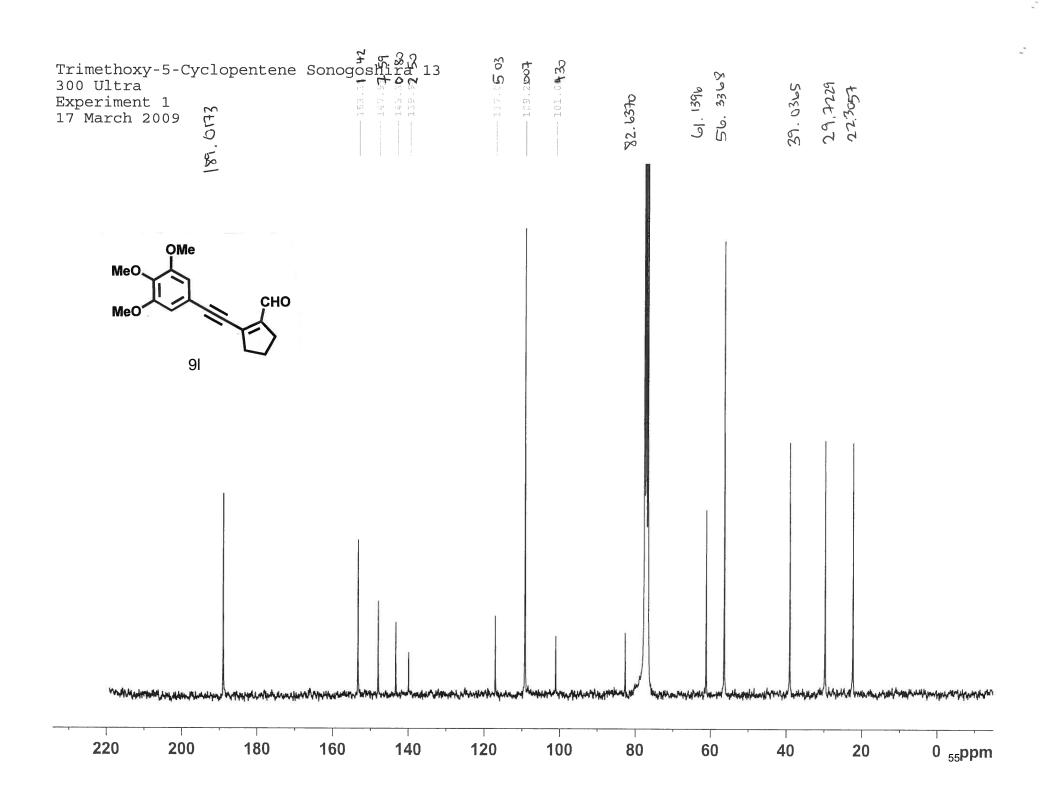


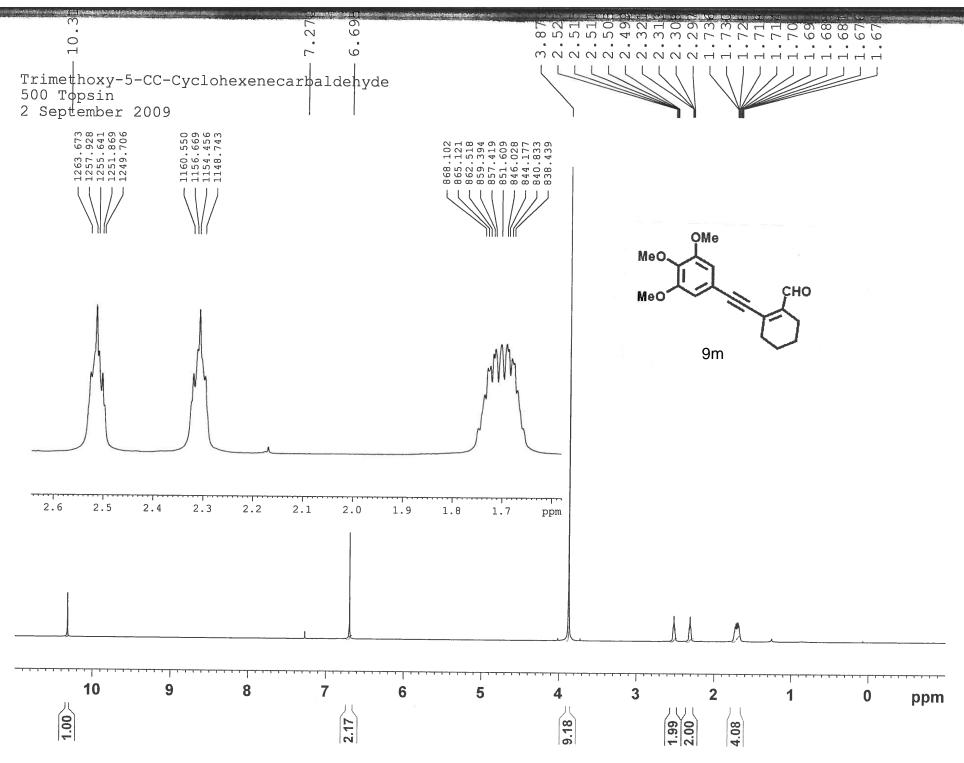


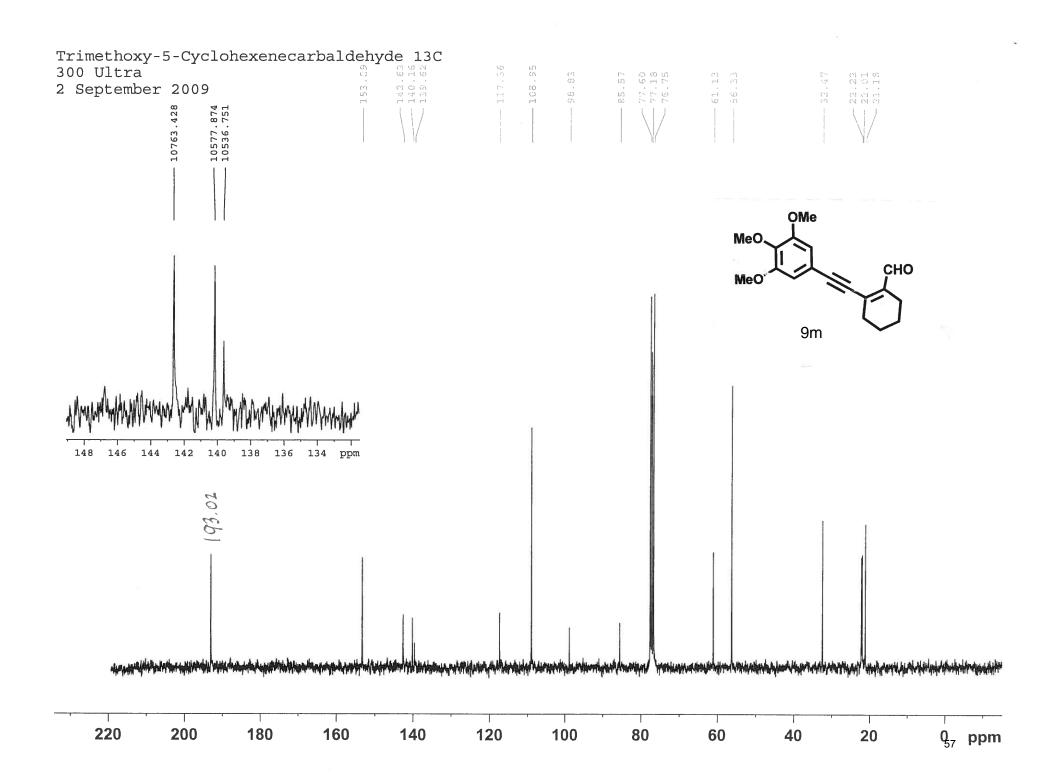


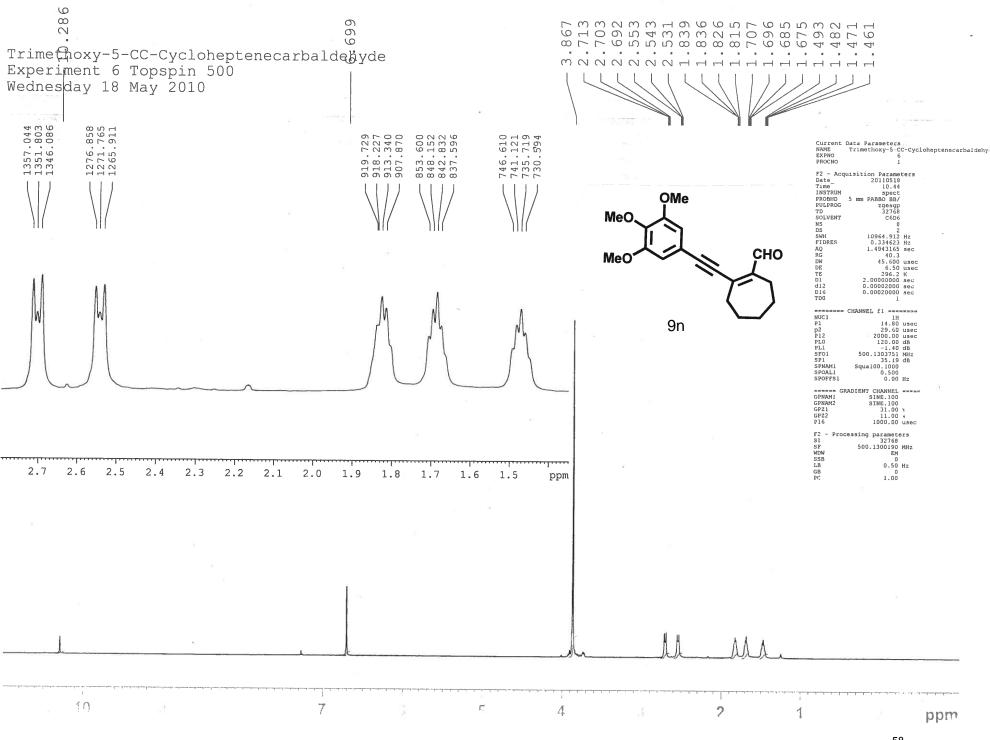




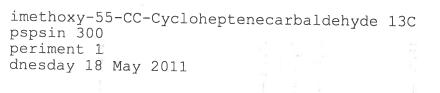


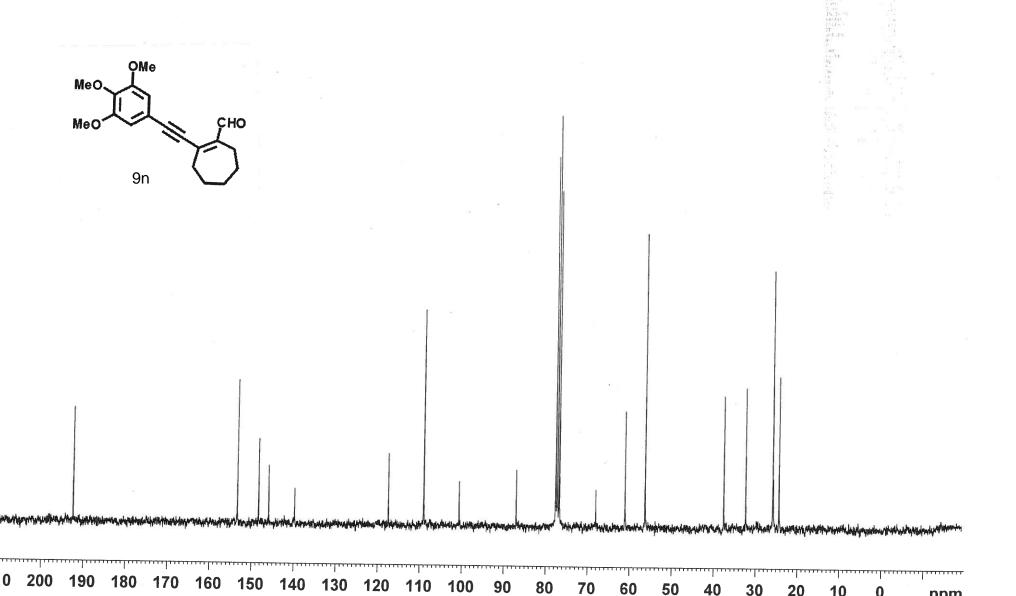




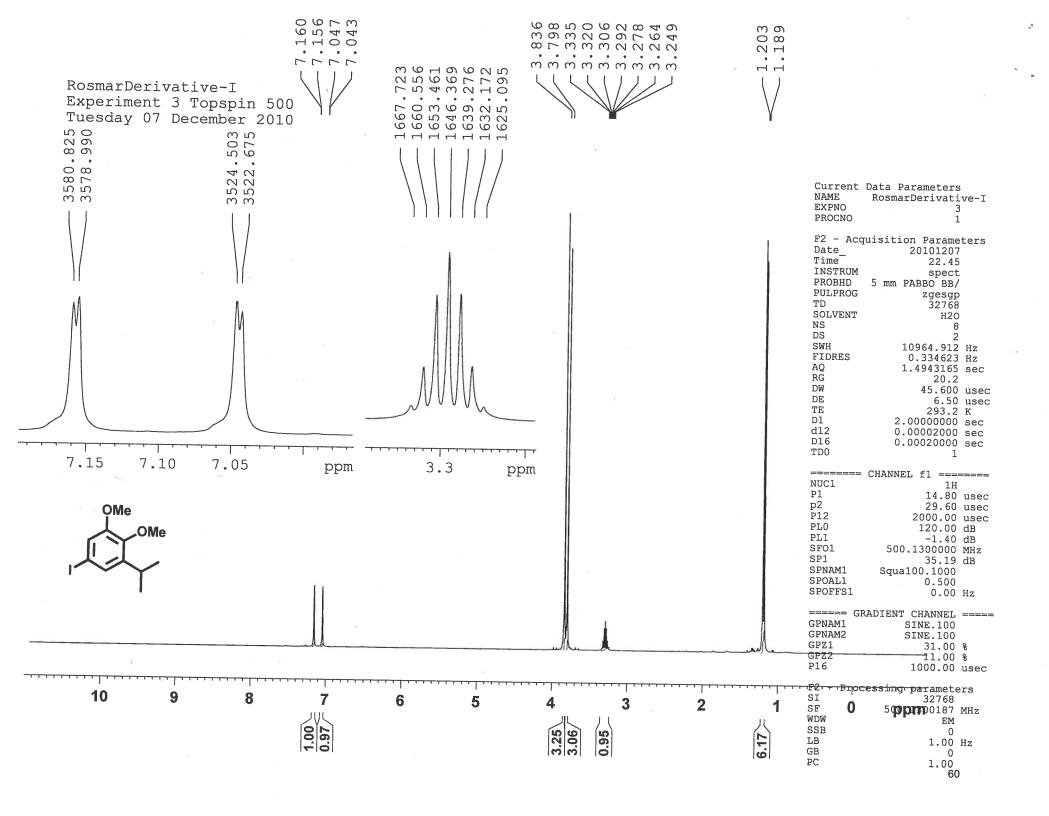


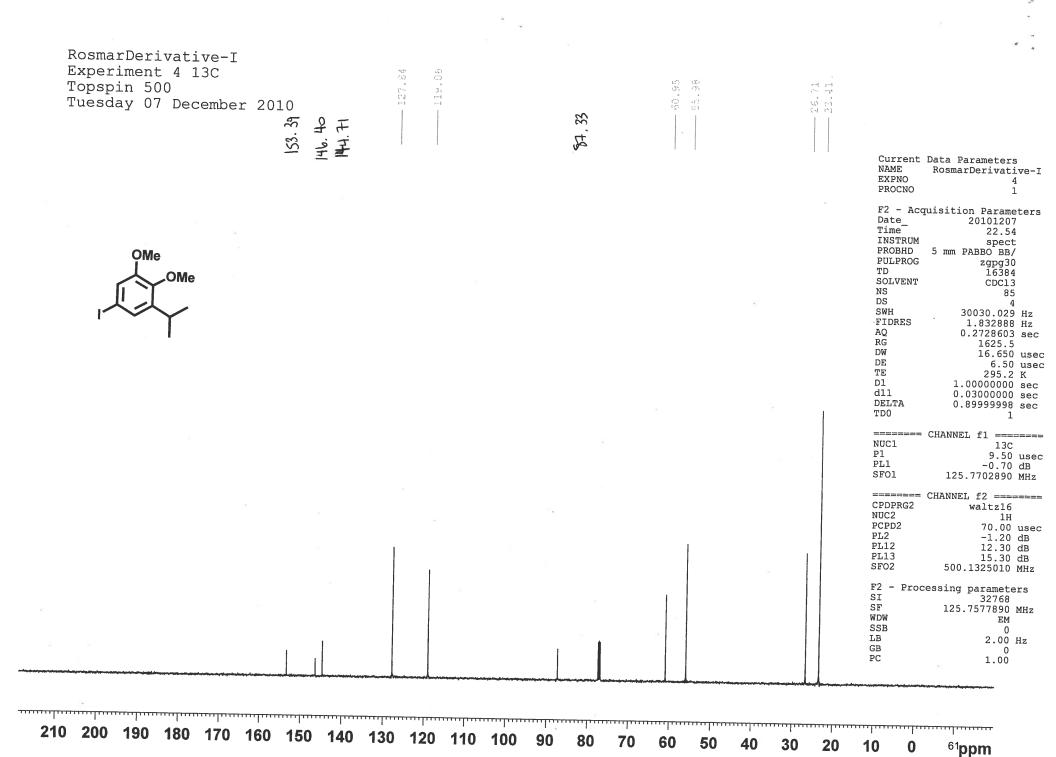
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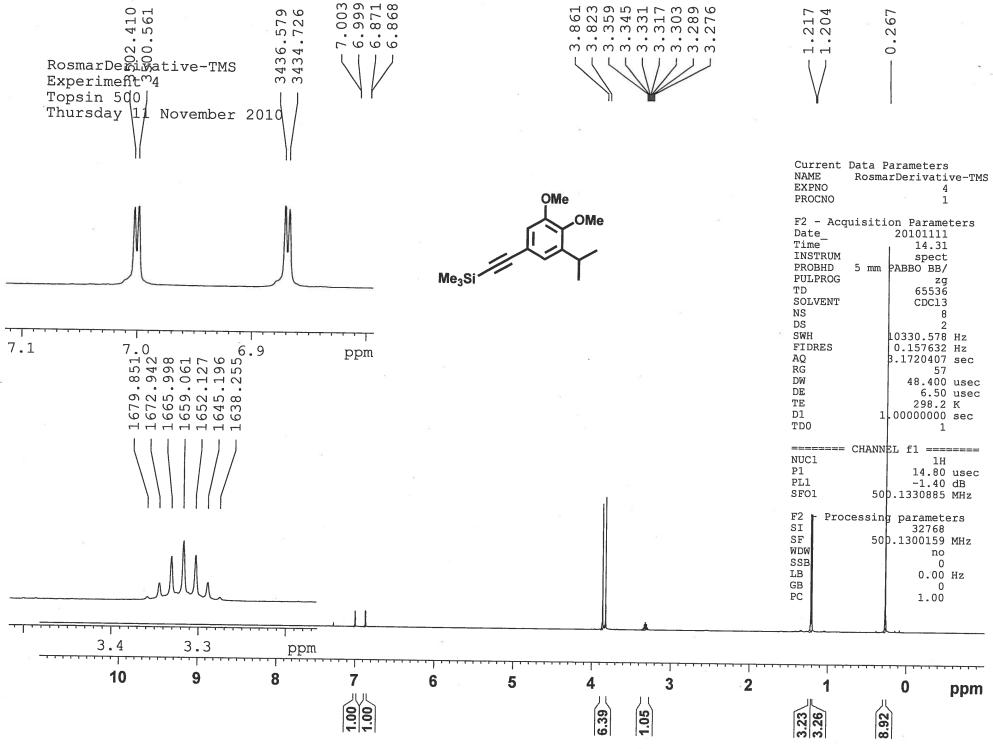


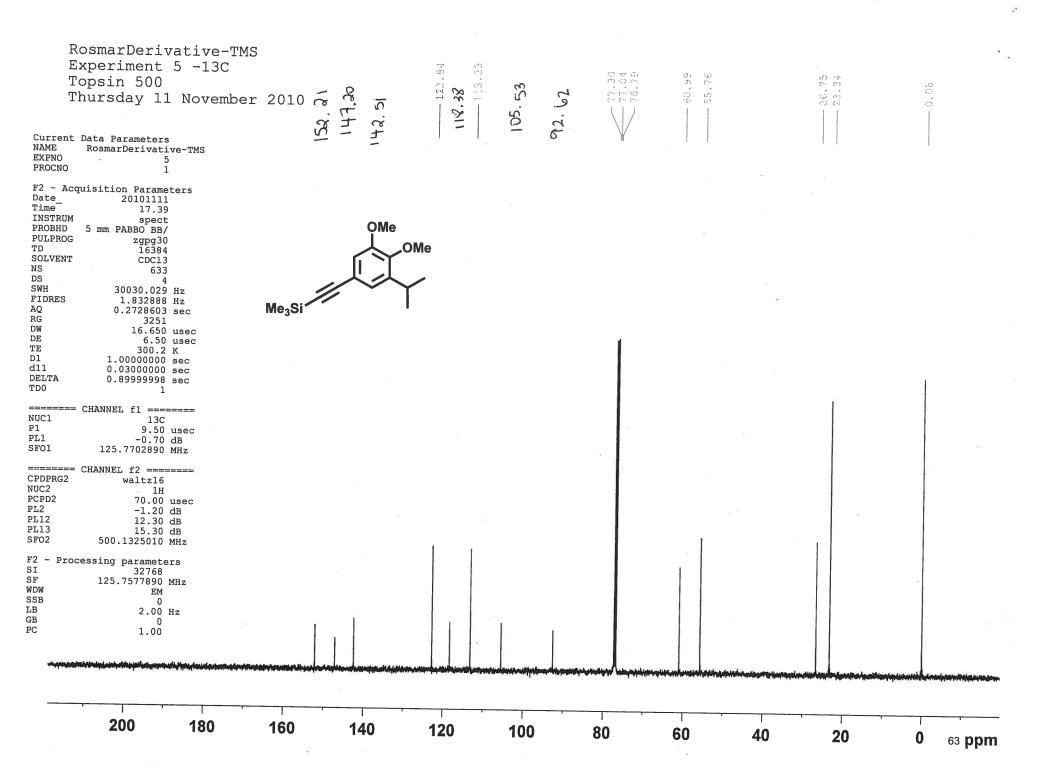


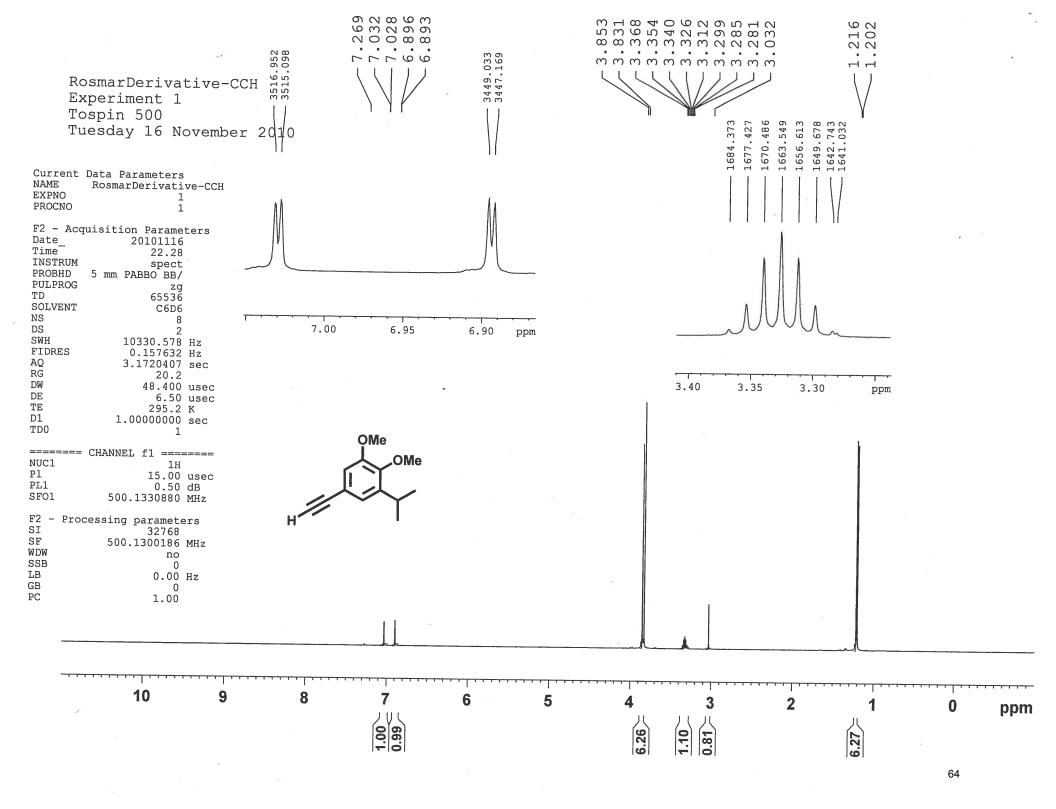
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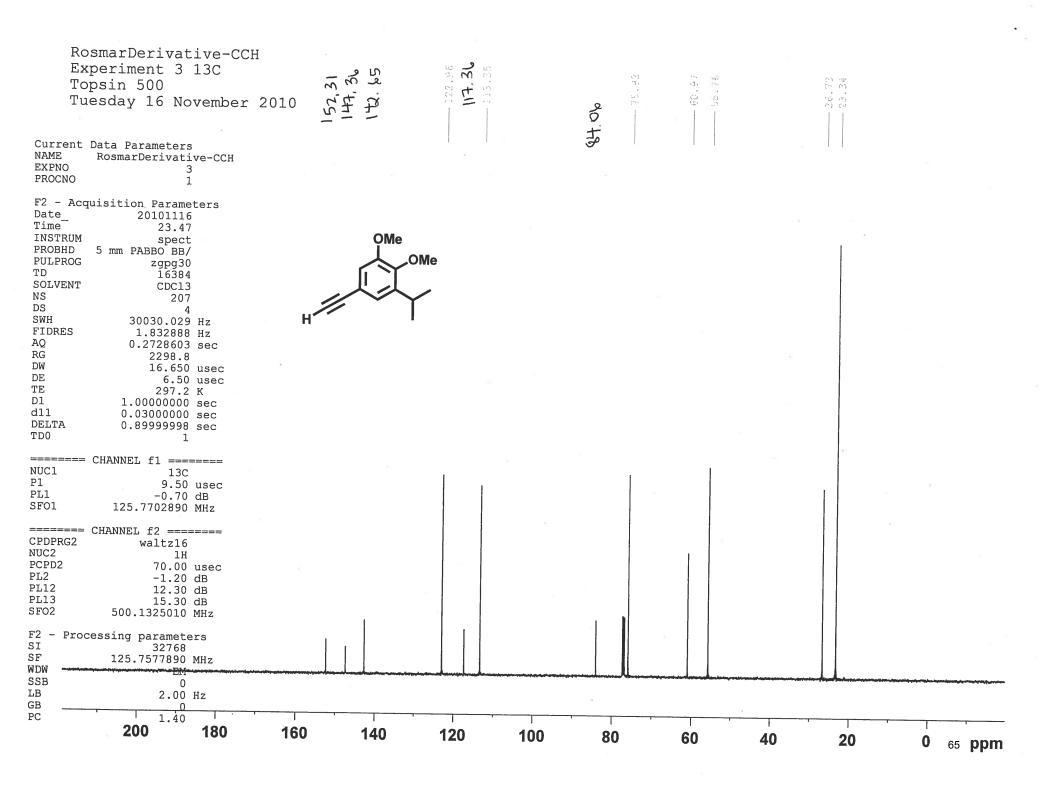


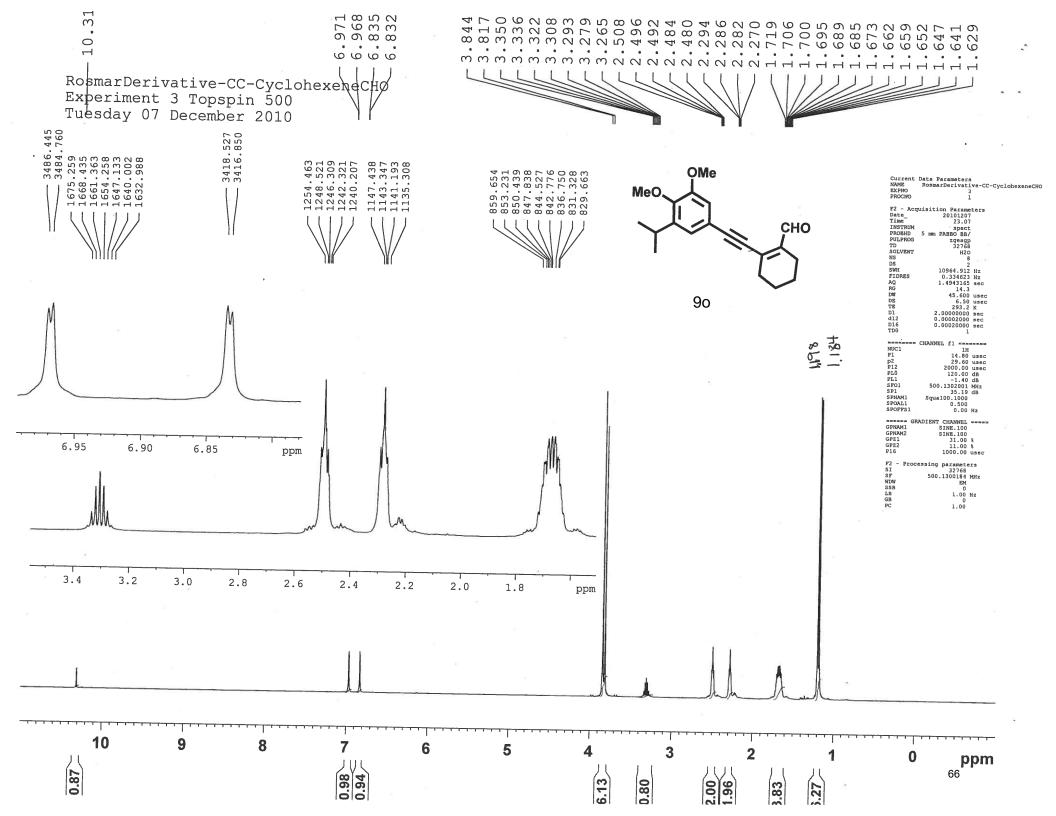


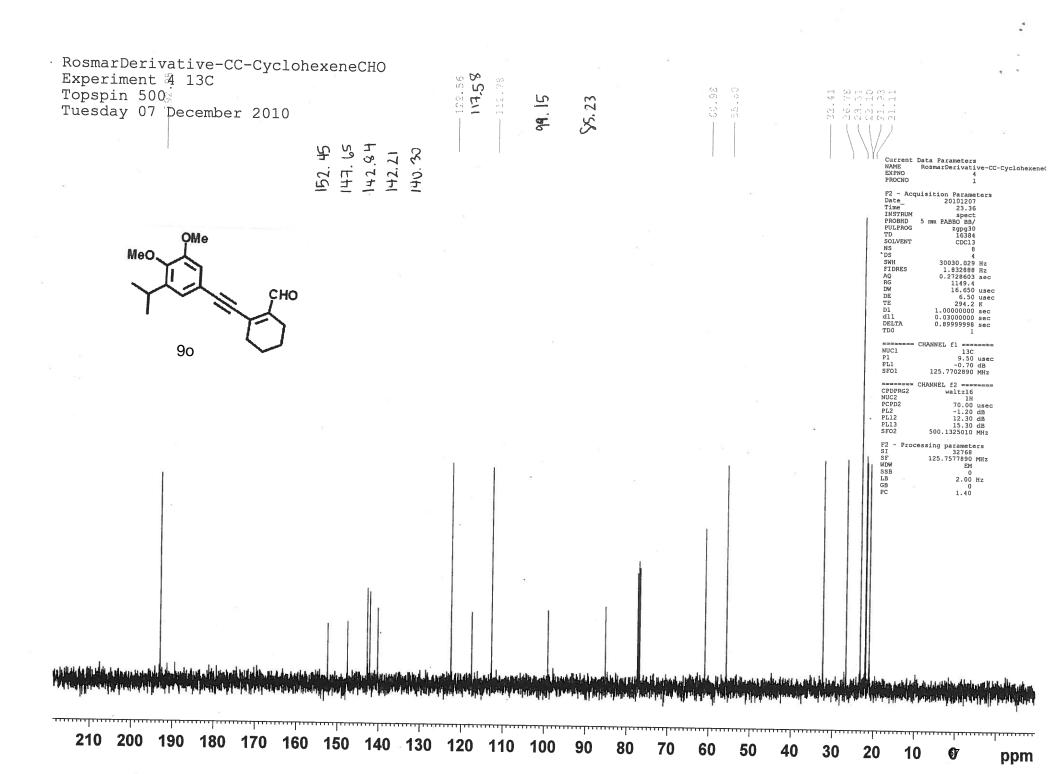


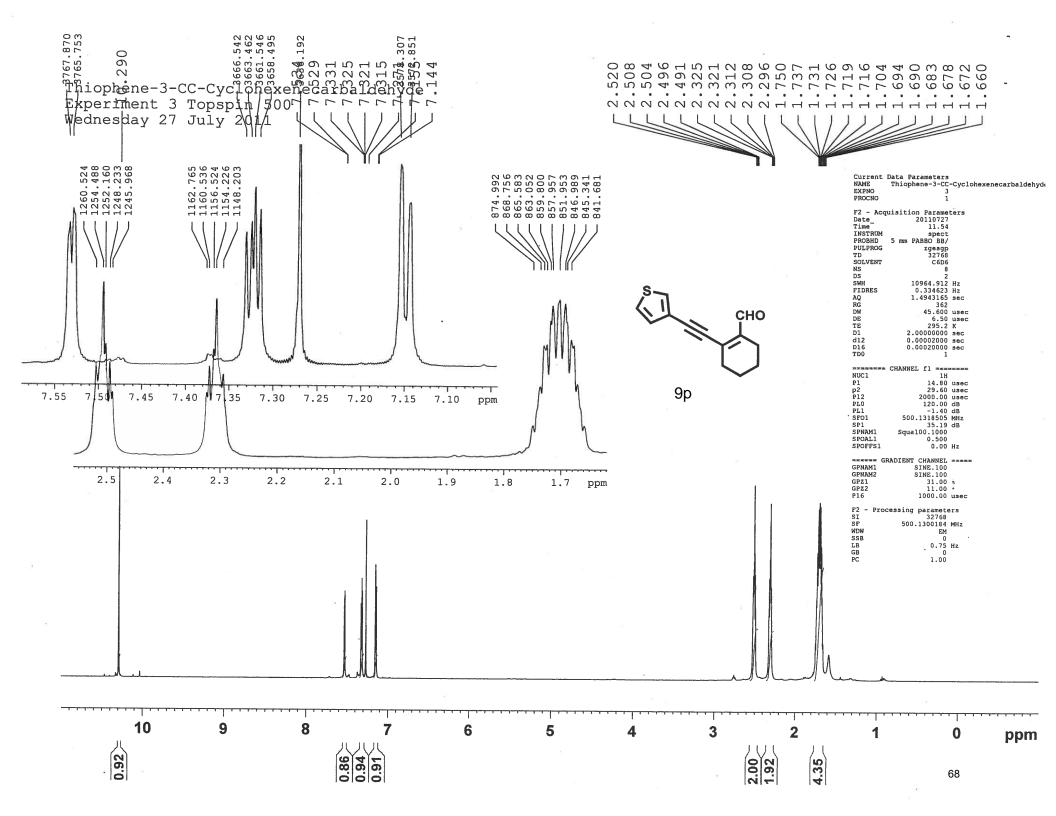




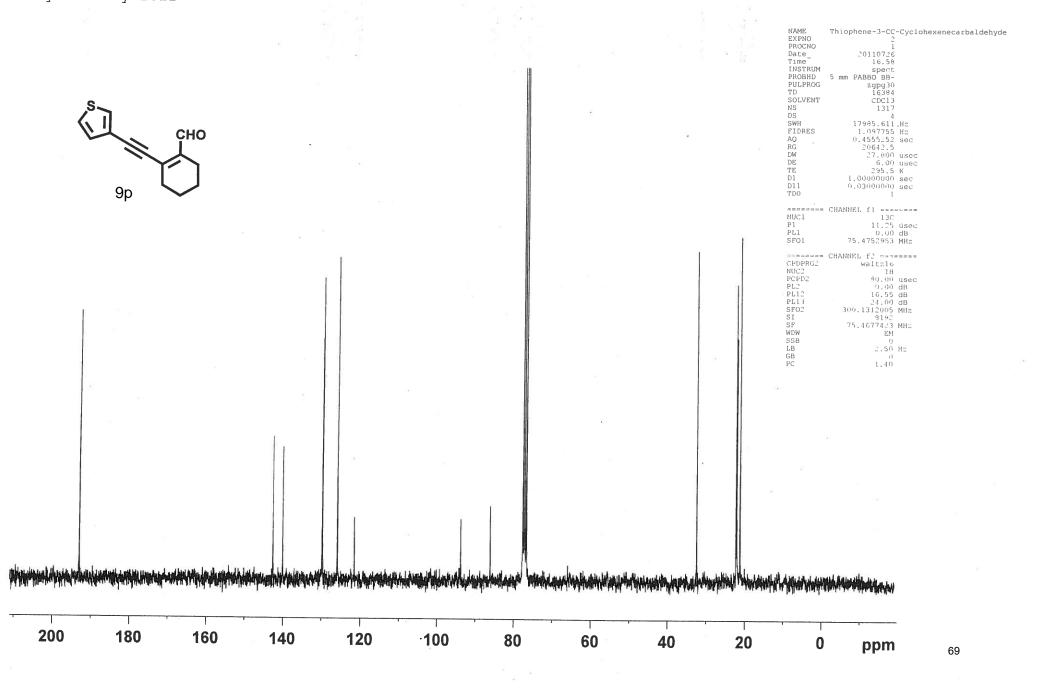


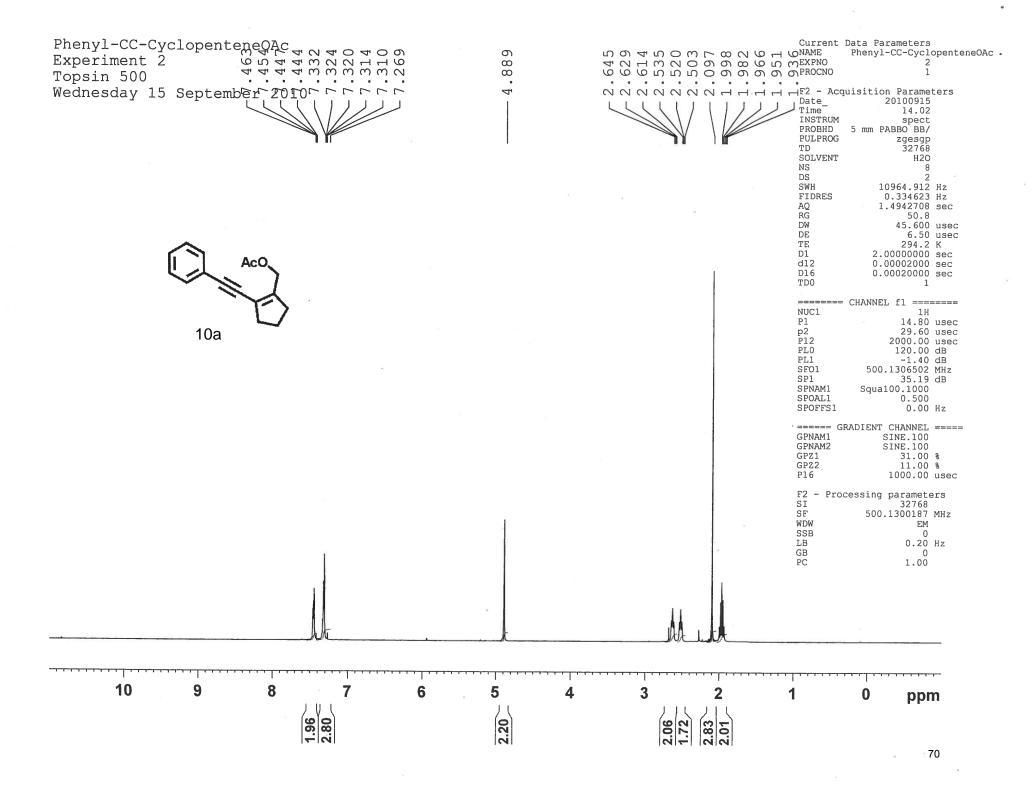


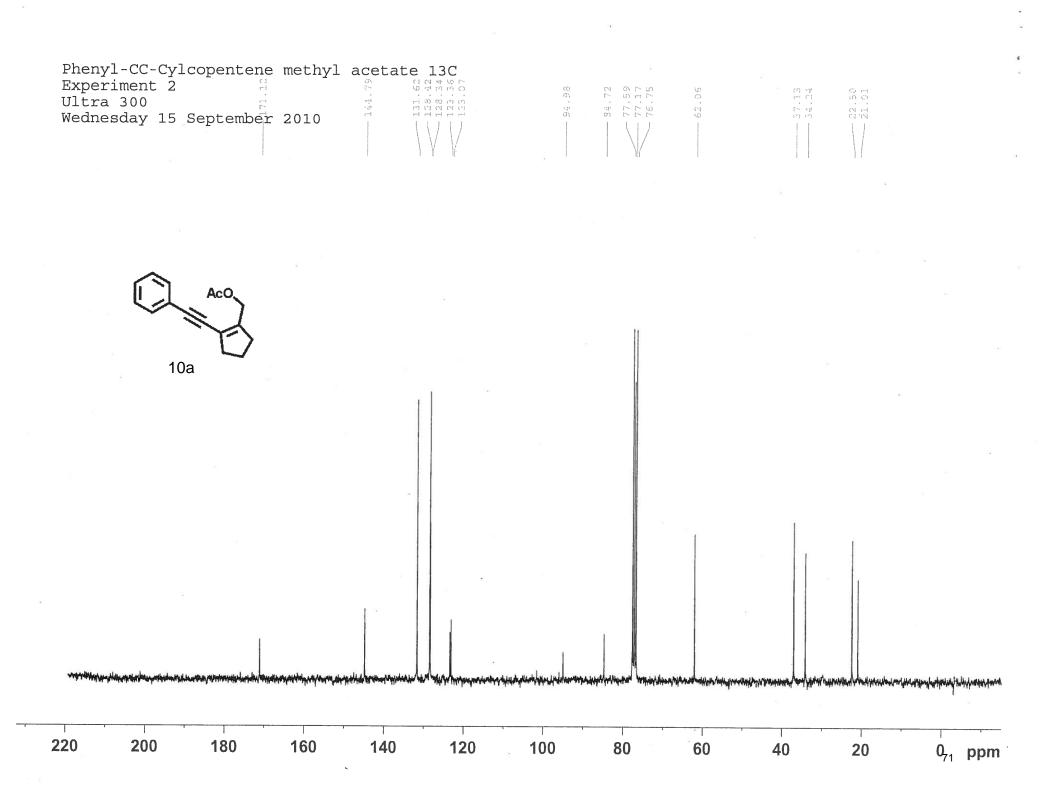


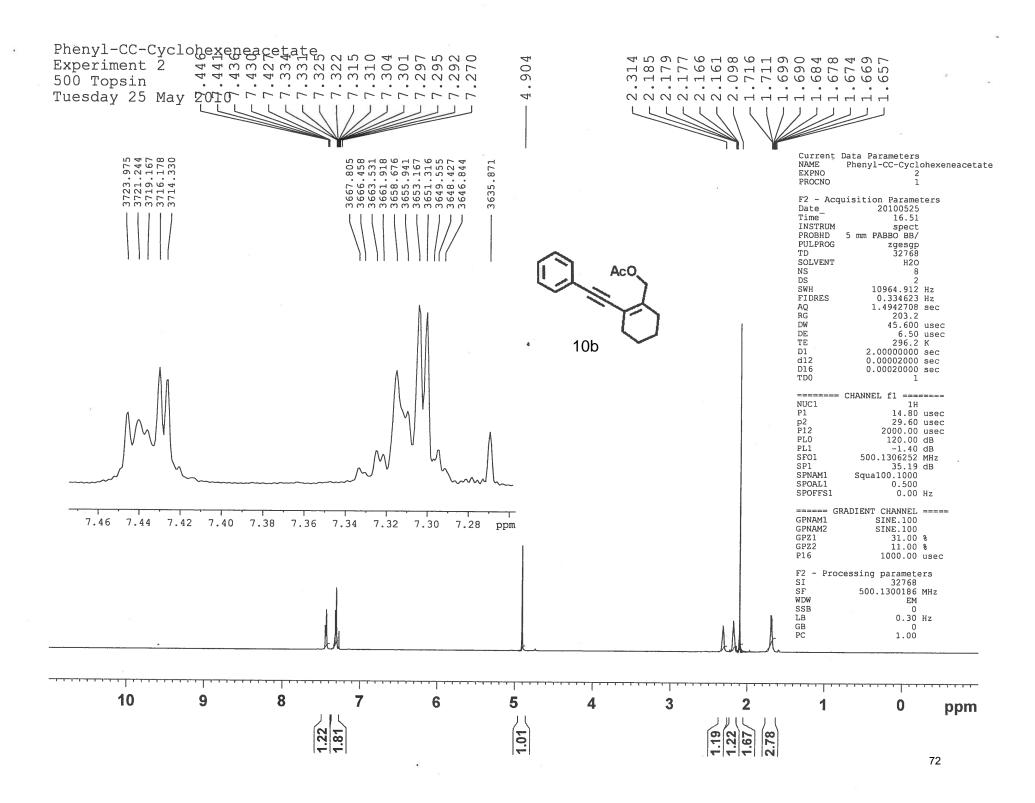


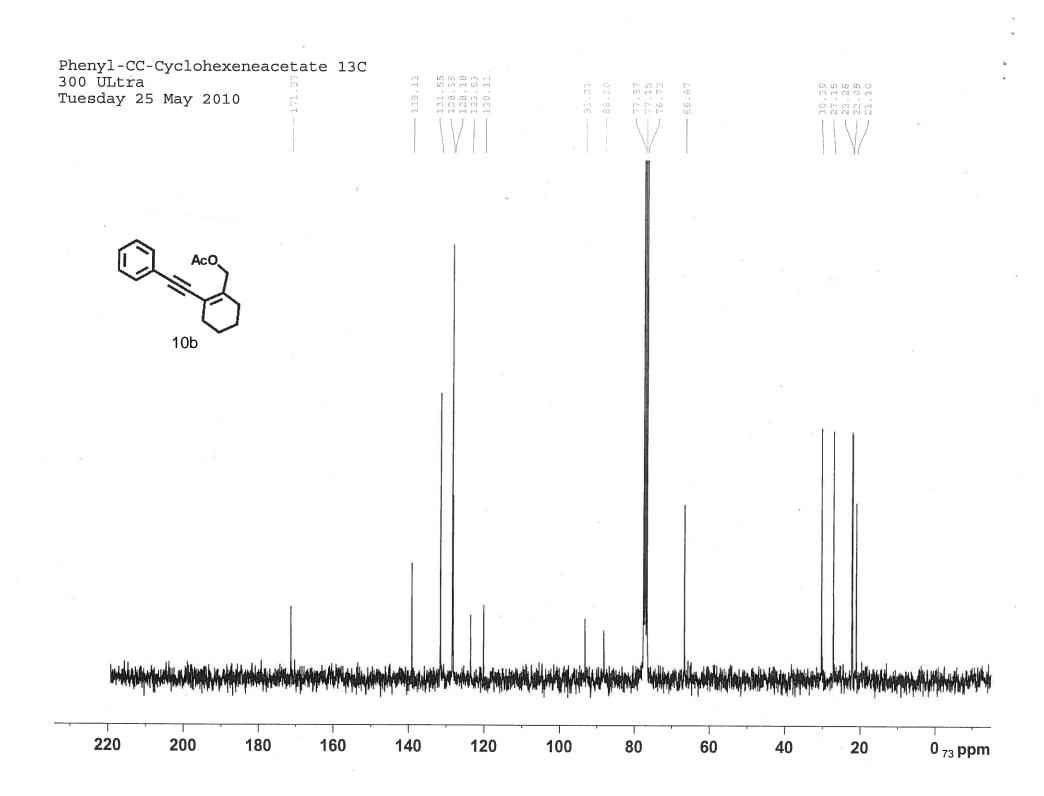
iophene-3-CC-Cyclohexenecarbaldehyde
periment 2
pspin 300 Ultra
esday 26 July 2011

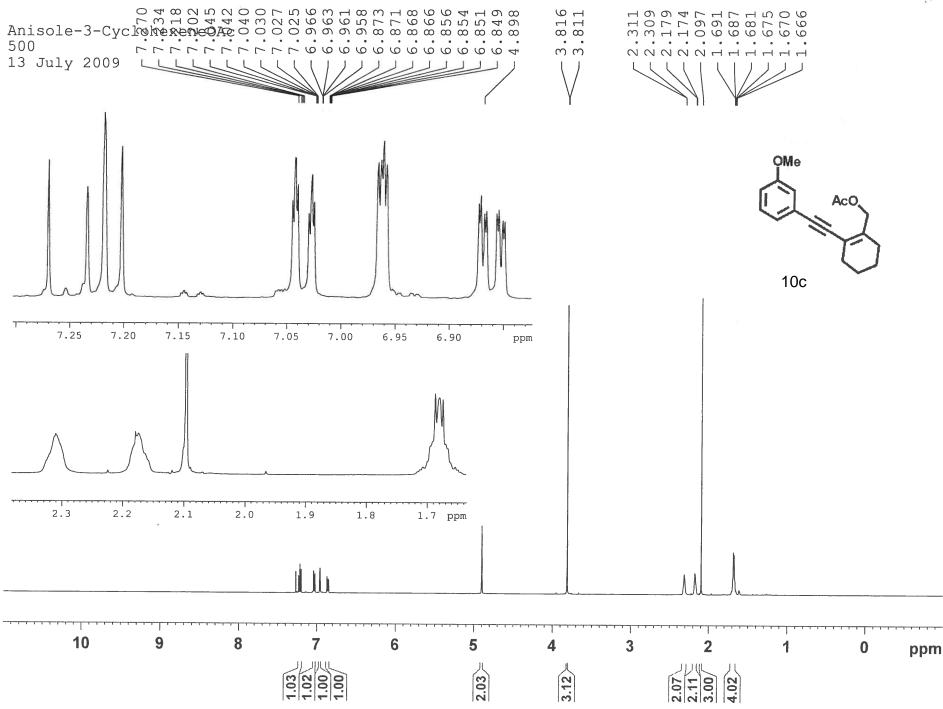


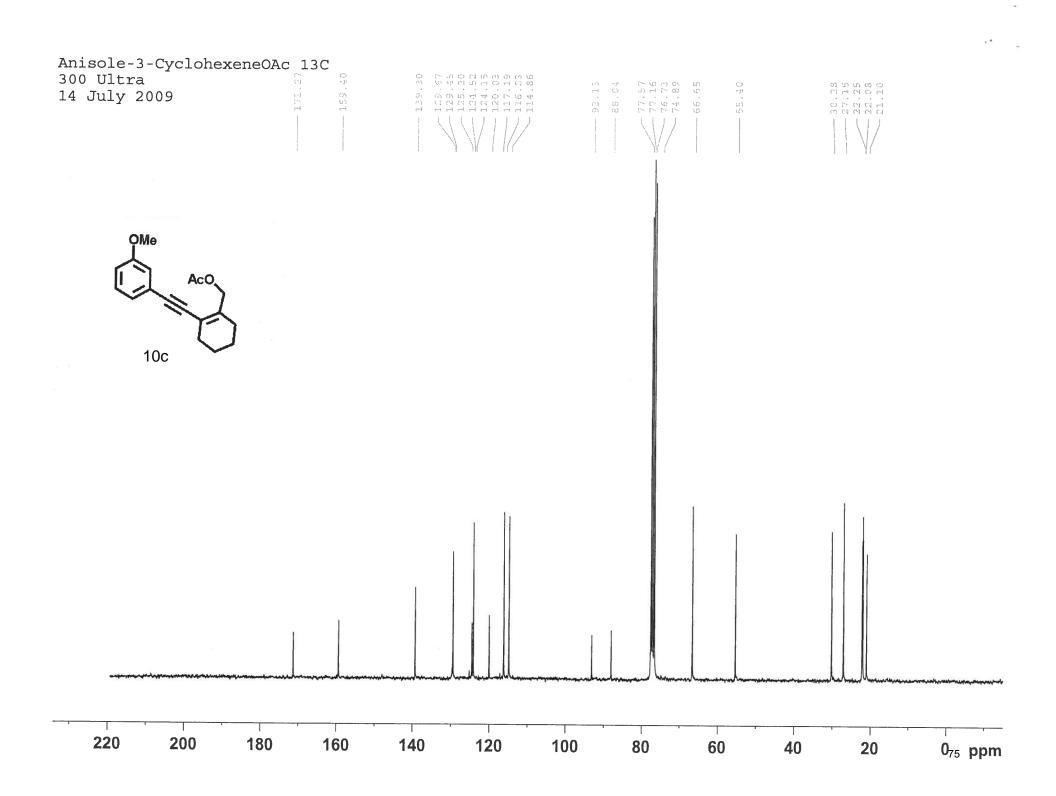


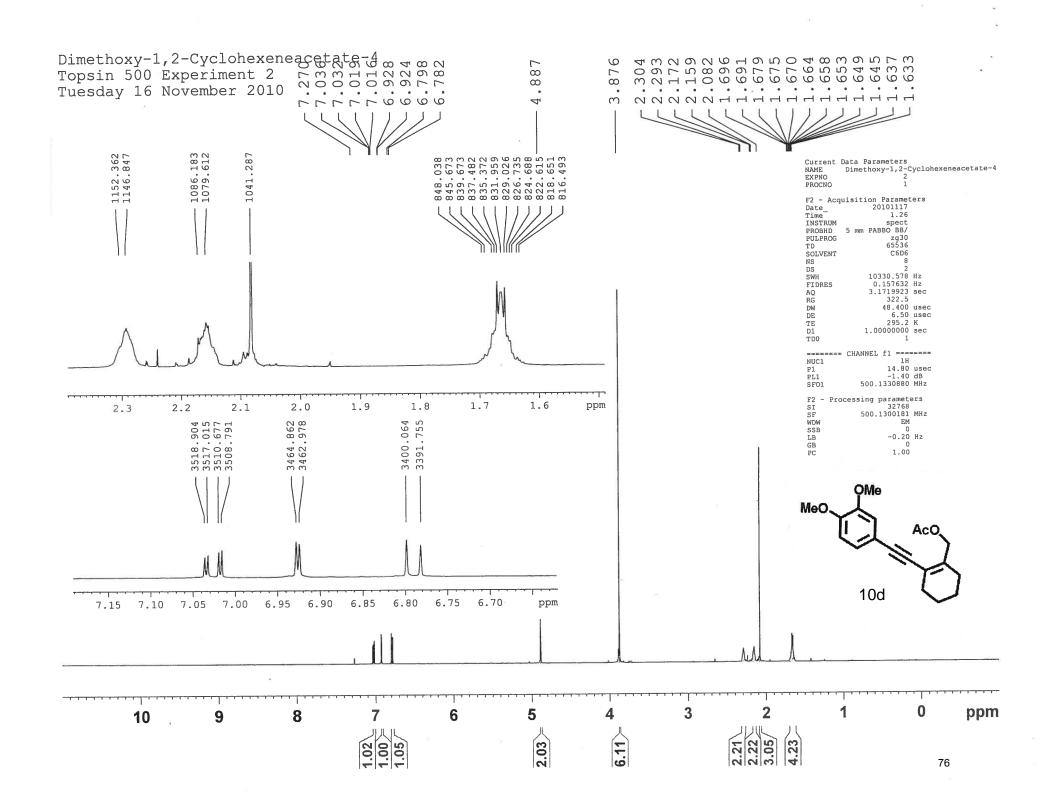


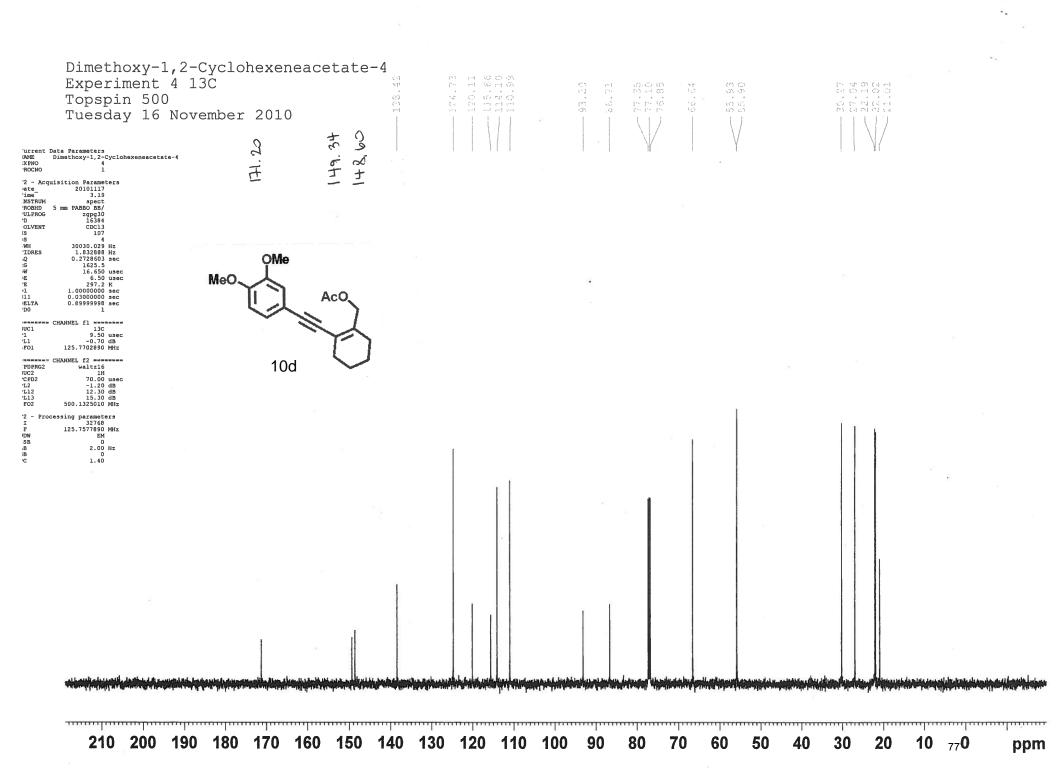


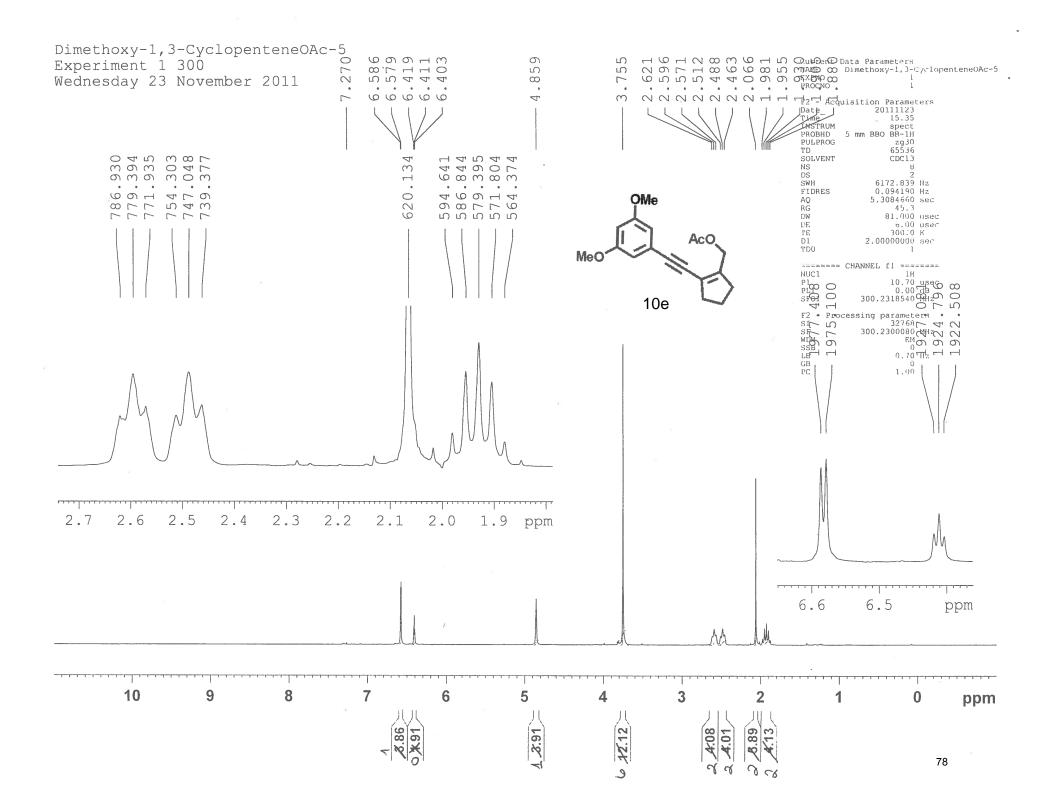


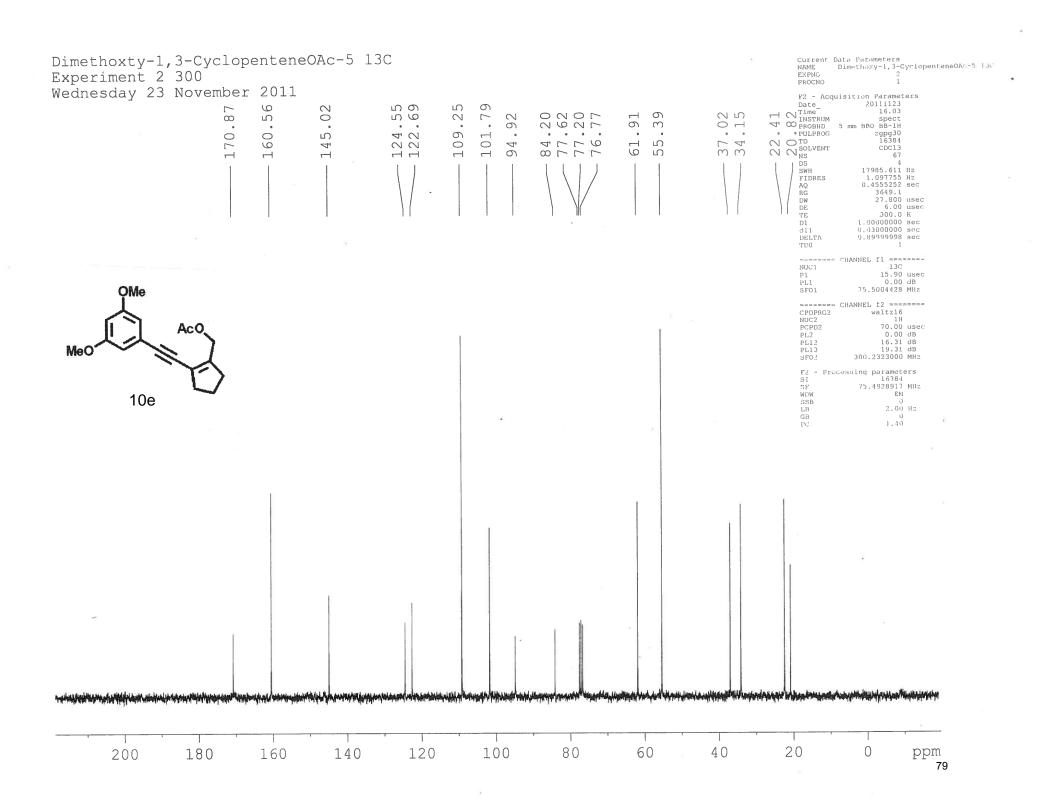


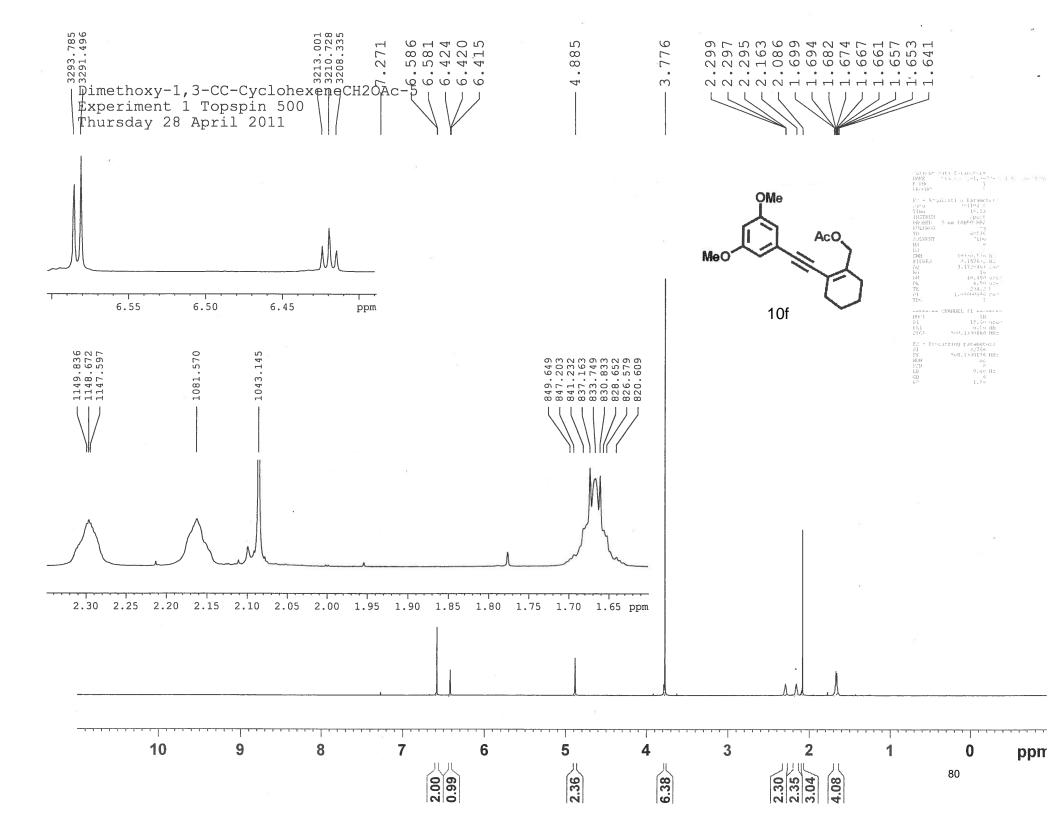






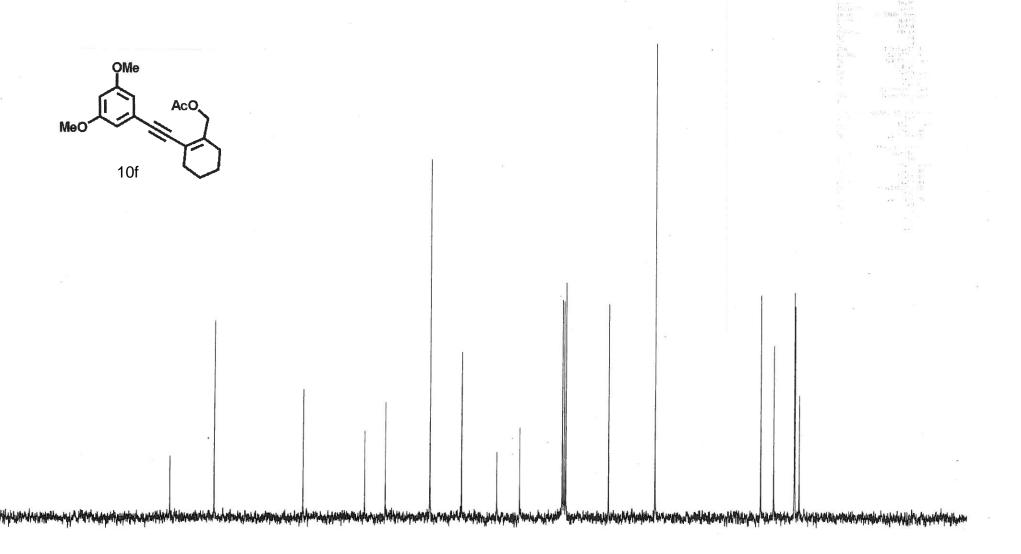




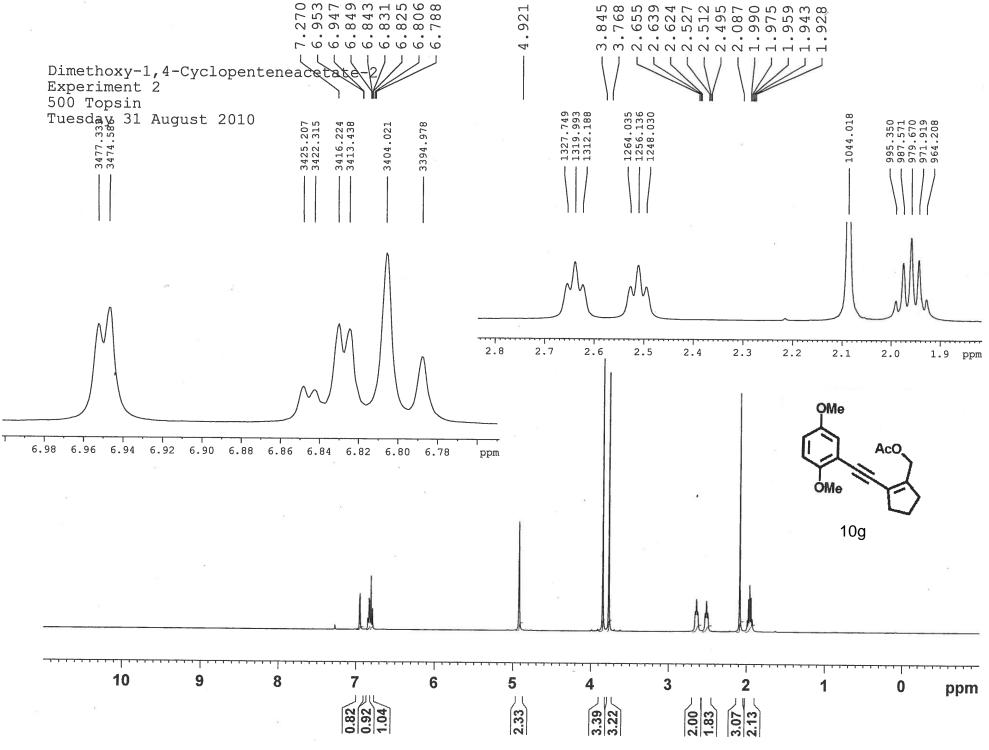


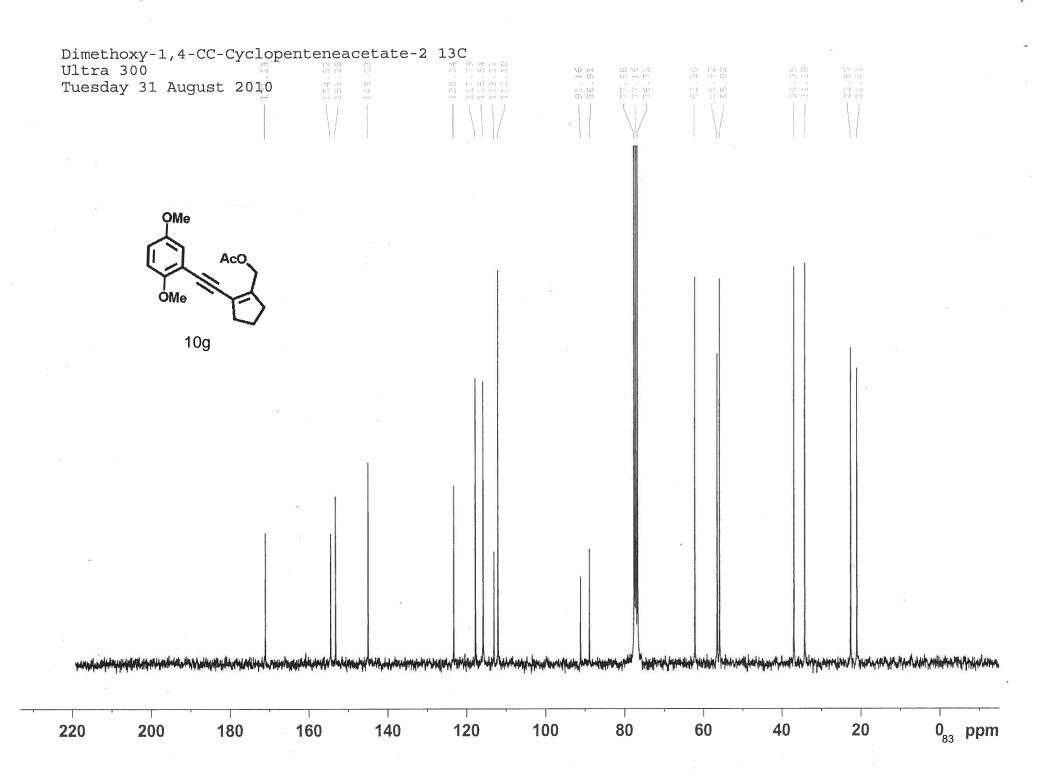
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pspin 300 Ultra
iday 29 April 2011
:periment 1

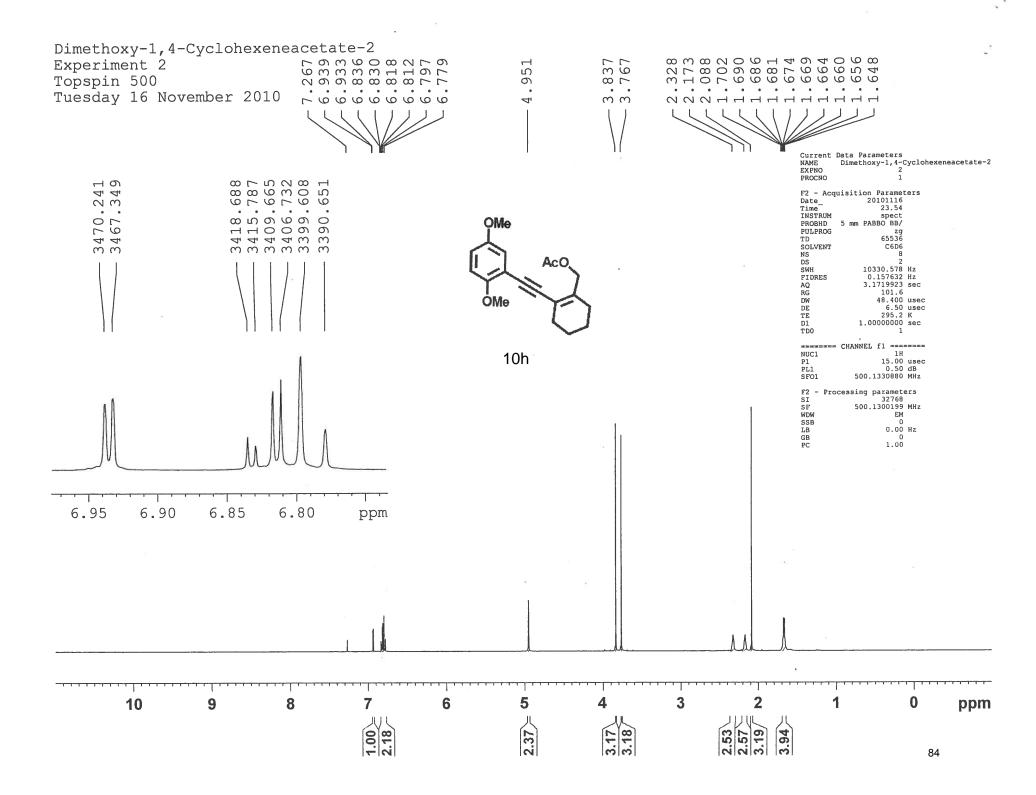
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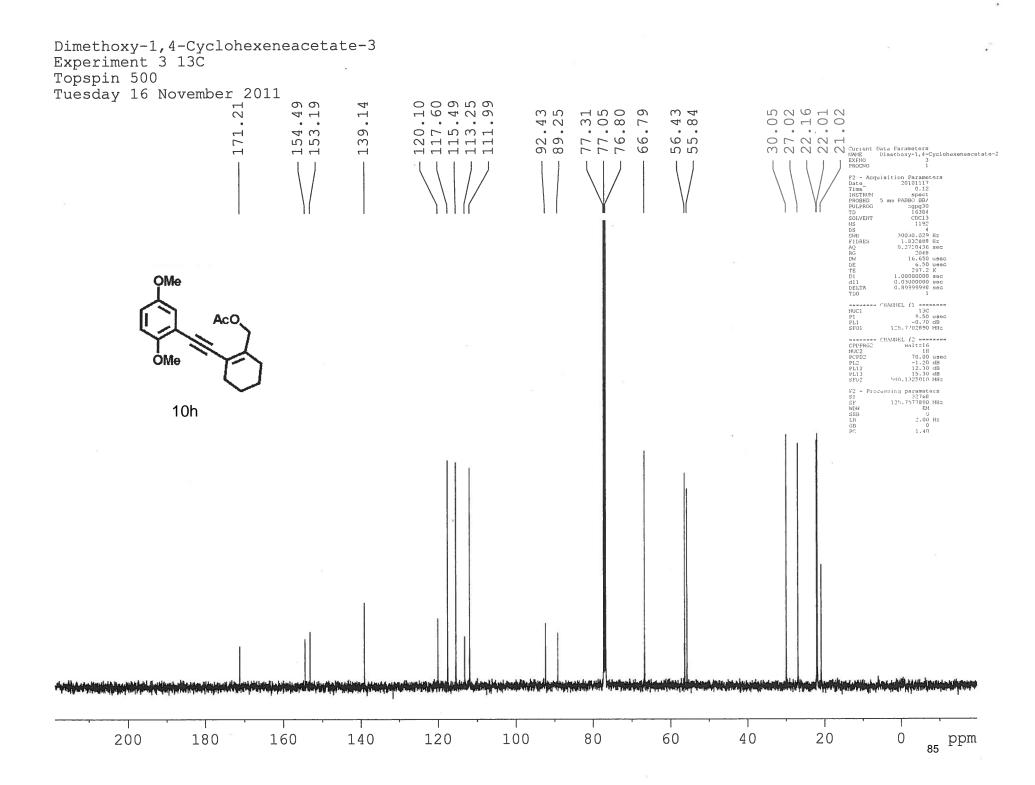


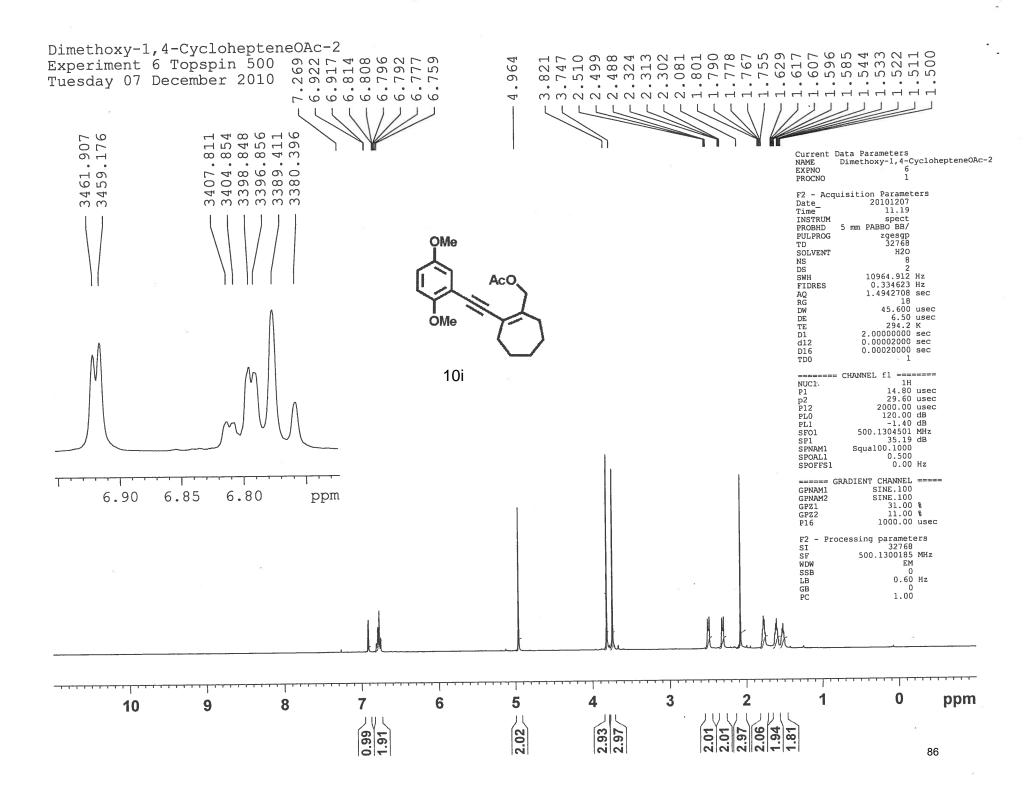
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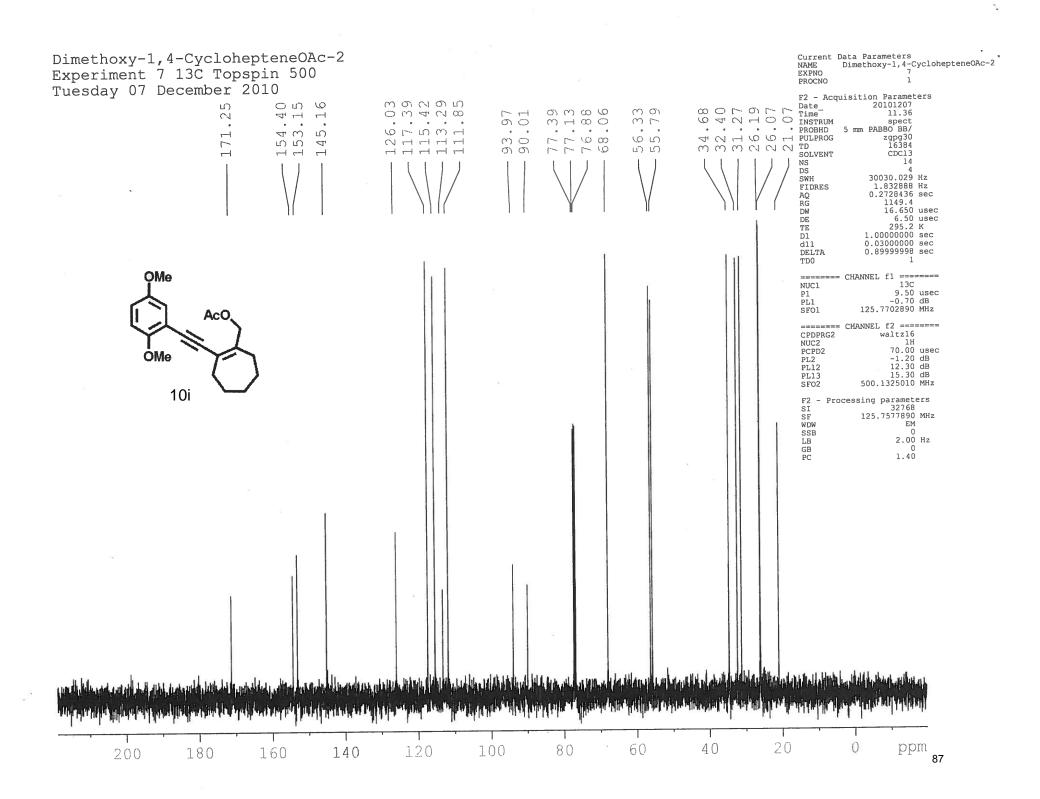


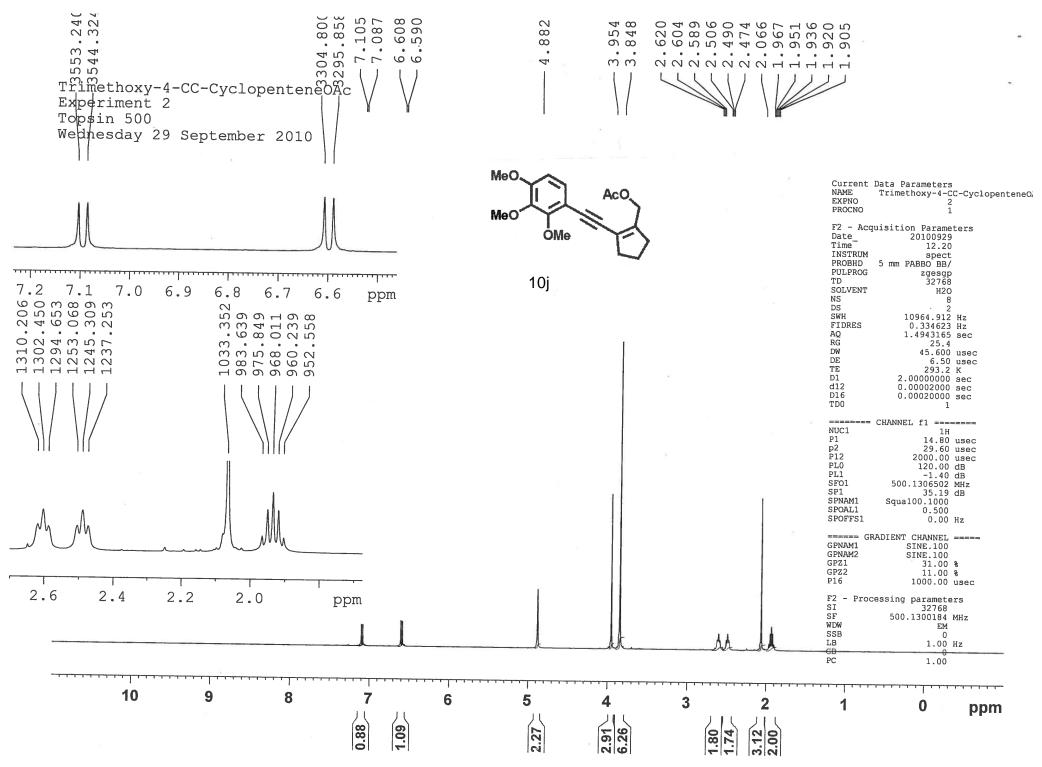


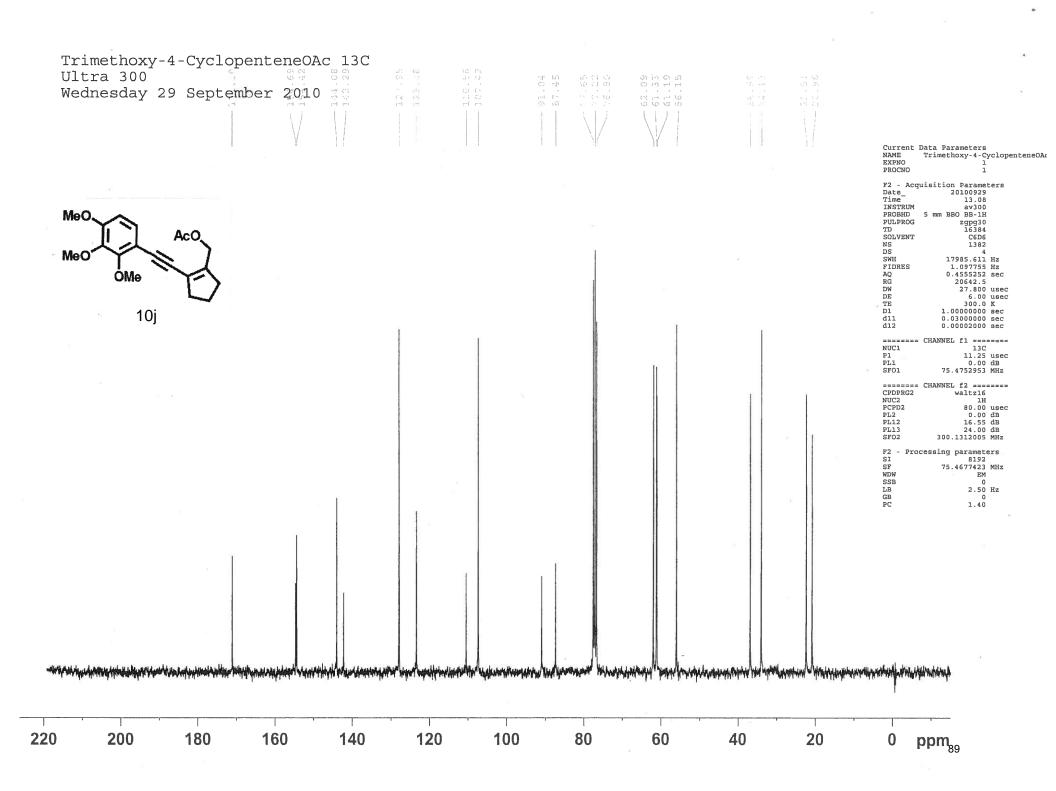




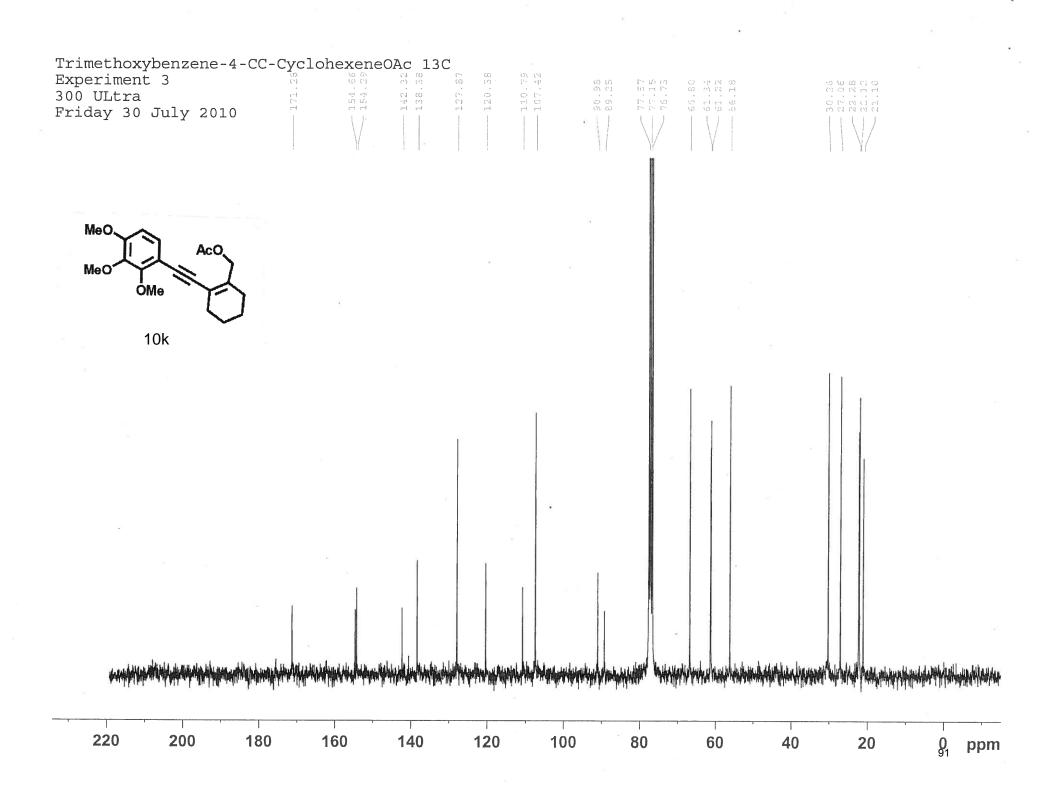


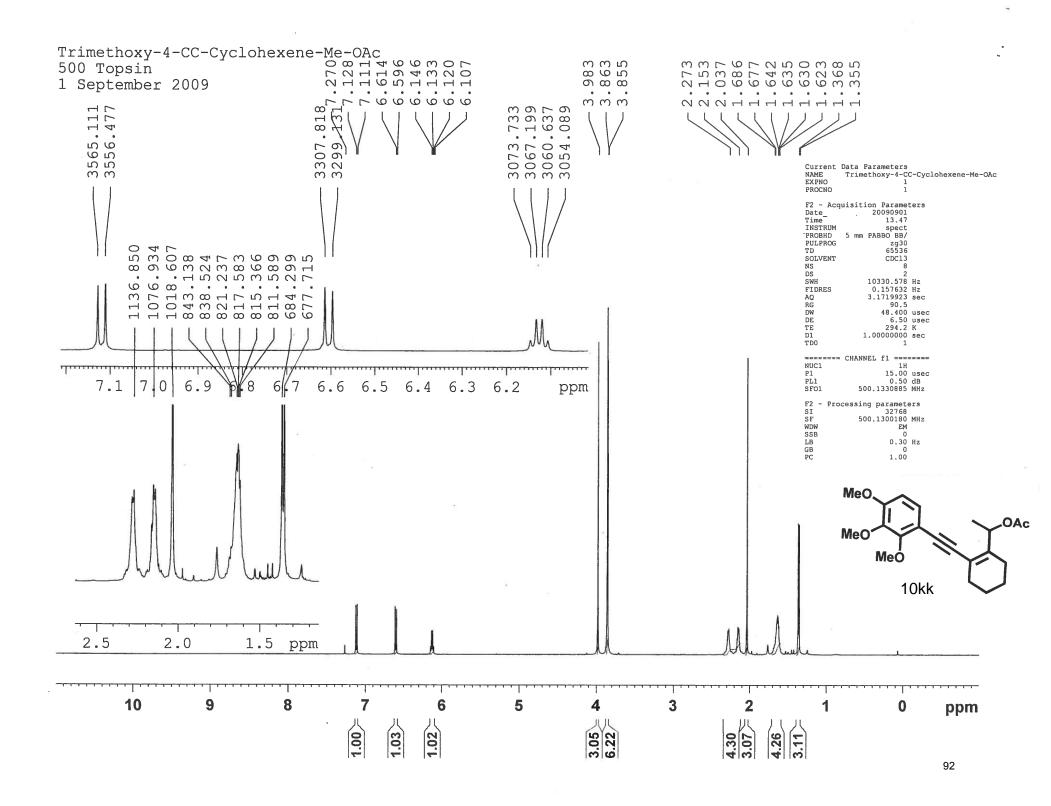


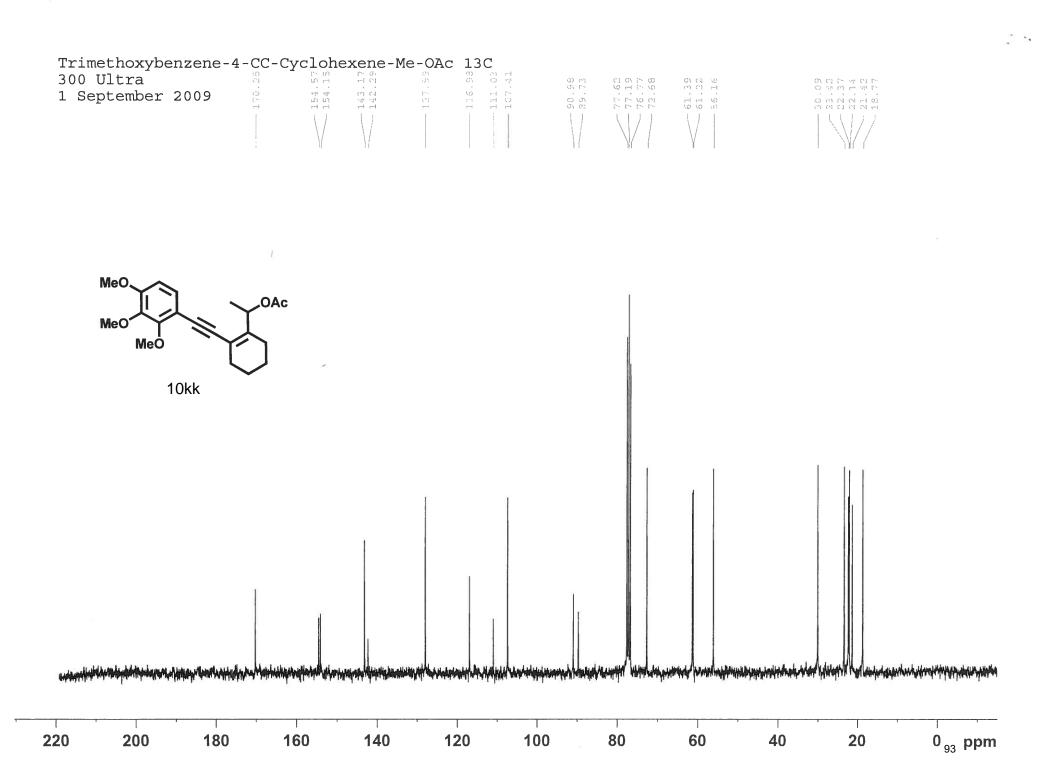


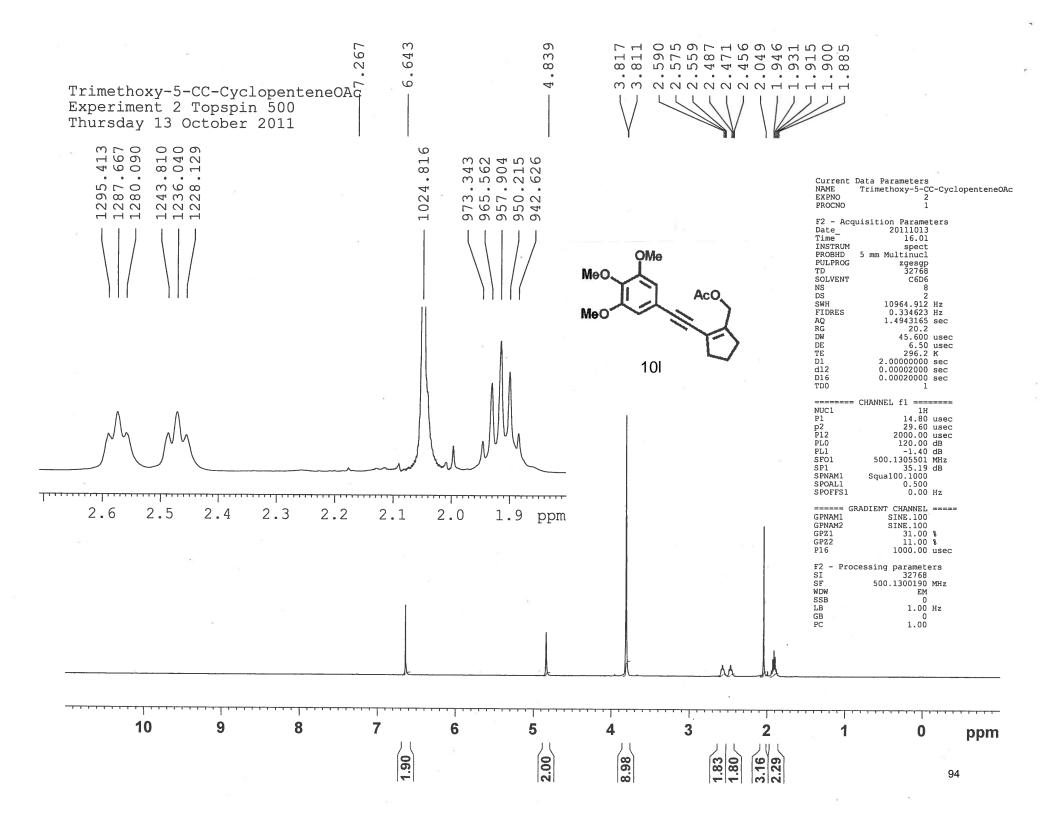




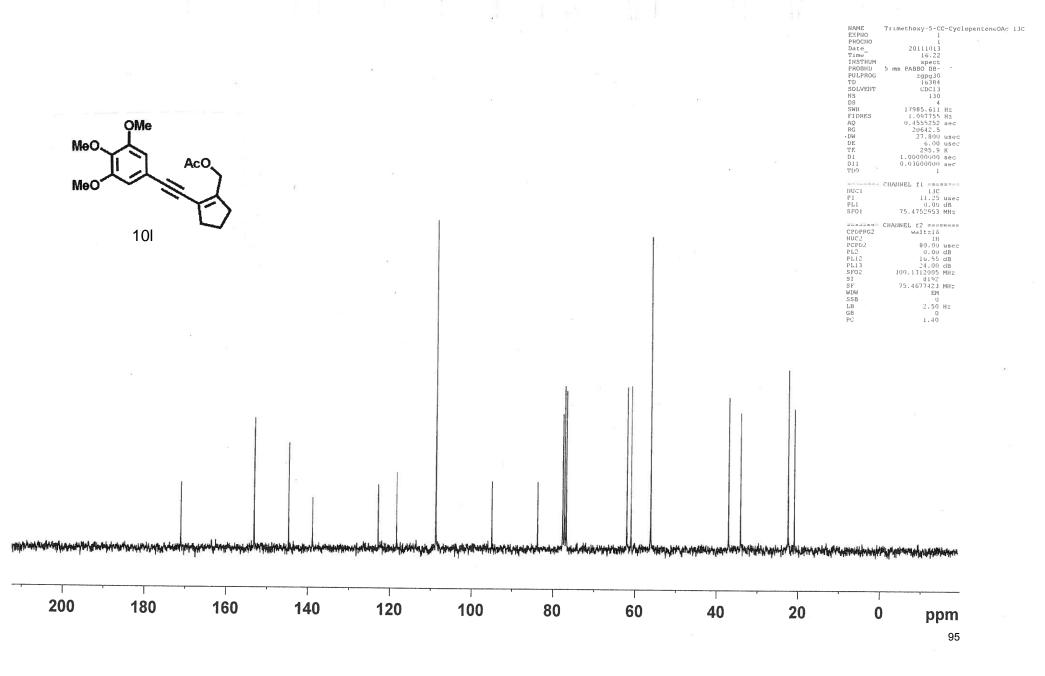


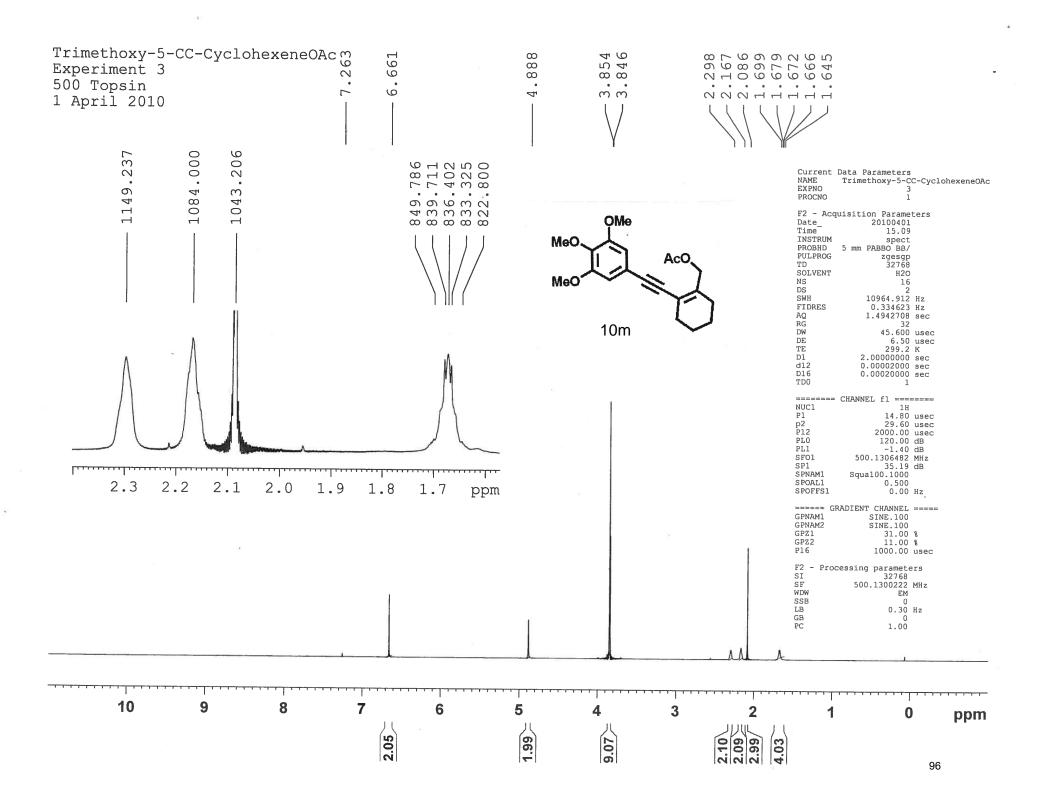


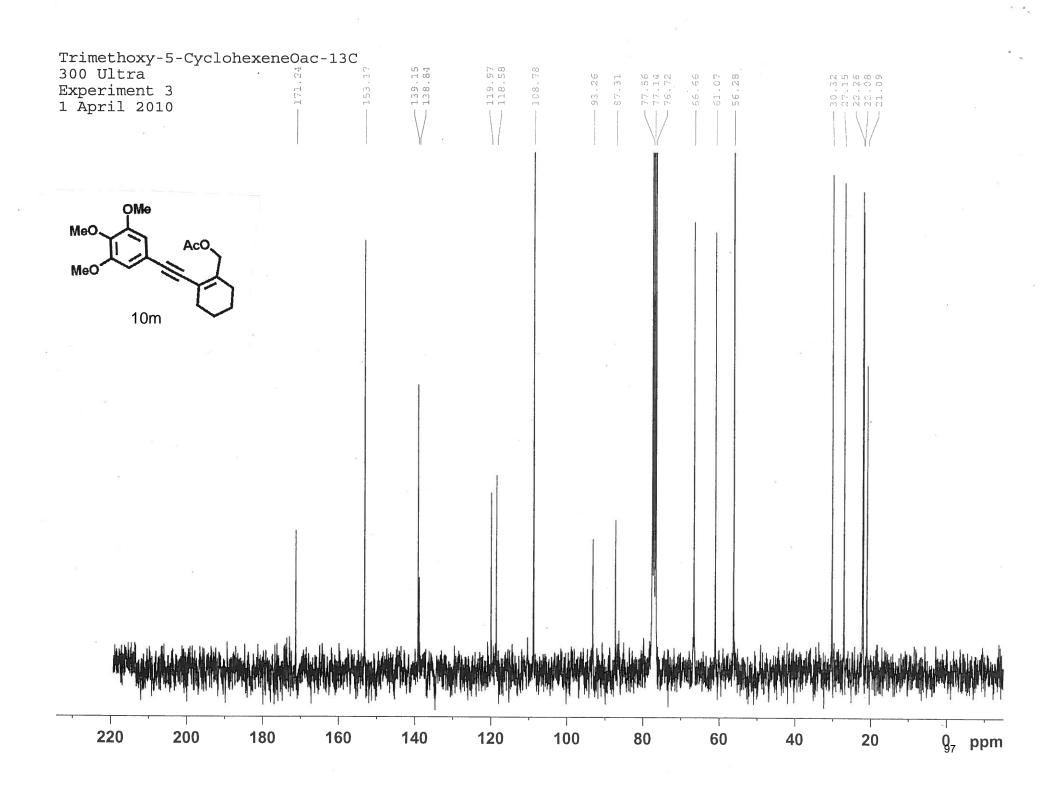


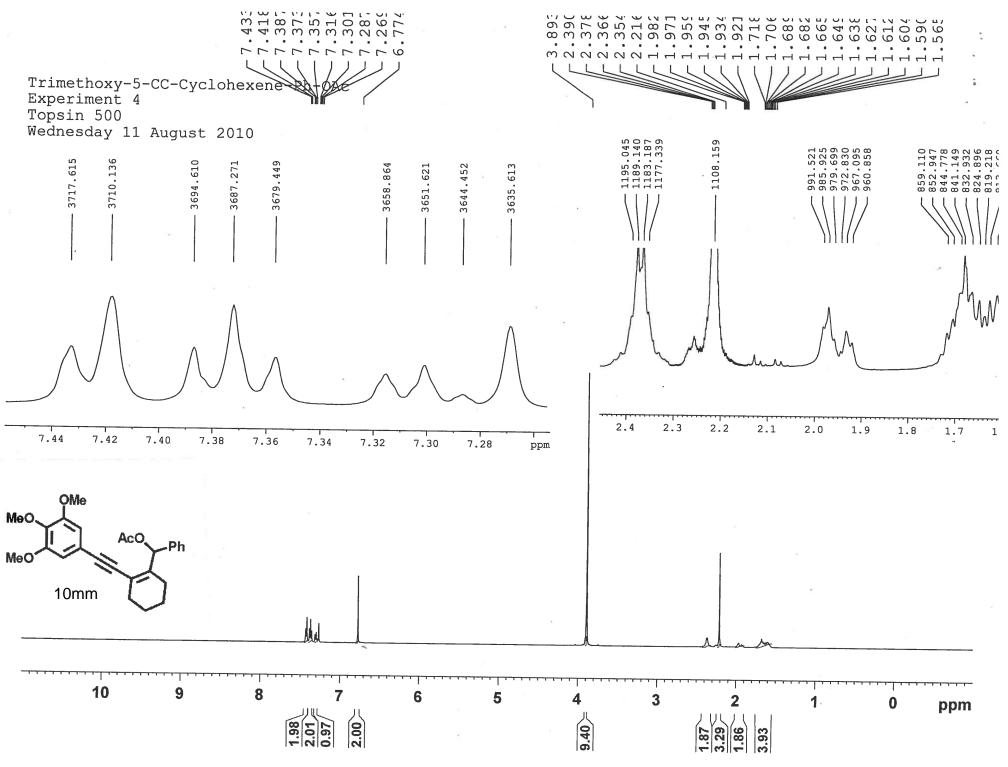


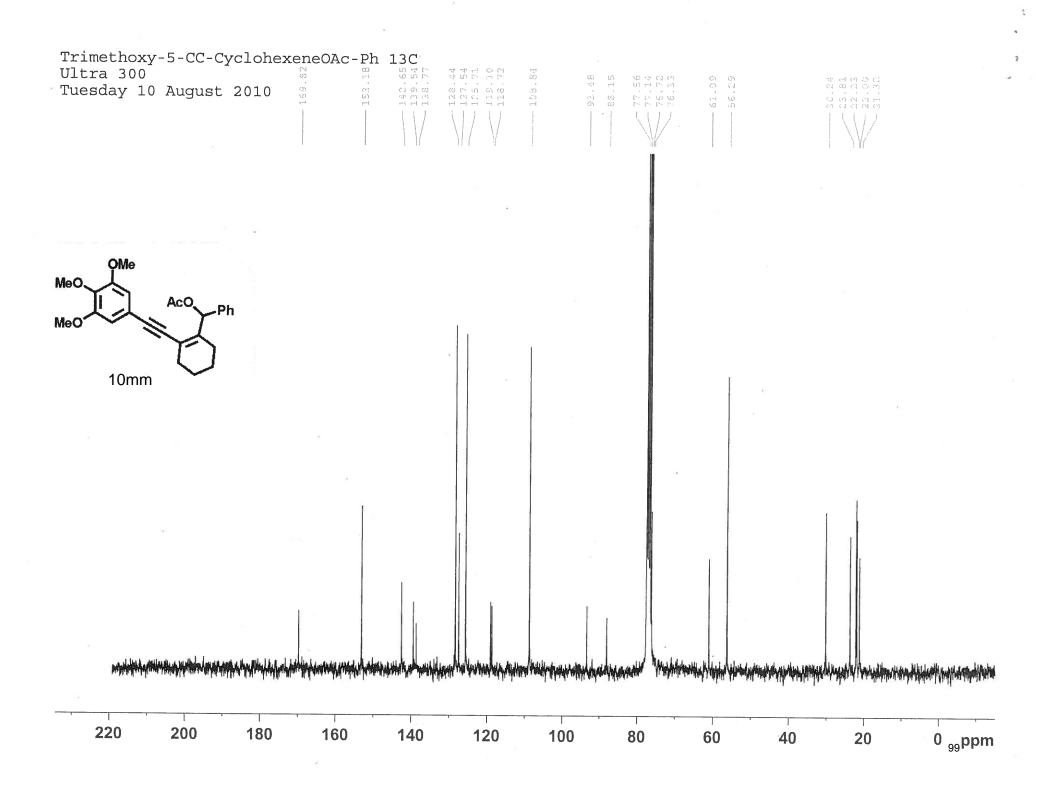
imethoxy-5-CC-CyclopenteneOAc 13C
periment 1 Topspin Ultra 300
ursday 13 October 2011

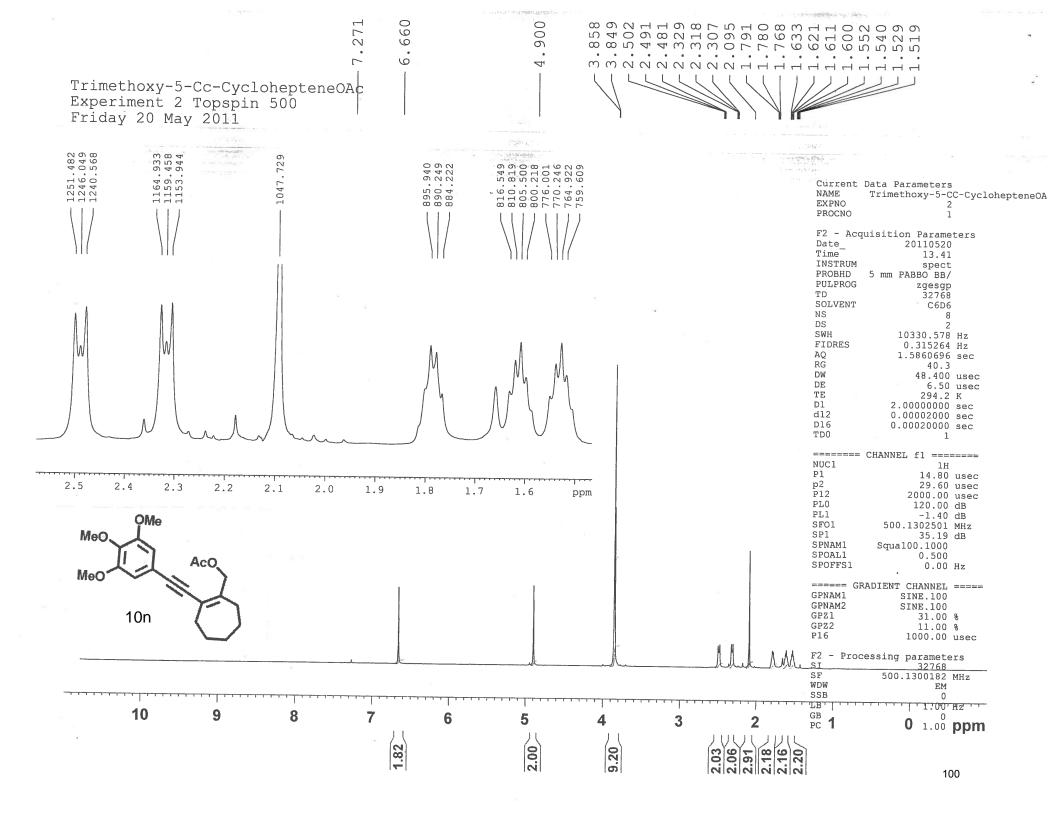




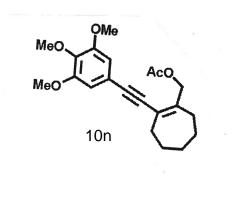


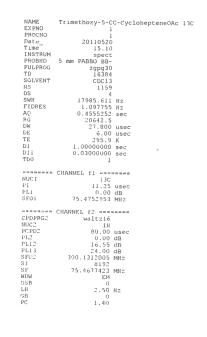


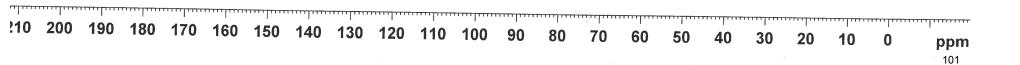


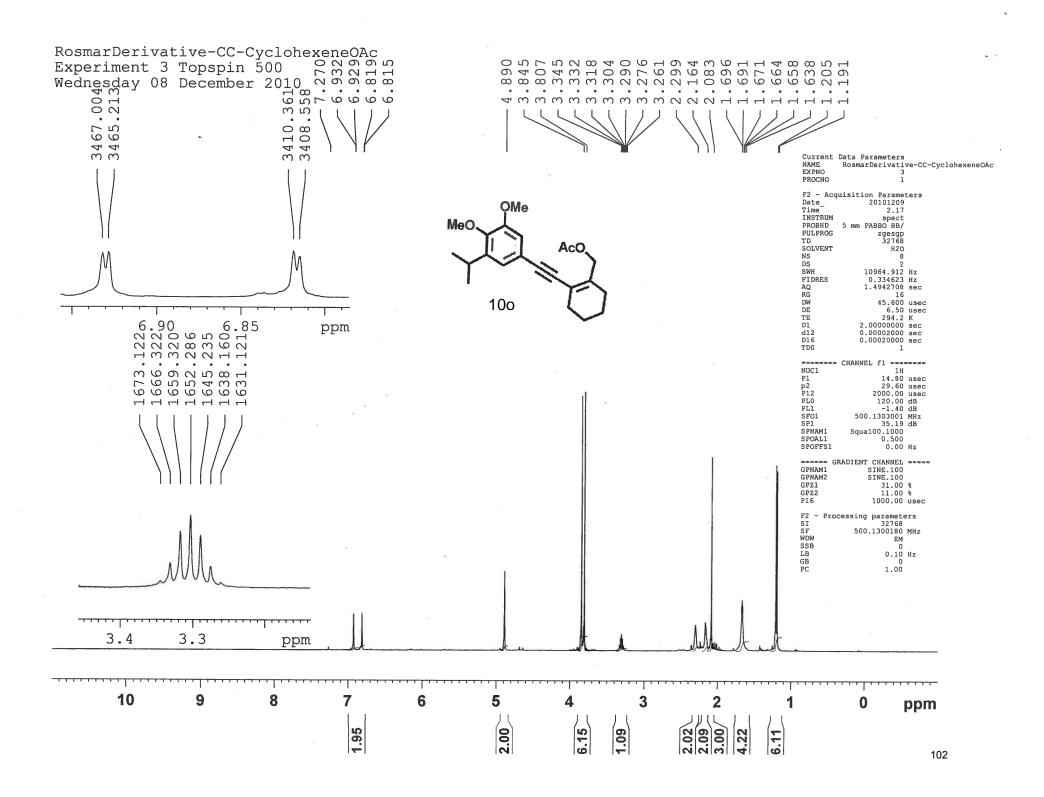


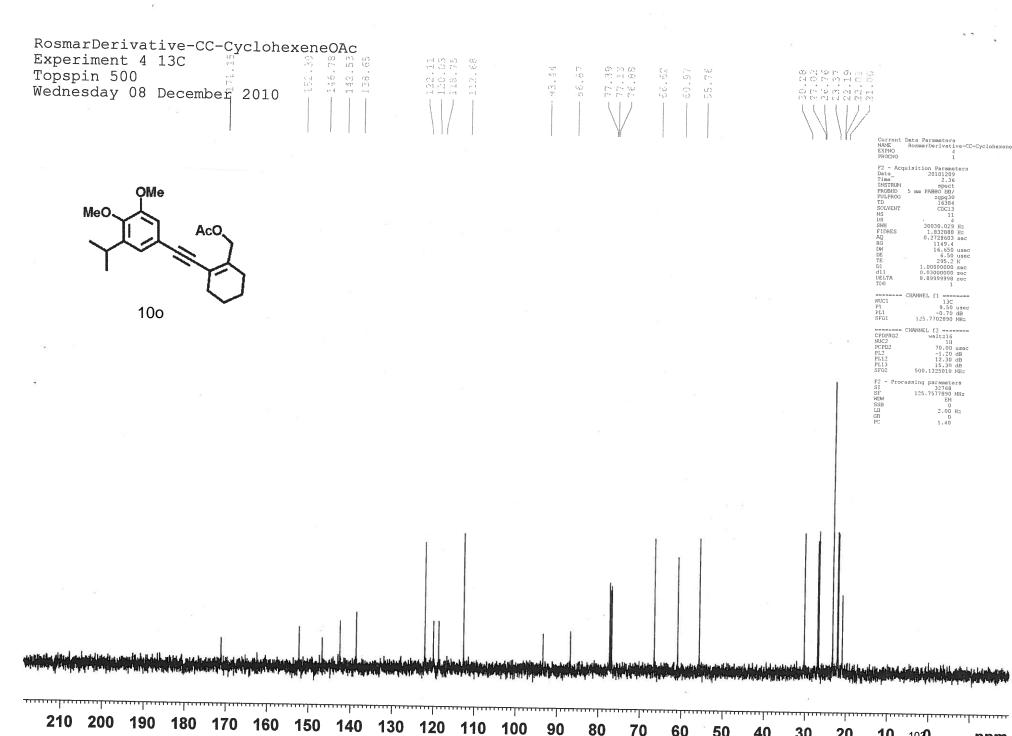
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:iday 20 May 2011





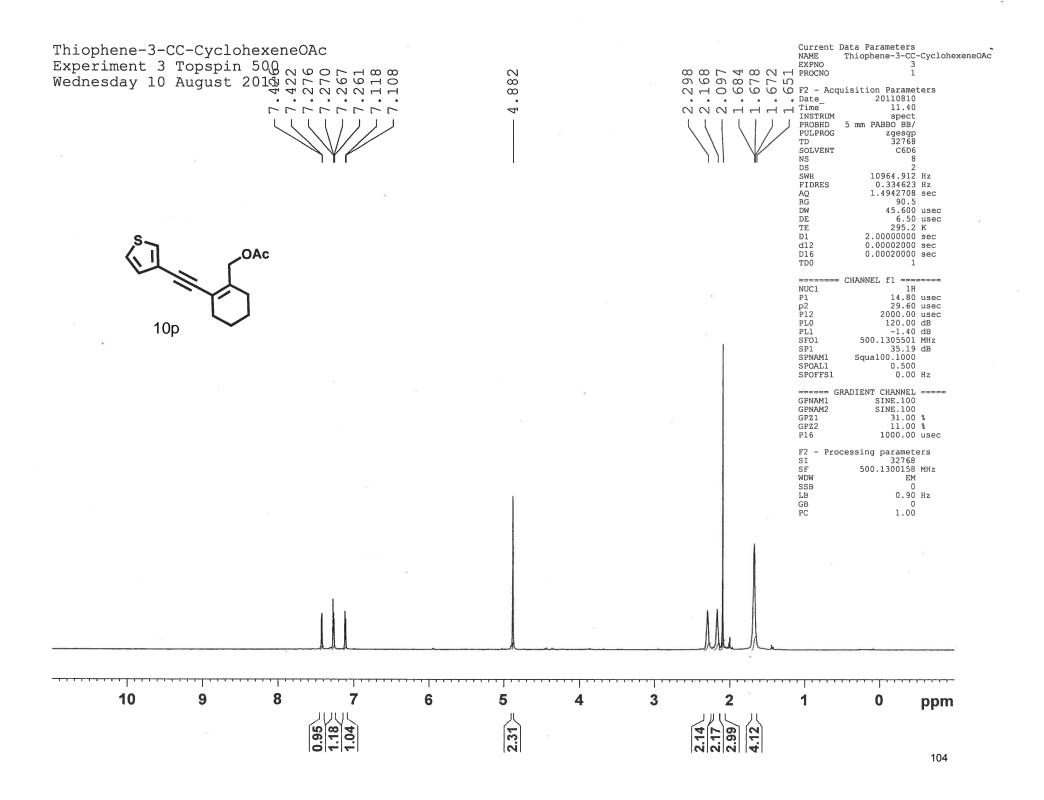


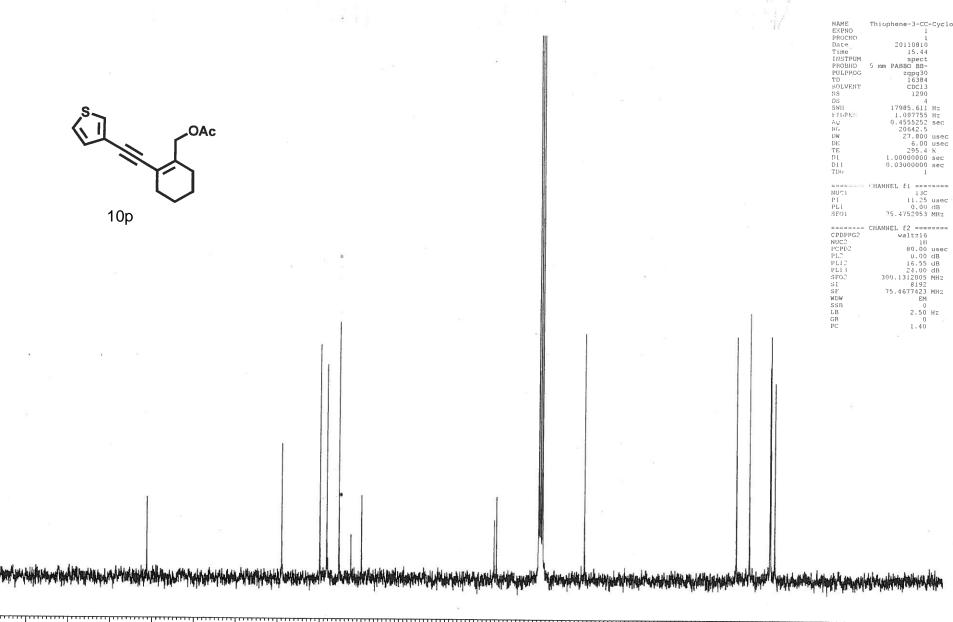




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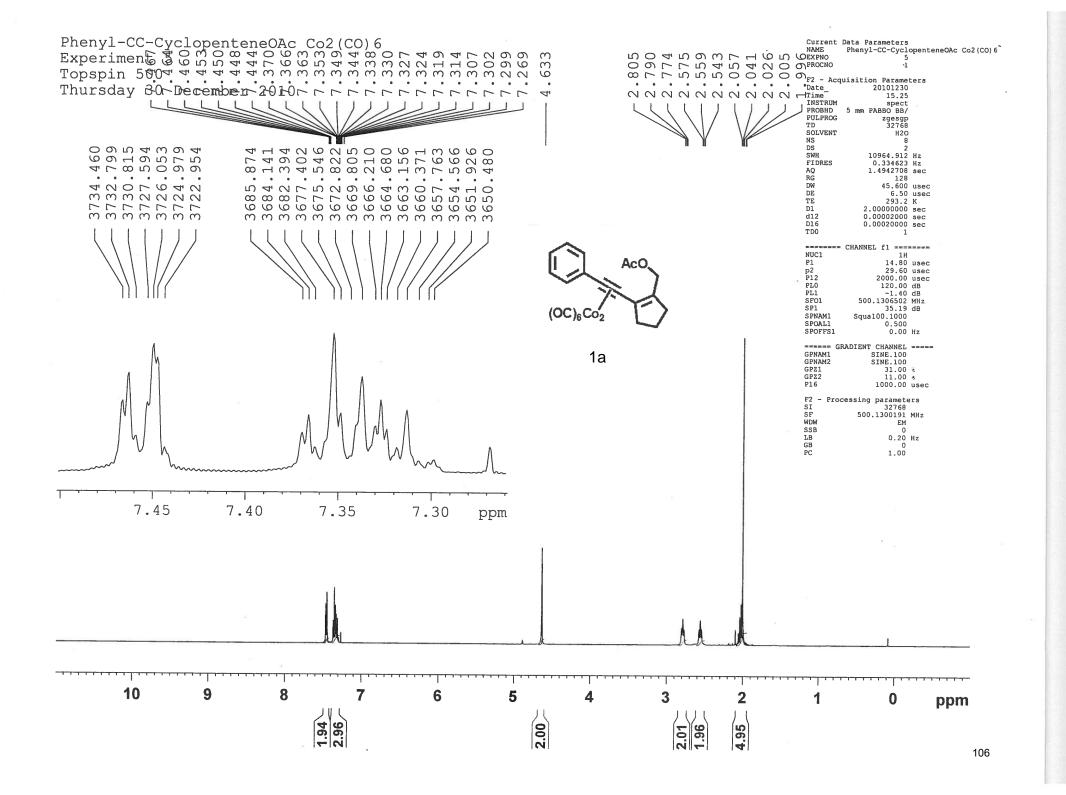
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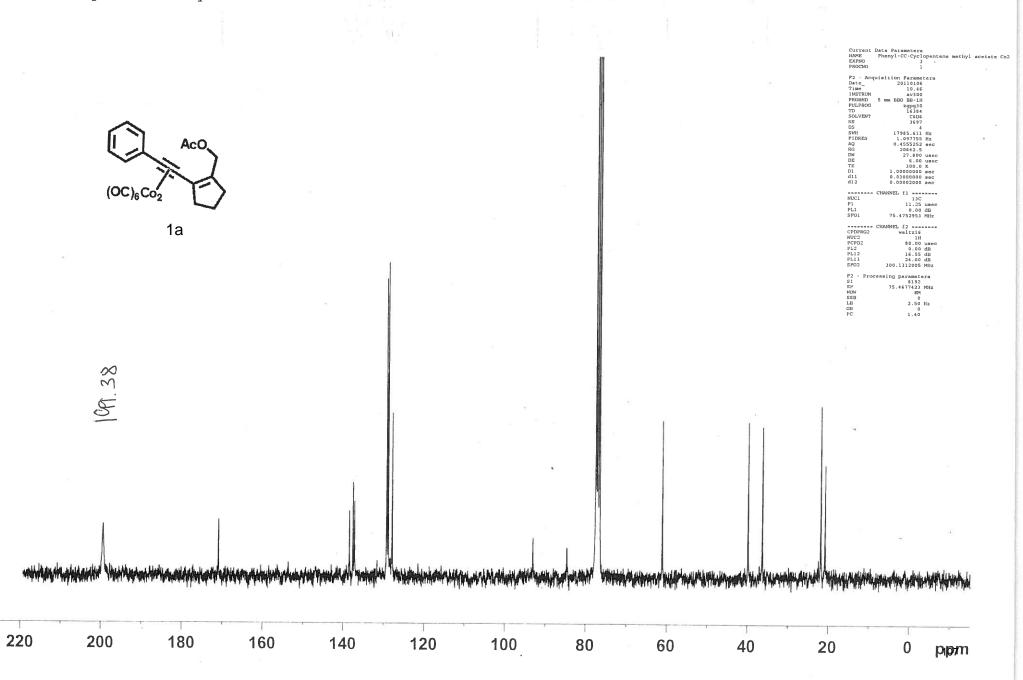


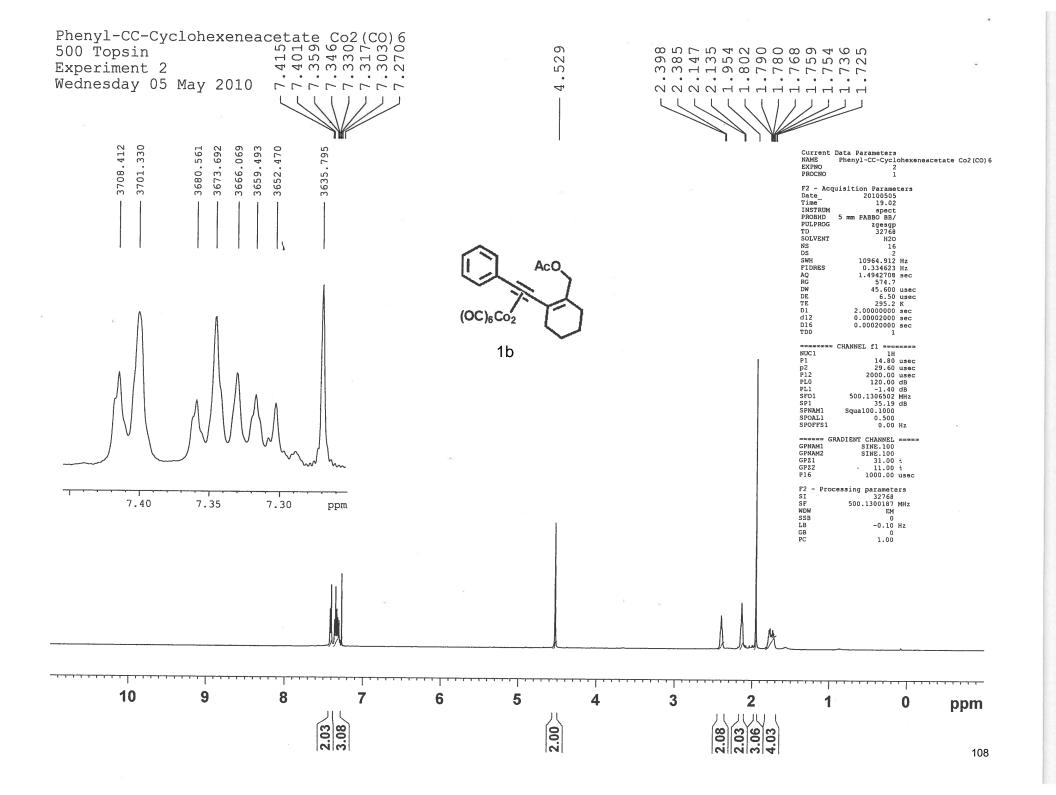
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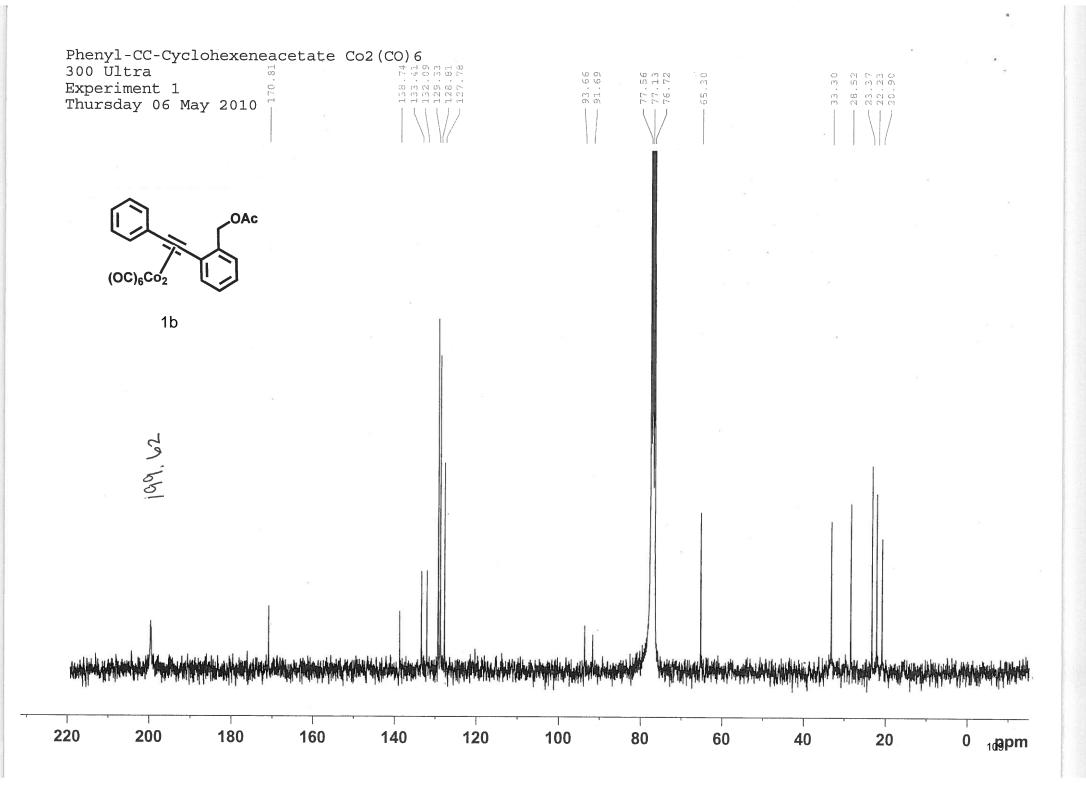
180 170 160 150 140 130 1**p**pm

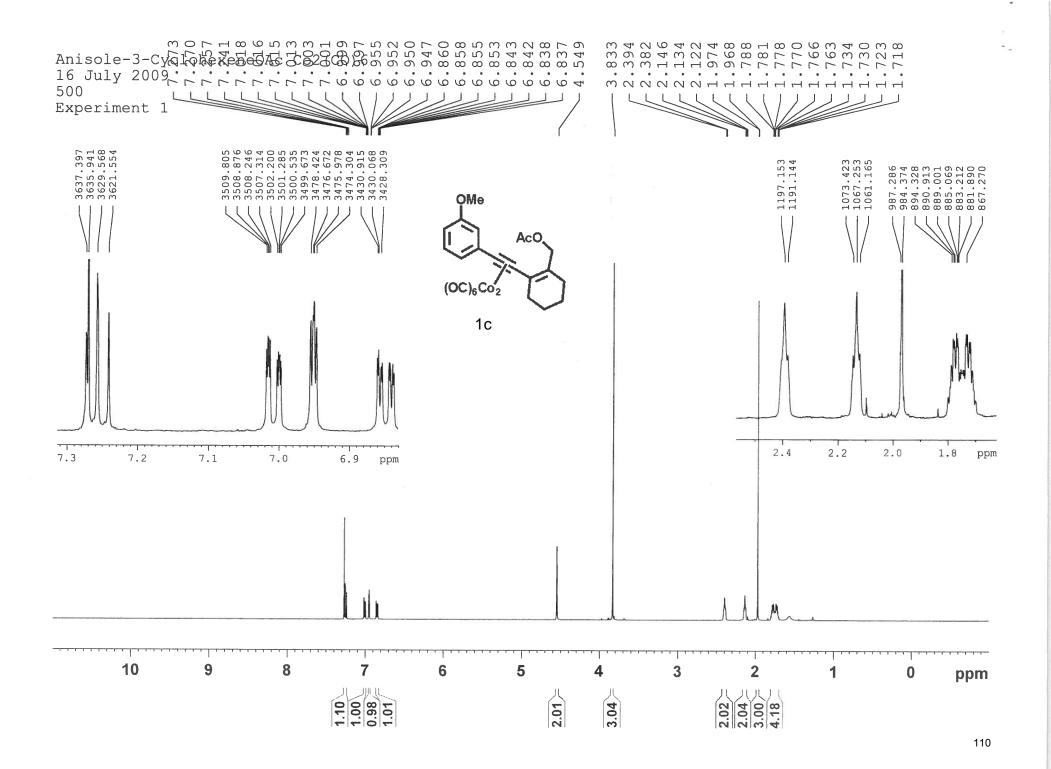


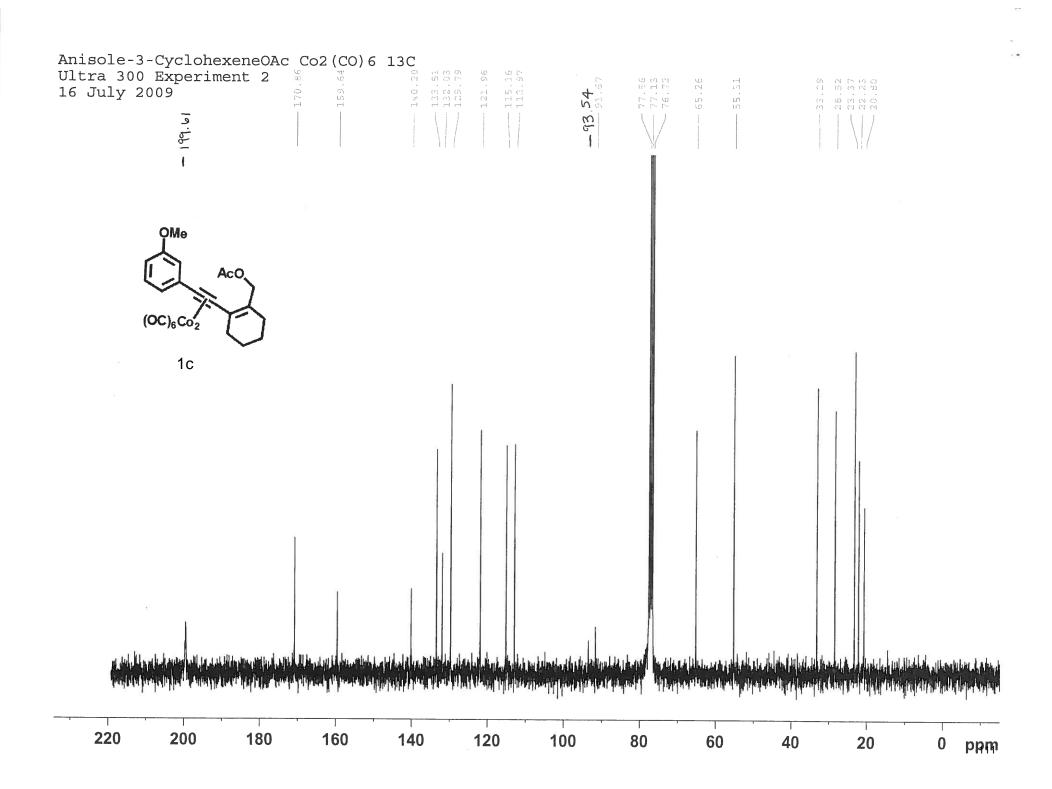
Phenyl-CC-Cyclopentene methyl acetate Co2(CO)6 13C Experiment 3 Ultra 300 Thursday 06 January 2011

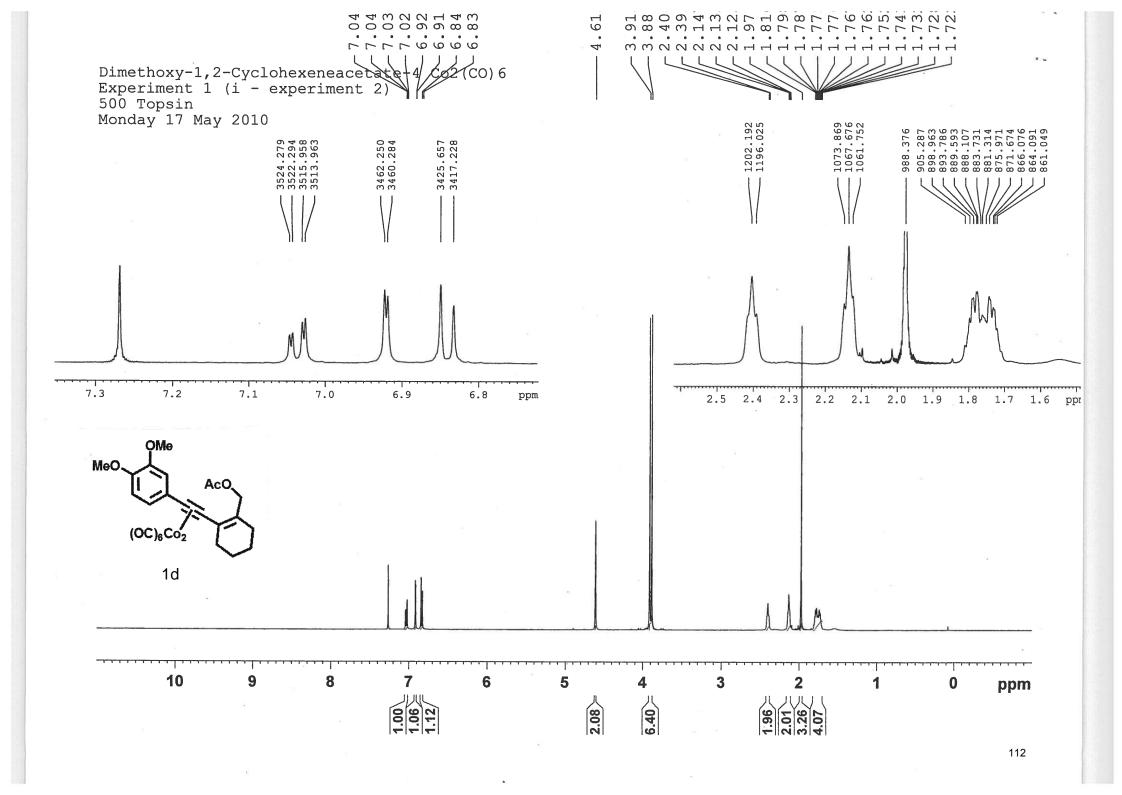


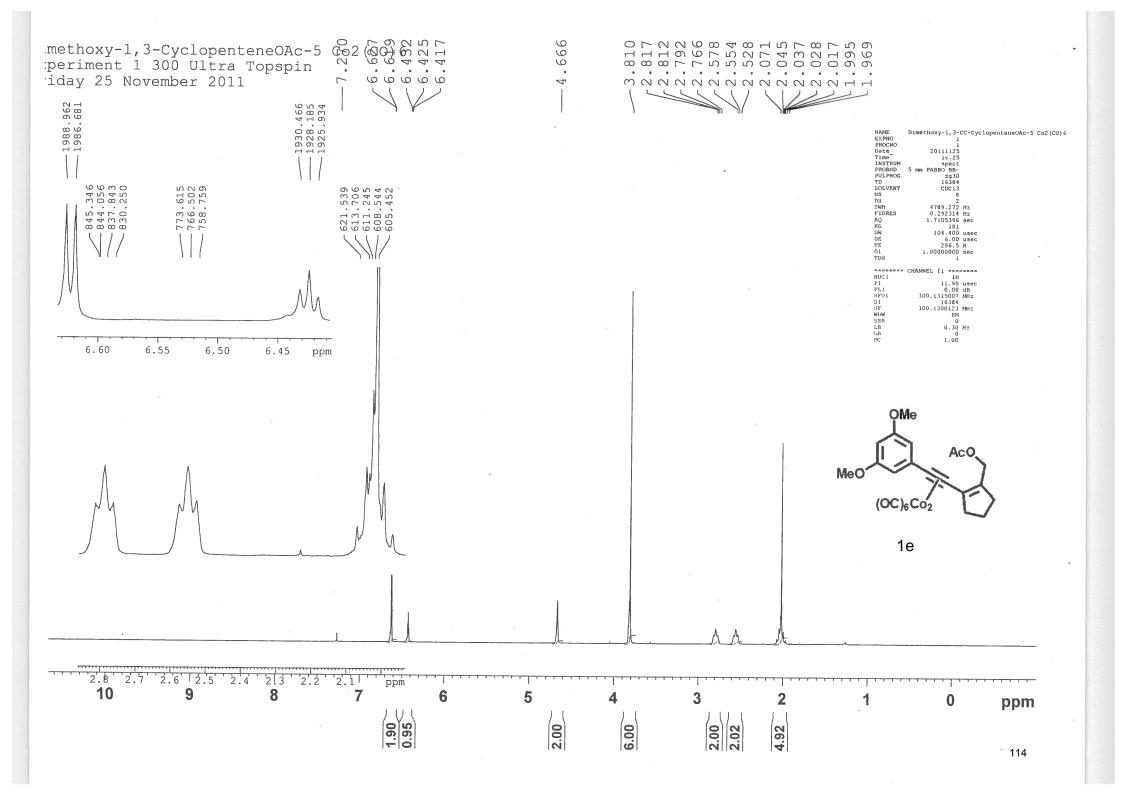


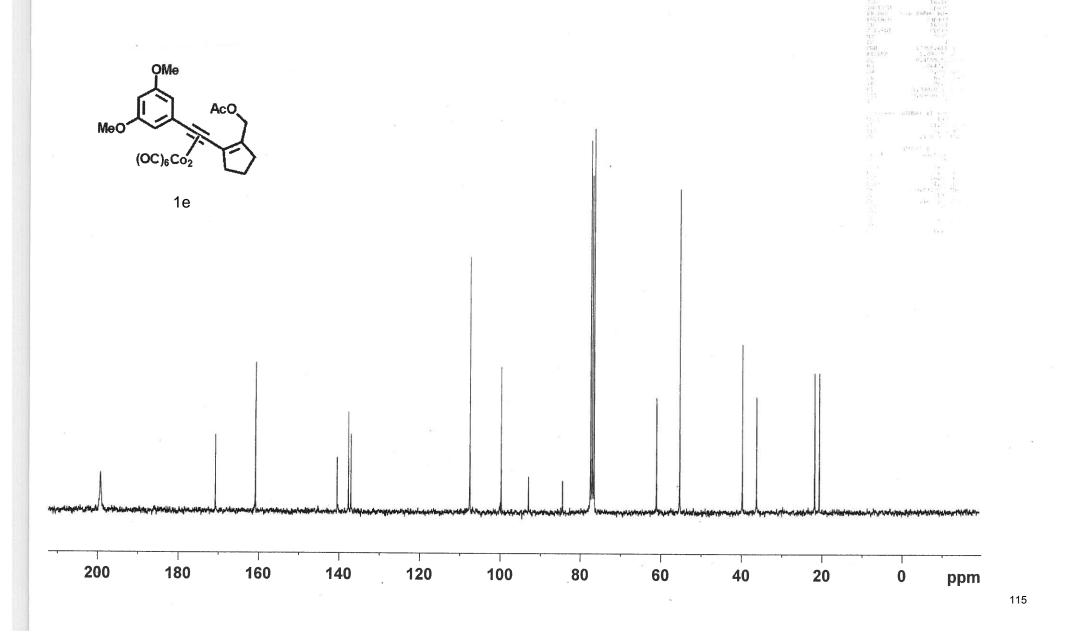


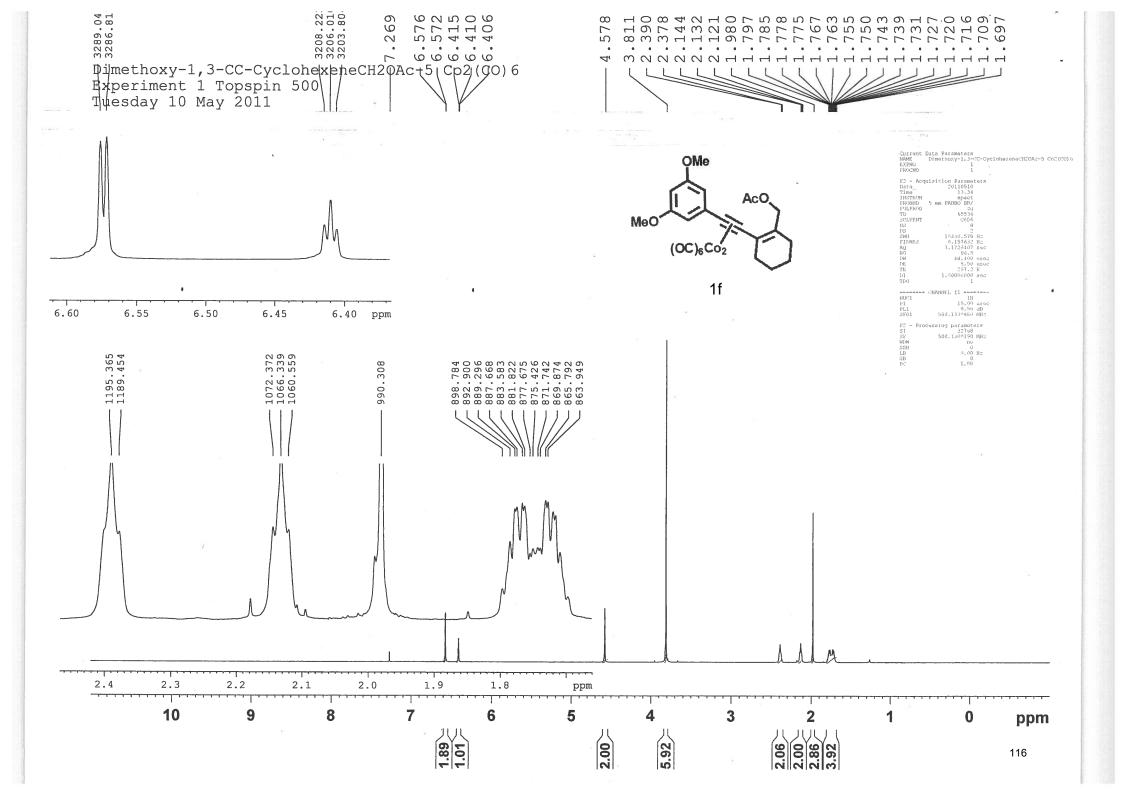


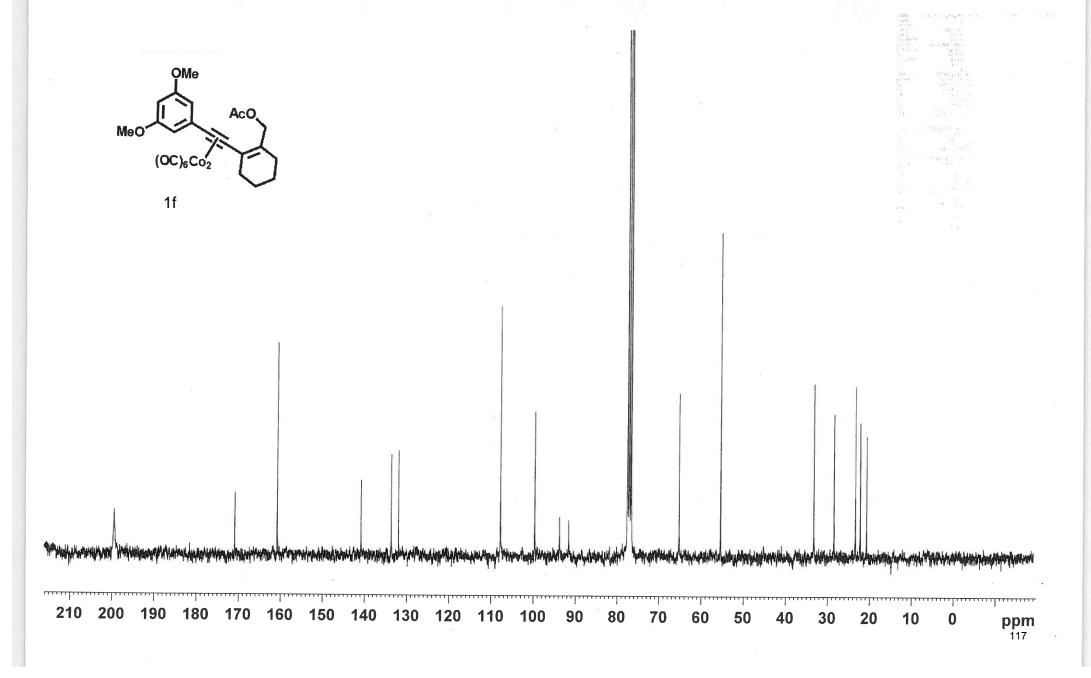


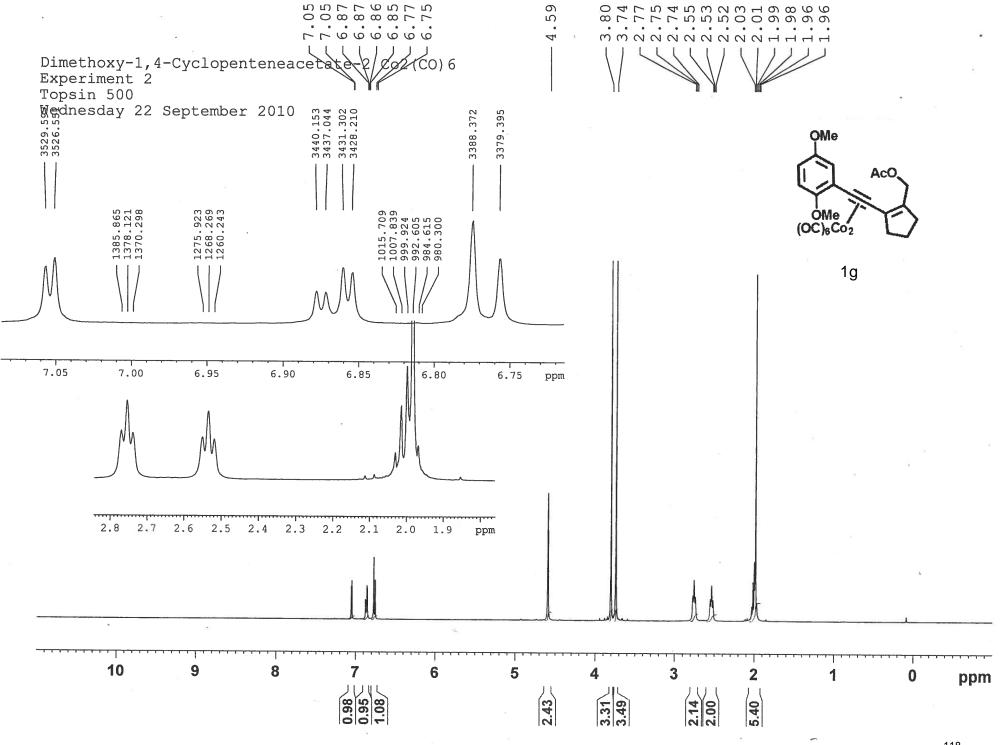


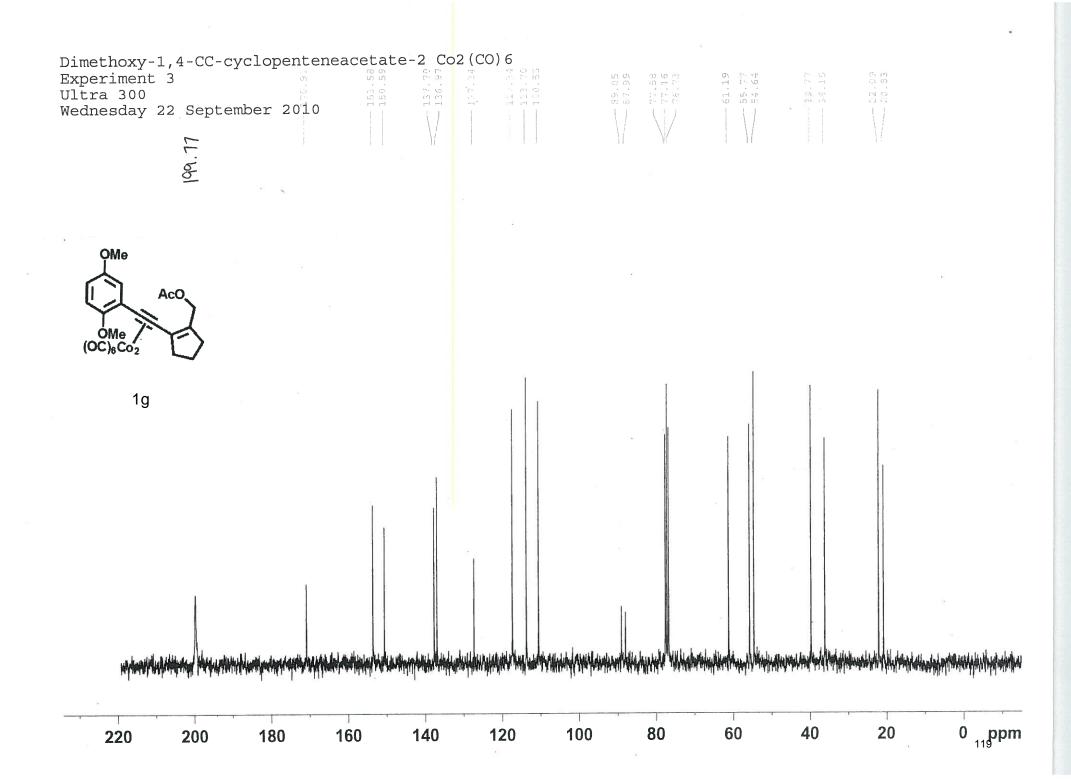


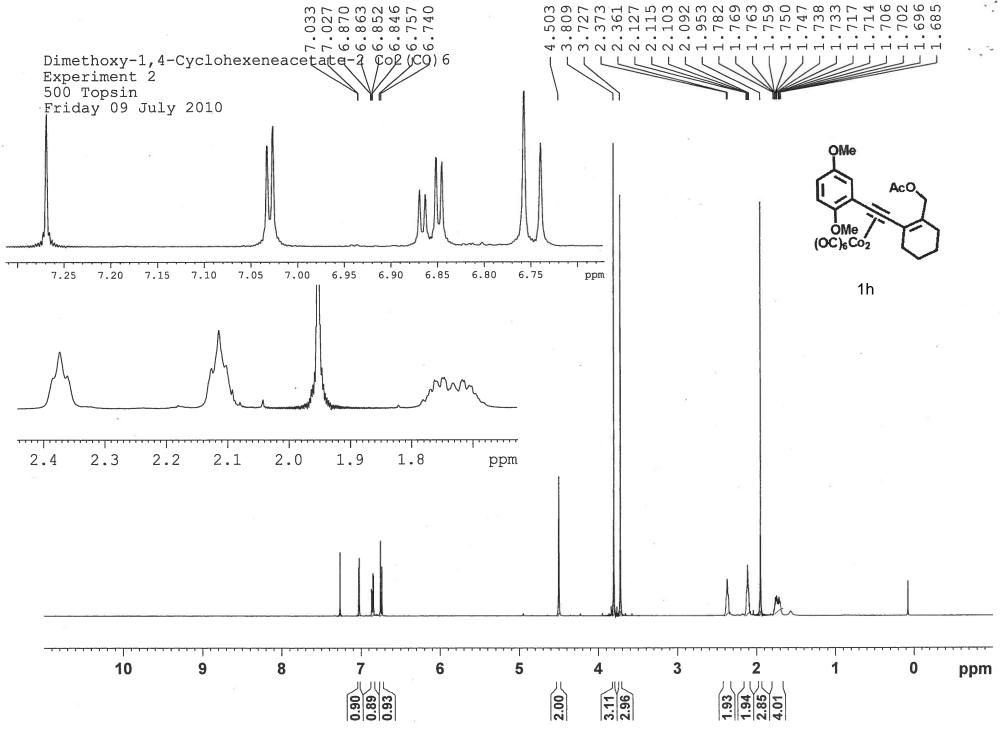


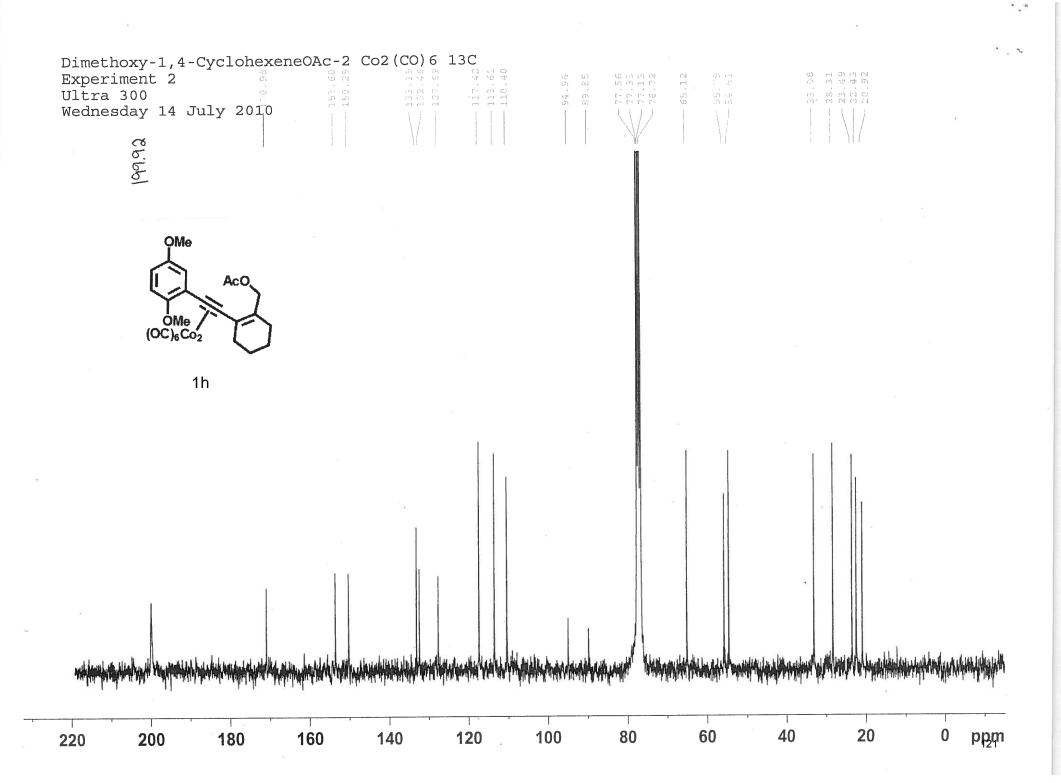


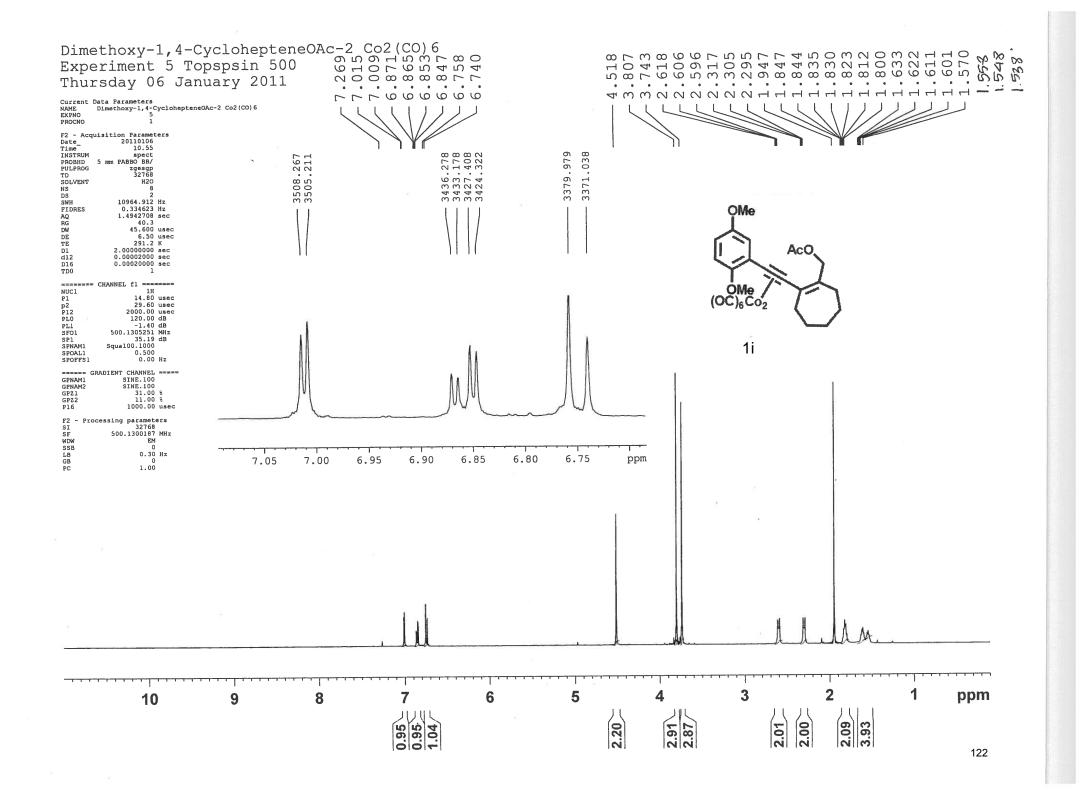


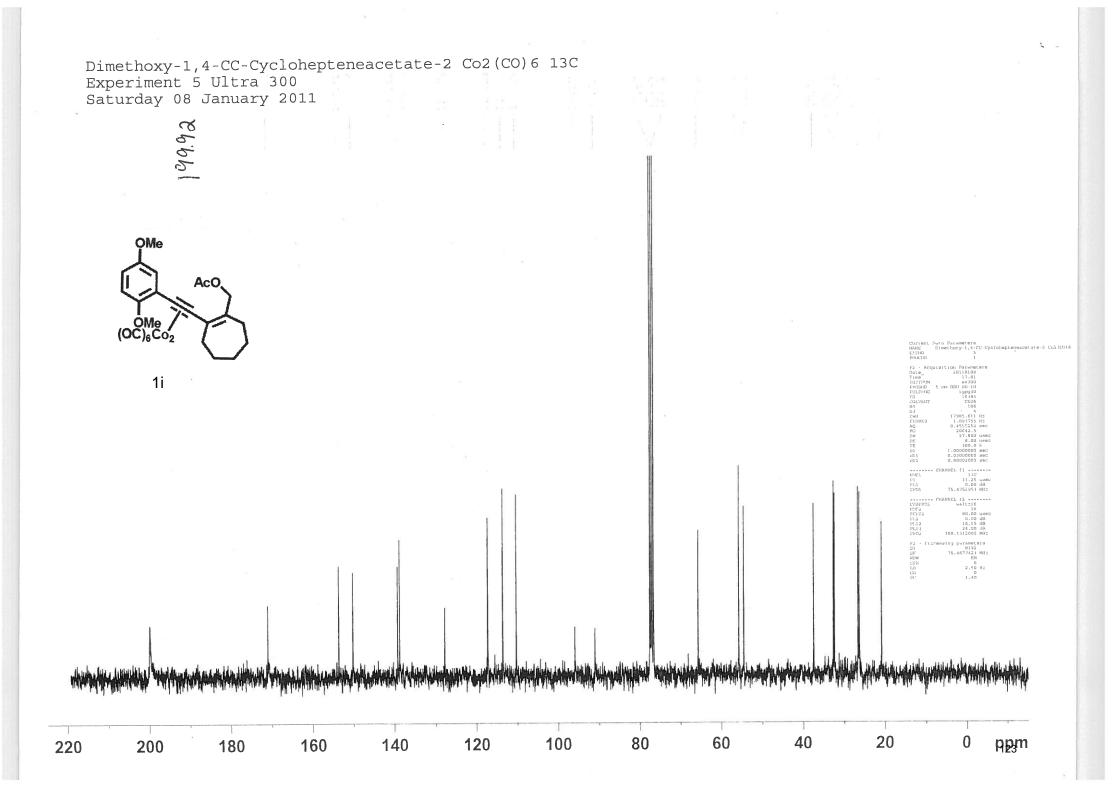


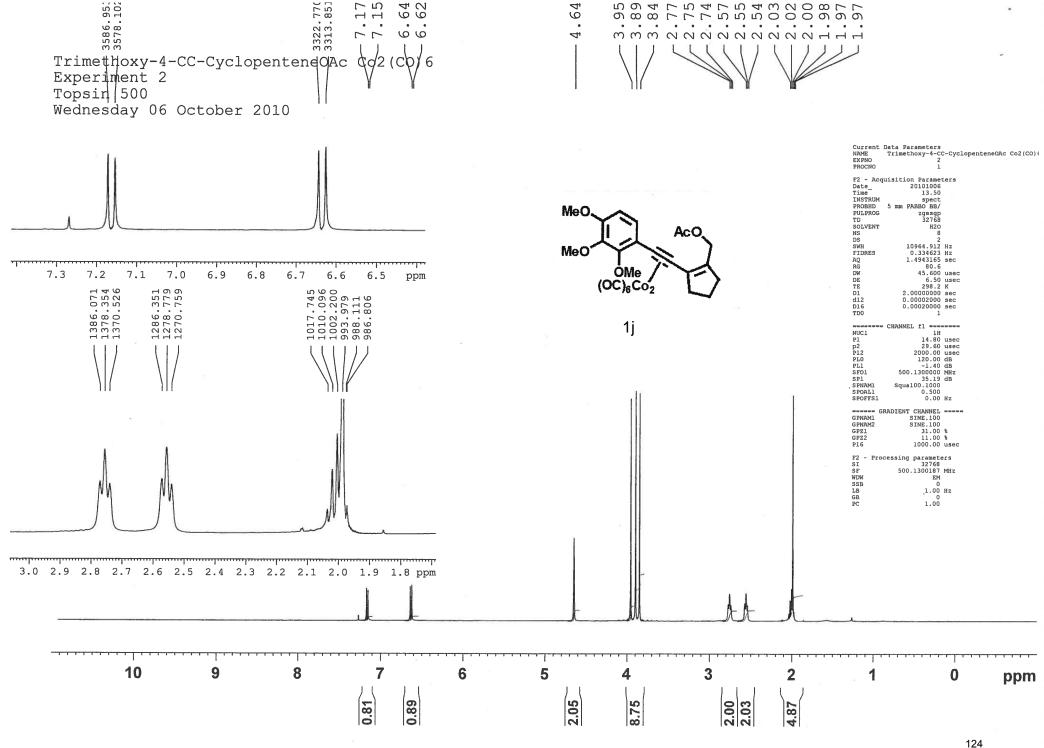


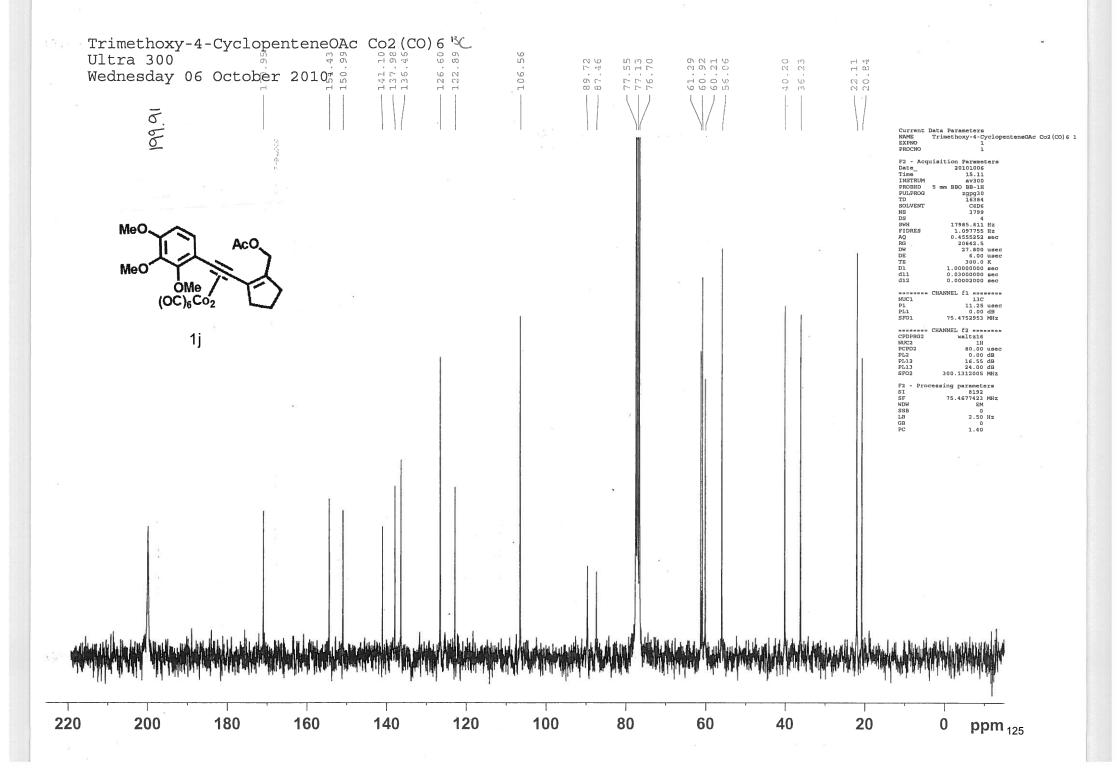


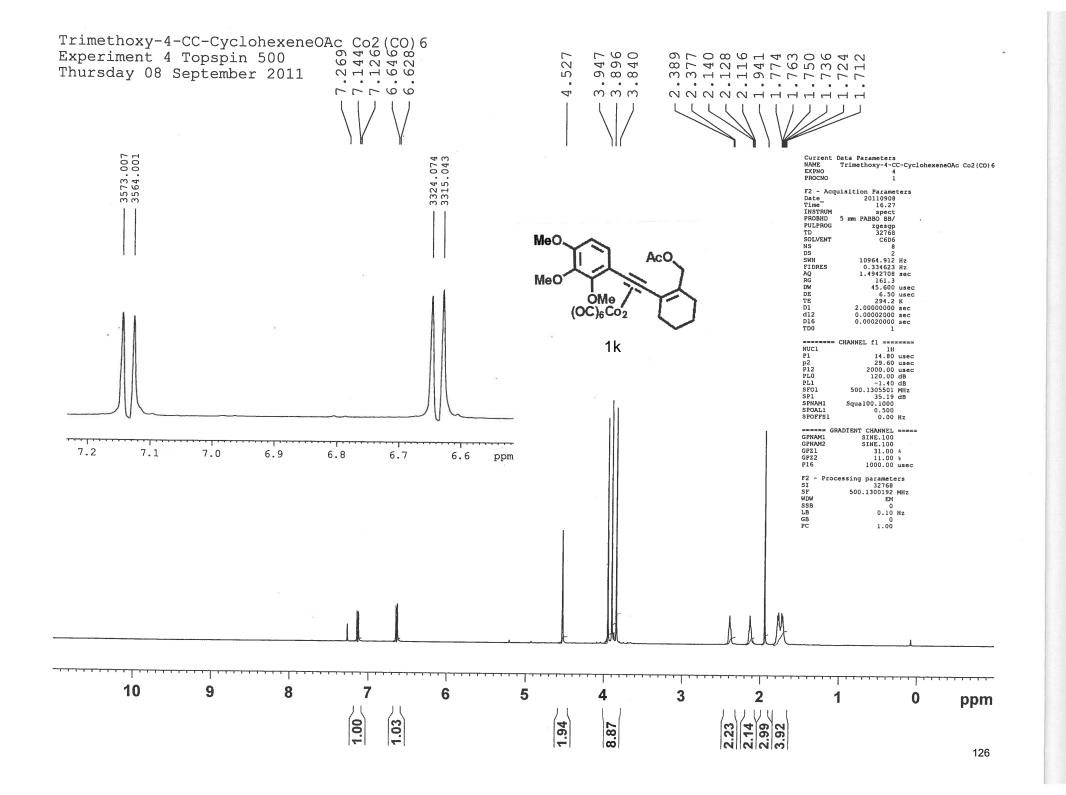


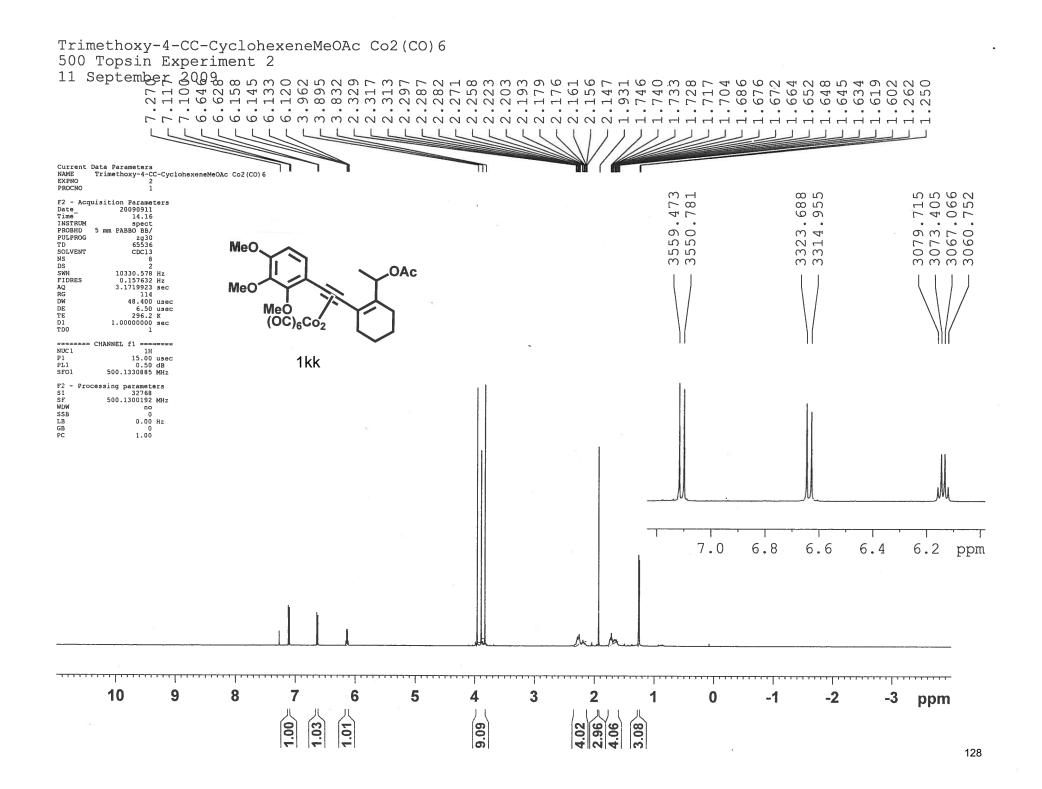


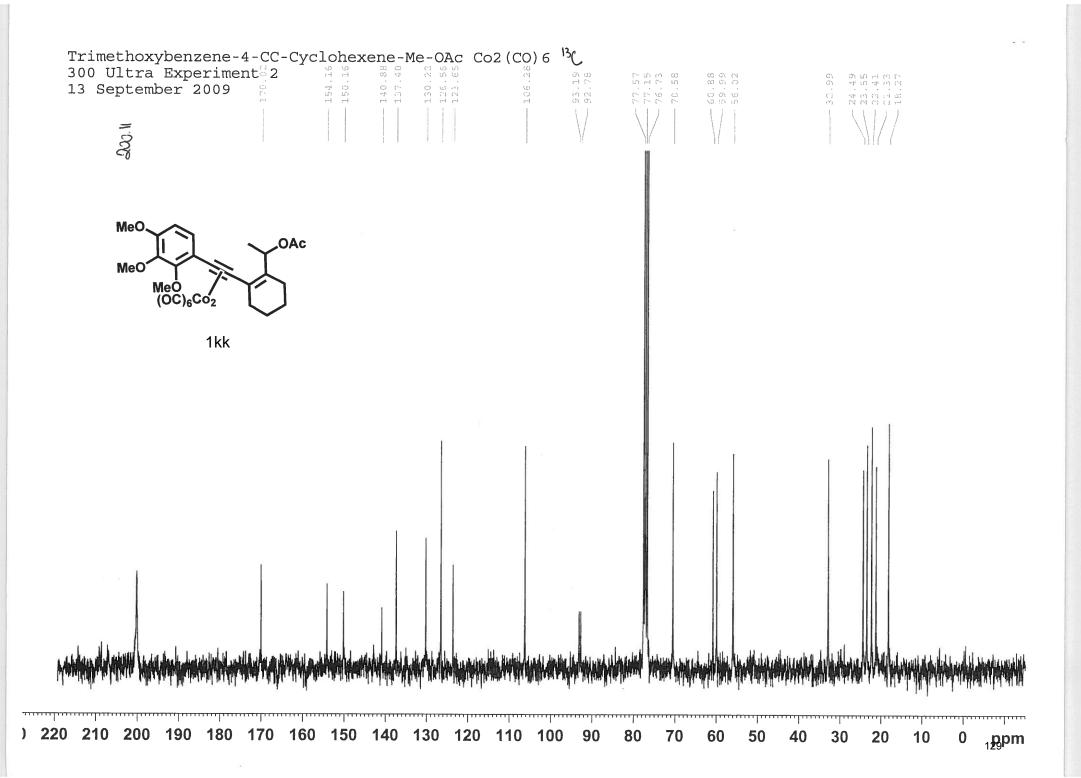


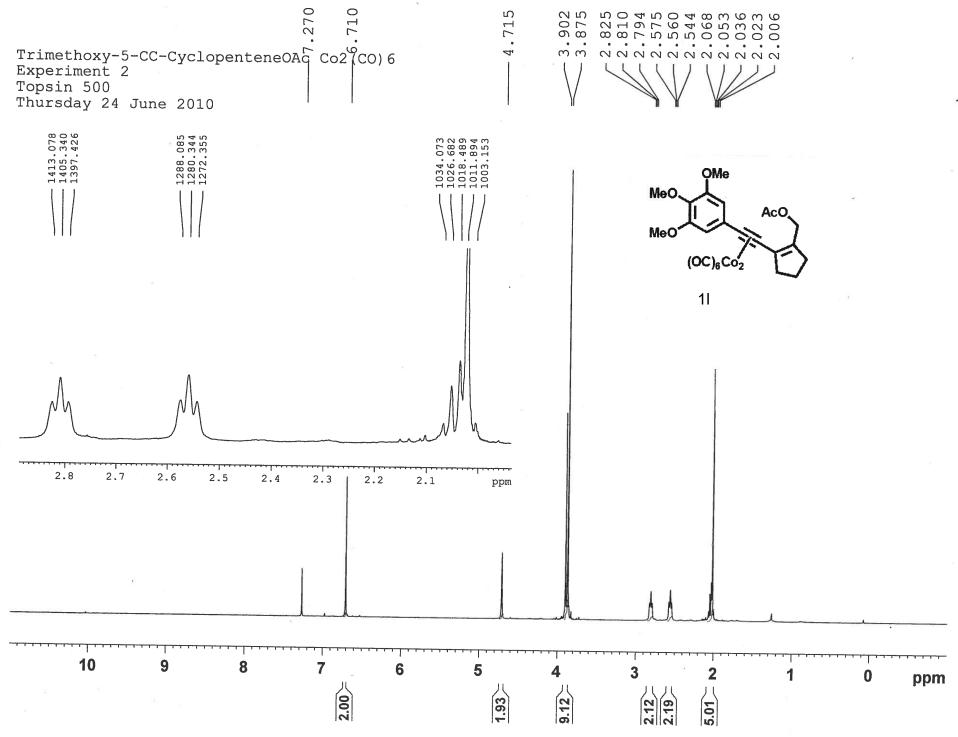


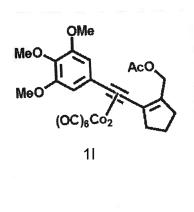




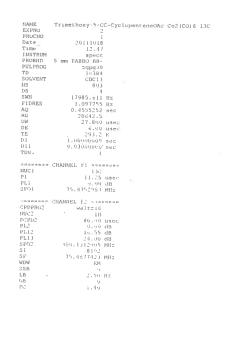






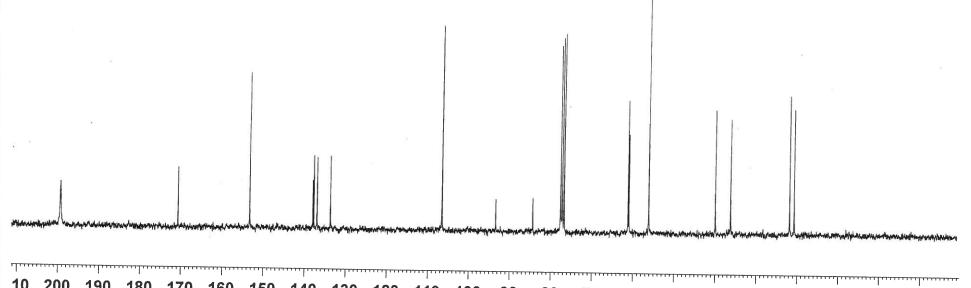


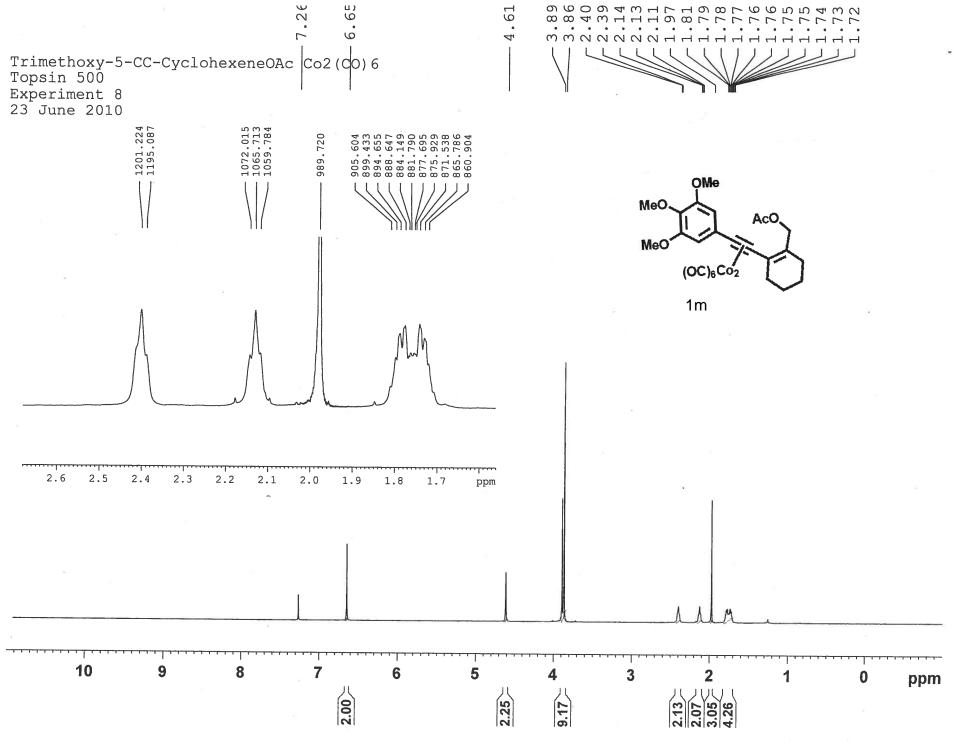
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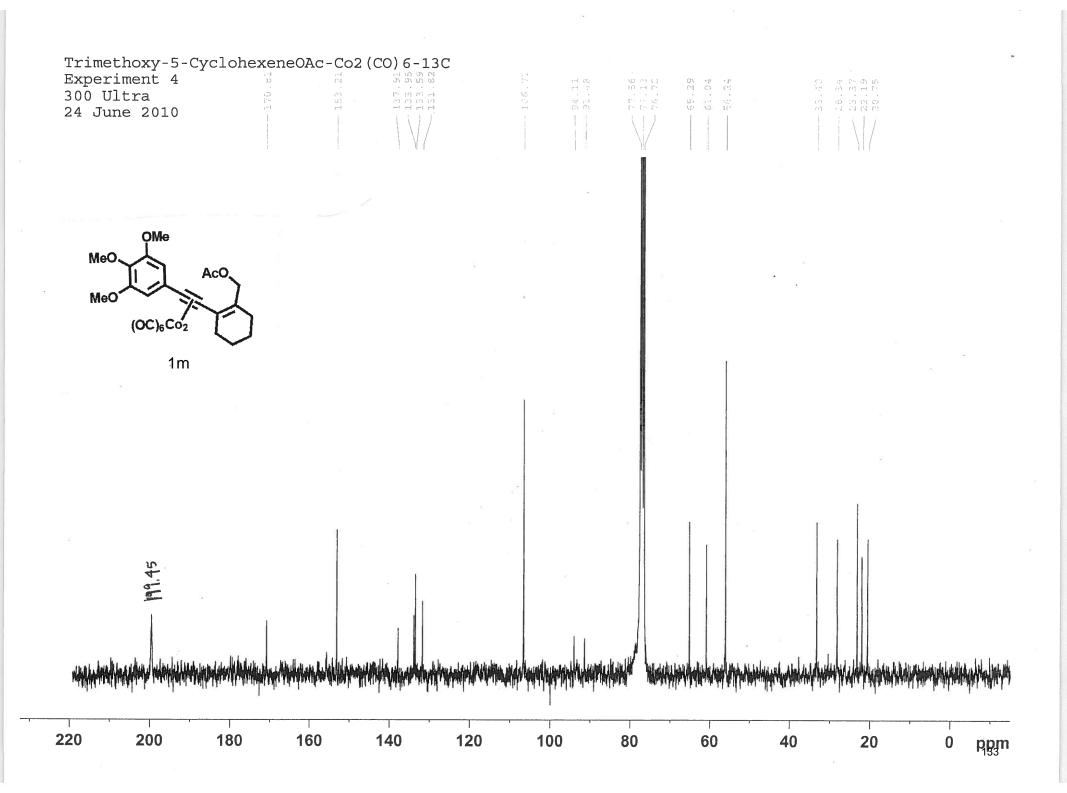


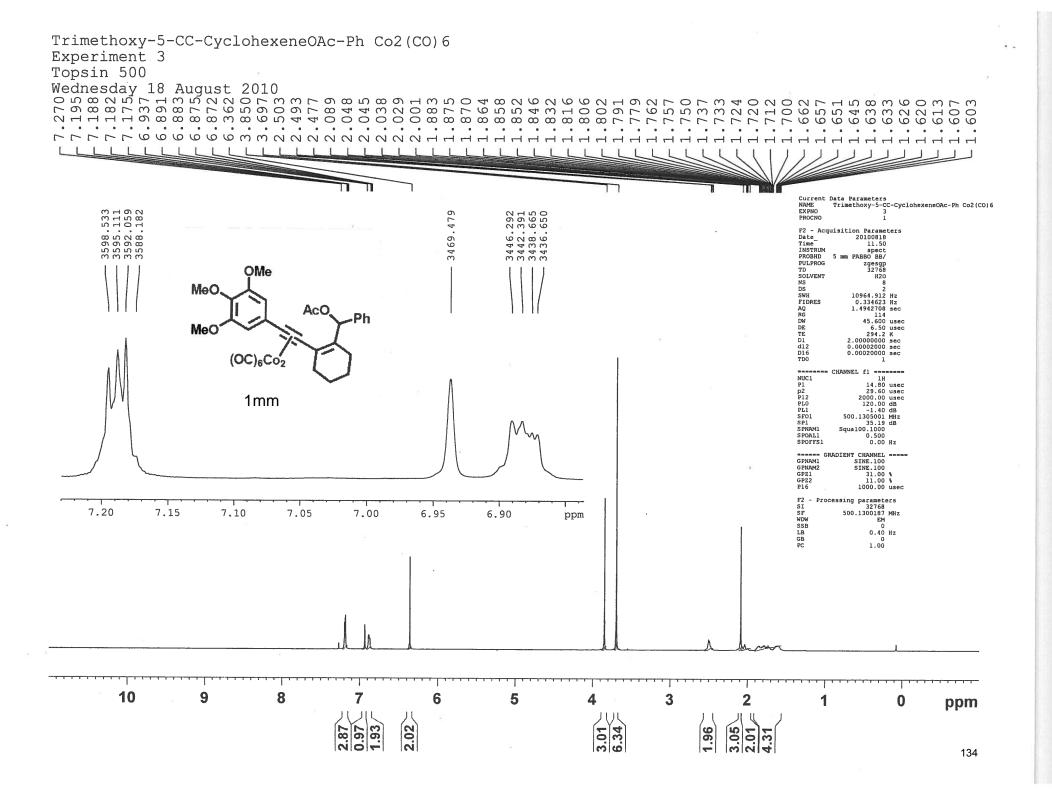
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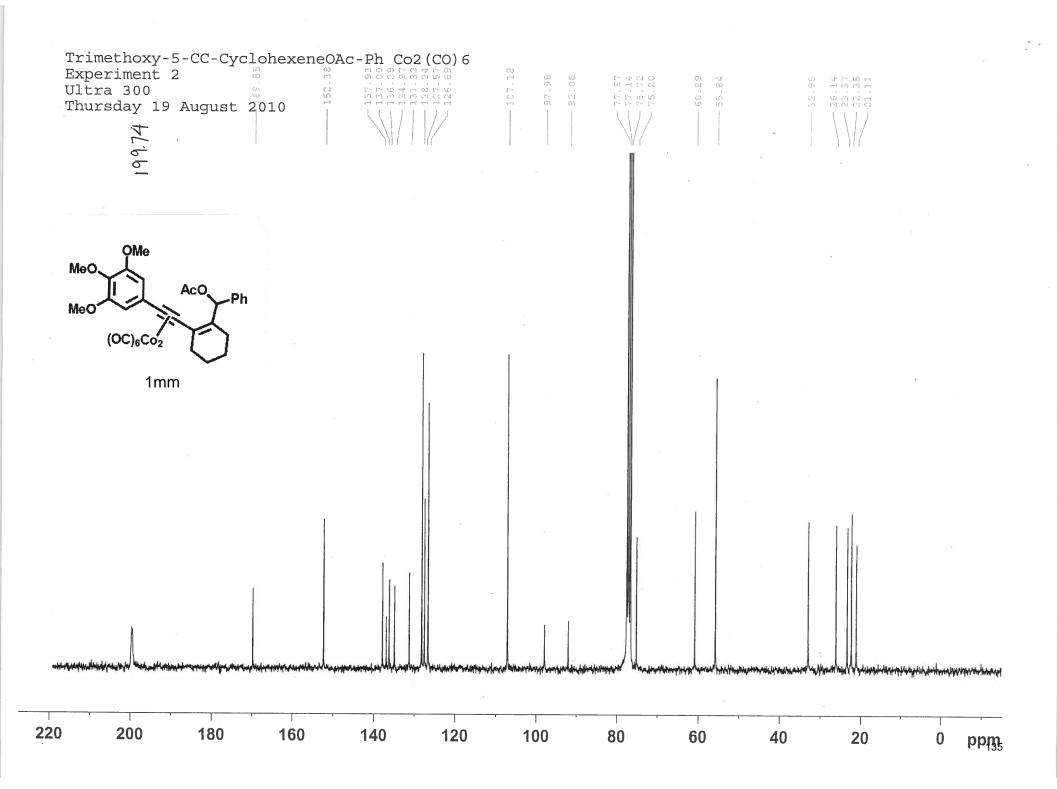
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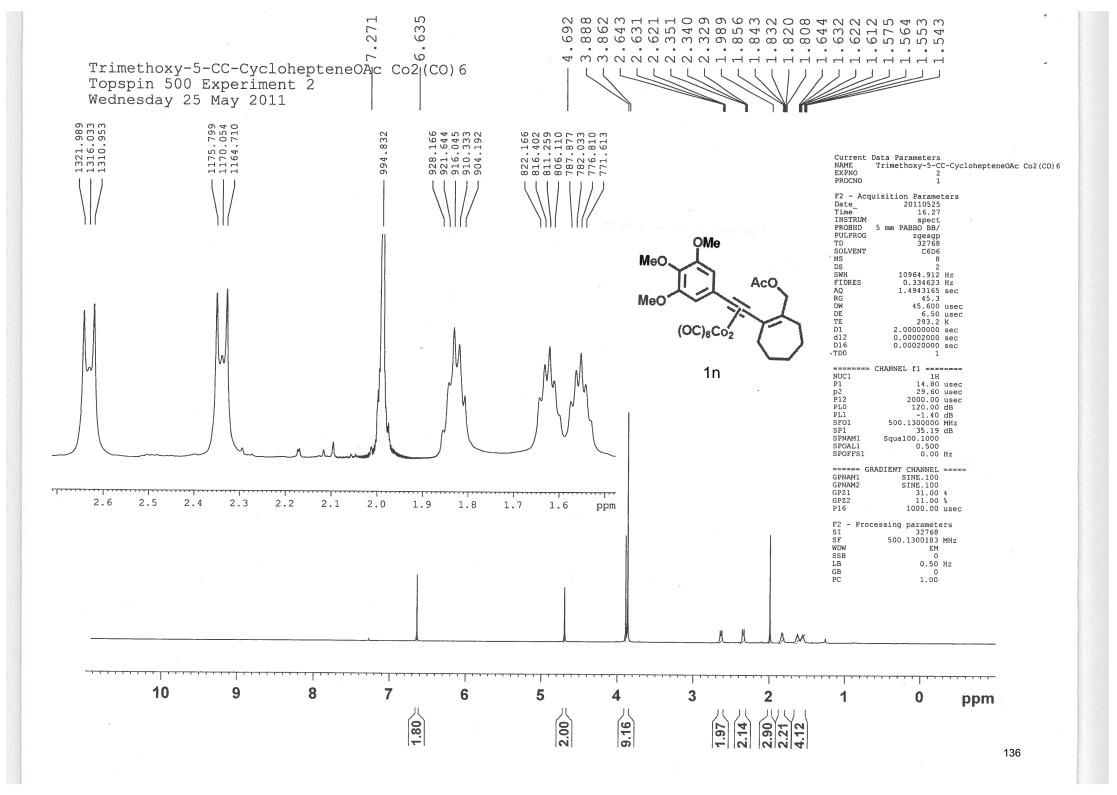


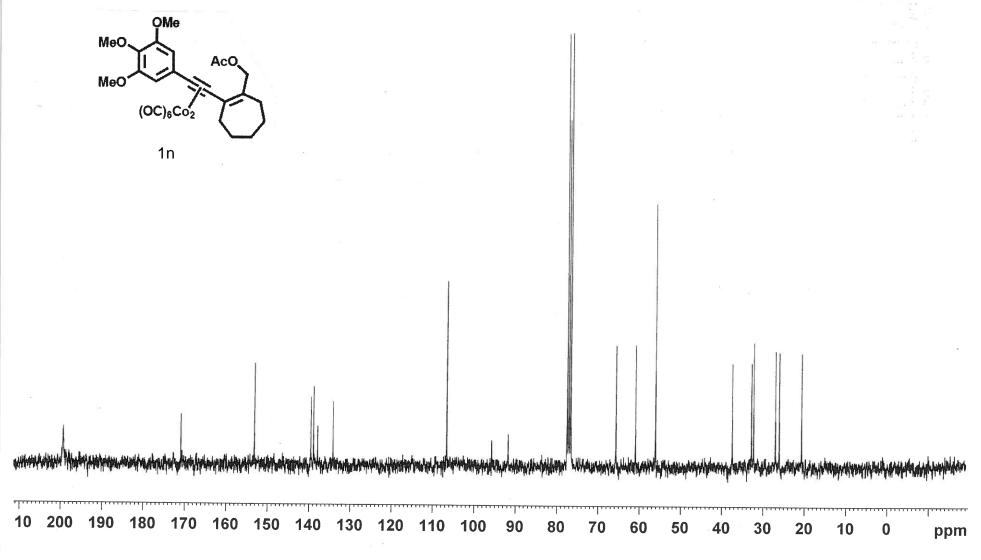


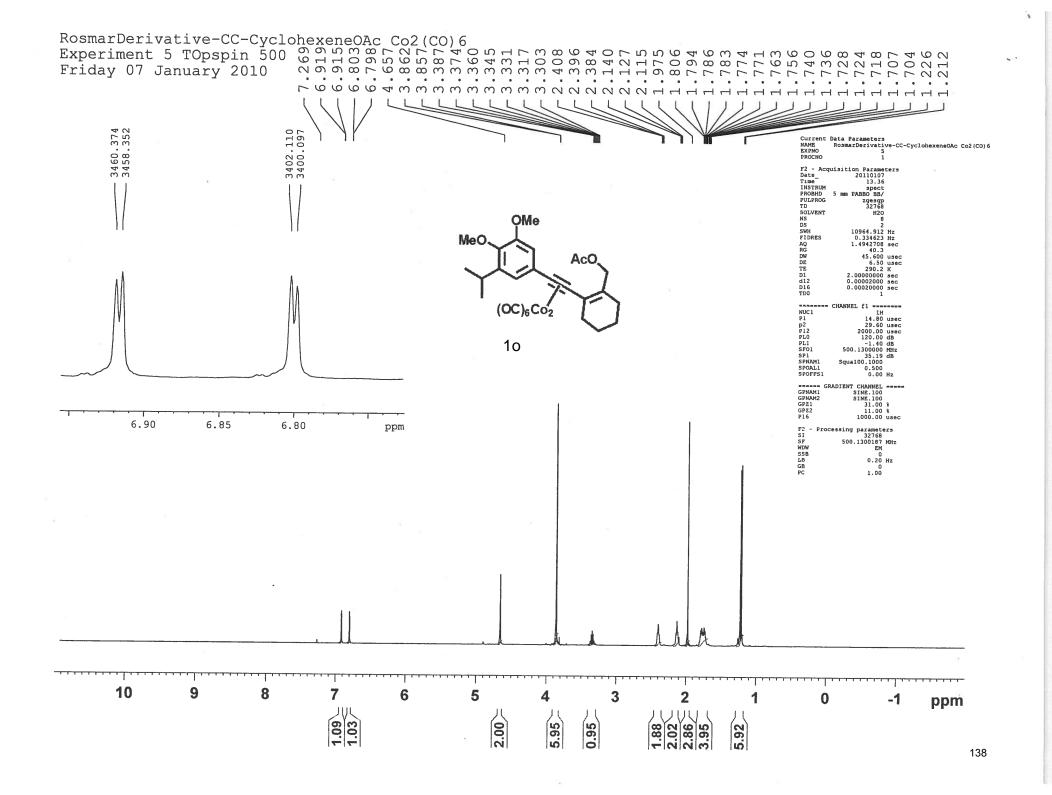


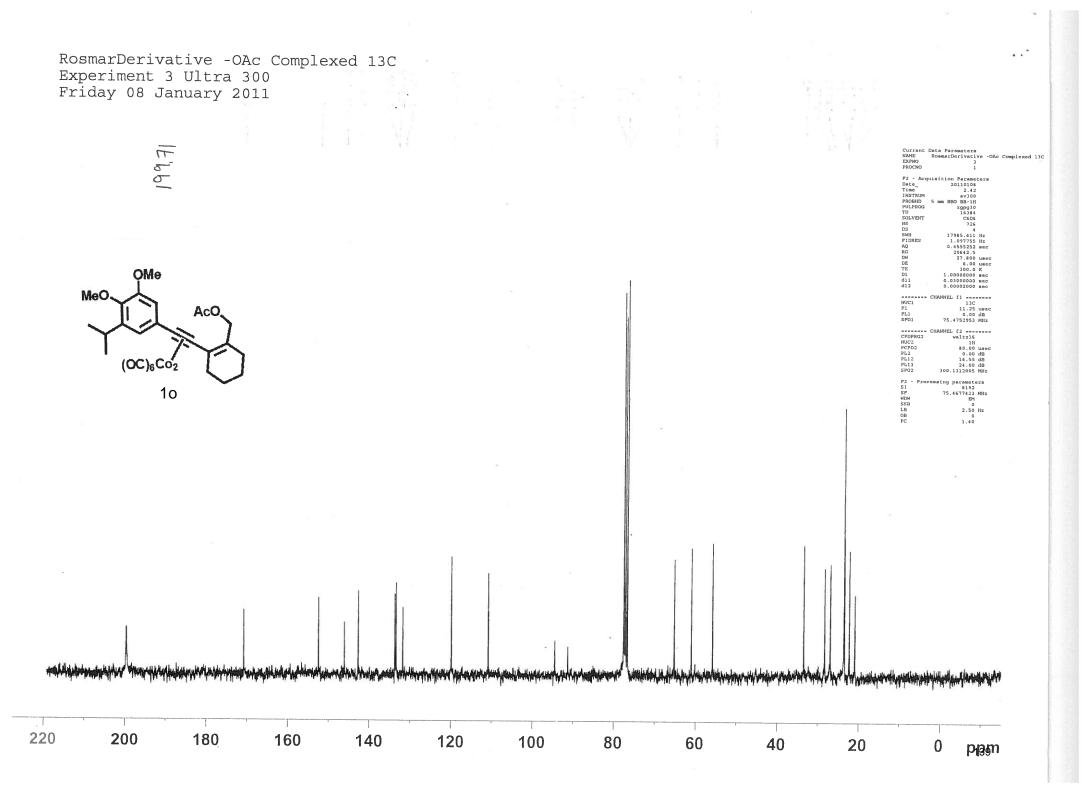


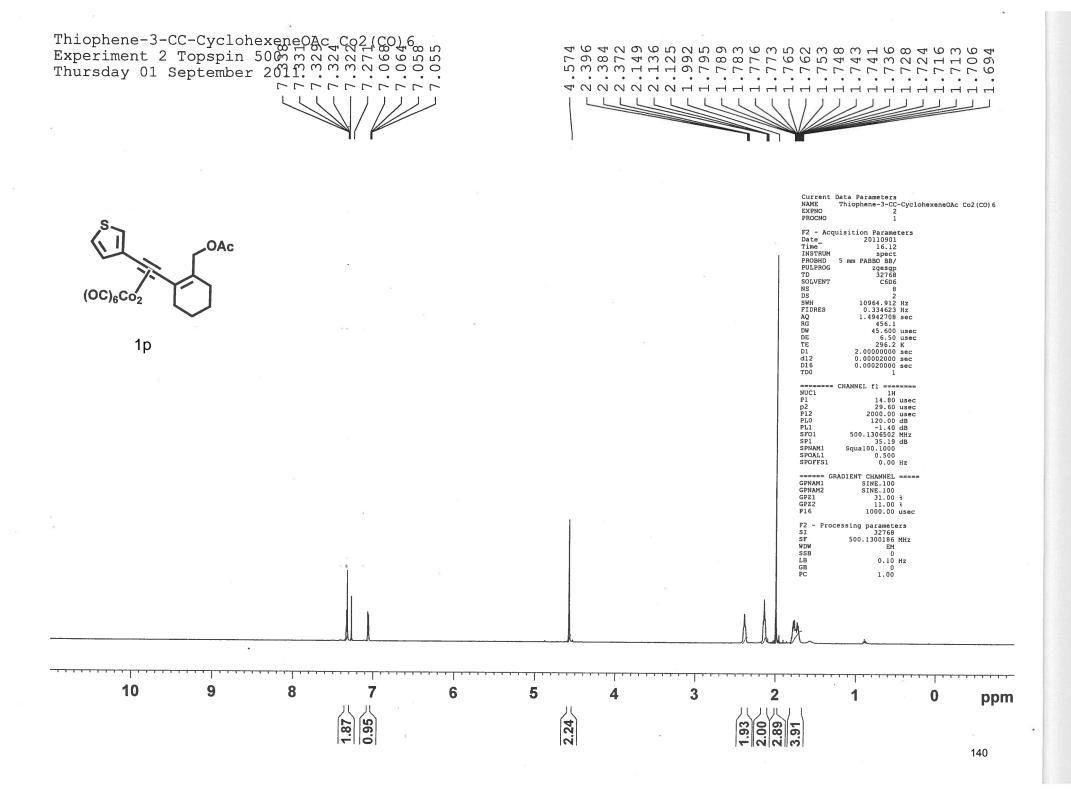


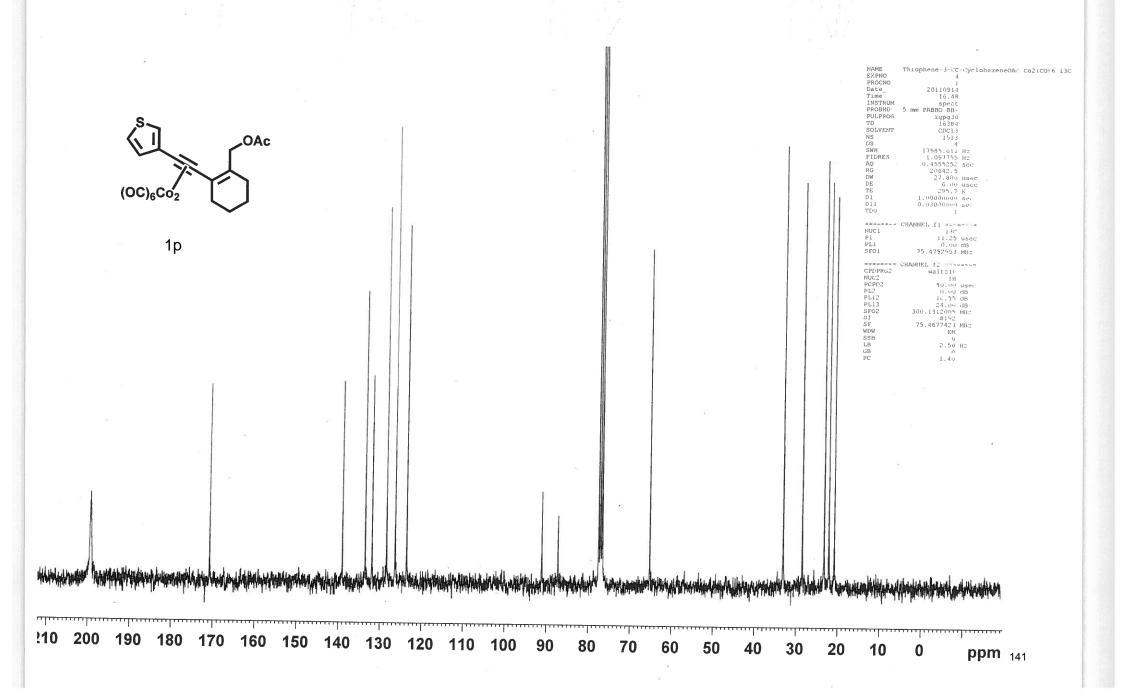


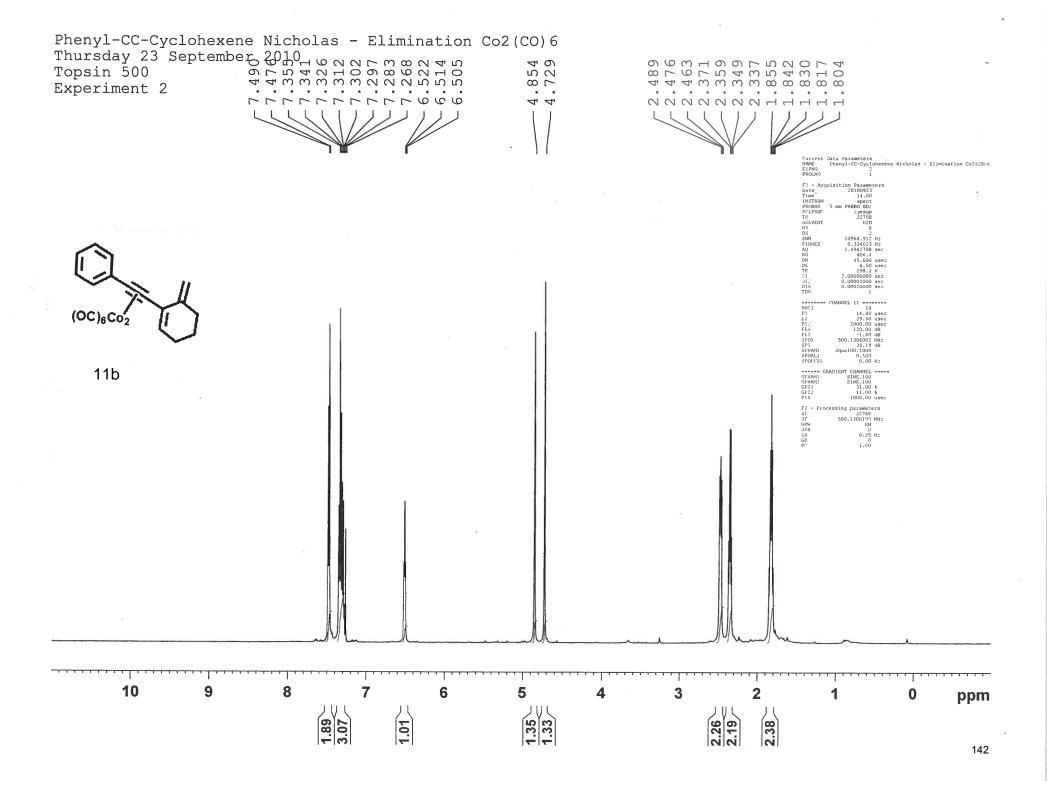


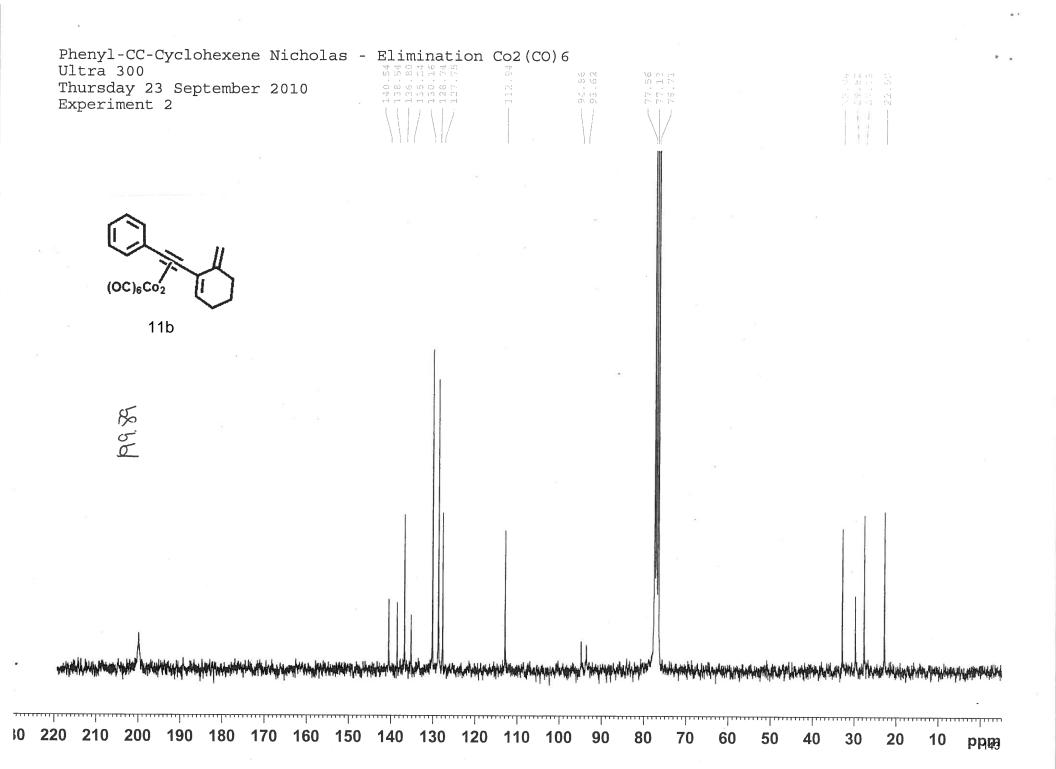


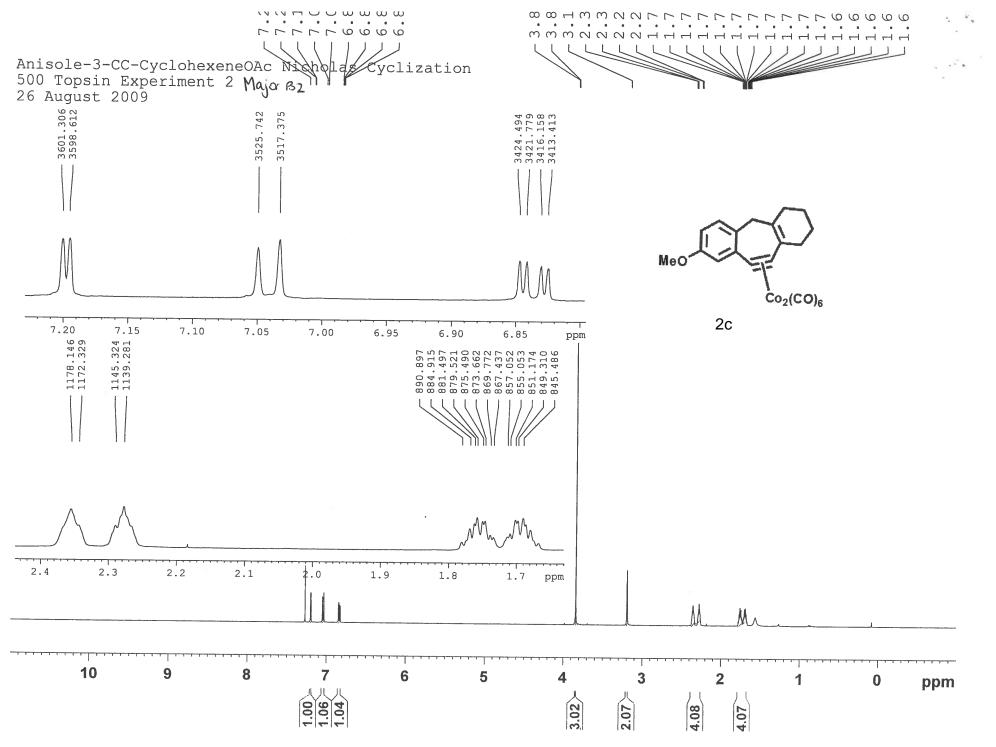


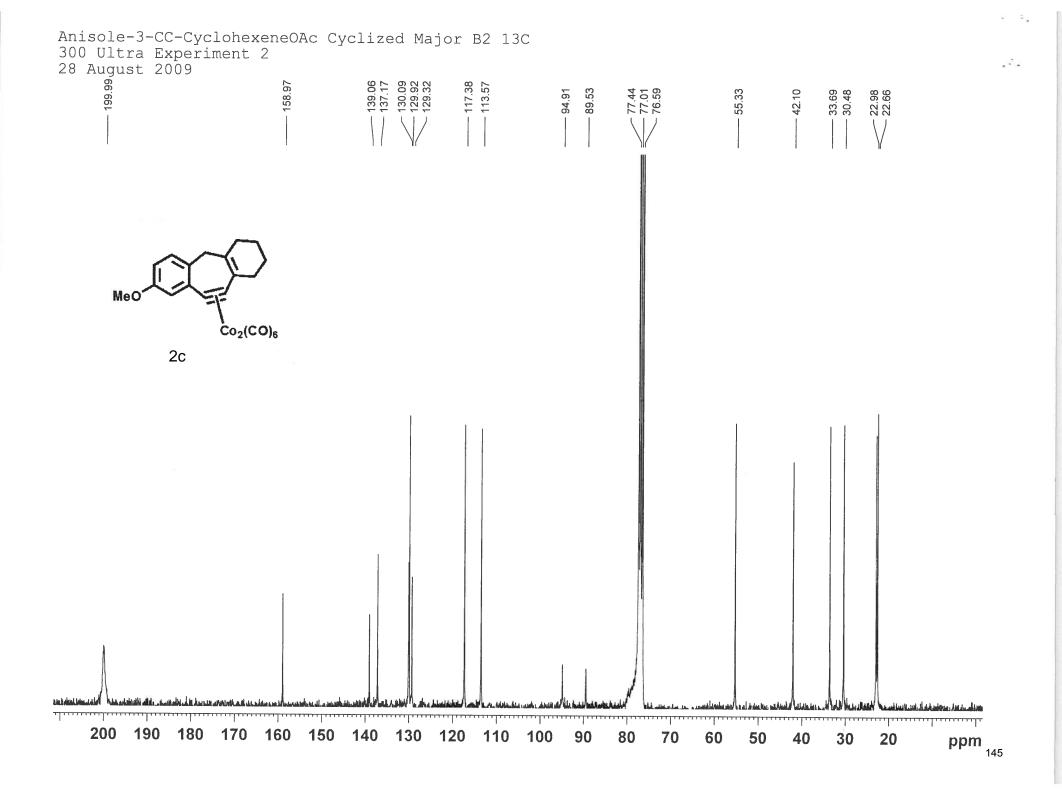


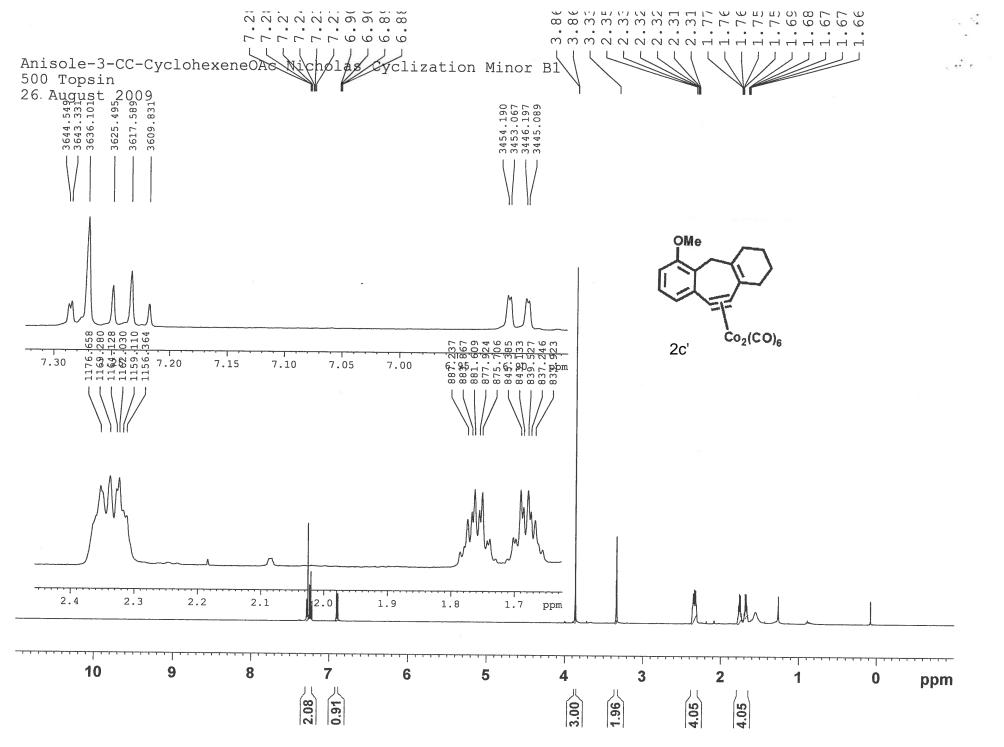


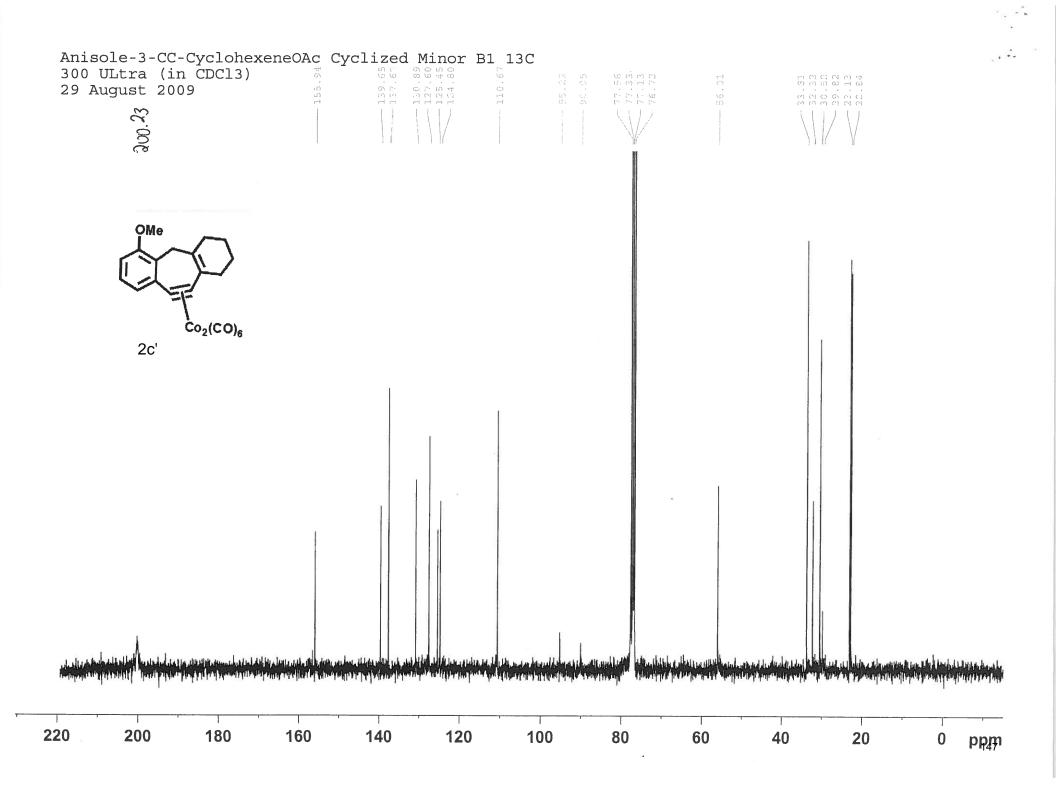


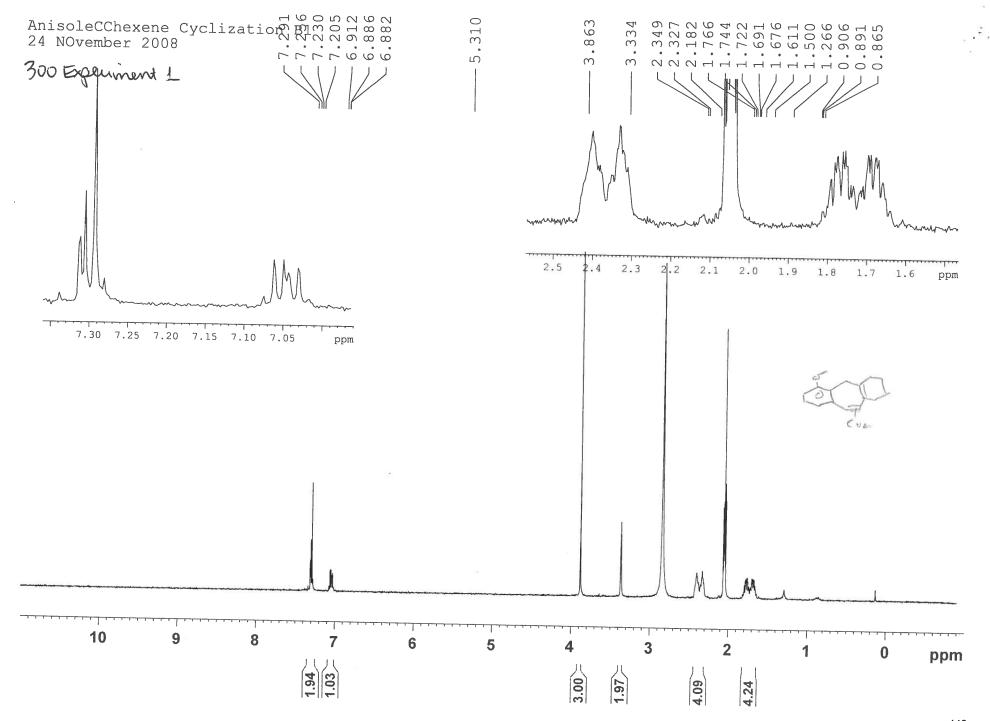


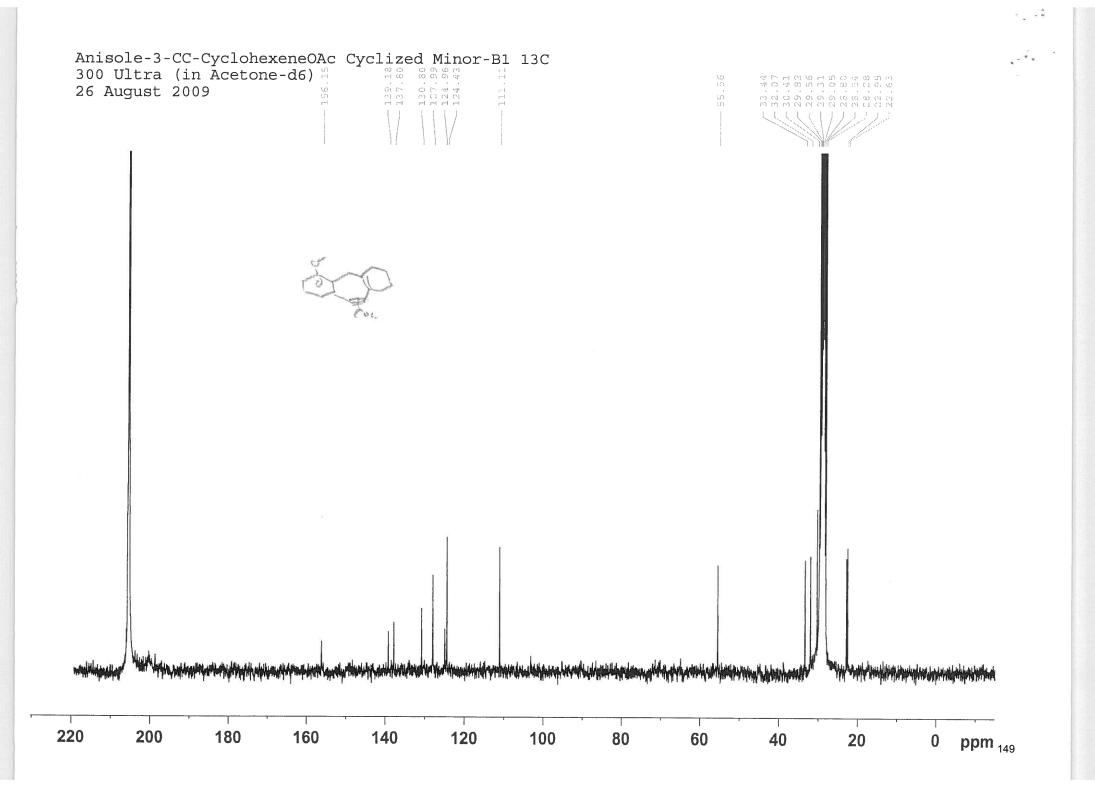


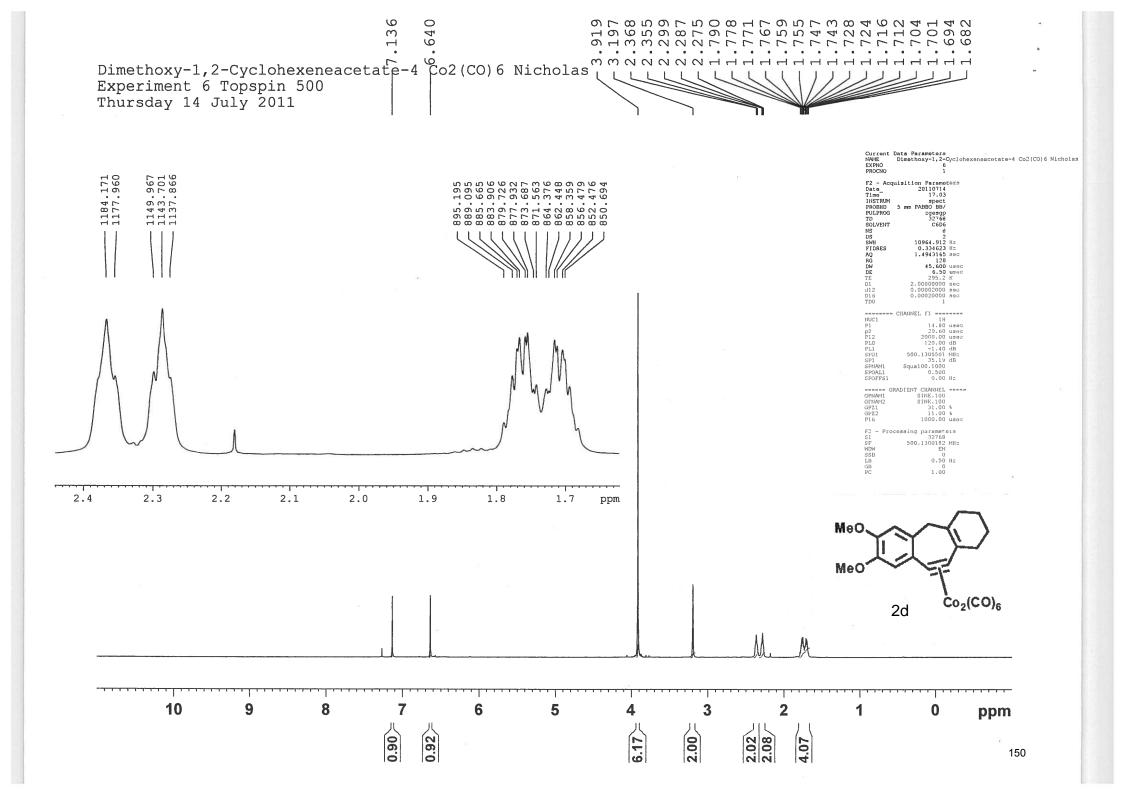


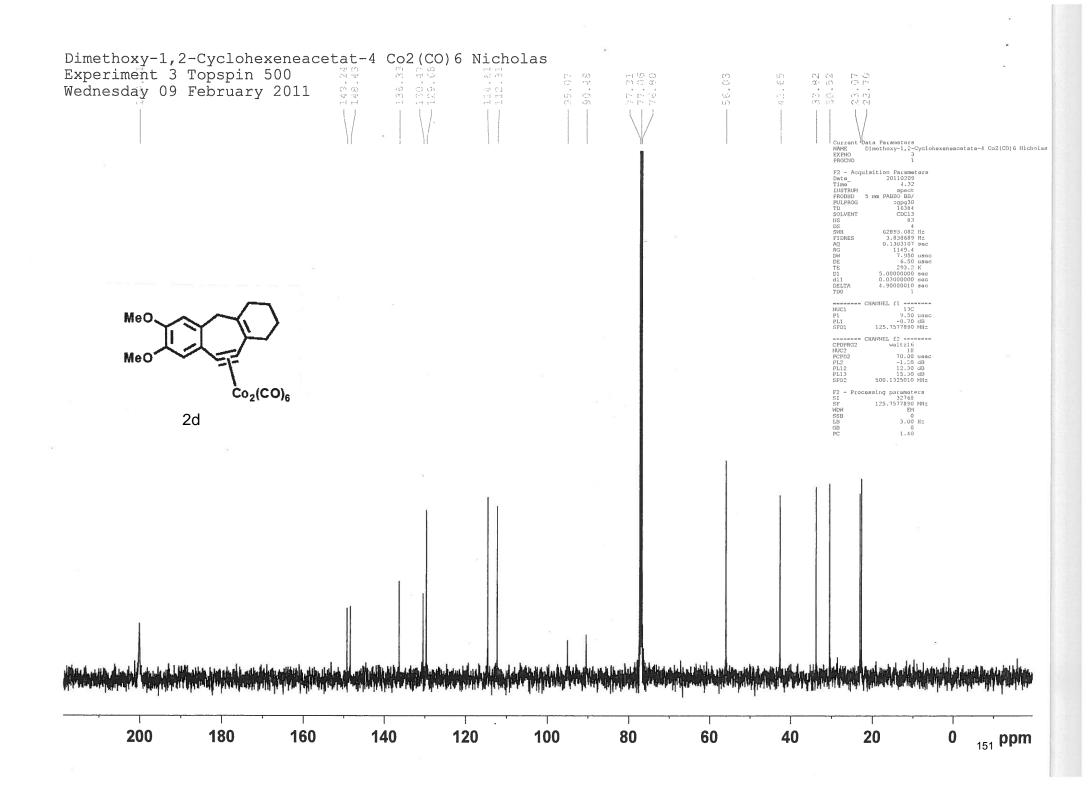


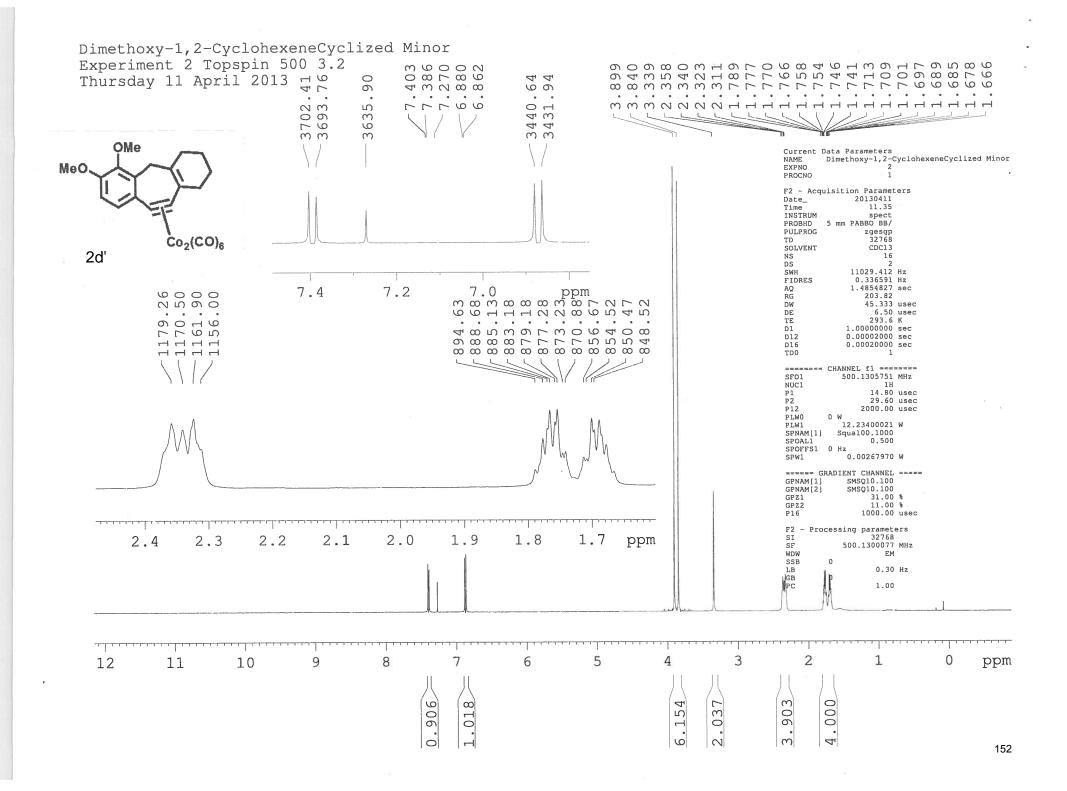


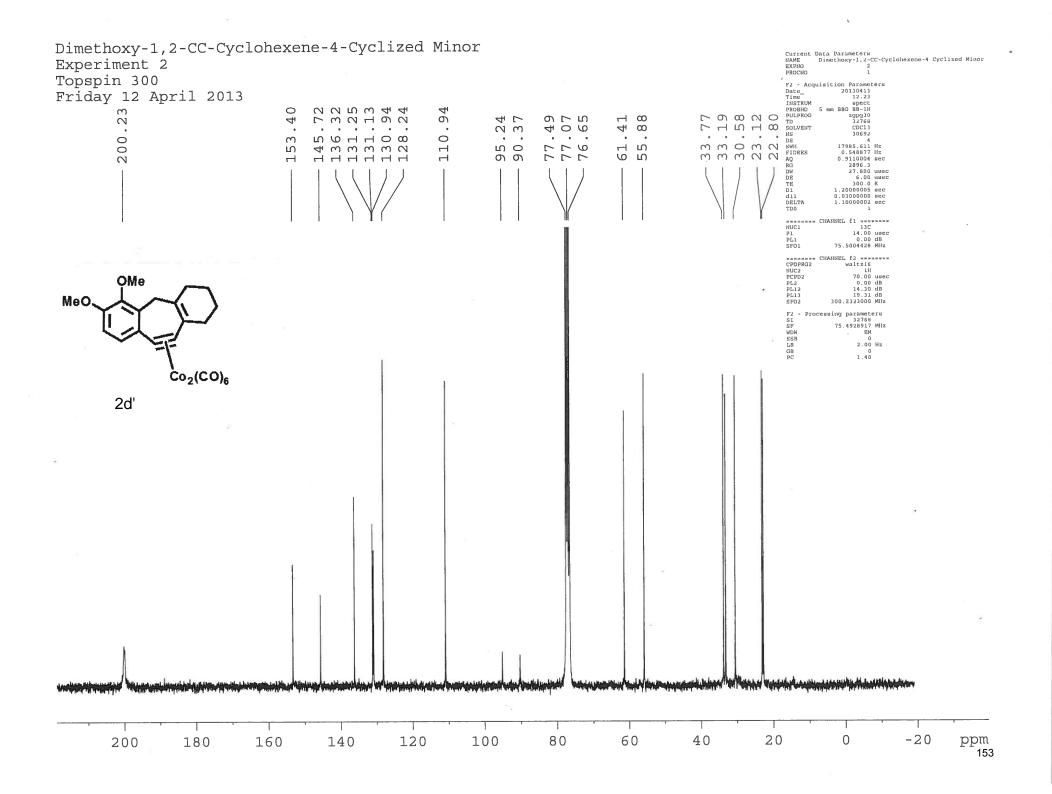


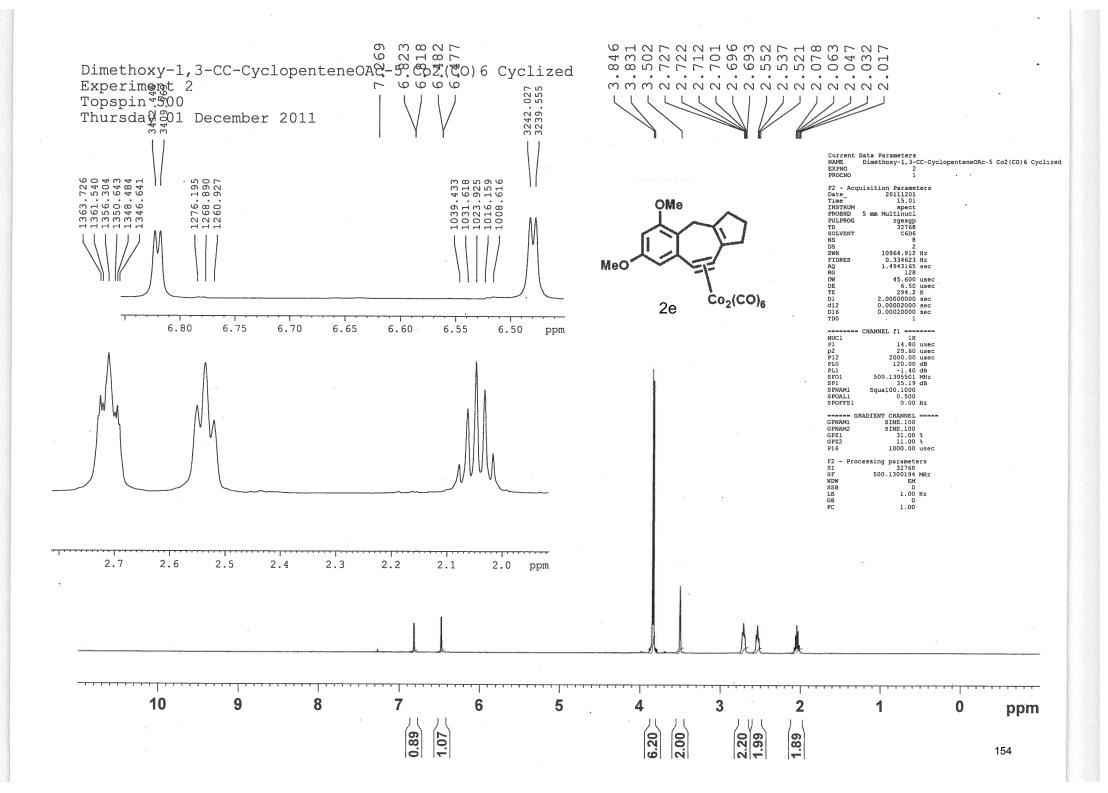


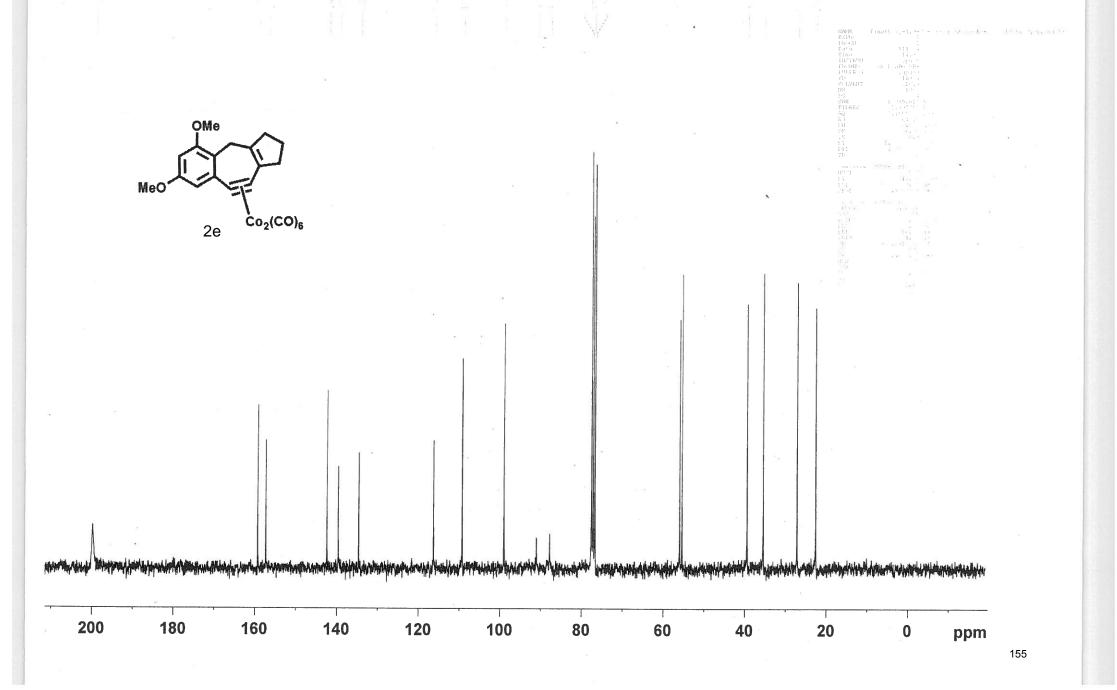


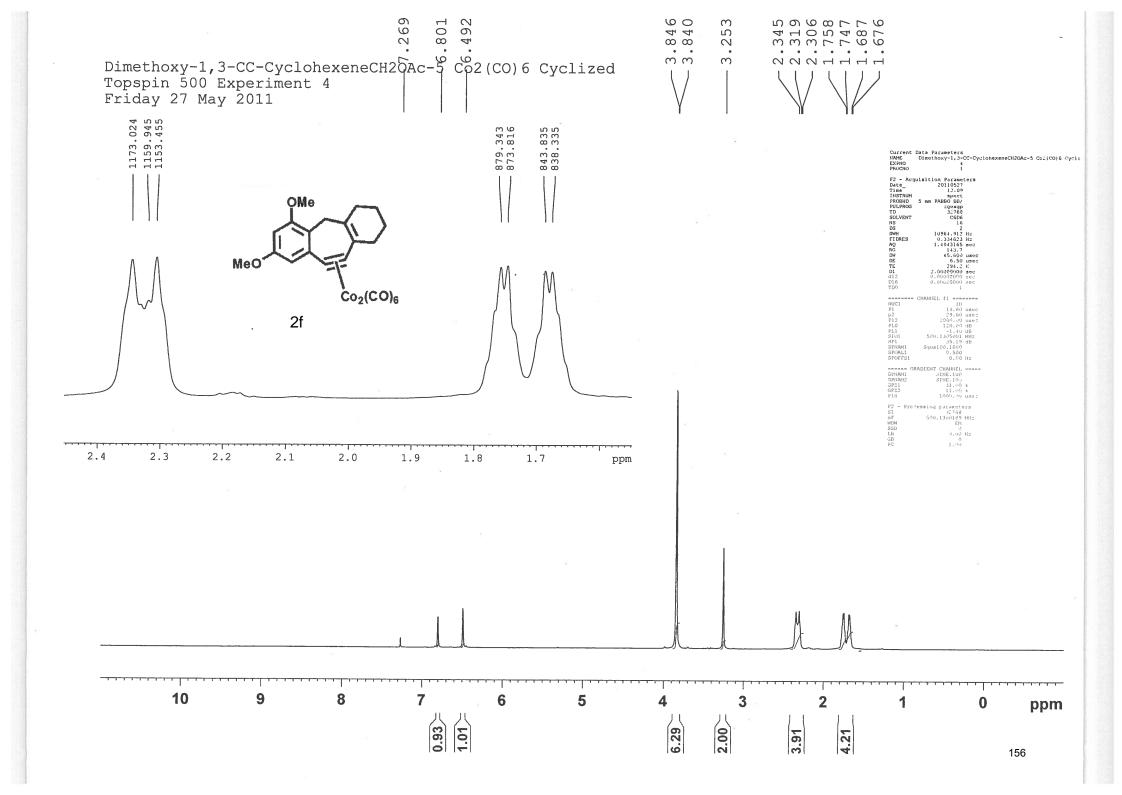


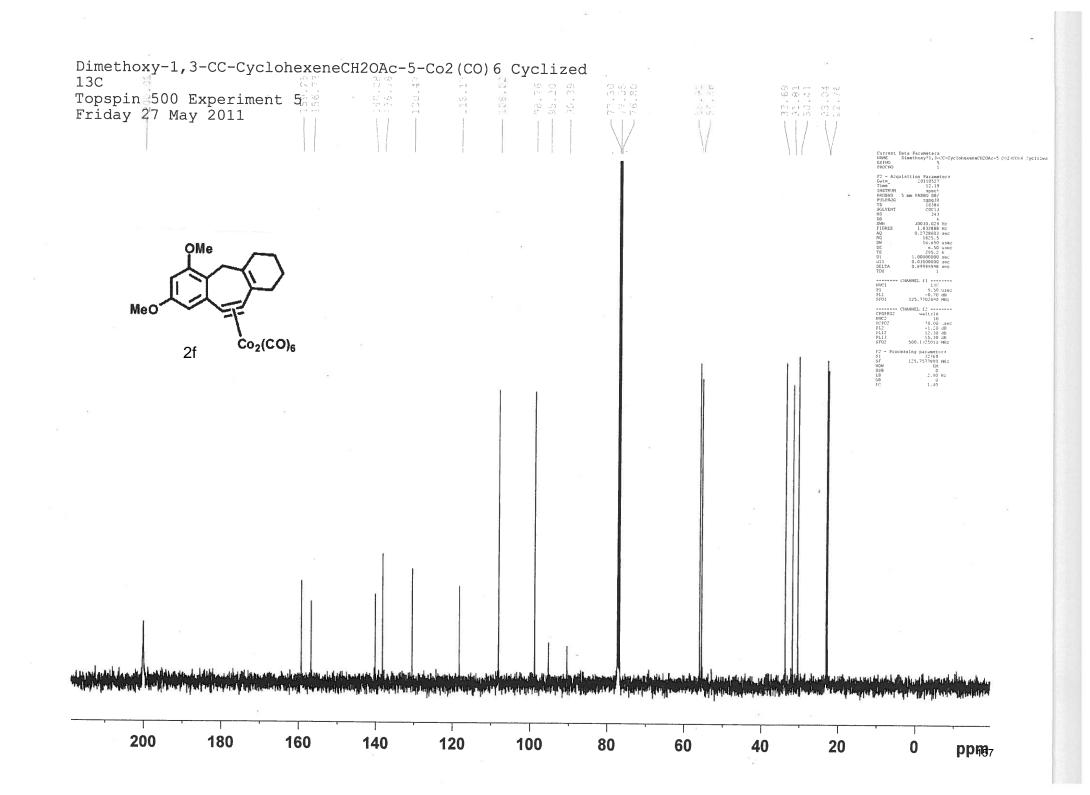


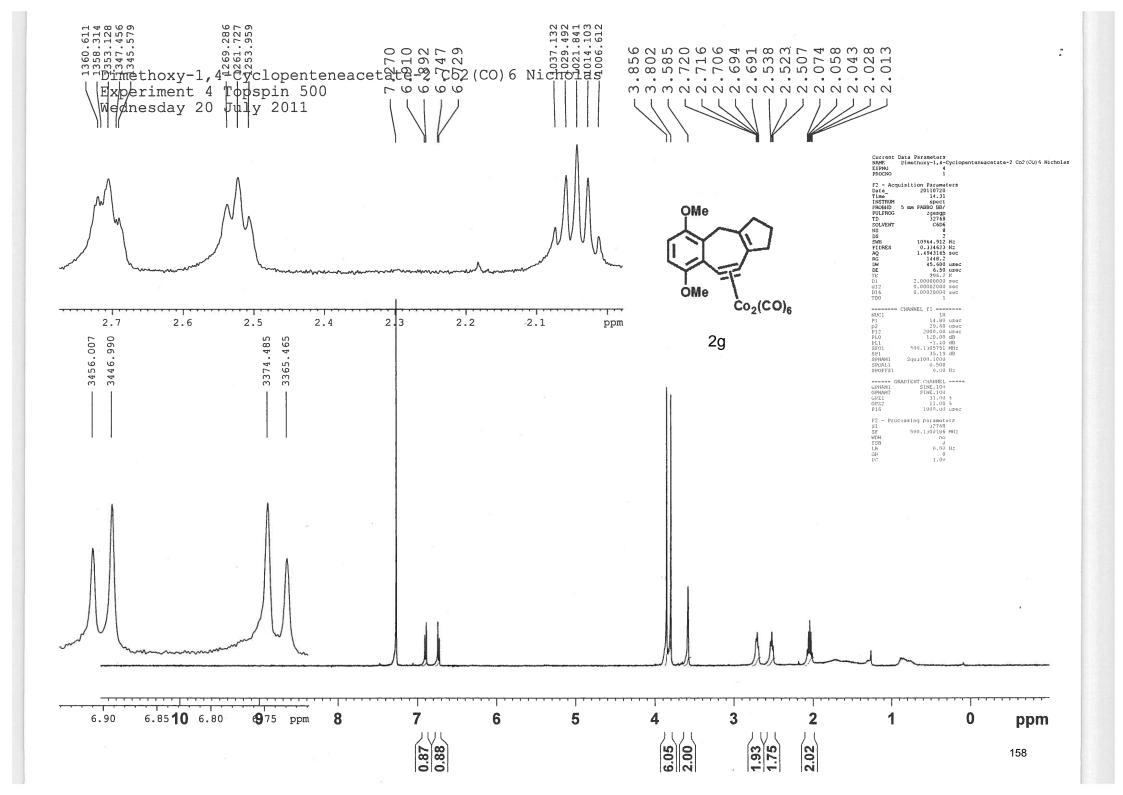


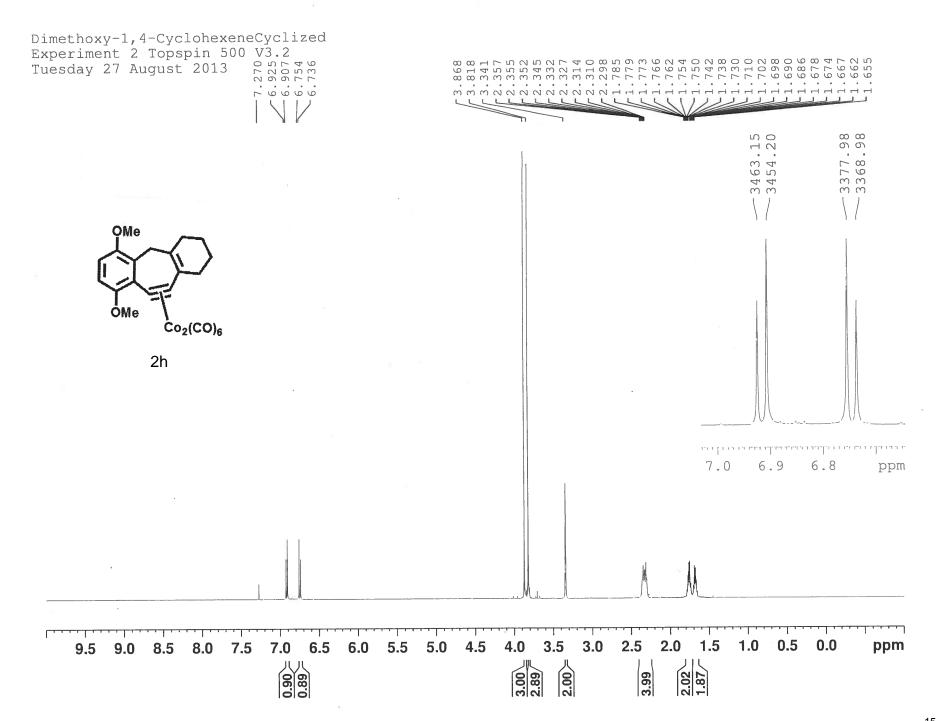


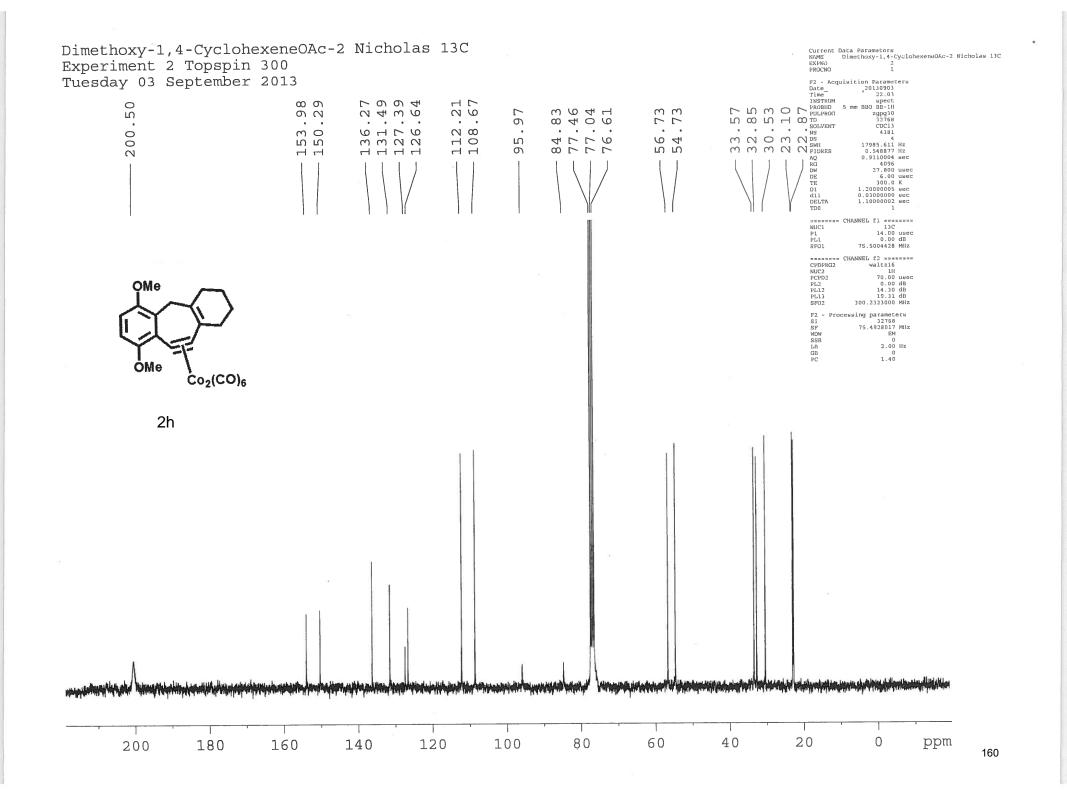


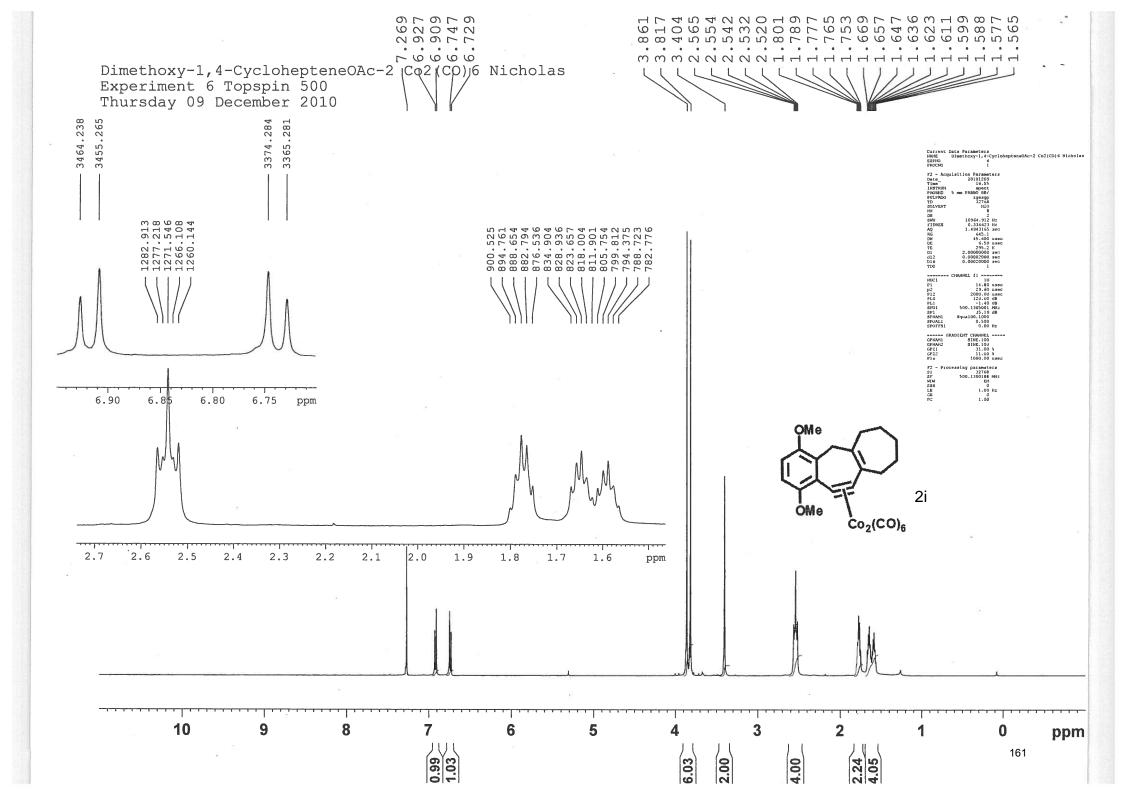


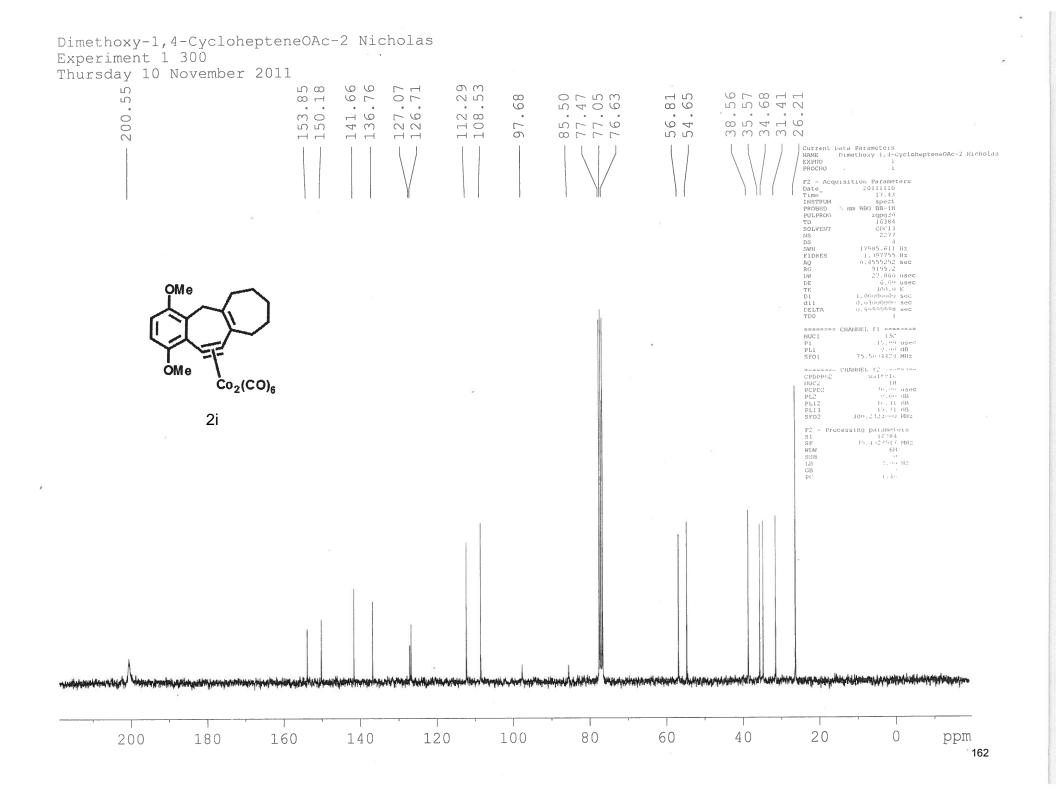


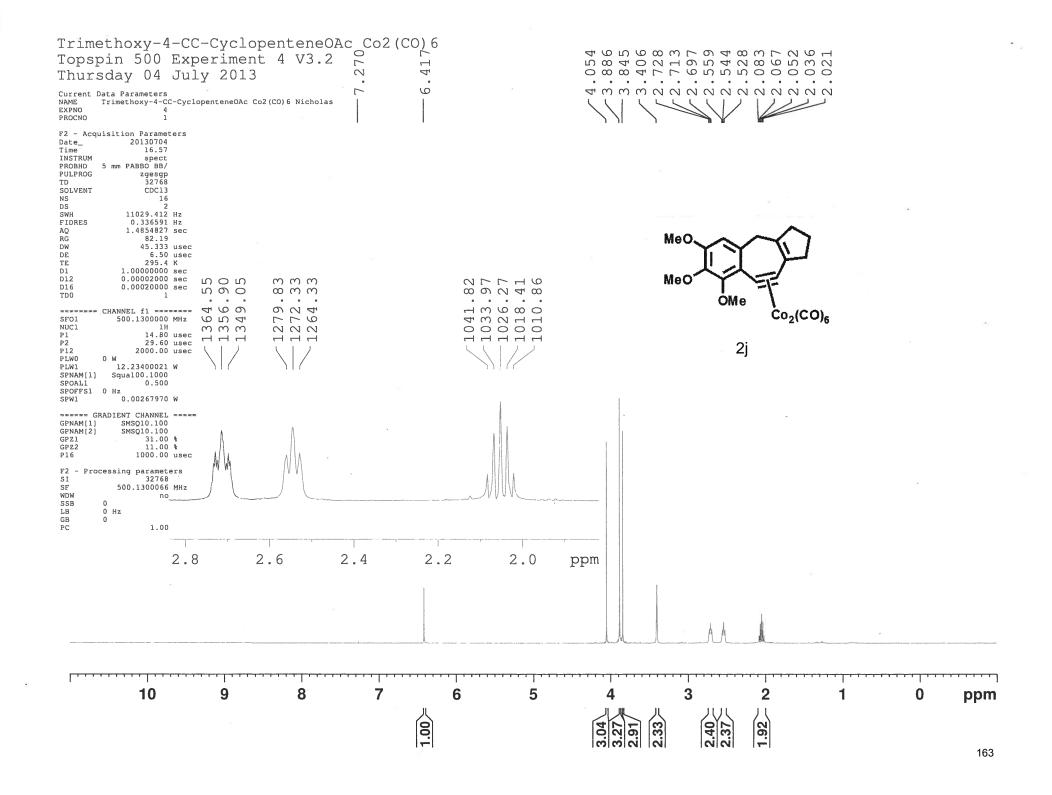


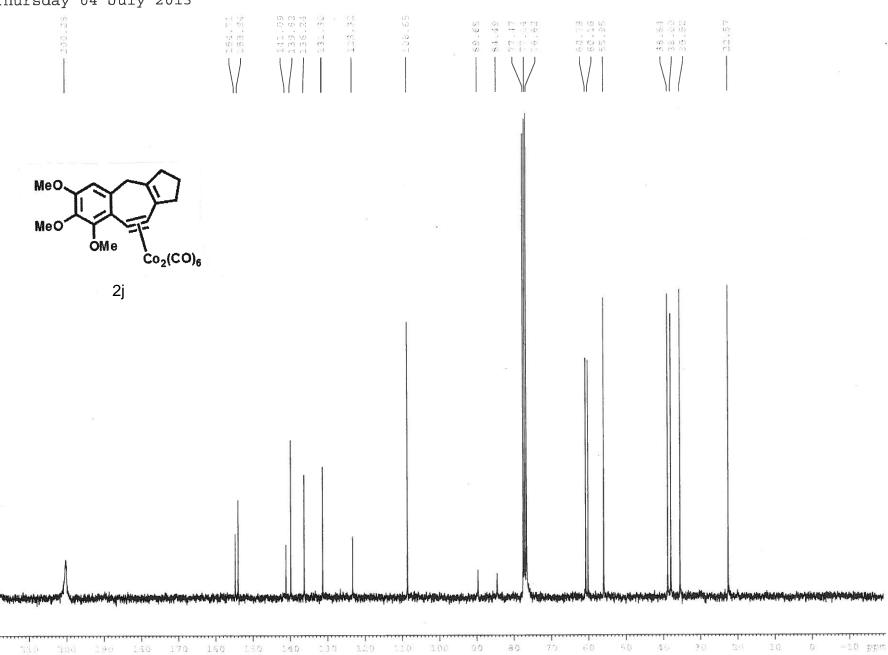


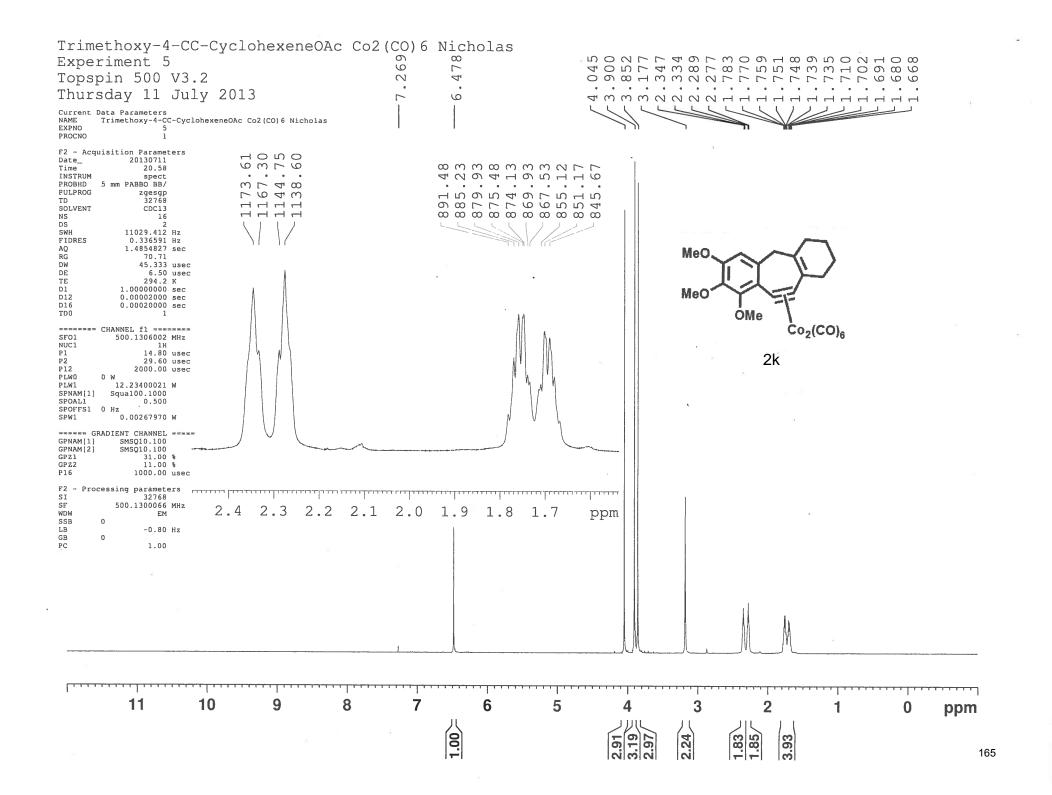


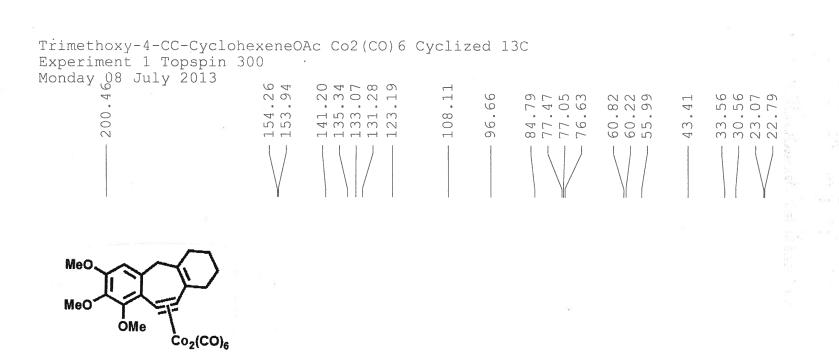


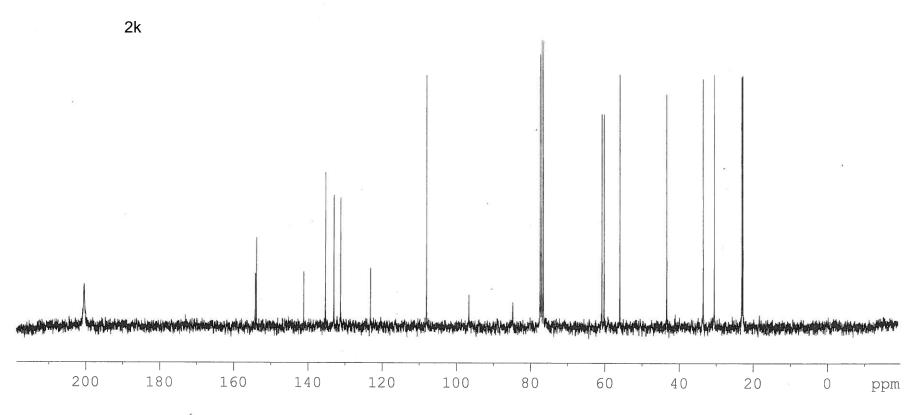


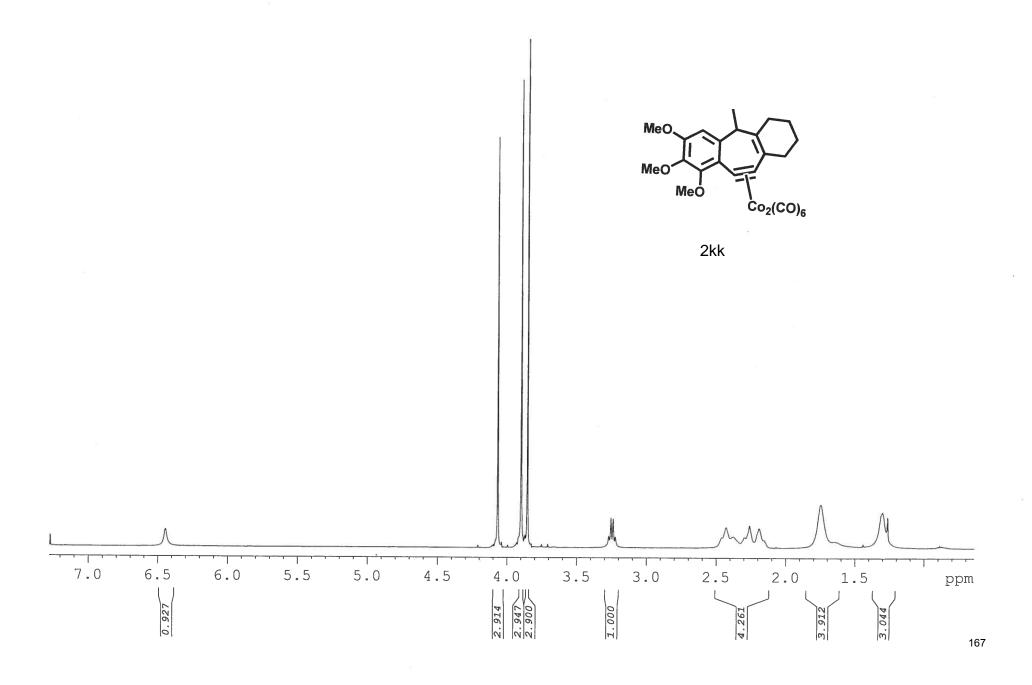


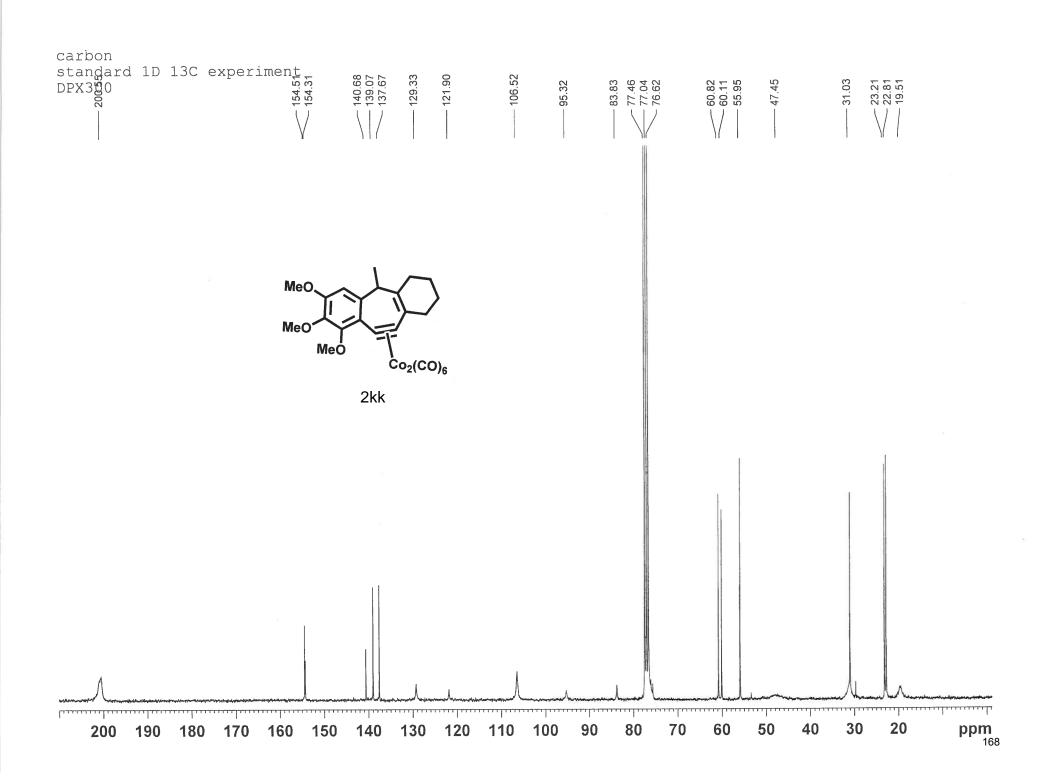


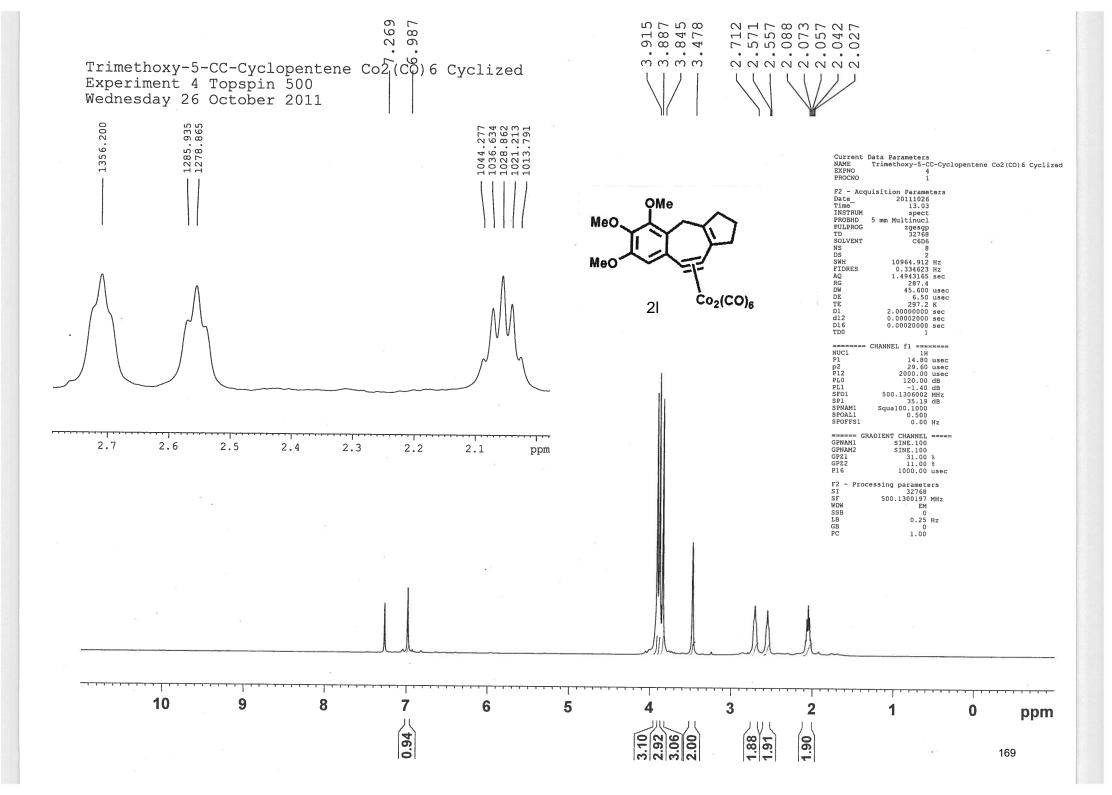


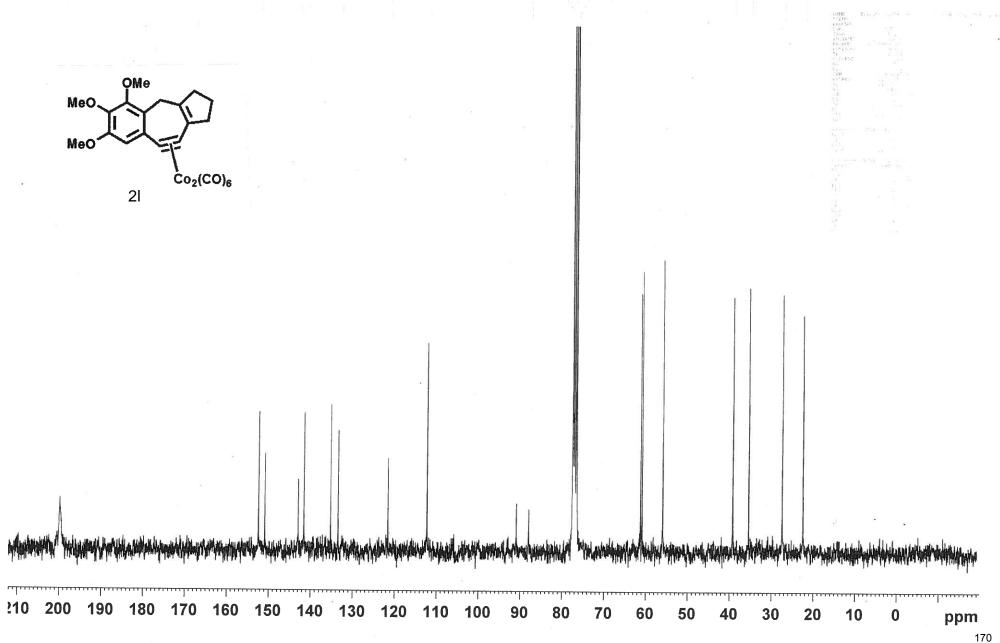


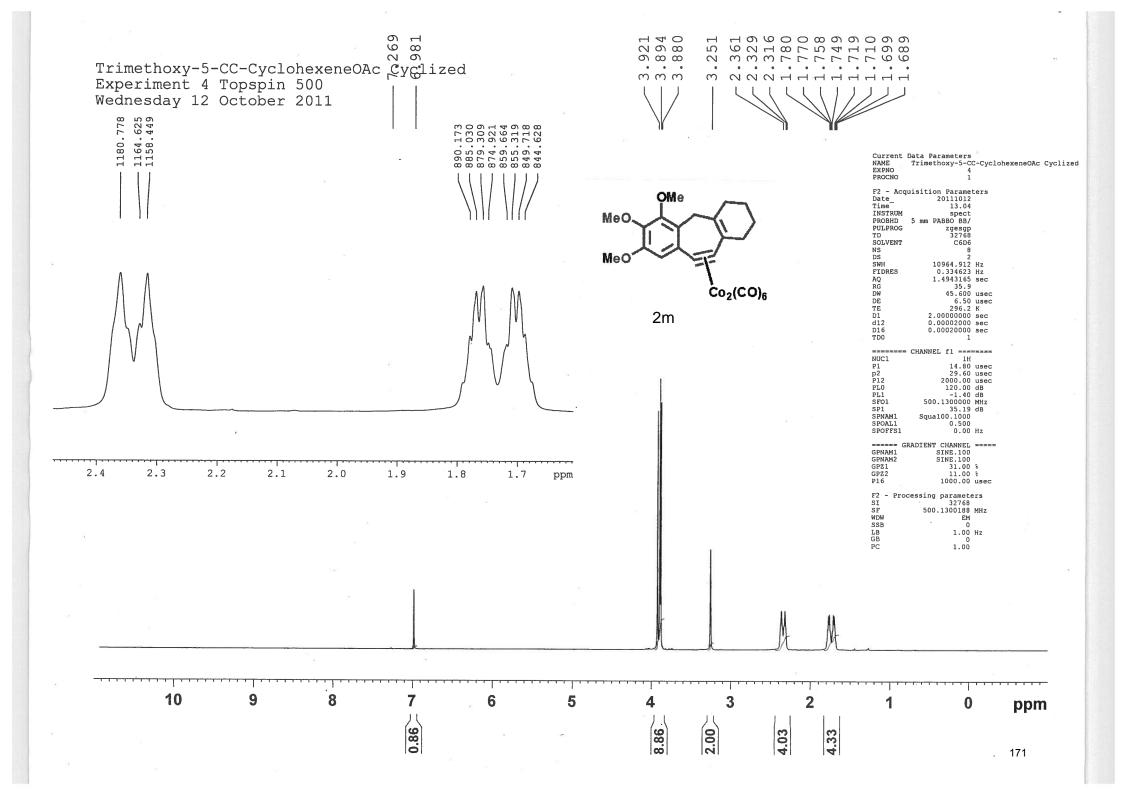


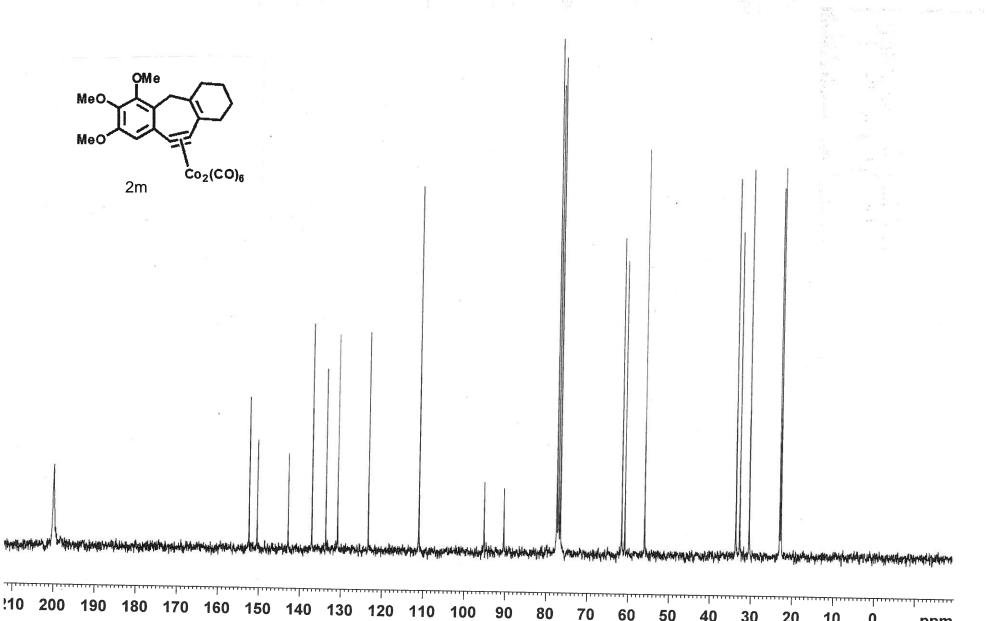


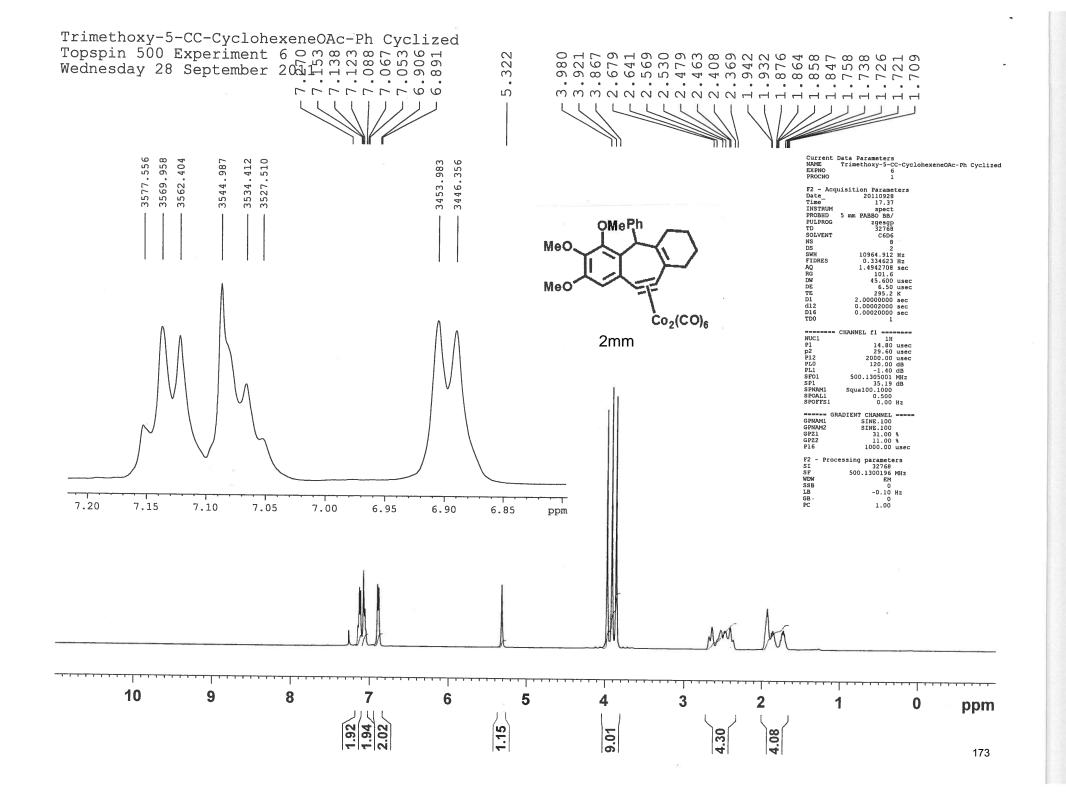


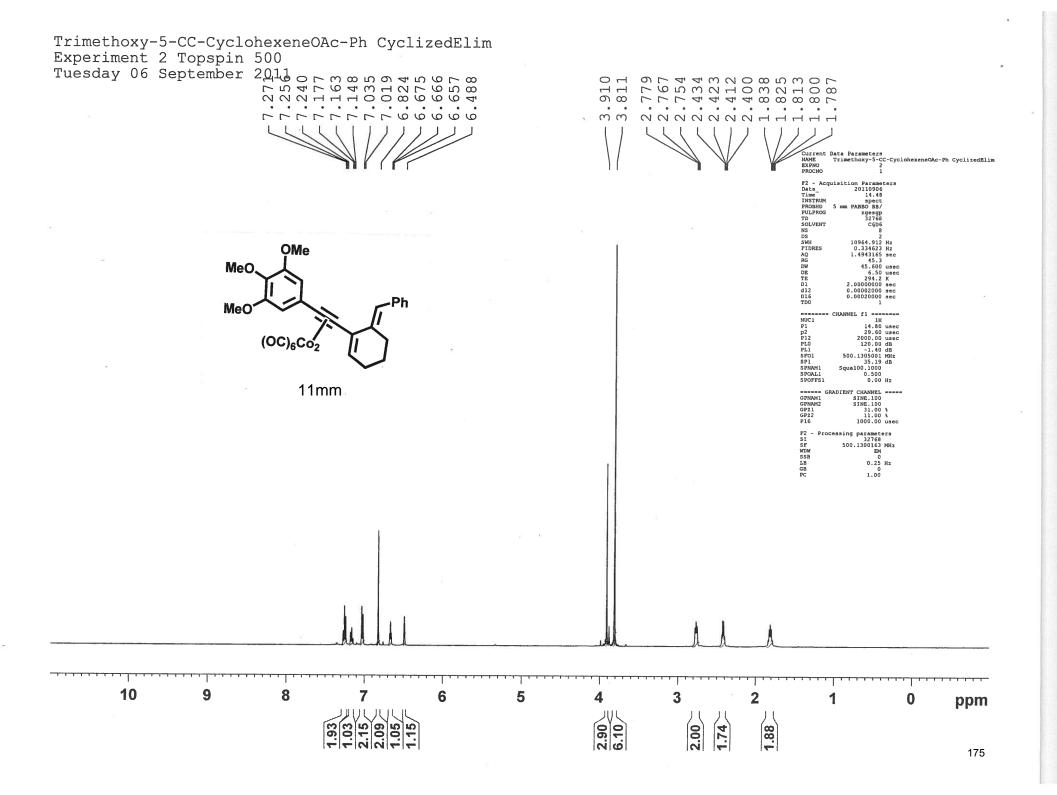


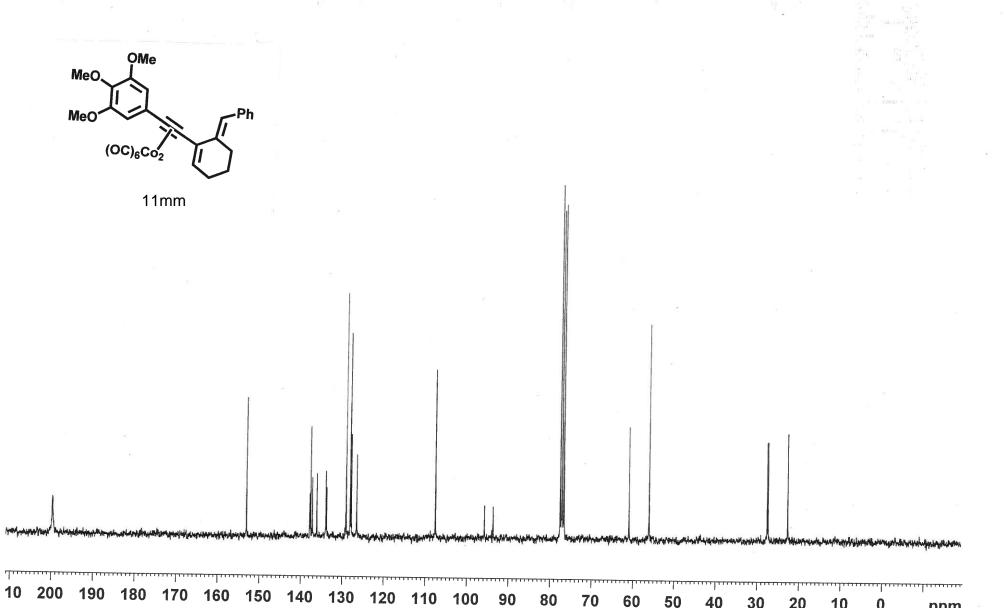


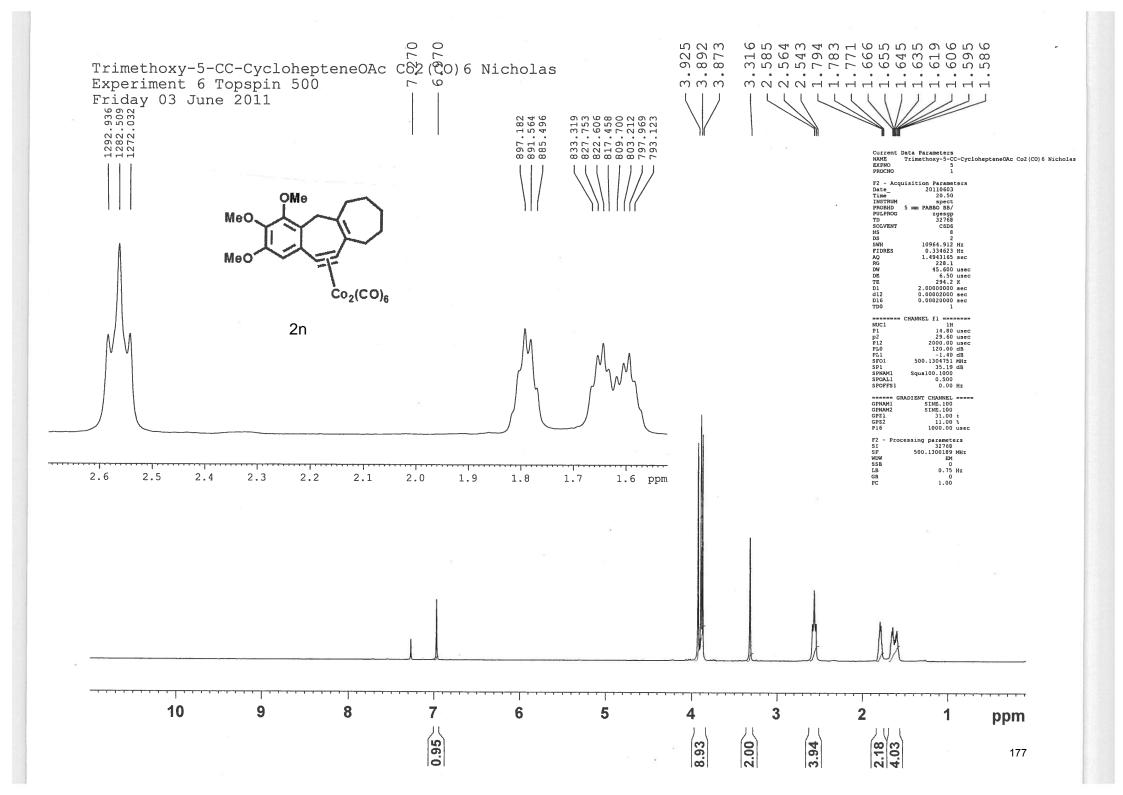


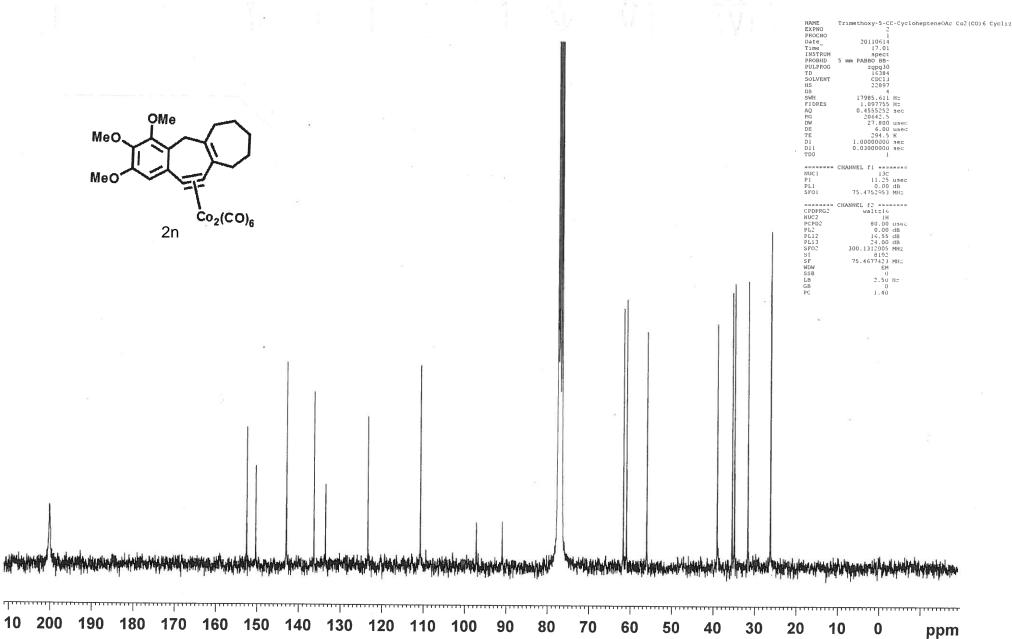






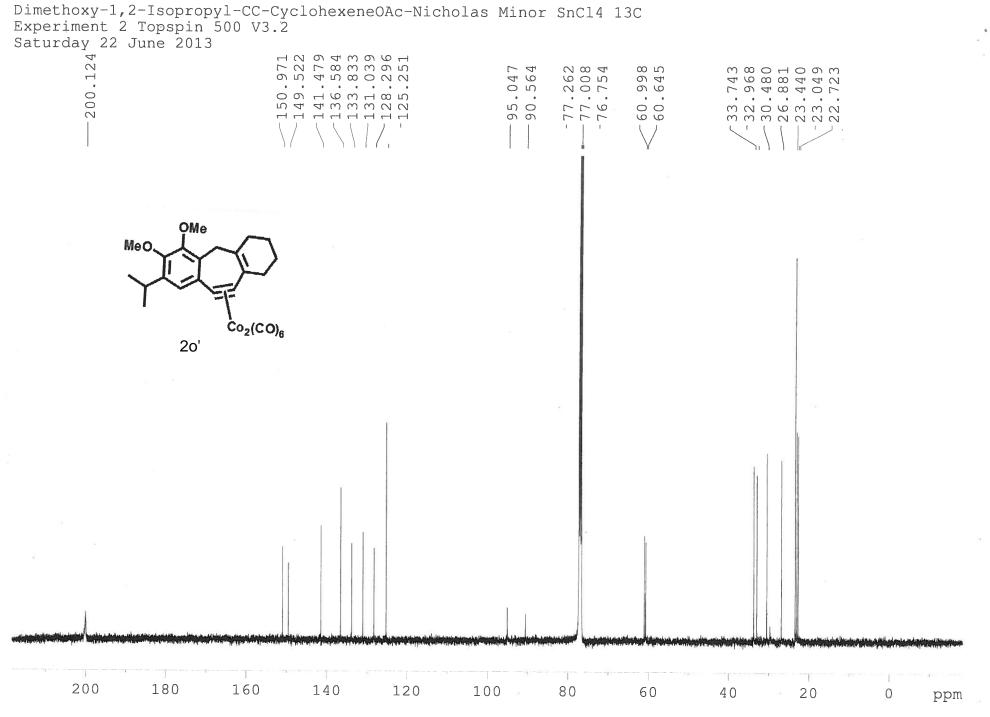


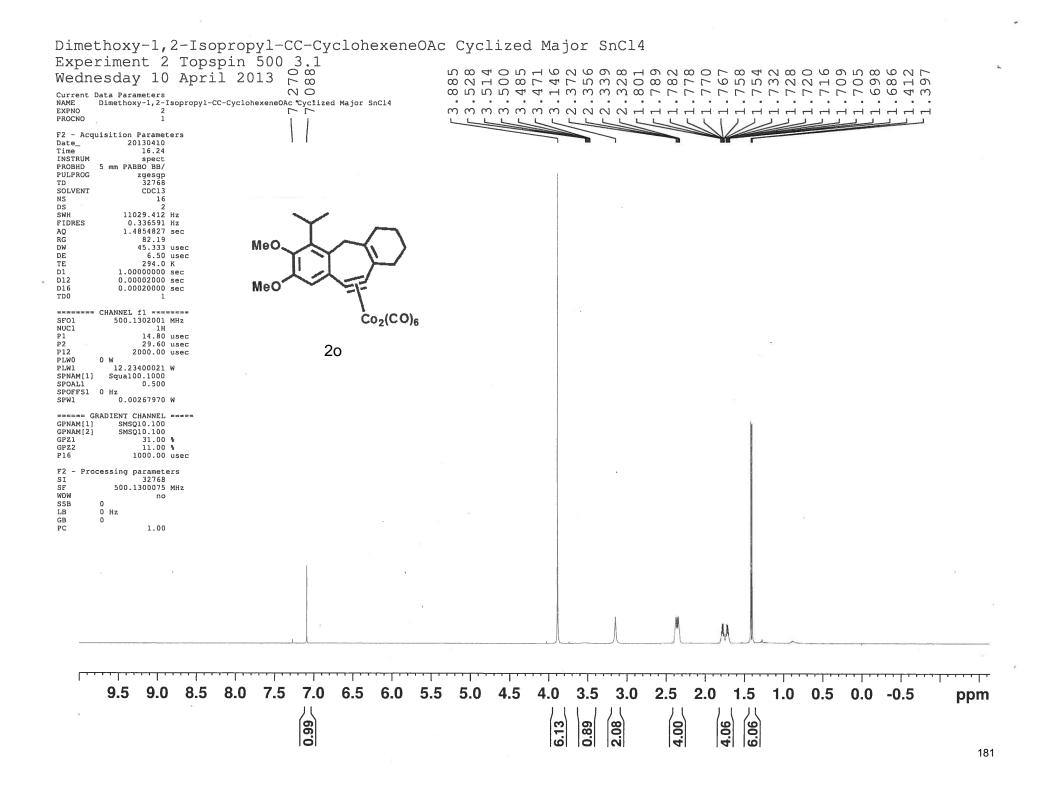


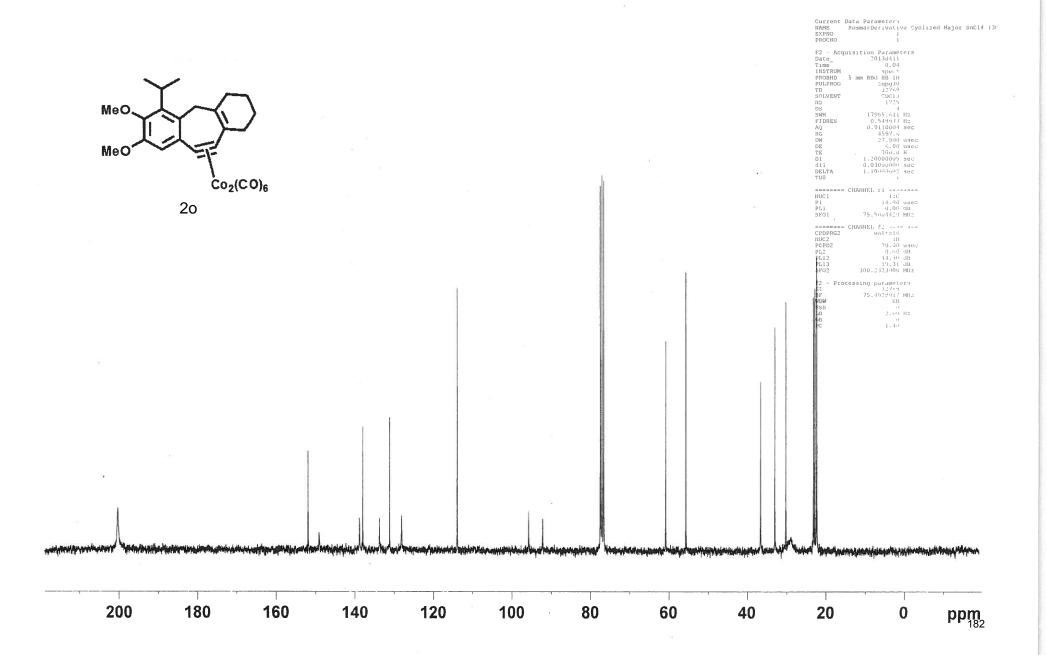


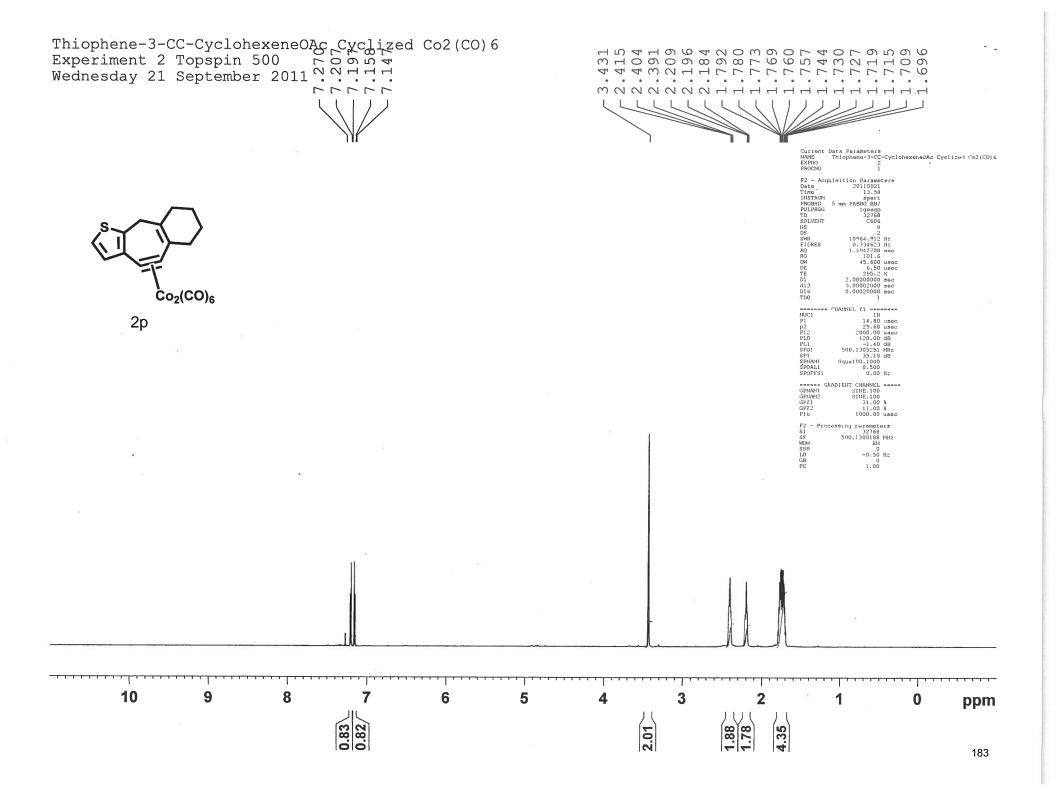
Experiment 2 Topspin 500 3.2 Wednesday 10 April 2013 Current Data Parameters Dimethoxy-1, 2-Isopropyl-CC-CyclohexeneOAc Cyclized Minor SnCl4 EXPNO 10 PROCNO 00 1 \sim 2266666777777777733333332222333333 F2 - Acquisition Parameters Date_ 20130410 r r INSTRUM spect PROBHD 5 mm PABBO BB/ PULPROG zgesgp 32768 SOLVENT CDC13 NS 16 DS SWH 11029.412 Hz FIDRES 0.336591 Hz 1.4854827 sec AQ RG 203.82 DW DE 45.333 usec 6.50 usec TE 294.0 K OMe 1.00000000 sec D1 D12 0.00002000 sec MeO D16 0.00020000 sec TD0 == CHANNEL f1 ======= SF01 500.1300000 MHz NUC1 14.80 usec P2 29.60 usec` 2000.00 usec PLWO Co2(CO)6 12.23400021 W SPNAM[1] Squa100.1000 0.500 20' SPOFFS1 0 Hz 0.00267970 W ===== GRADIENT CHANNEL ===== GPNAM[1] SMSQ10.100 GPNAM[2] SMSQ10.100 GPZ1 31.00 % GPZ2 11.00 % P16 1000.00 usec F2 - Processing parameters 32768 SF 500.1300076 MHz WDW no SSB 0 LB 0 Hz GB 1.00 9.0 8.5 8.0 7.5 5.0 6.5 6.0 5.5 4.5 4.0 3.5 3.0 2.5 2.0 ppm 2.93 4.00 6.15 (ru) 4.11 6.1 179

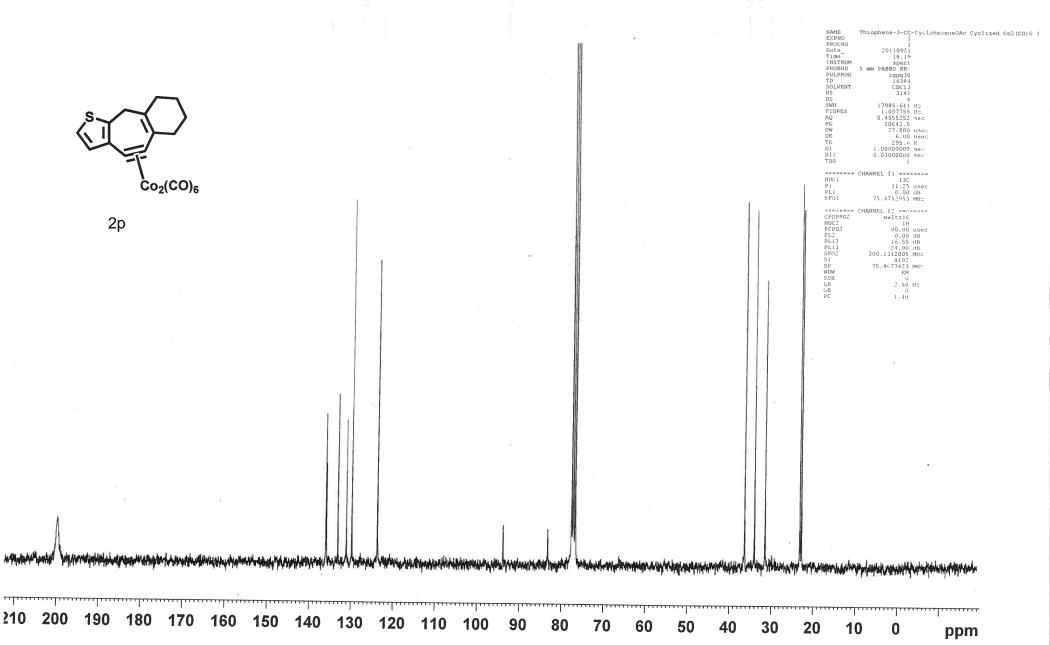
Dimethoxy-1,2-Isopropyl-CC-CyclohexeneOAc Cyclized Minor SnCl4

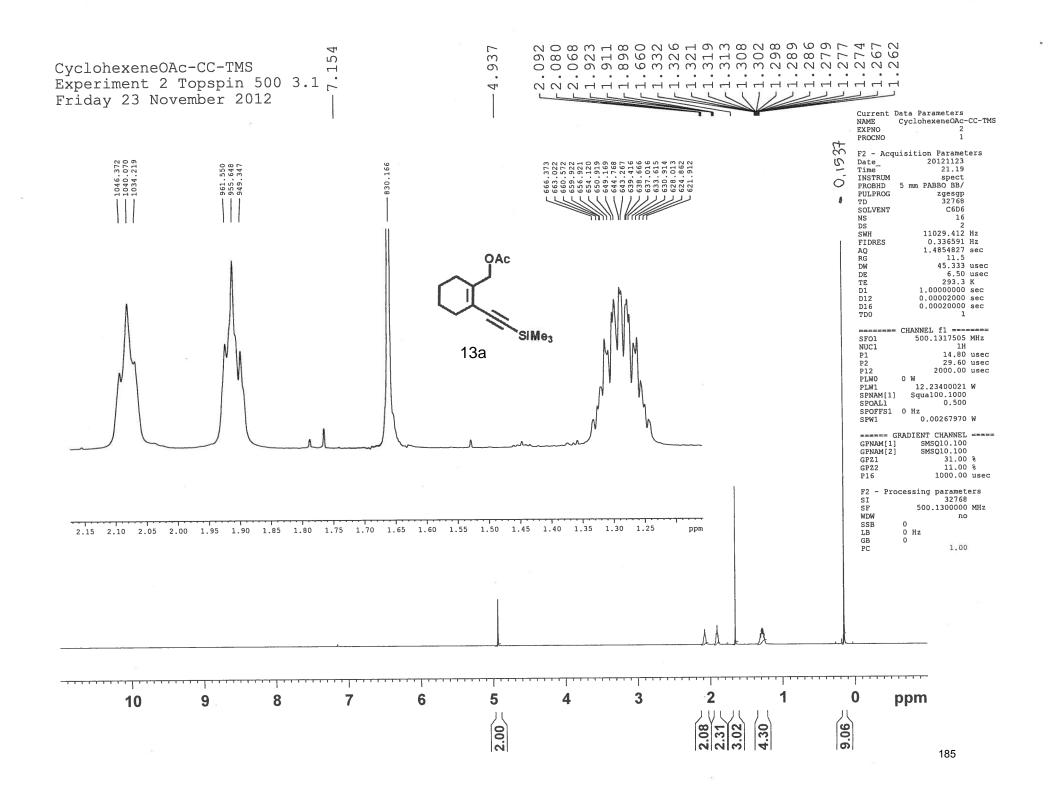


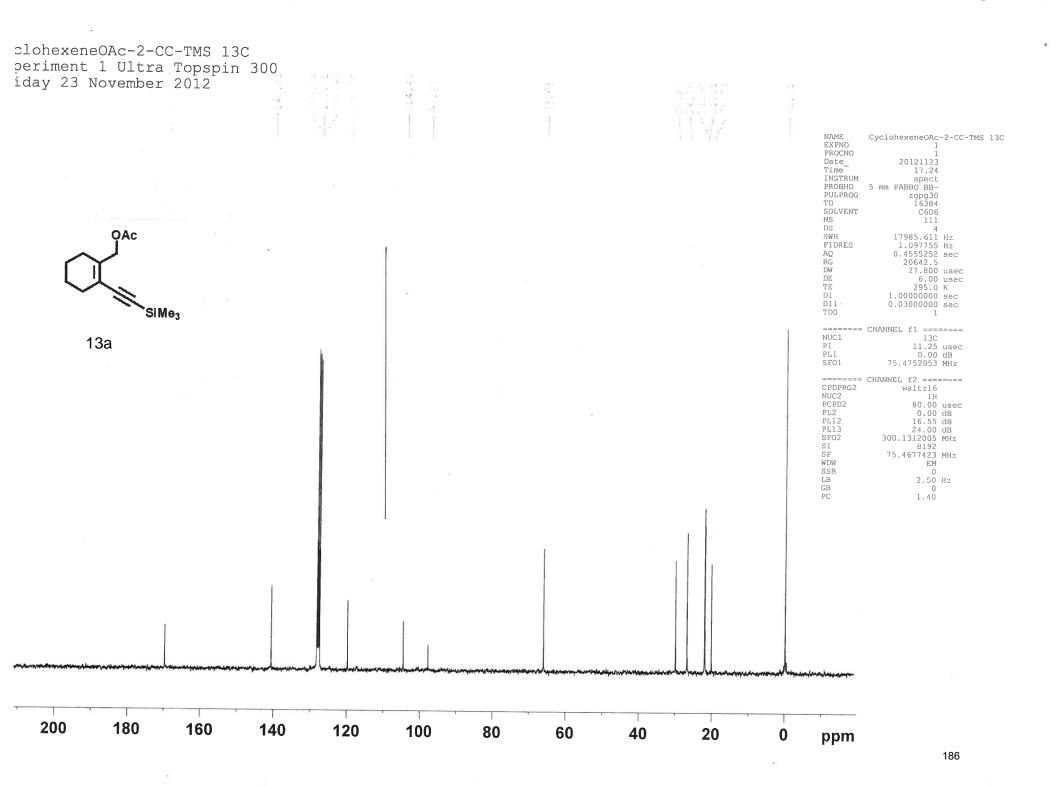


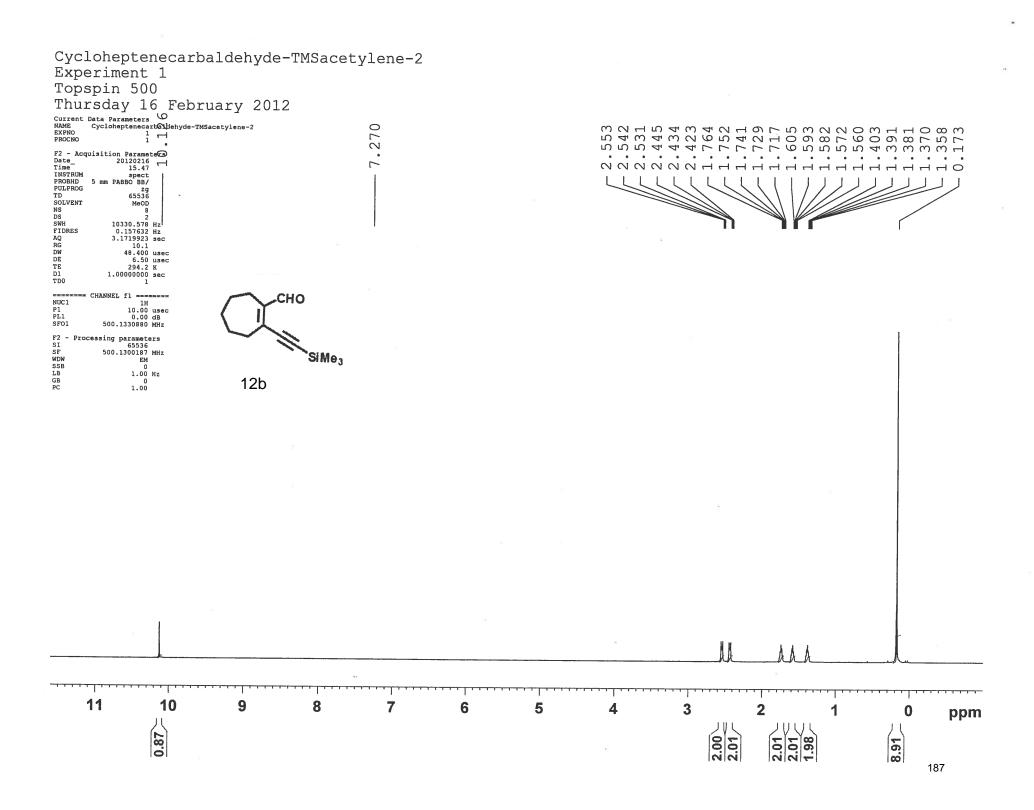


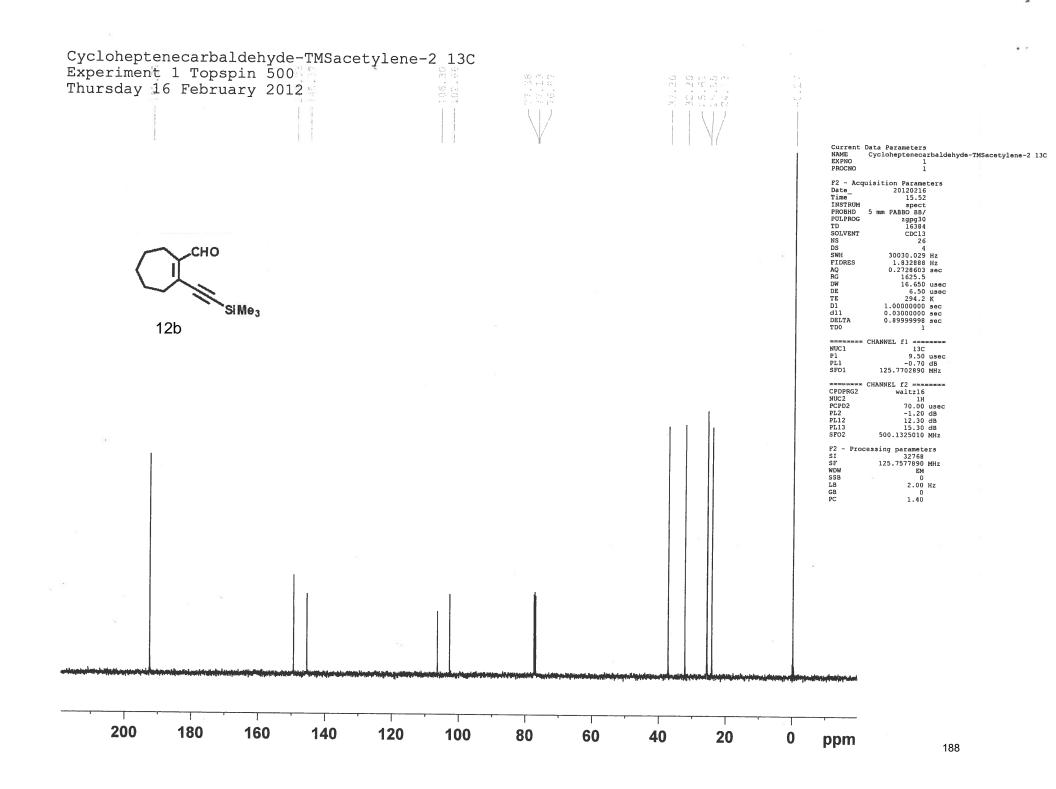


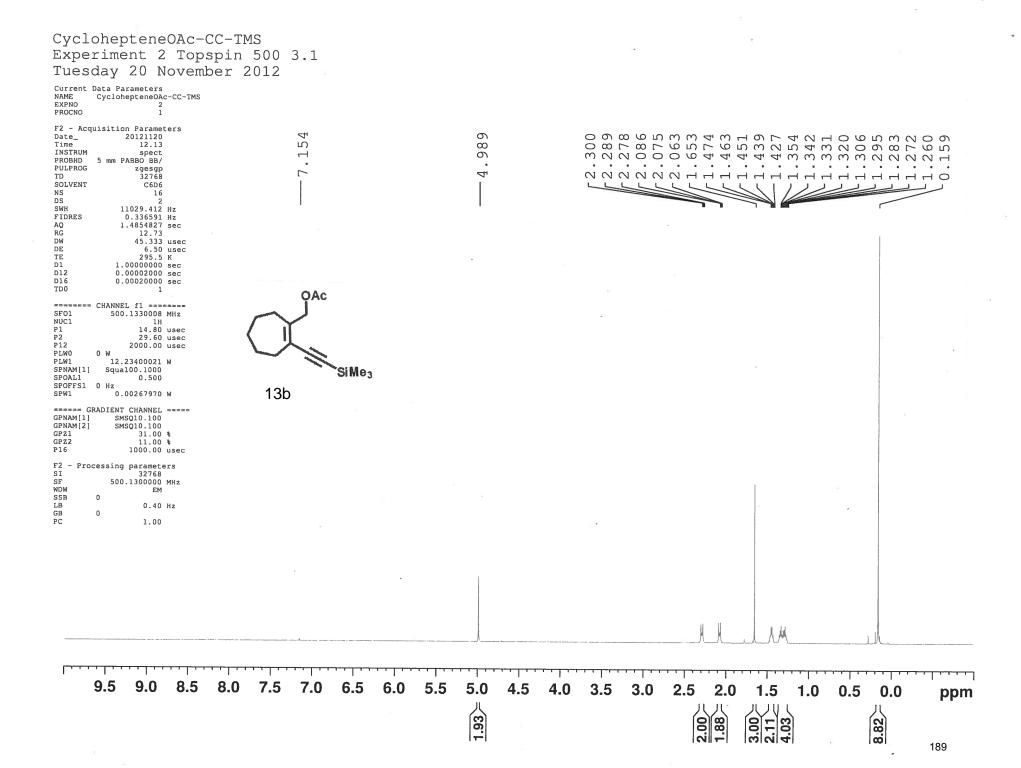


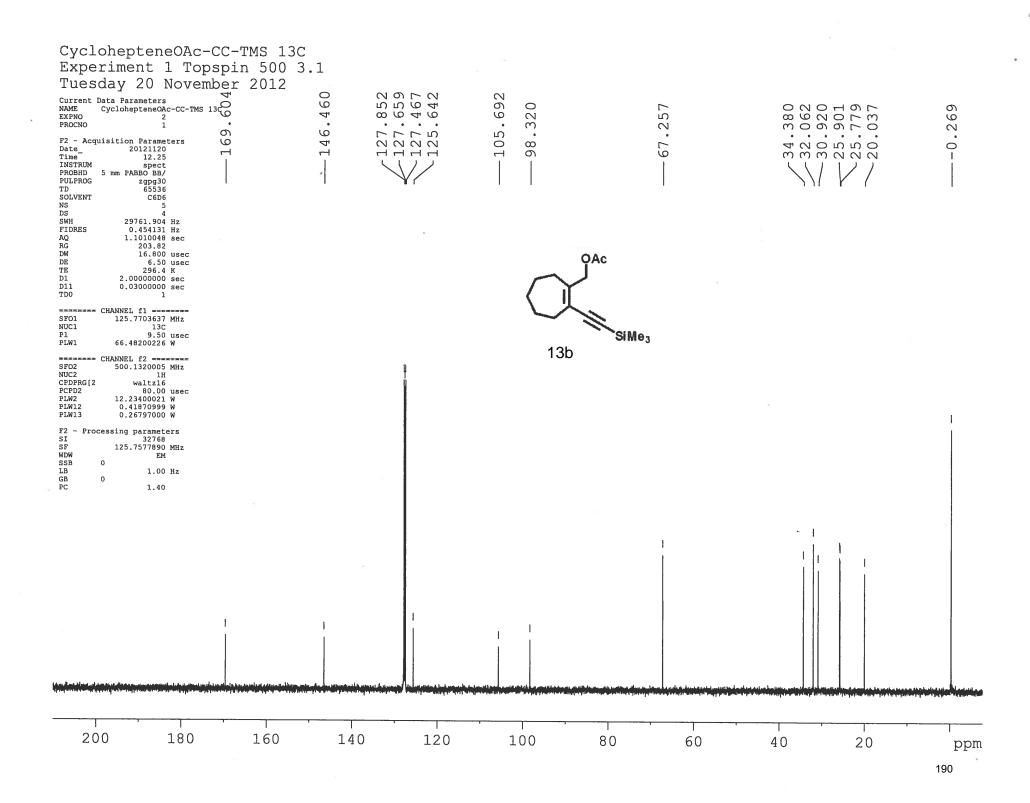


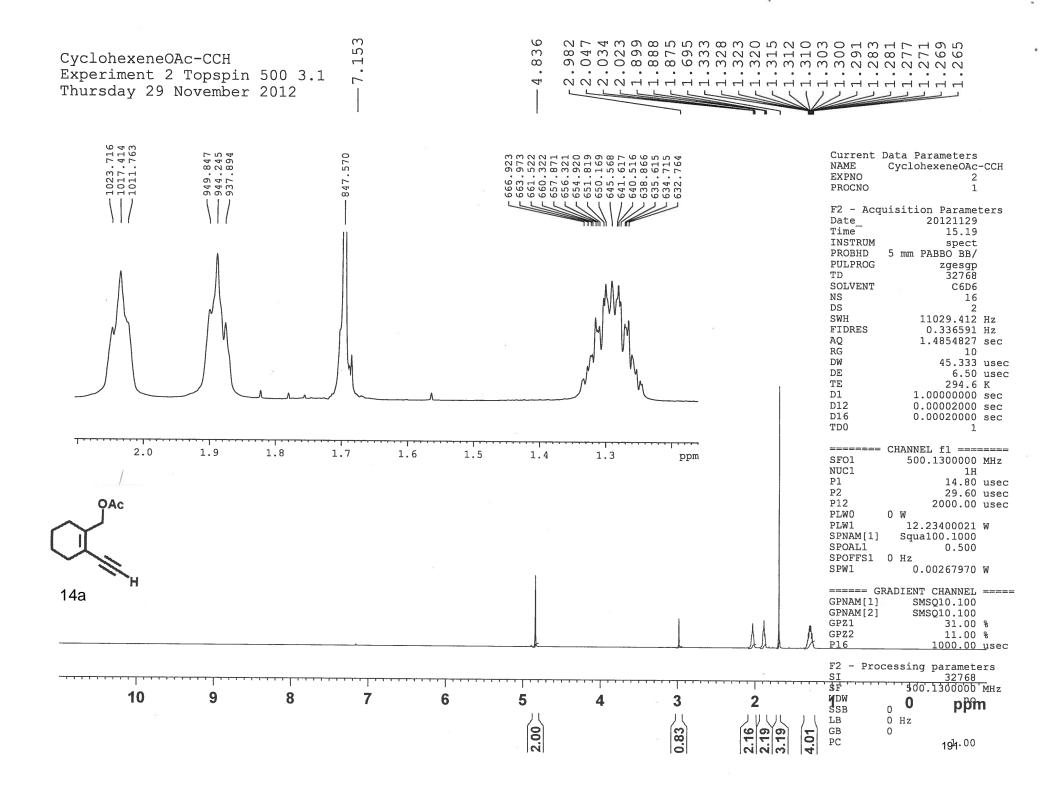








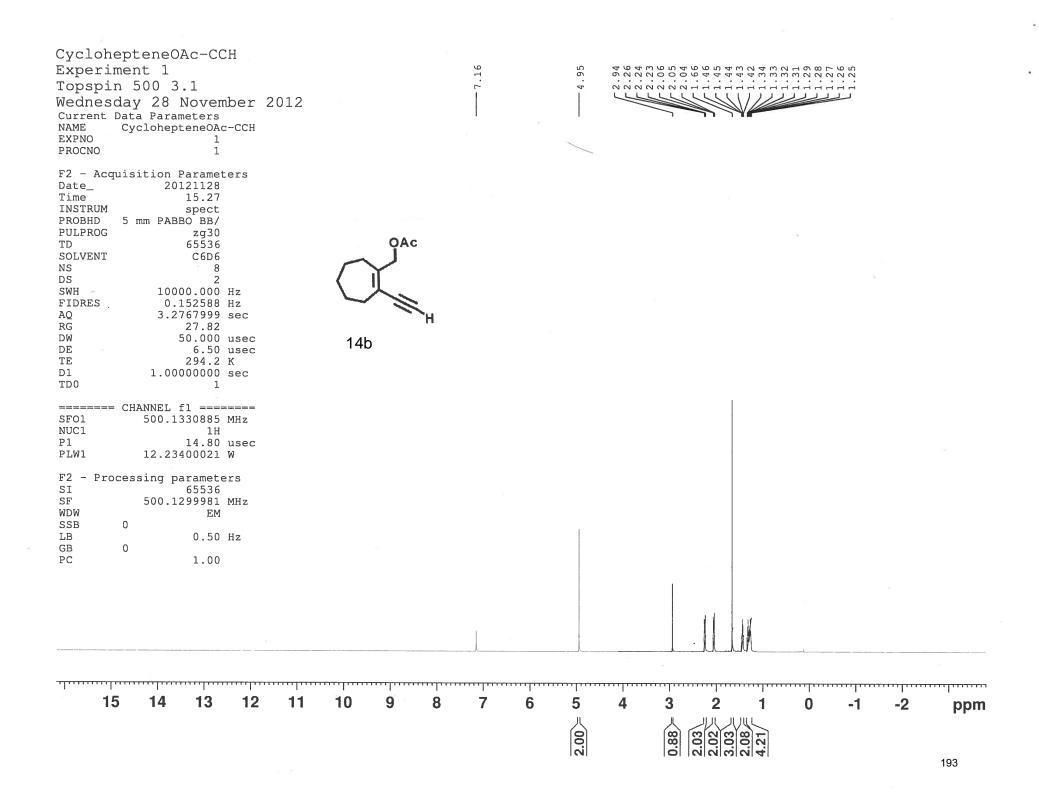




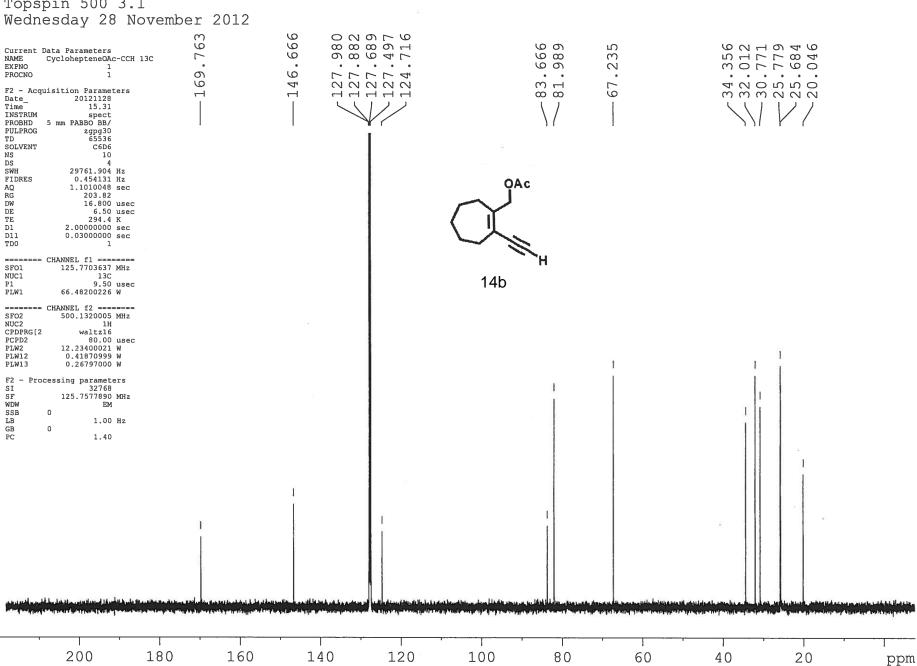
yclohexeneOAc-2-CCH xperiment 1 ra 300 Topspin nursday 29 November 2012 NAME CyclohexeneOAc-2-CCH 13C EXPNO PROCNO 1 Date 20121129 Time Time 16.33 INSTRUM spect 5 mm PABBO BB-PROBHD PULPROG zgpg30 QAc TD 16384 SOLVENT C6D6 NS 16 DS 4 SWH 17985.611 Hz FIDRES 1.097755 Hz ΑQ 0.4555252 sec RG 20642.5 27.800 usec 6.00 usec 14a TE 295.5 к D1 1.00000000 sec D11 0.03000000 sec TD0 1 ====== CHANNEL f1 ====== NUC1 13C P1 11.25 usec 0.00 dB SFO1 75.4752953 MHz ====== CHANNEL f2 ====== CPDPRG2 waltz16 NUC2 1H PCPD2 80.00 usec PL2 0.00 dB PL12 16.55 dB PL13 24.00 dB SFO2 300.1312005 MHz SI 8192 SF 75.4677423 MHz WDW EM SSB 0 LB 2.50 Hz GB 0 PC 1.40 200 180 160 140 120 100 80 60 40 20 0

ppm

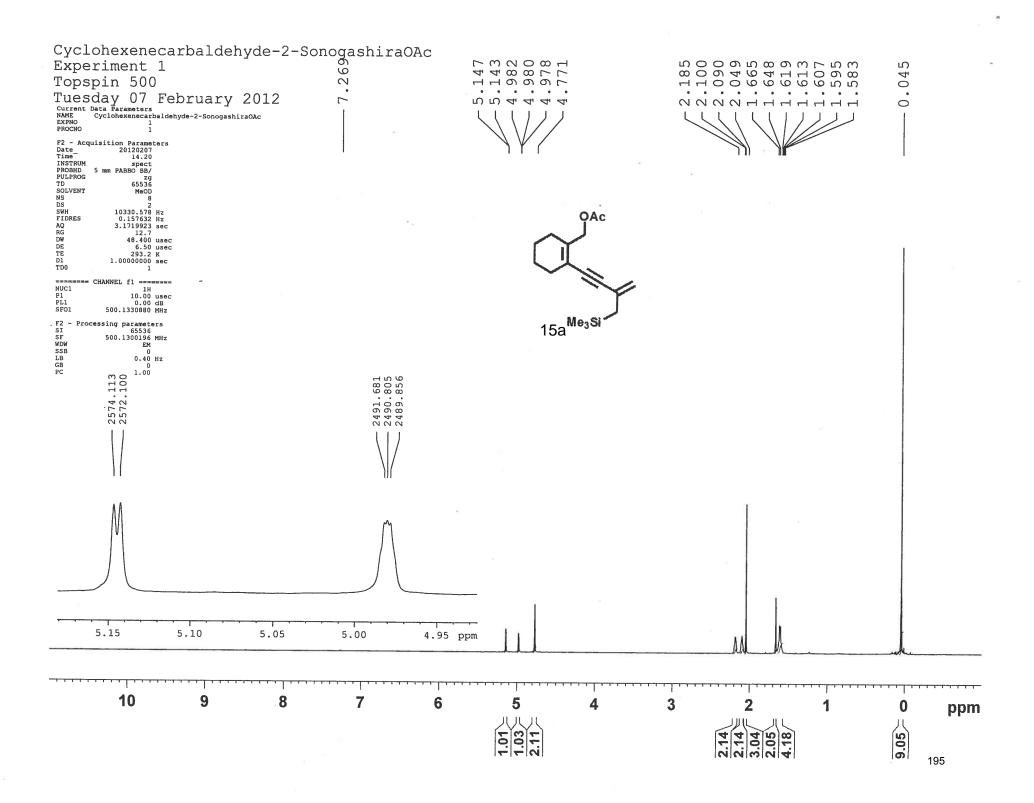
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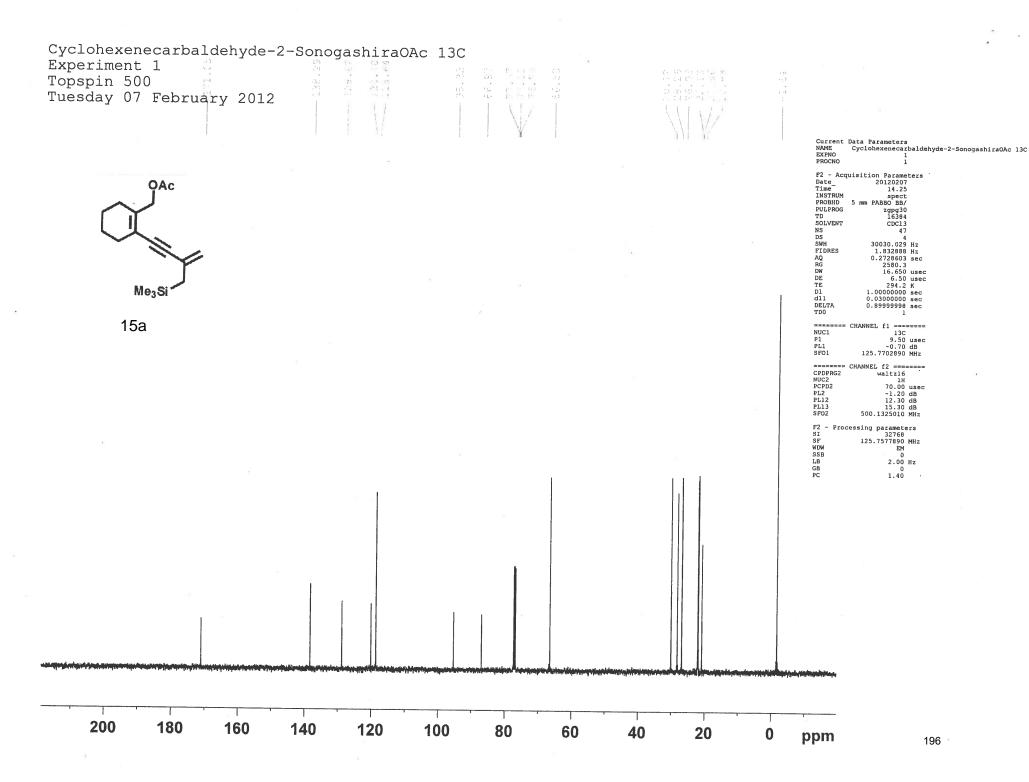


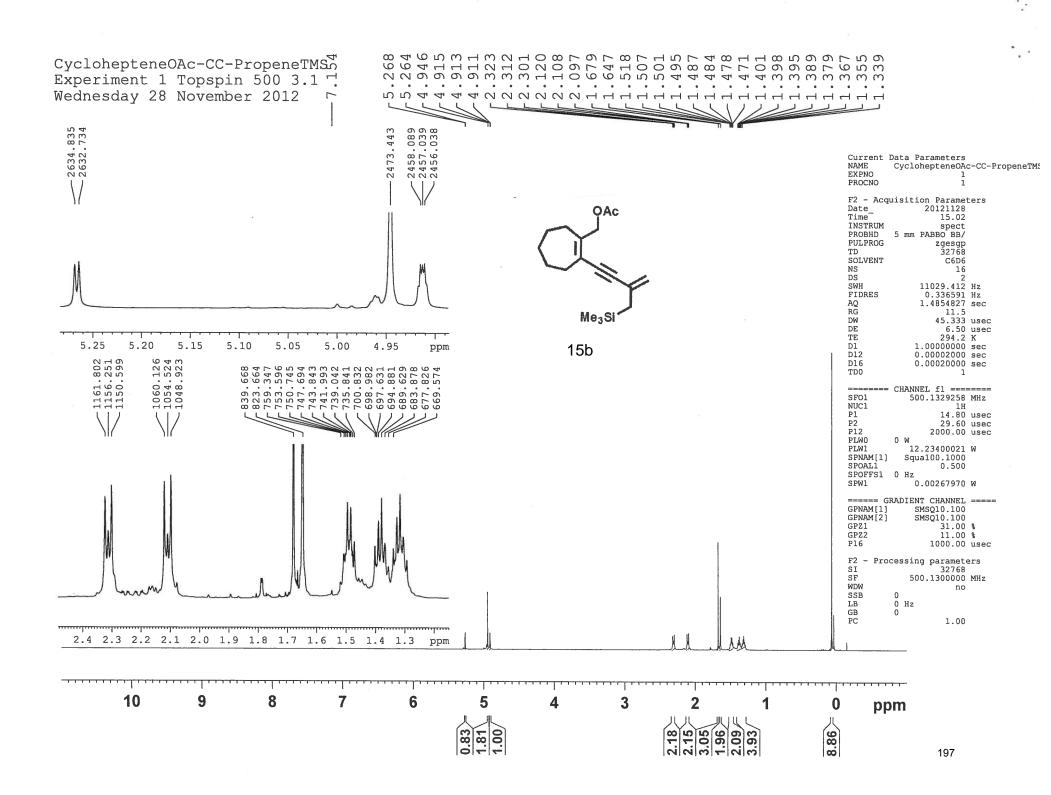
CyclohepteneOAc-CCH 13C Experiment 1 Topspin 500 3.1

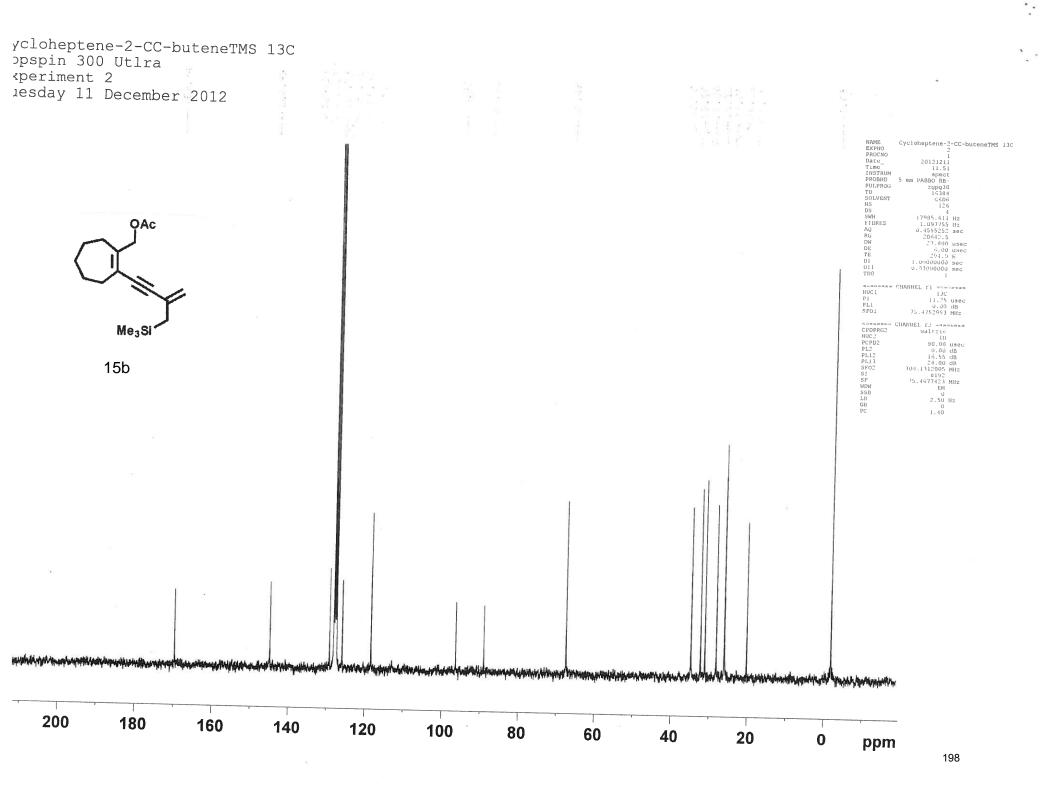


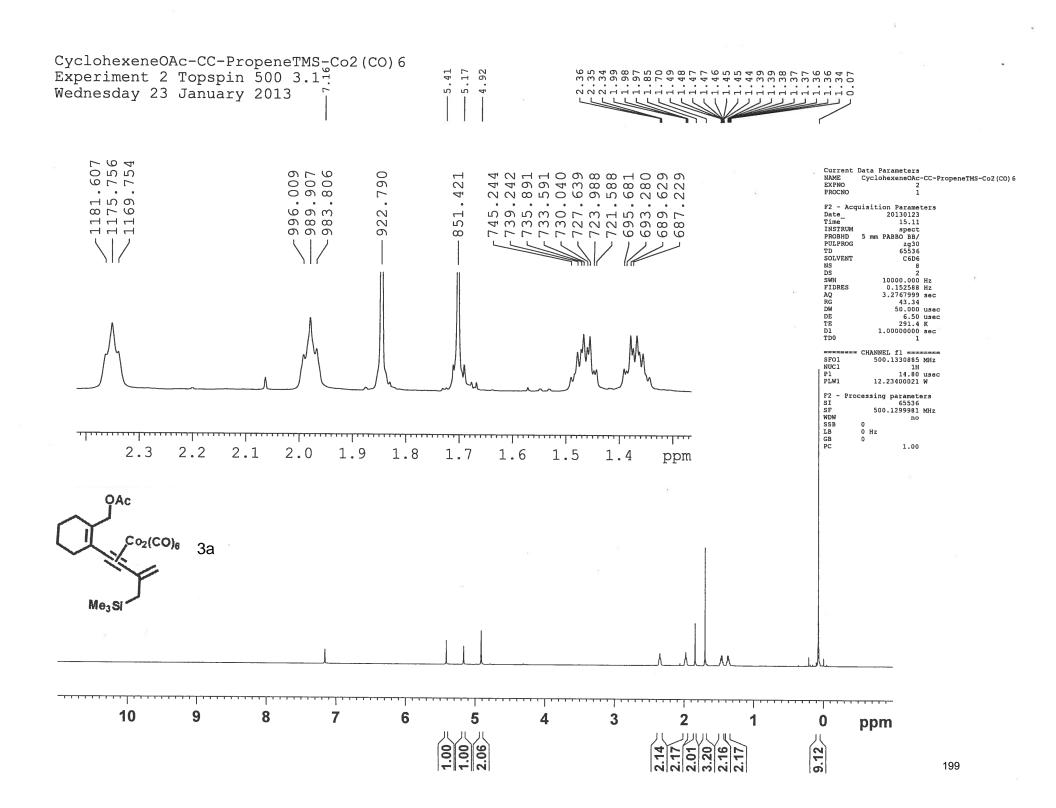
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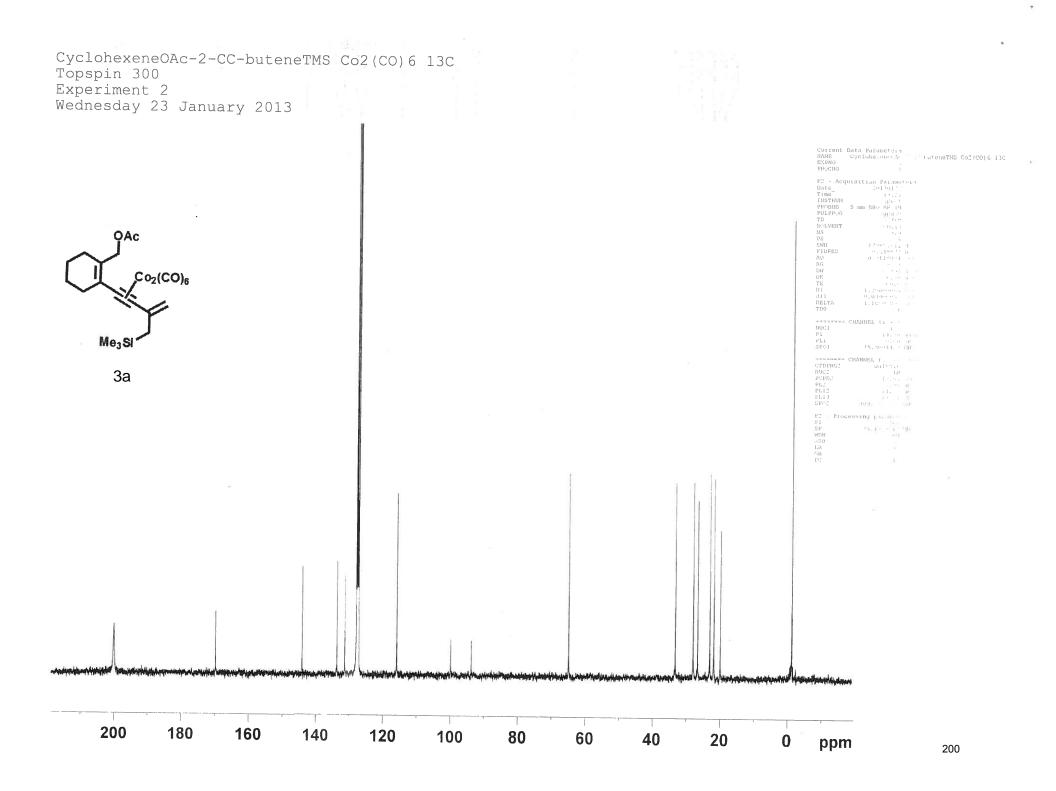


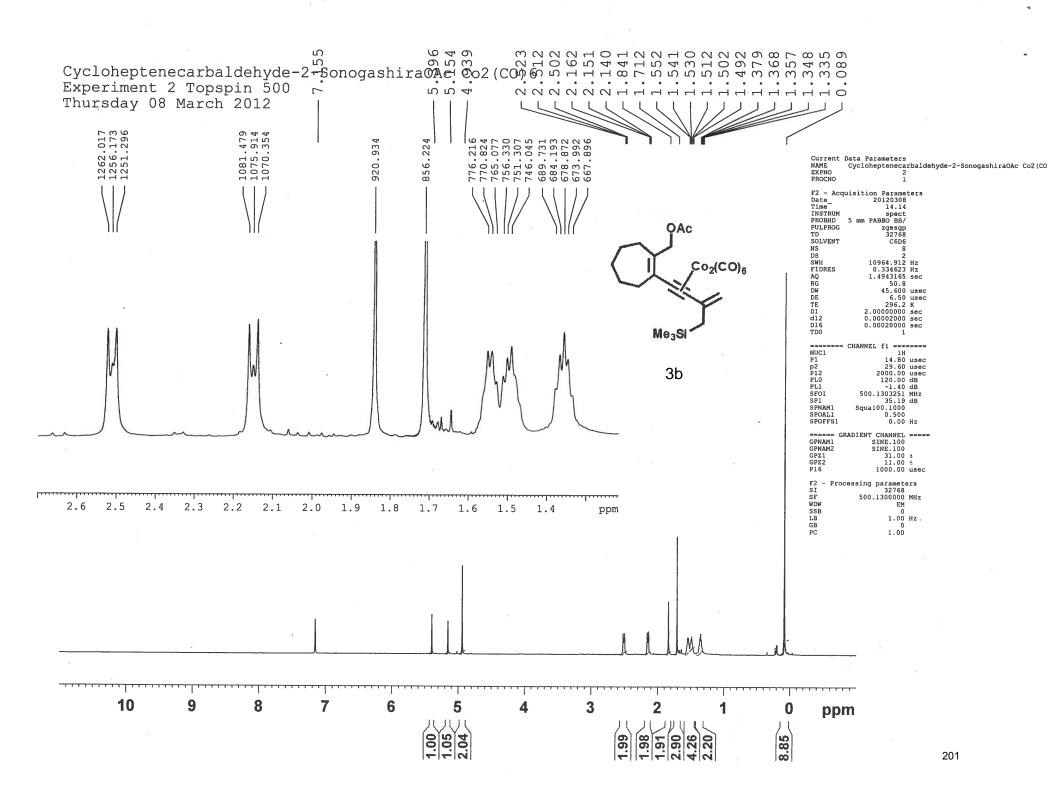


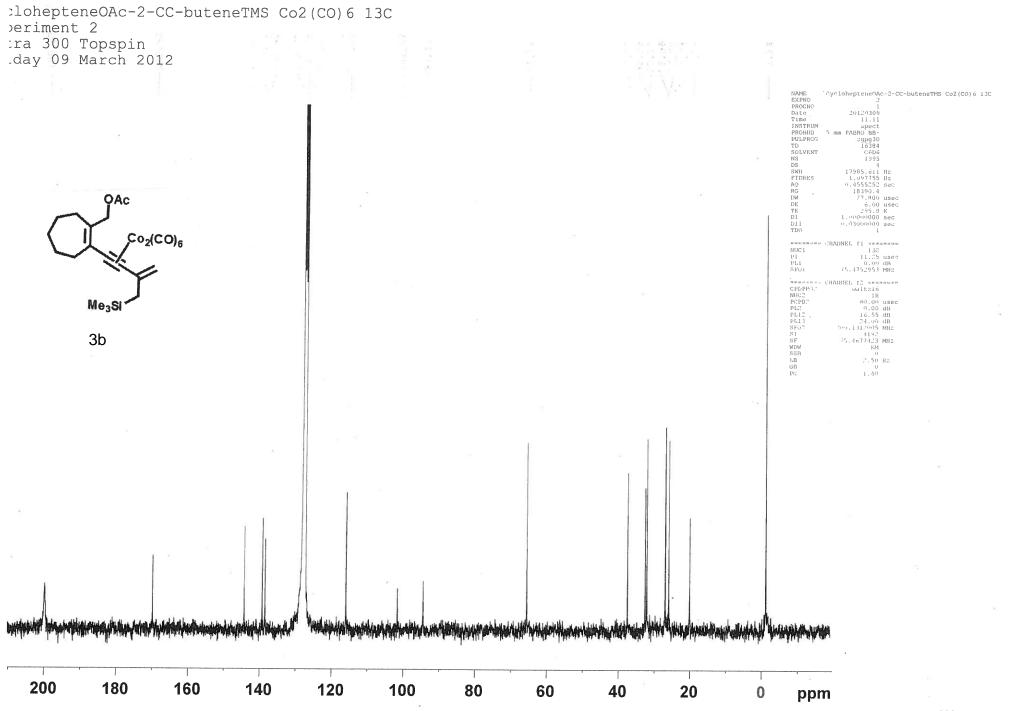




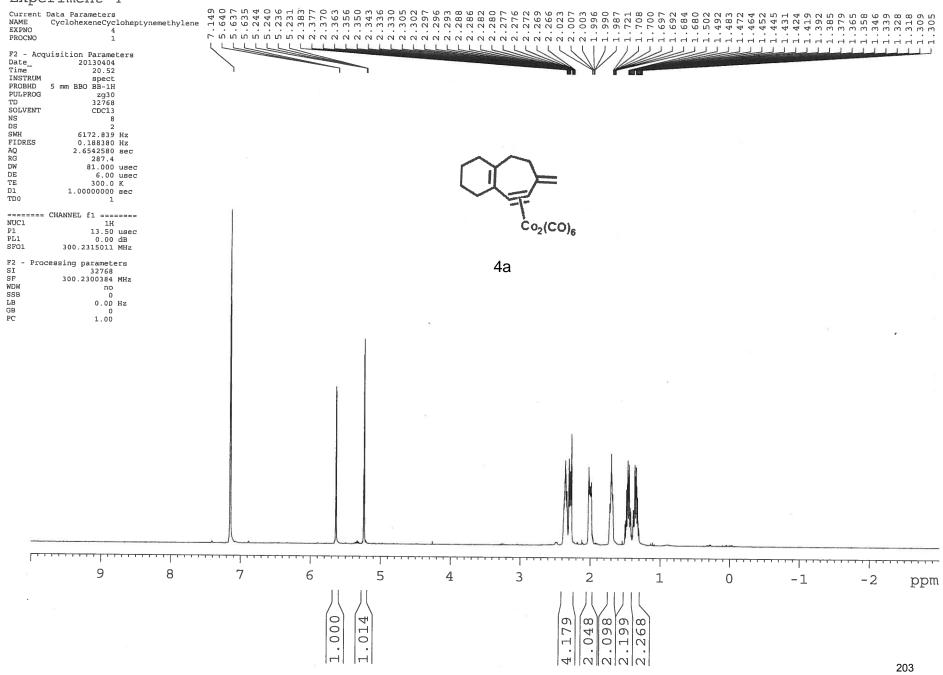


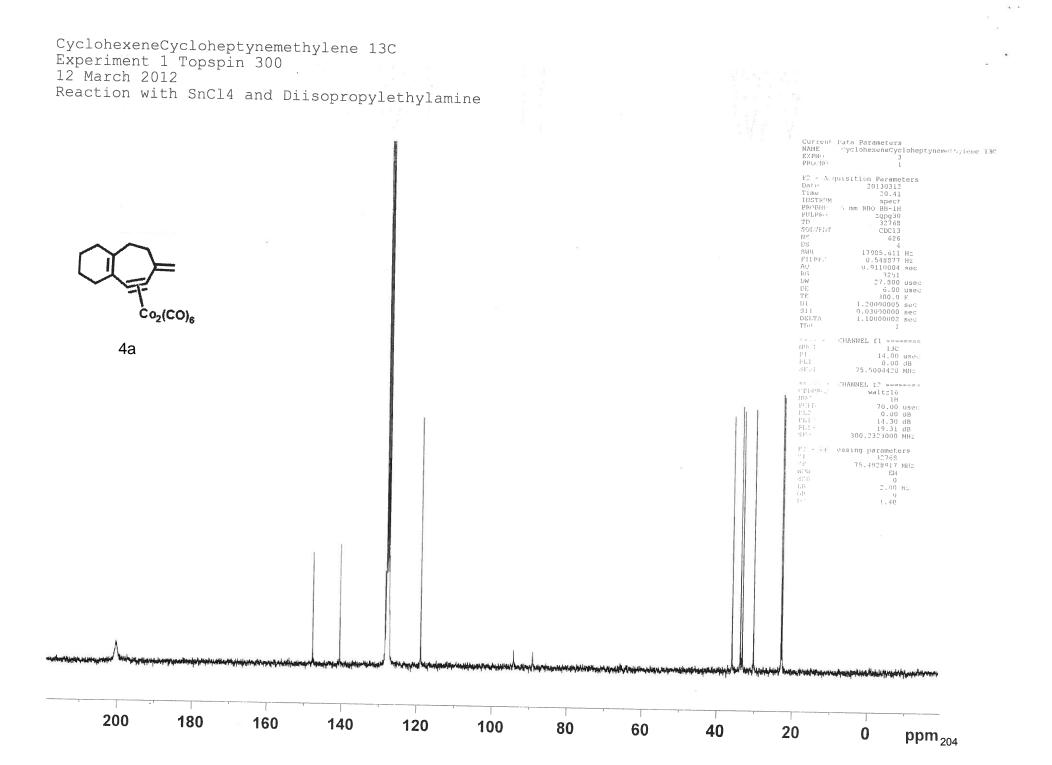


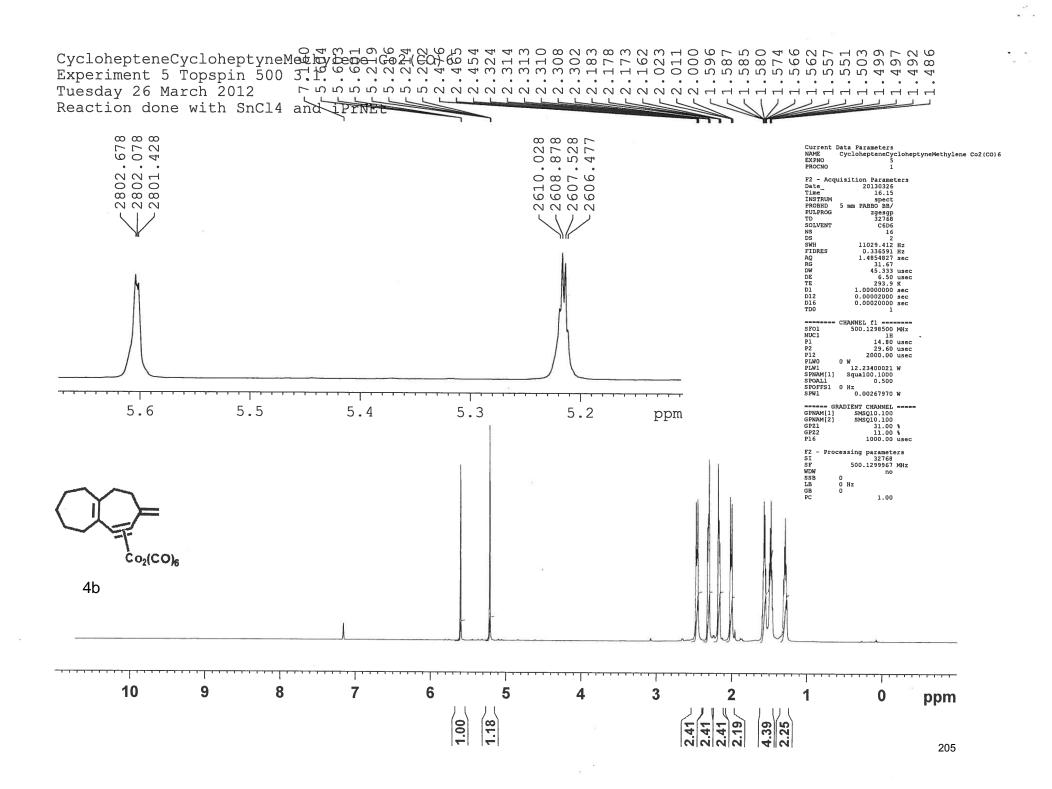


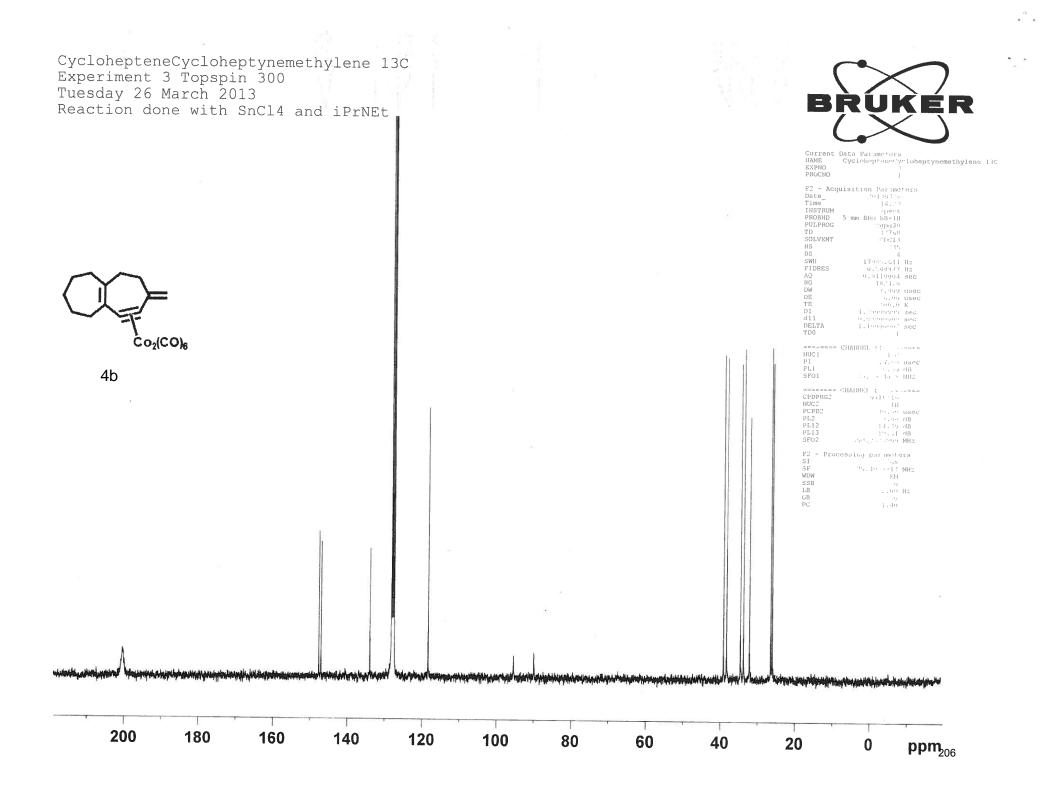


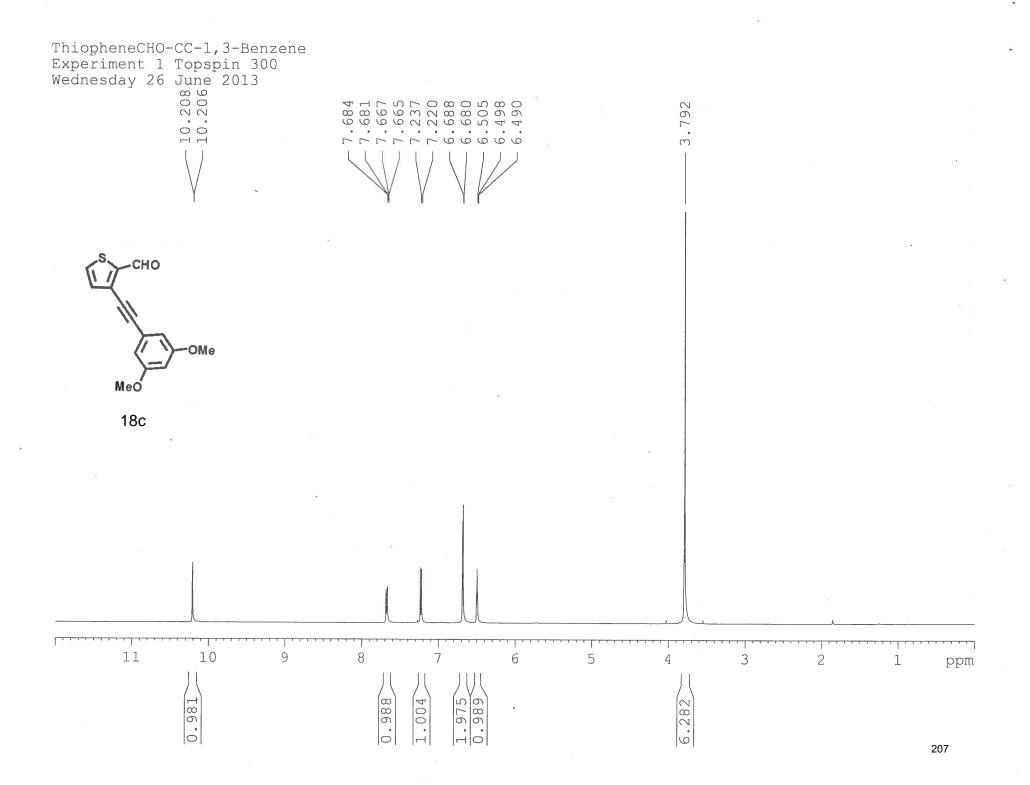
CyclohexeneCycloheptynemethylene Thursday 04 April 2013 Topspin 300 Experiment 4

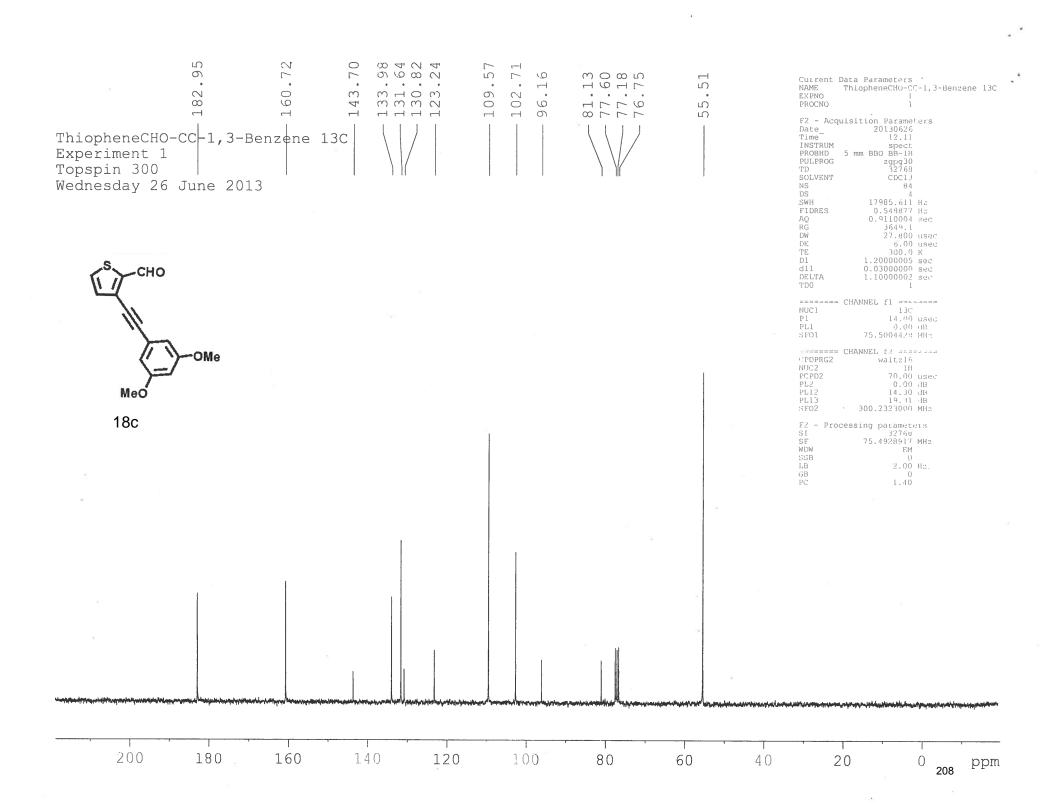


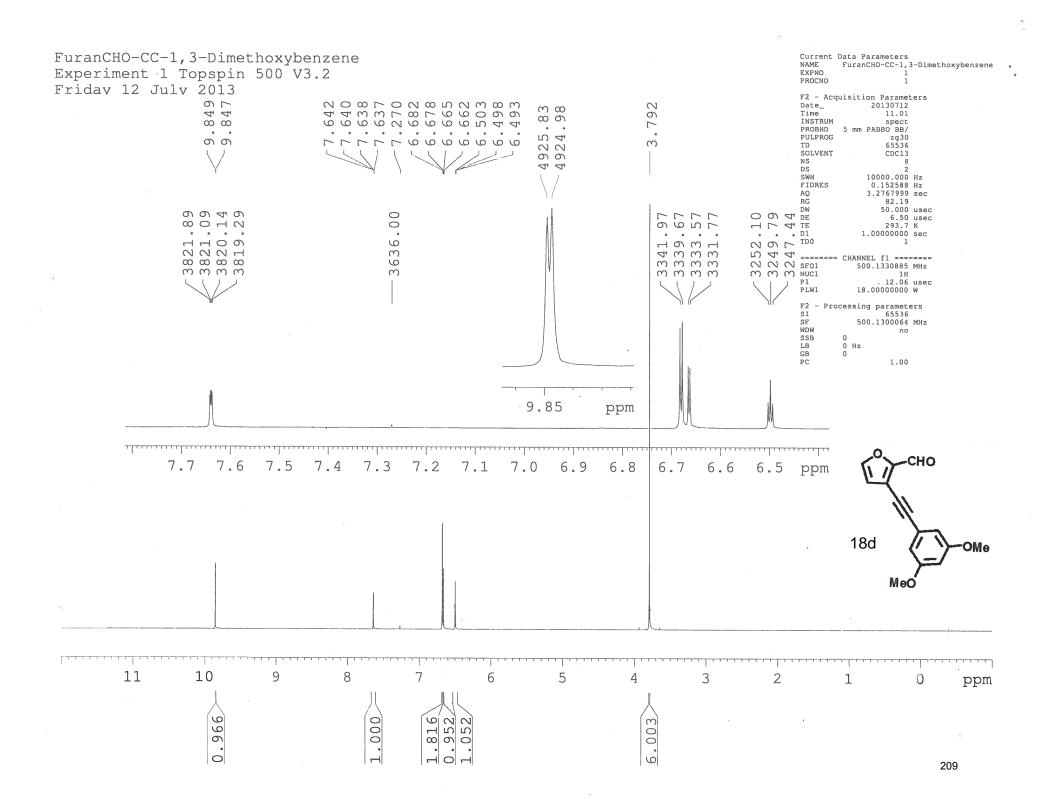


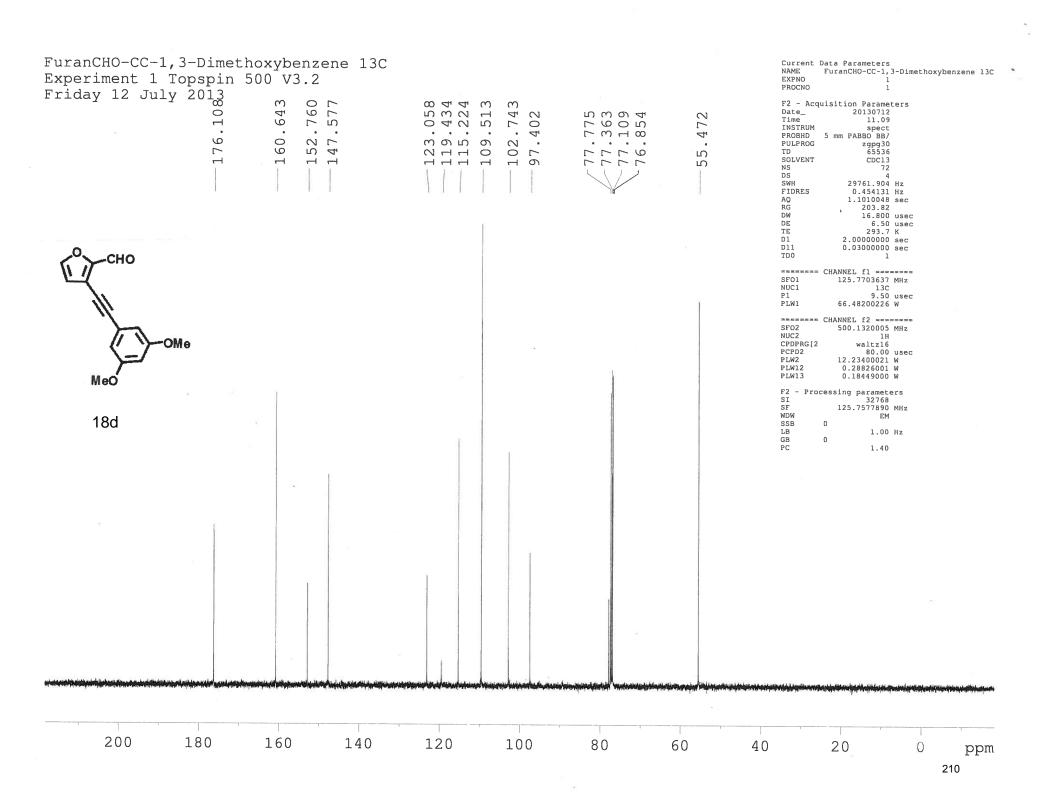


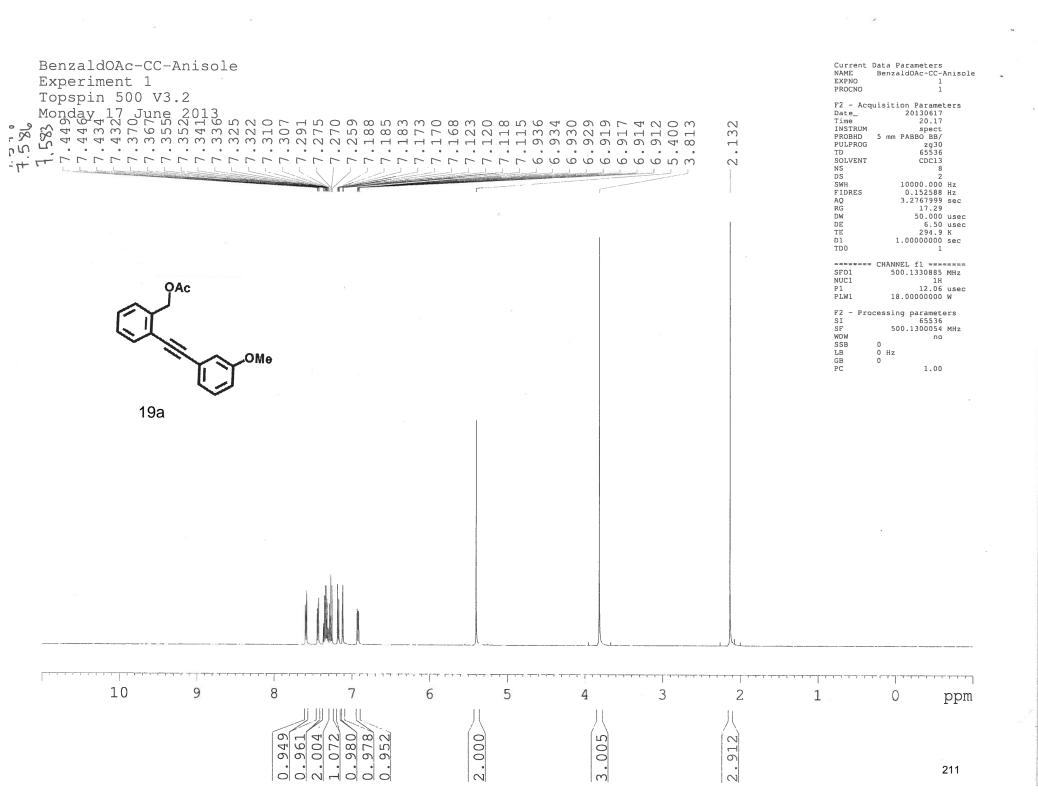


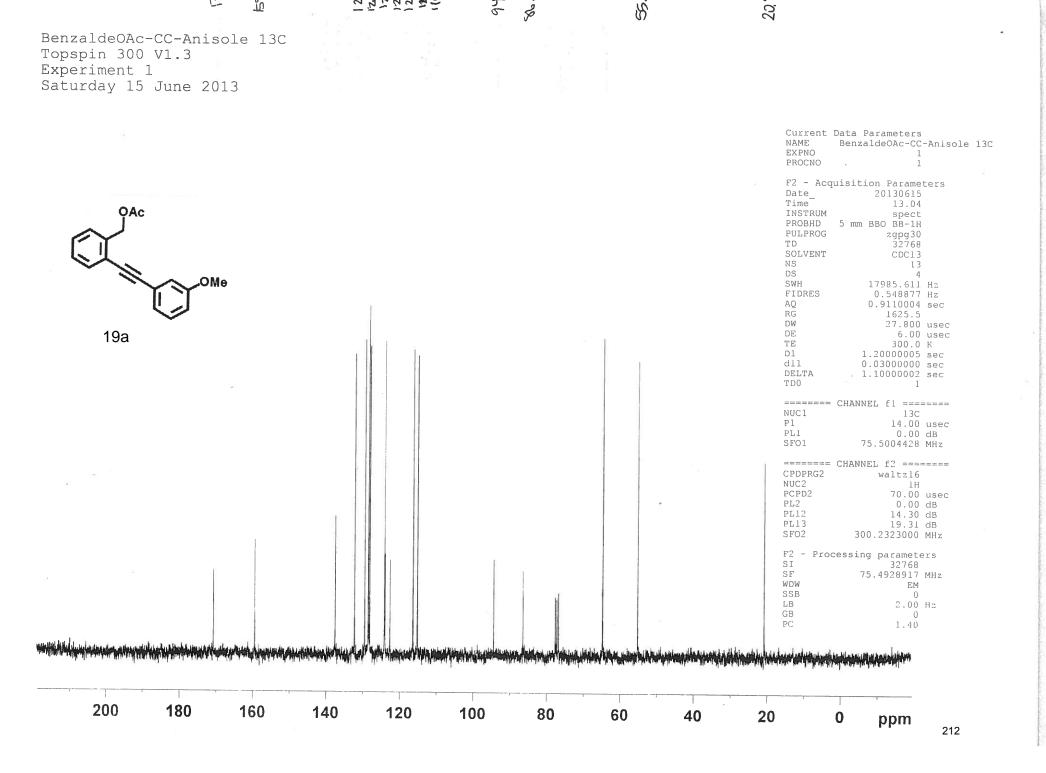


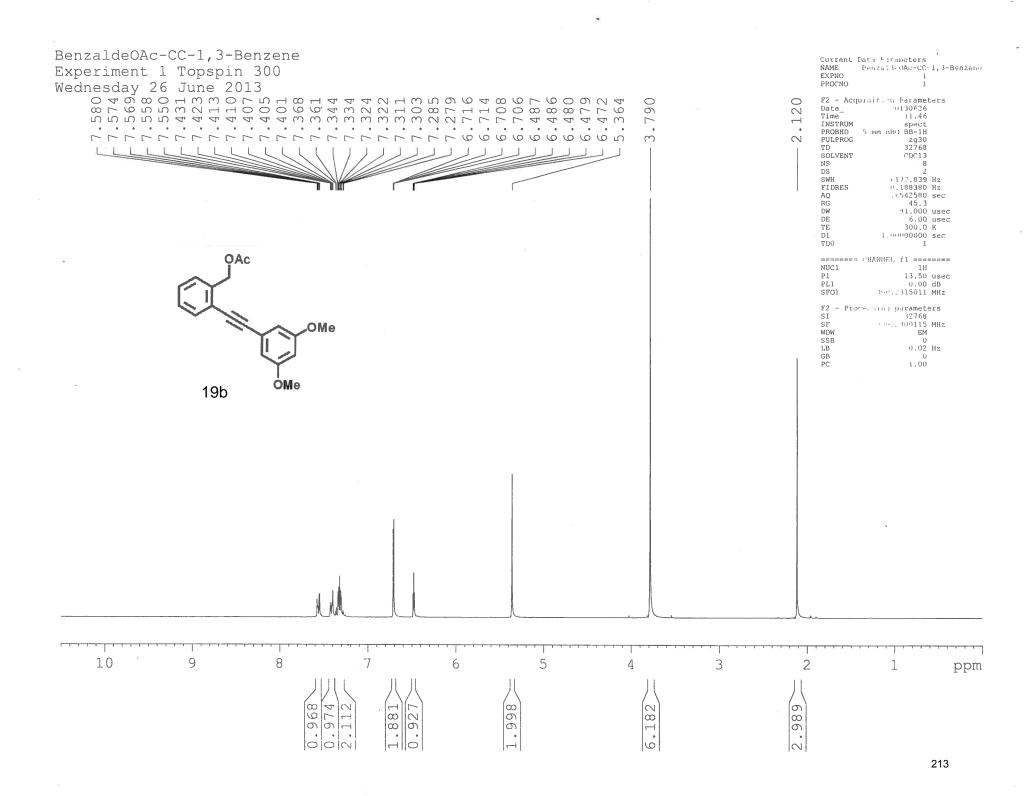


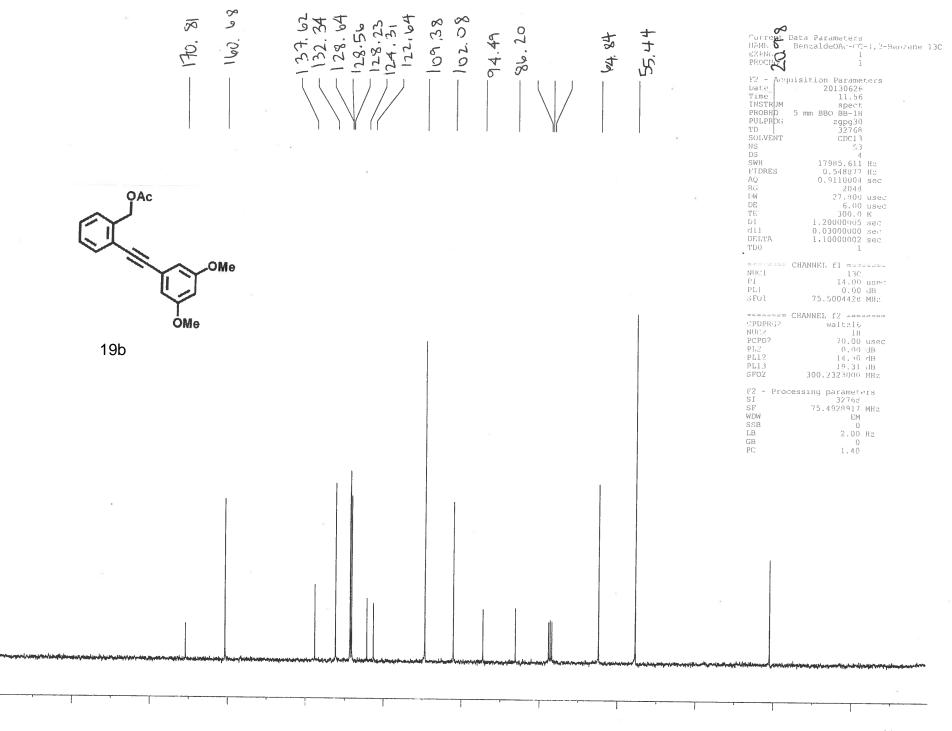


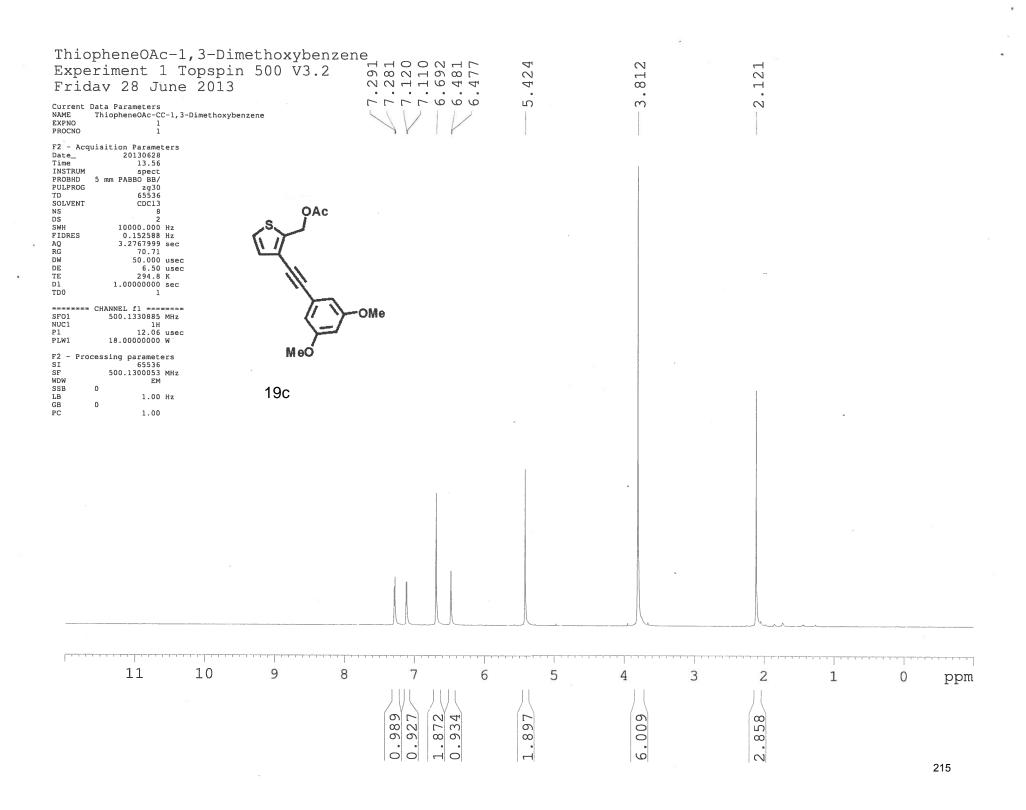


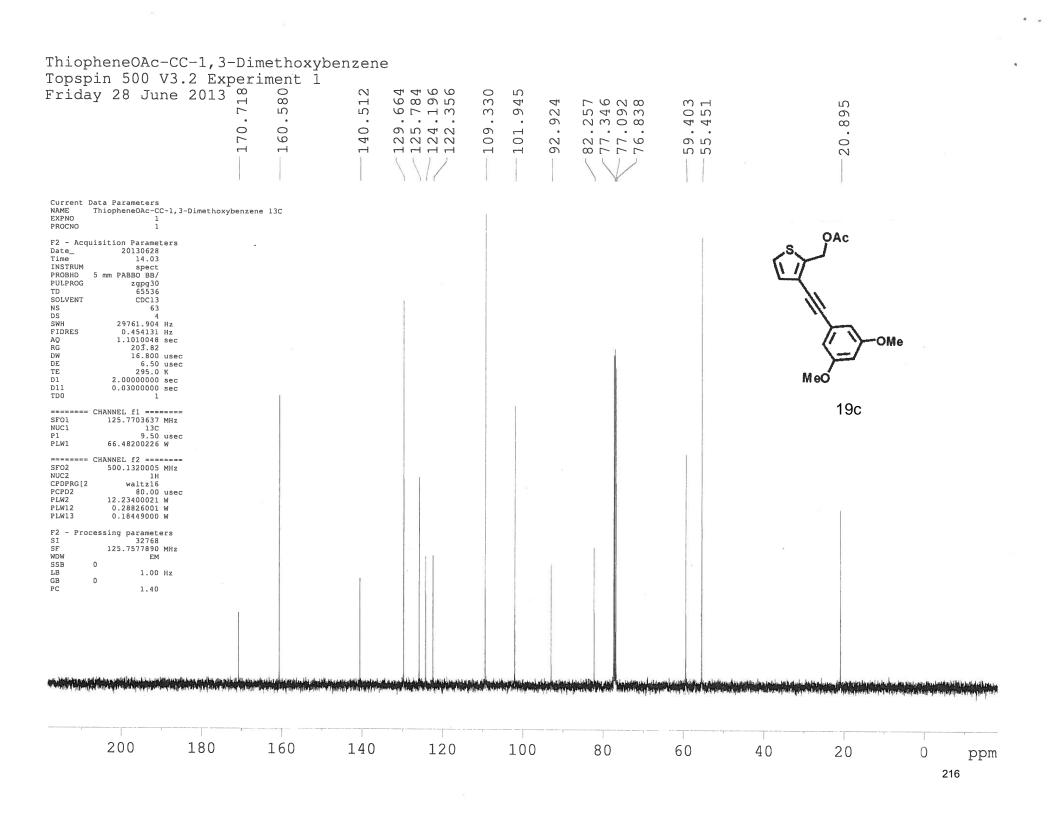


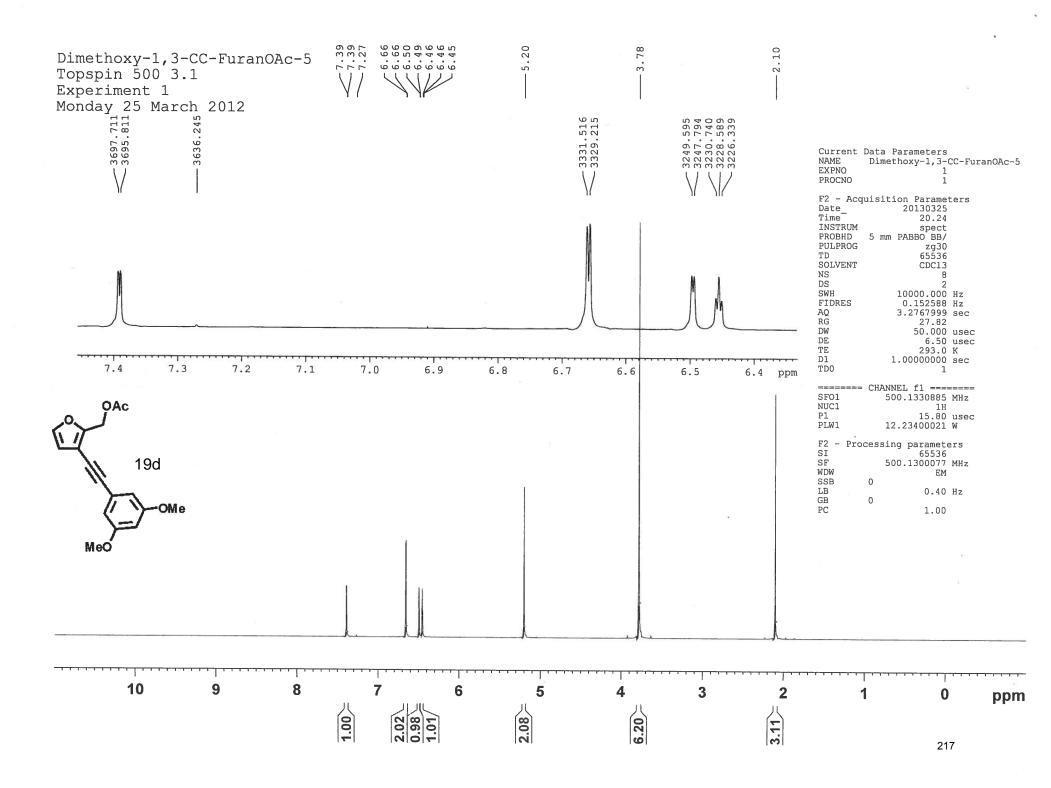


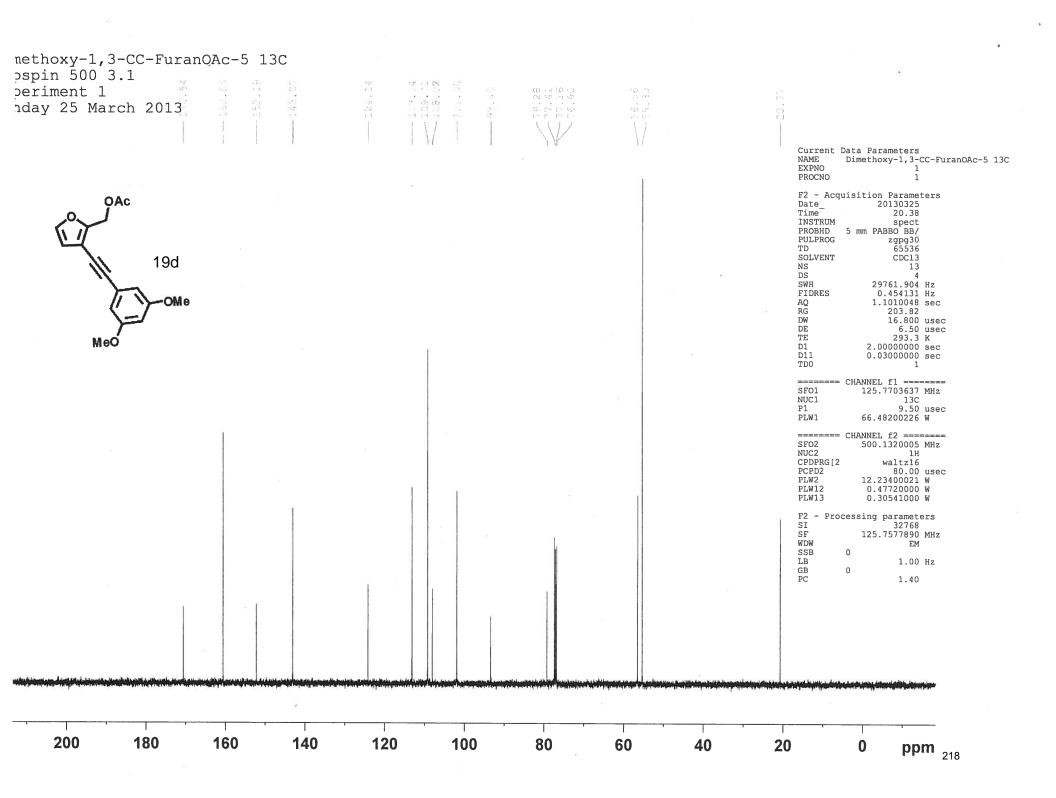


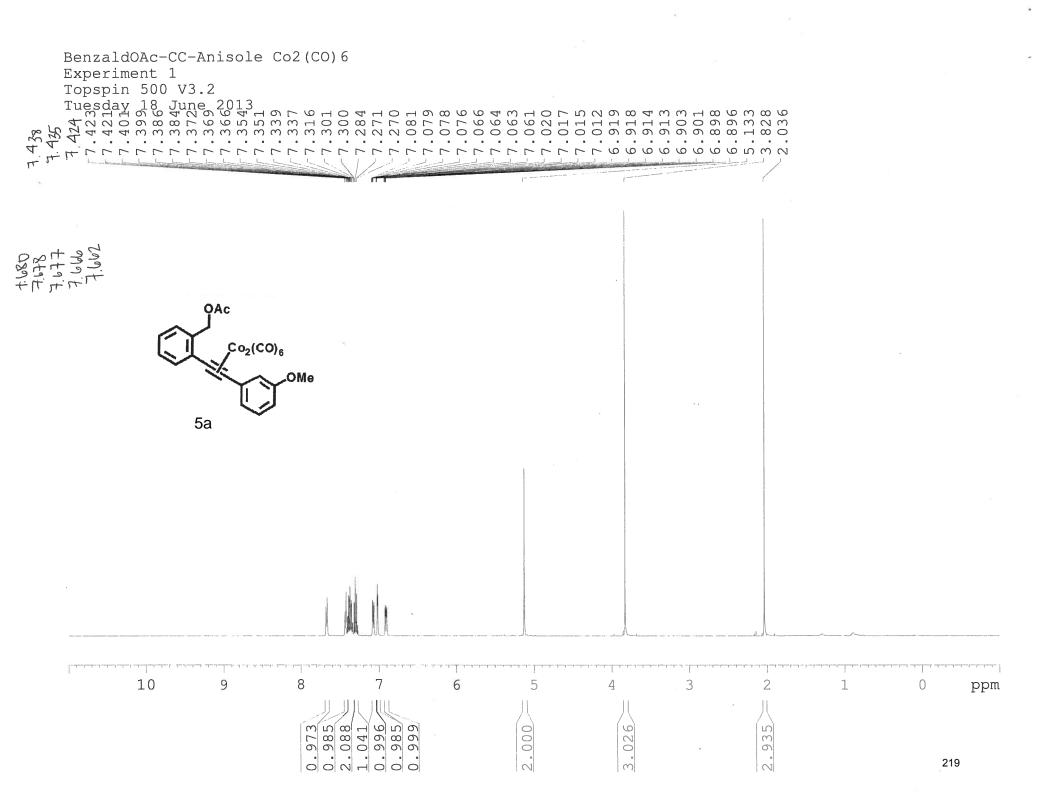


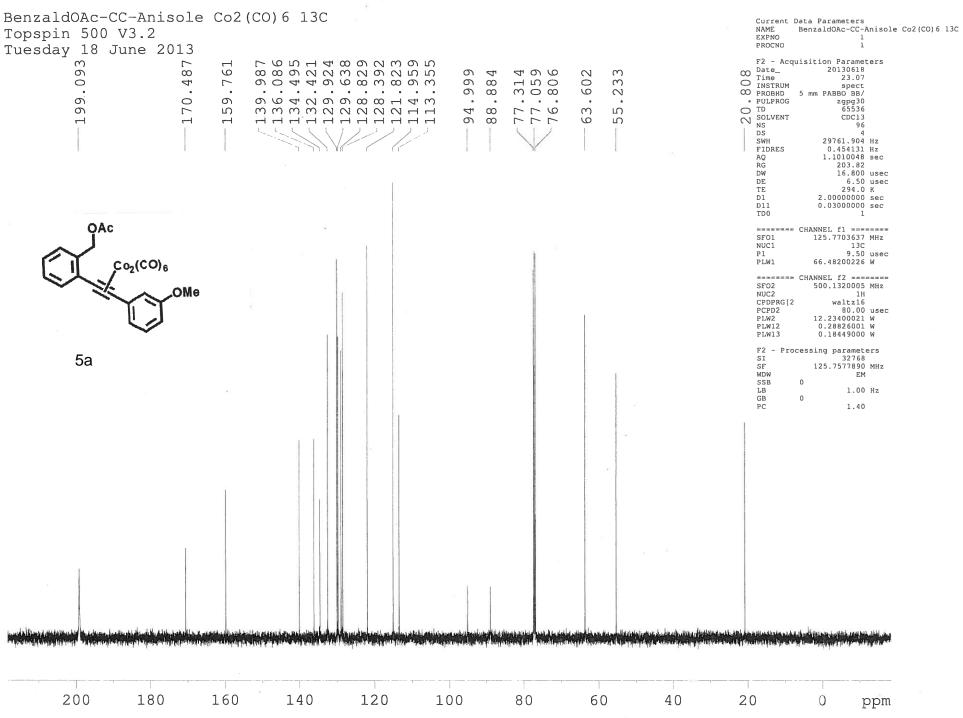


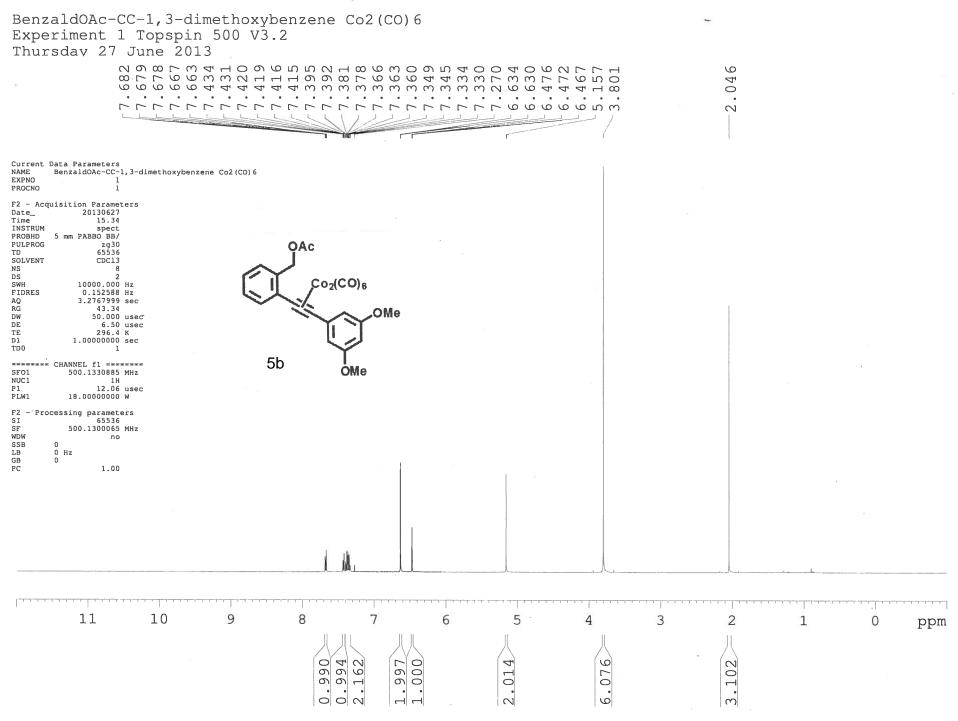


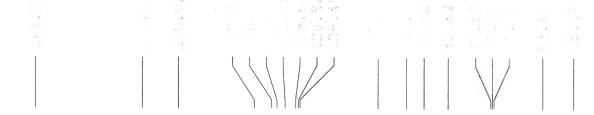




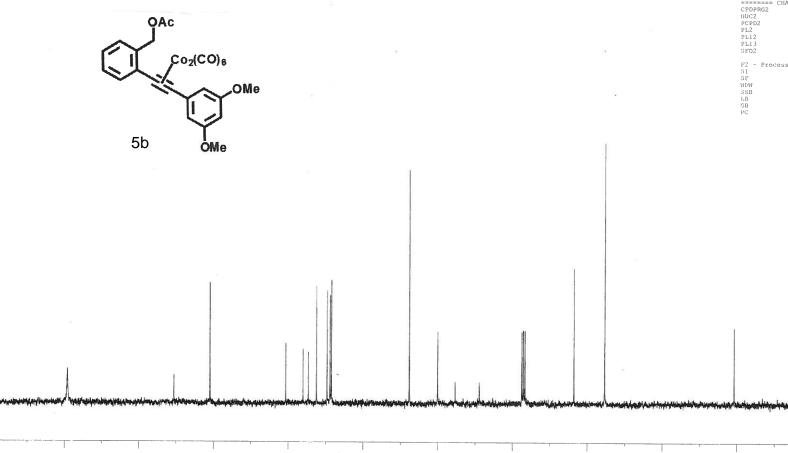


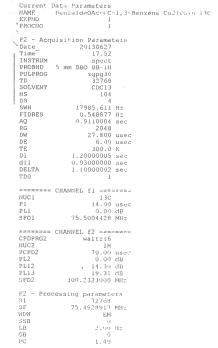


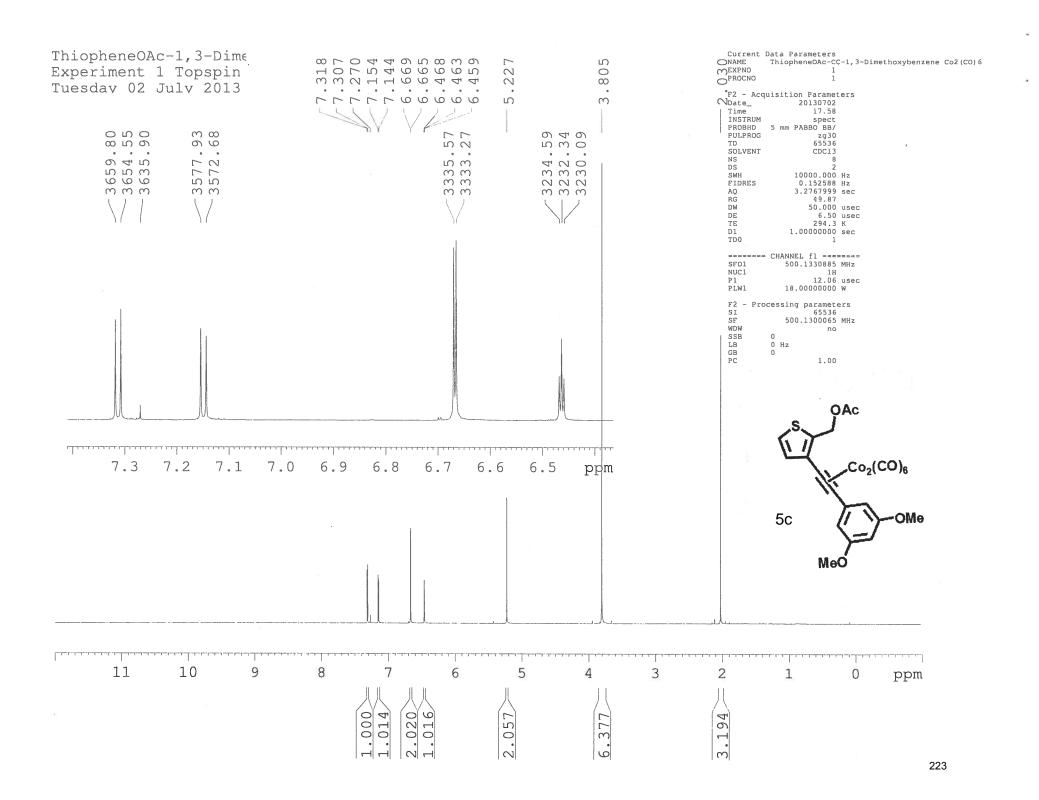


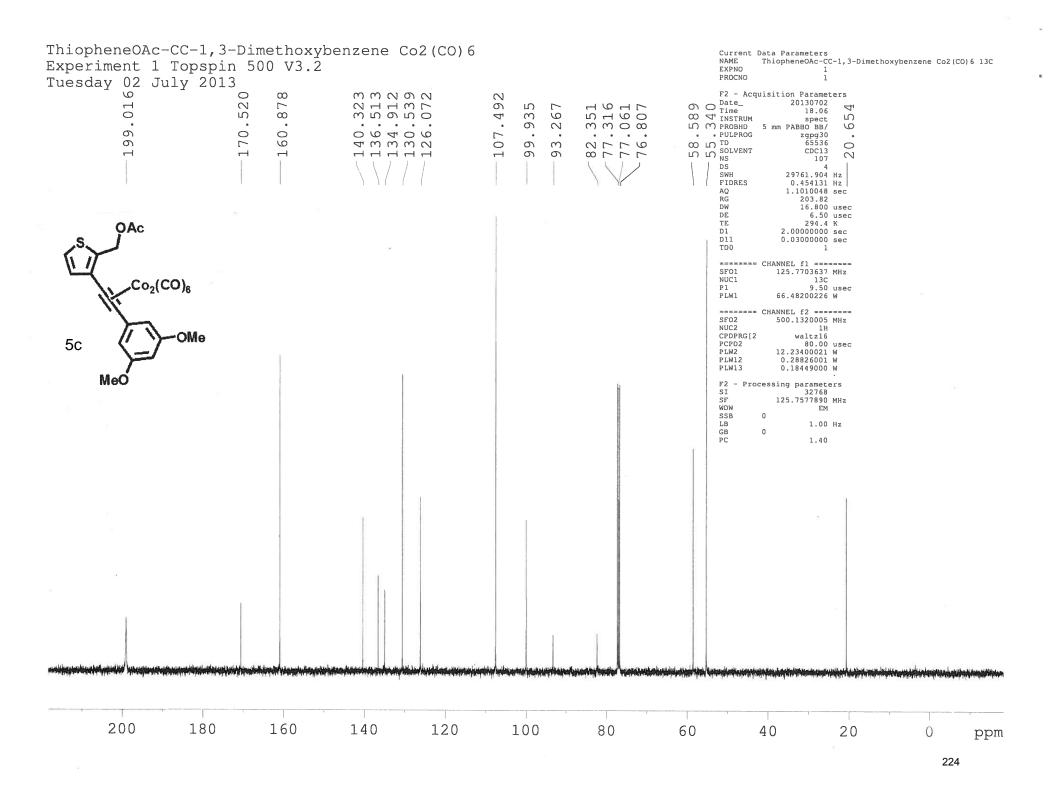


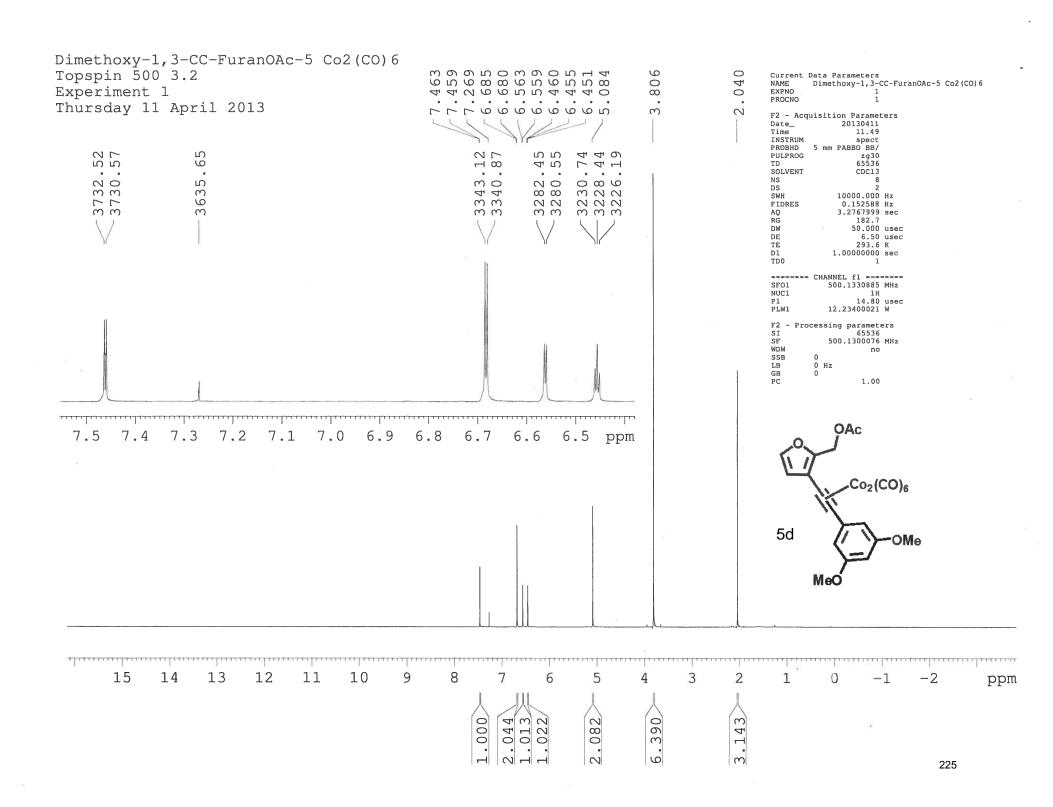
BenzaldeOAc-CC-1,3-Benzene Co2(CO)6 13C Topspin 300 Experiment 1 Thursday 27 June 2013

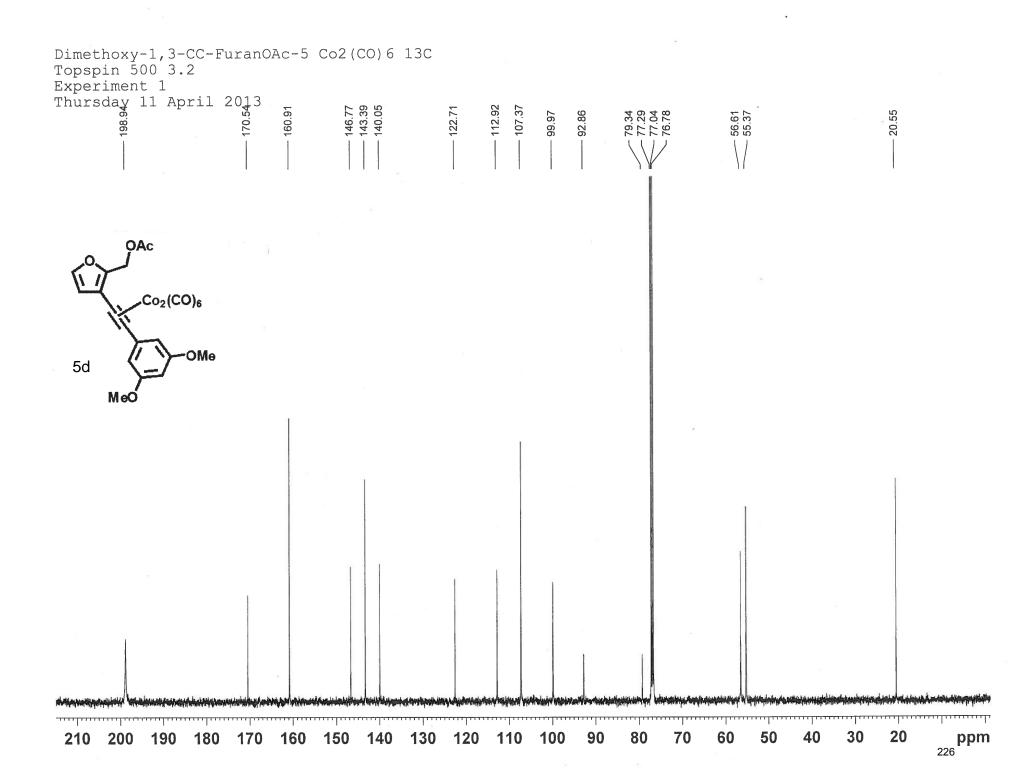


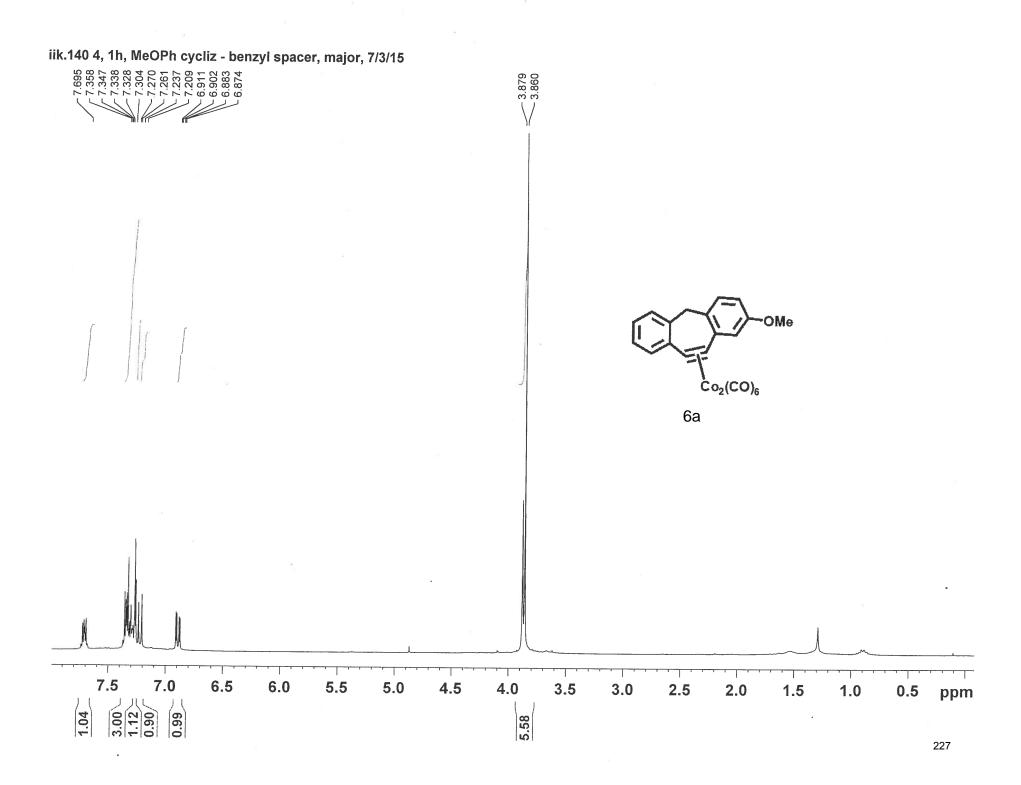




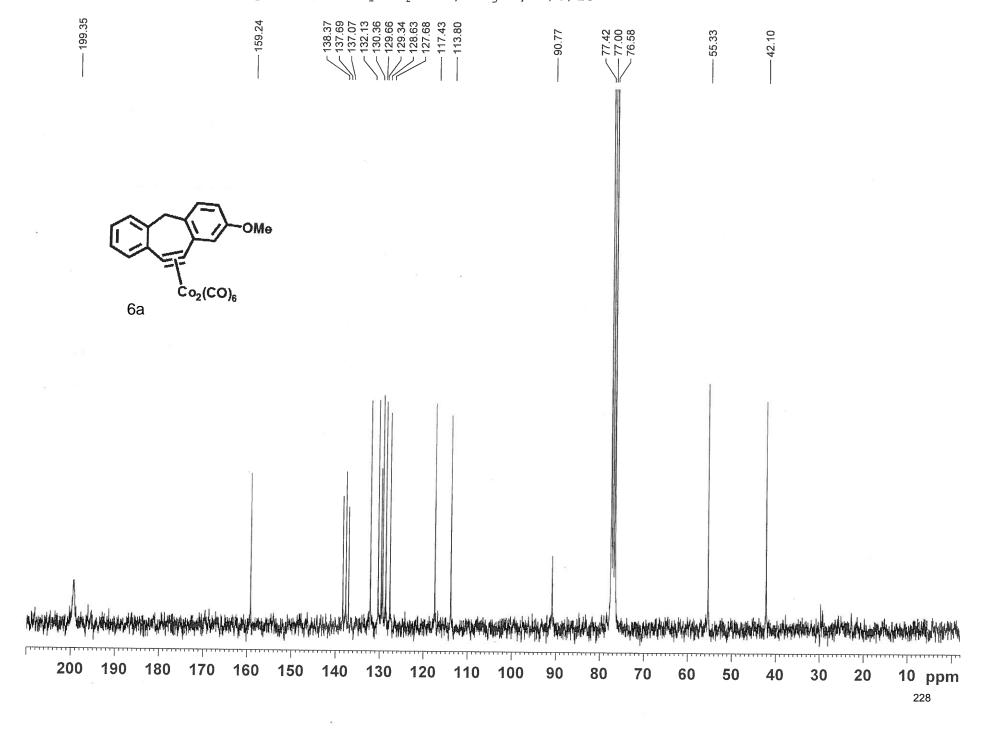


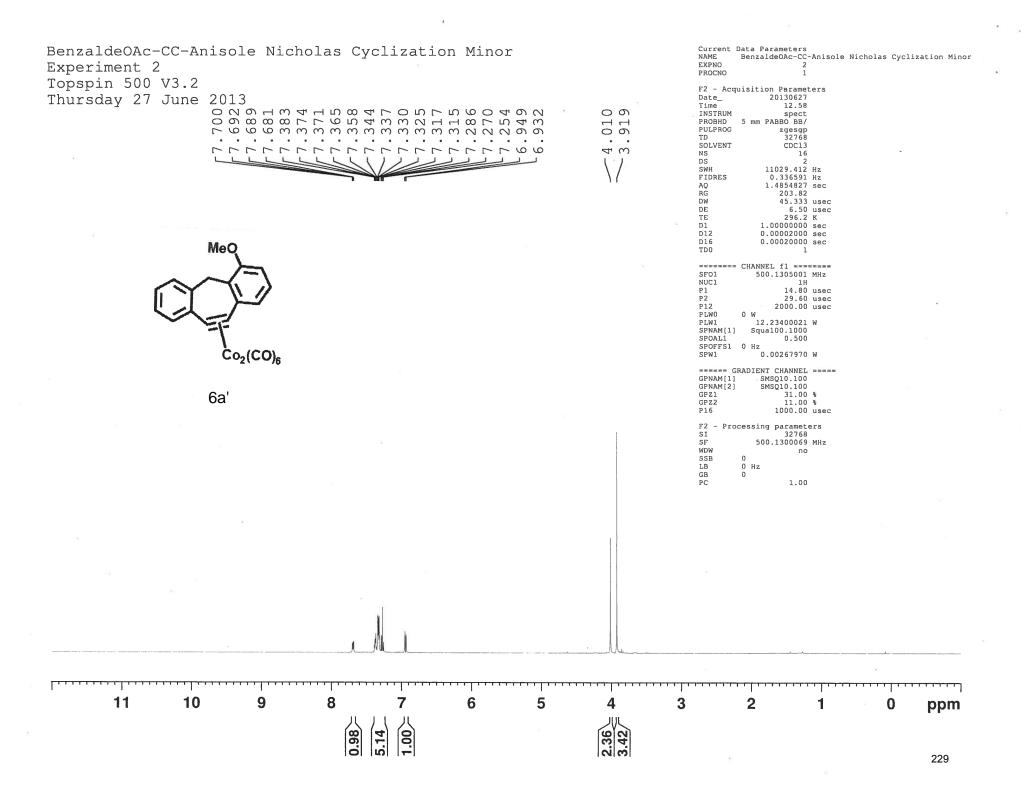


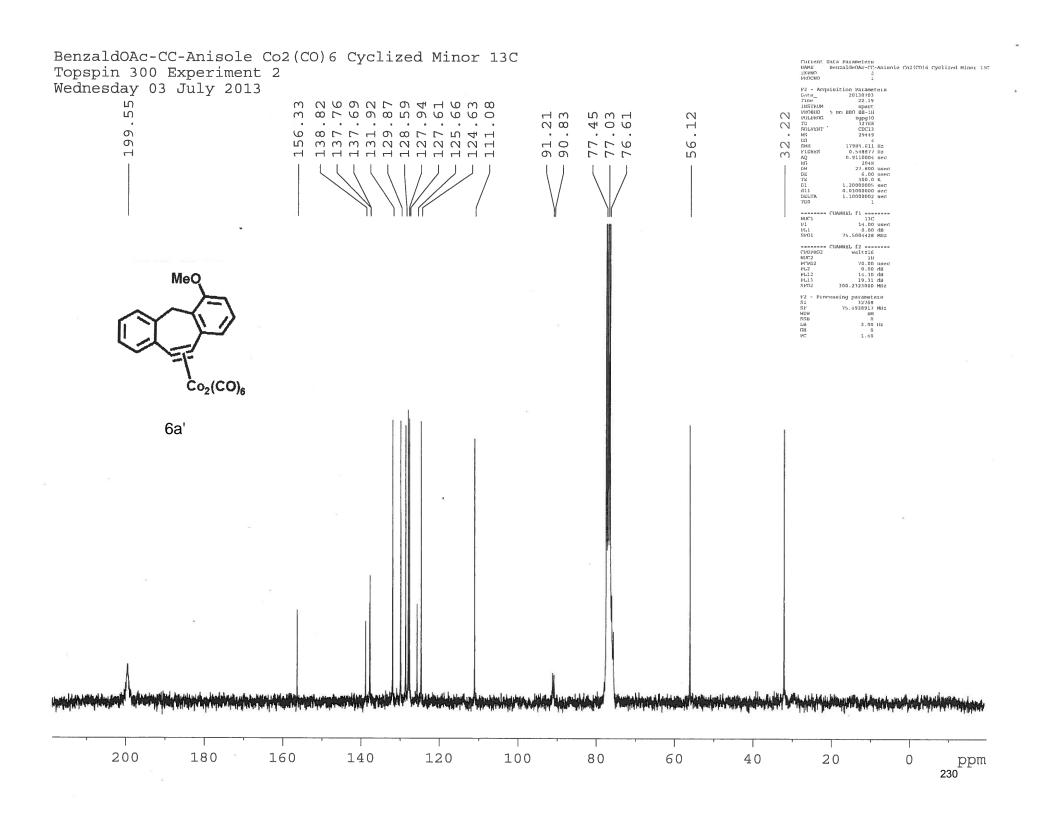


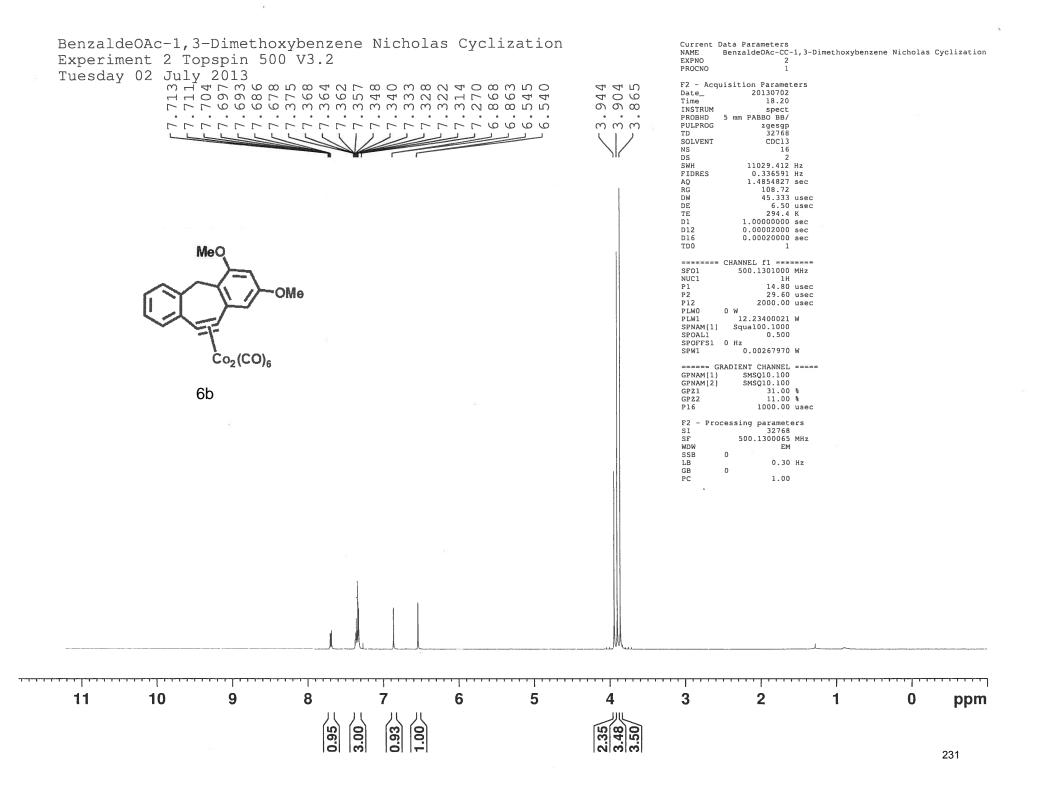


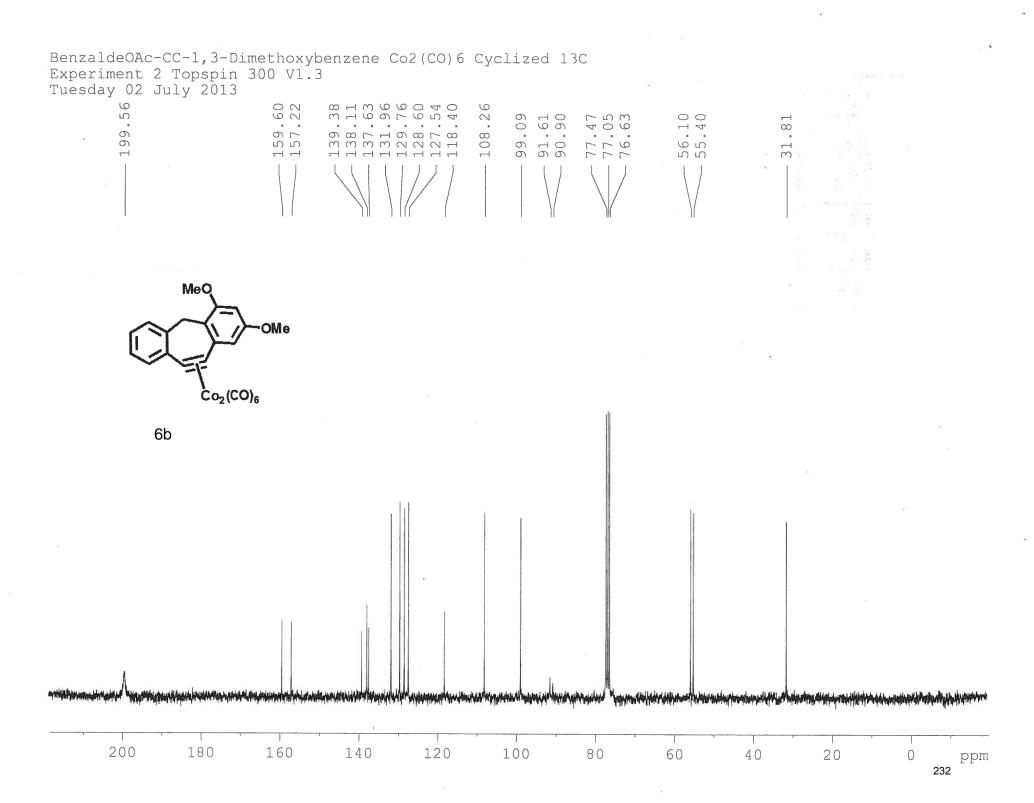
iik.140 5, 13C, MeOPh cycliza, benzyl spacer, major, 7/3/15

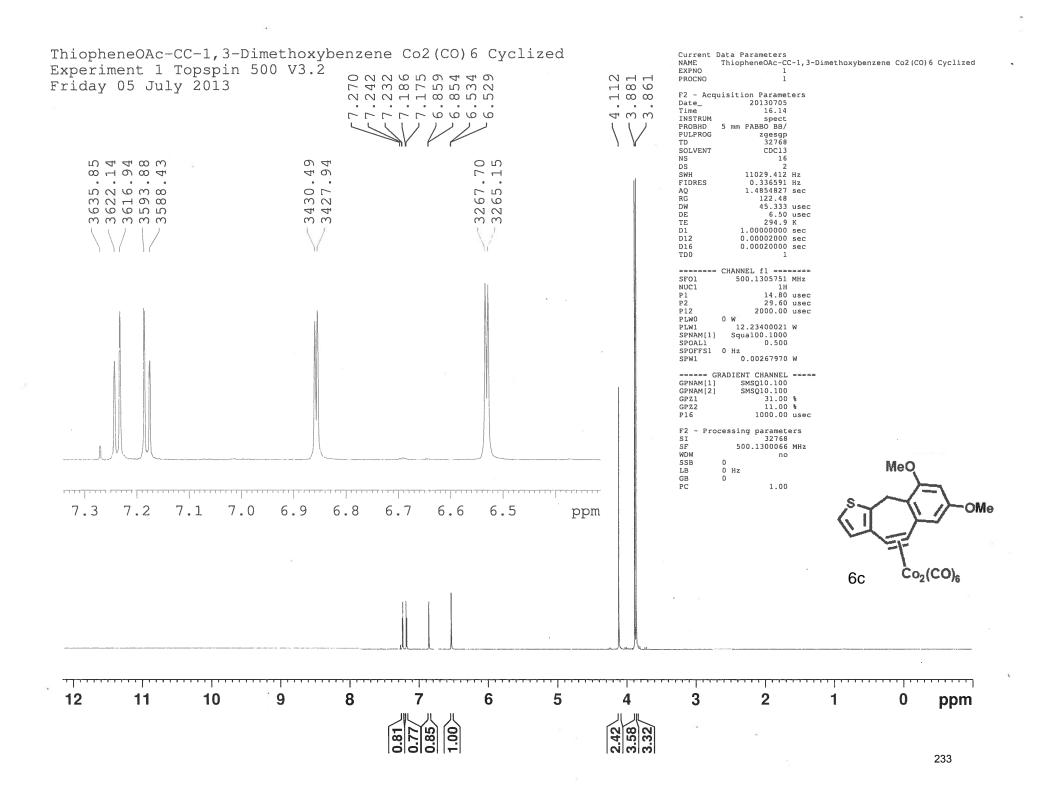


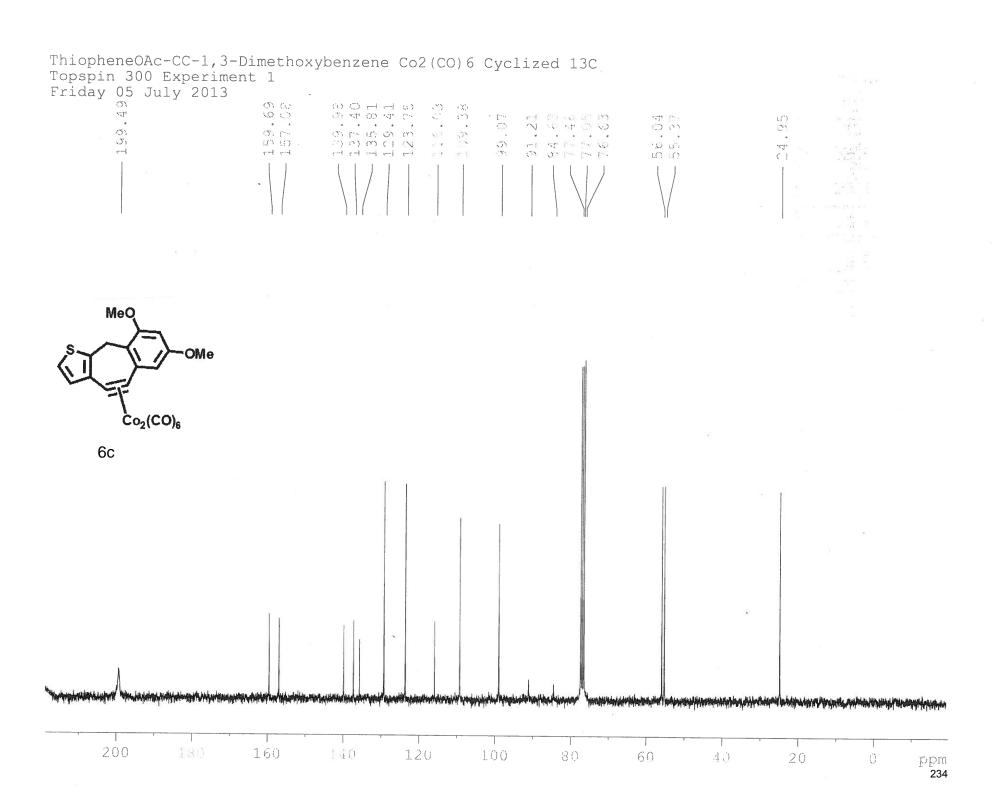


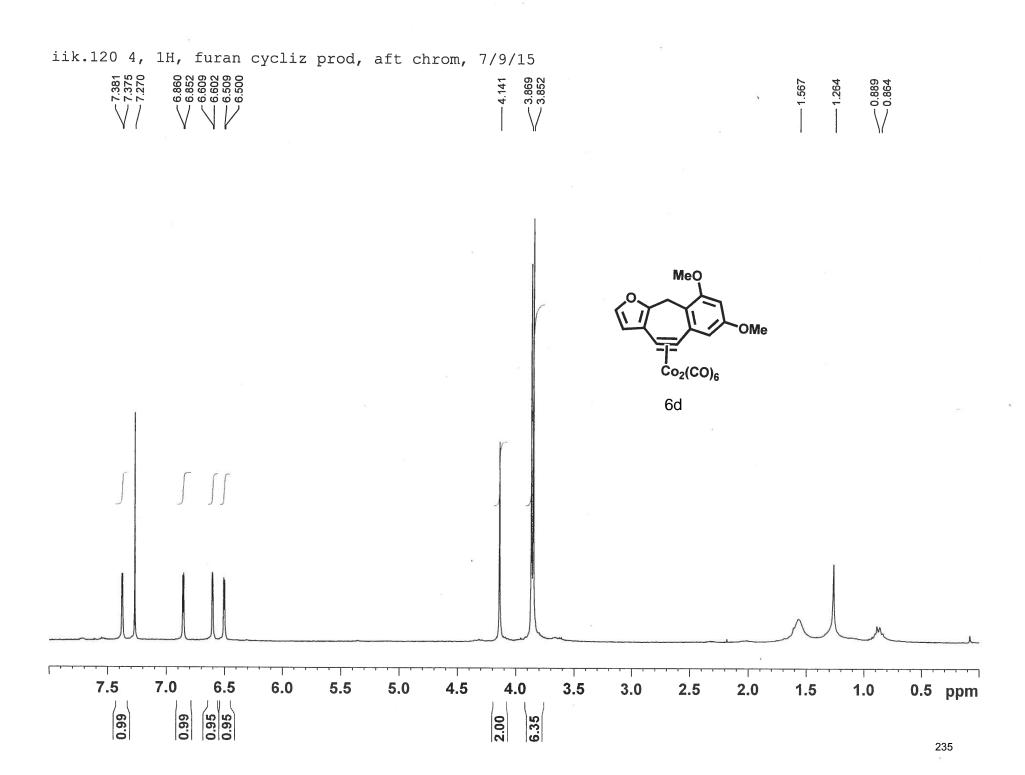


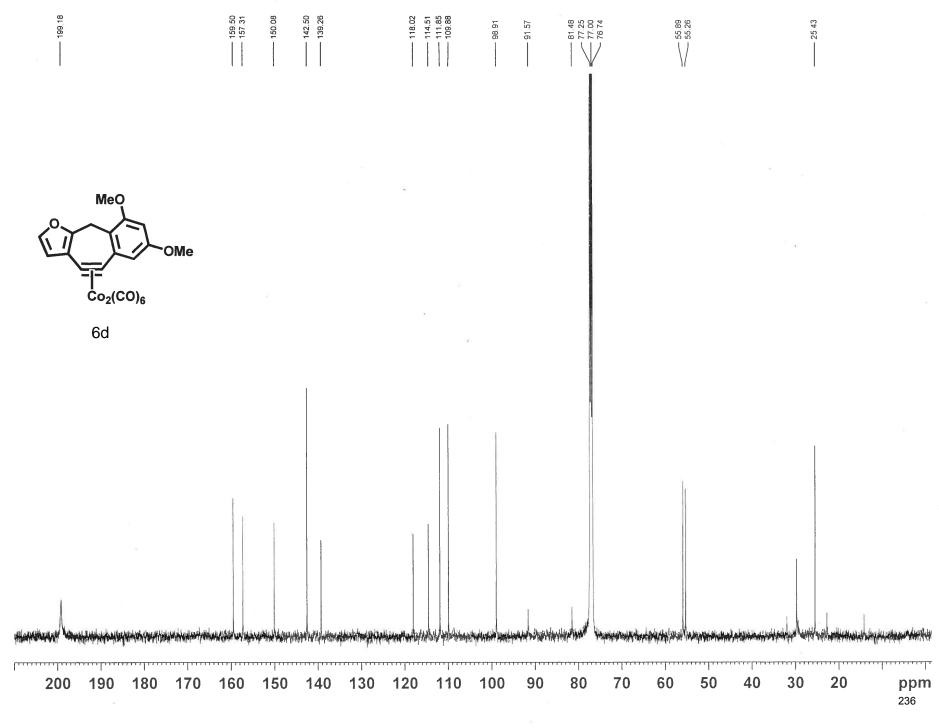


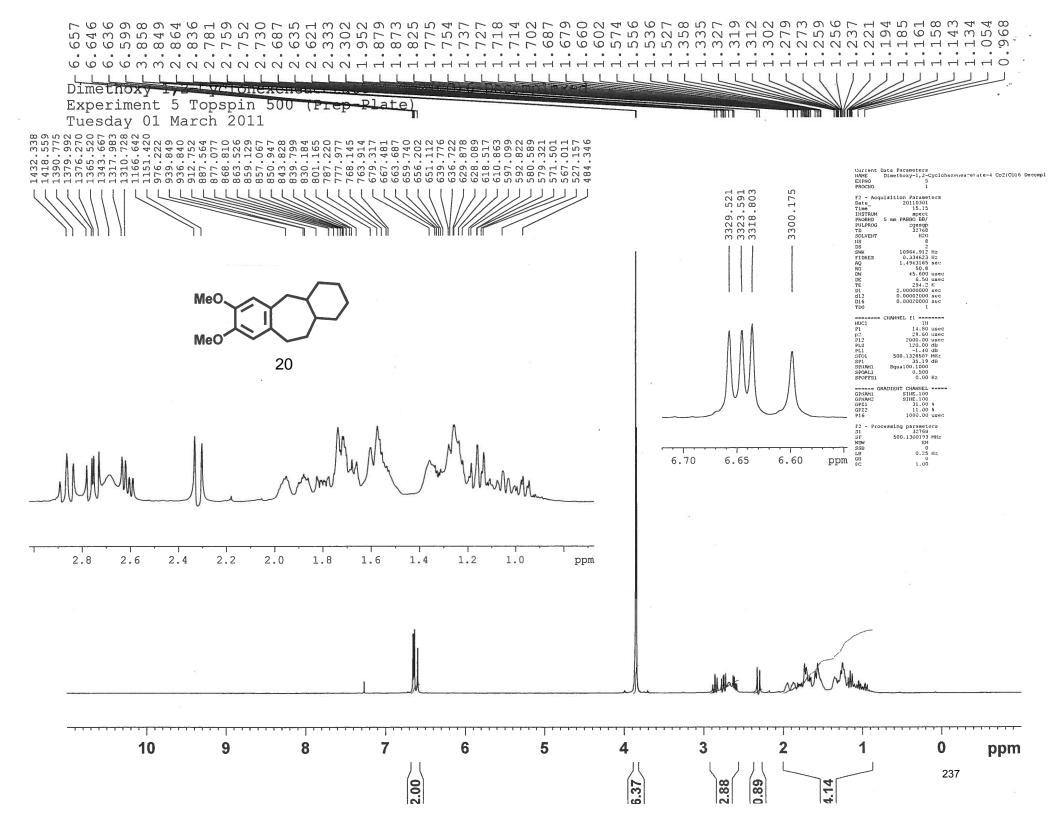


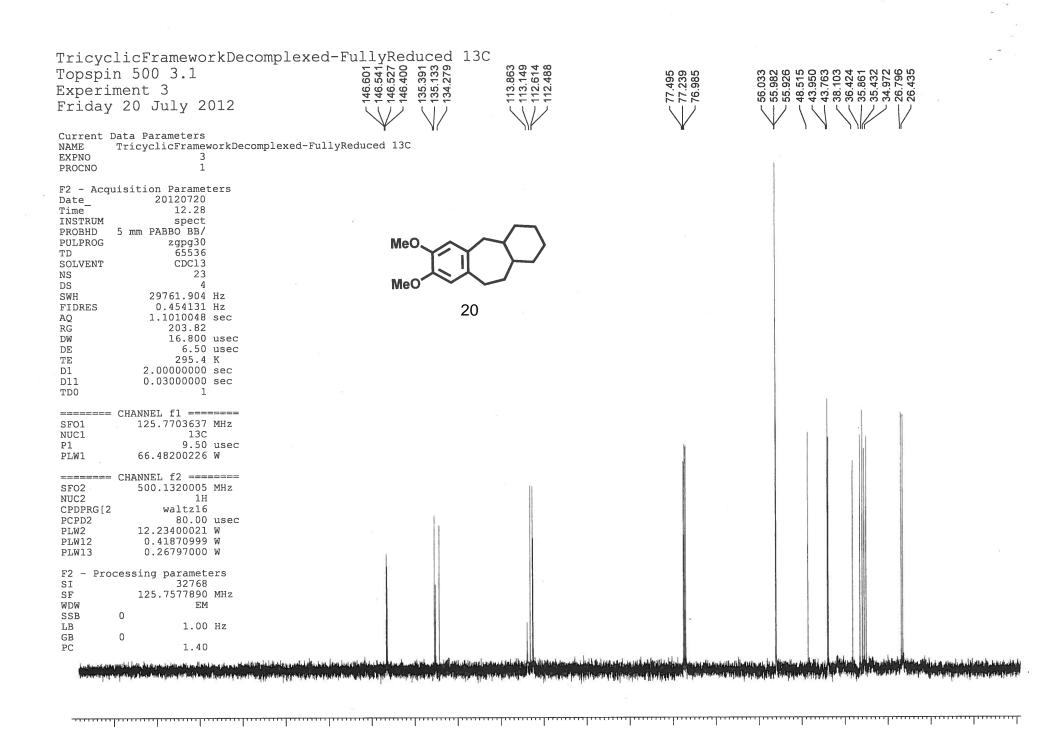








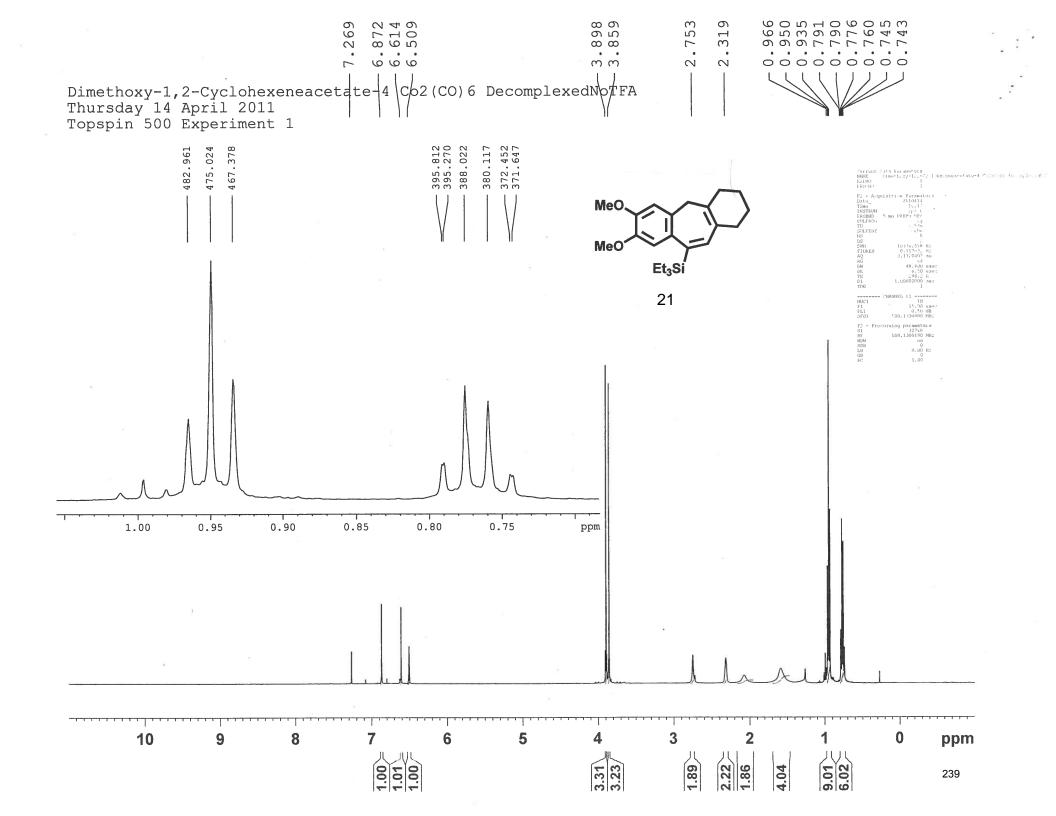




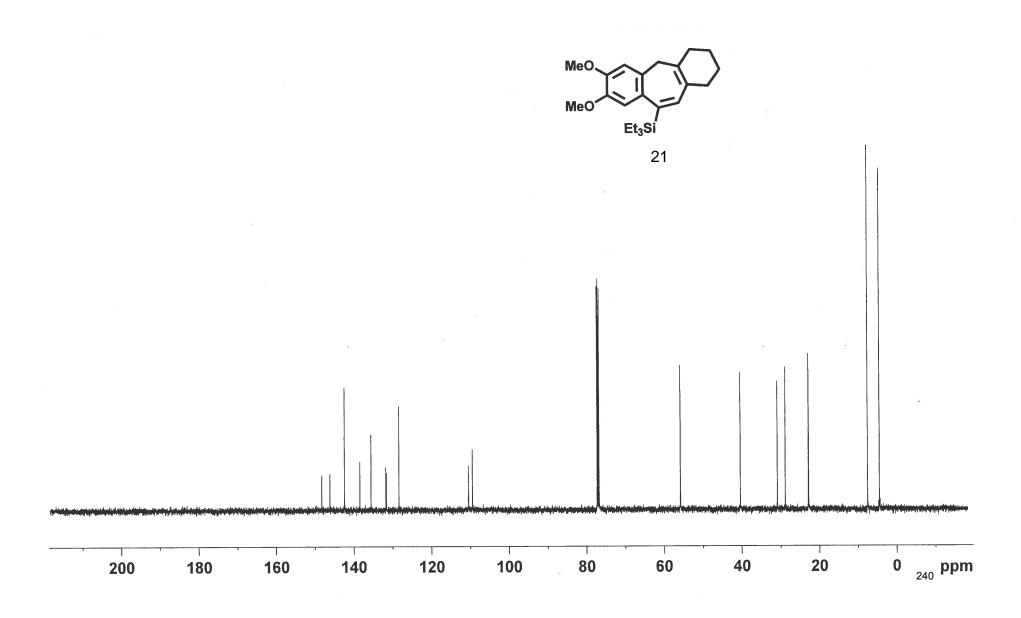
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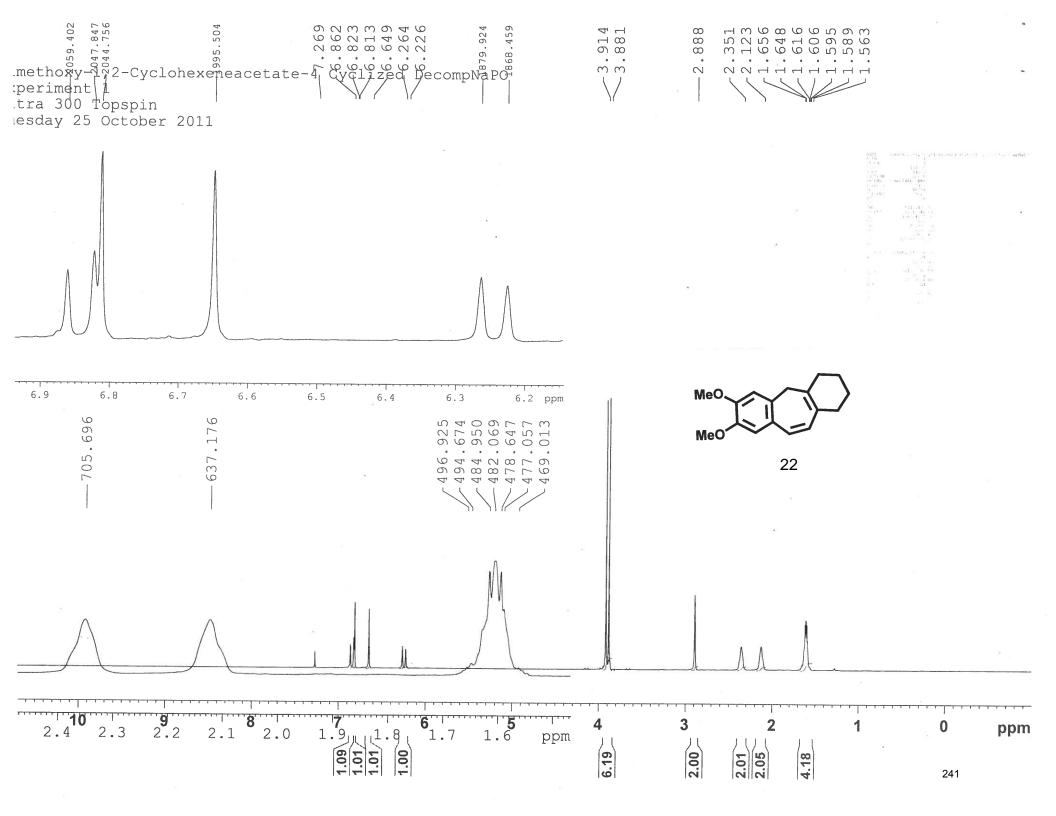
38

ppm

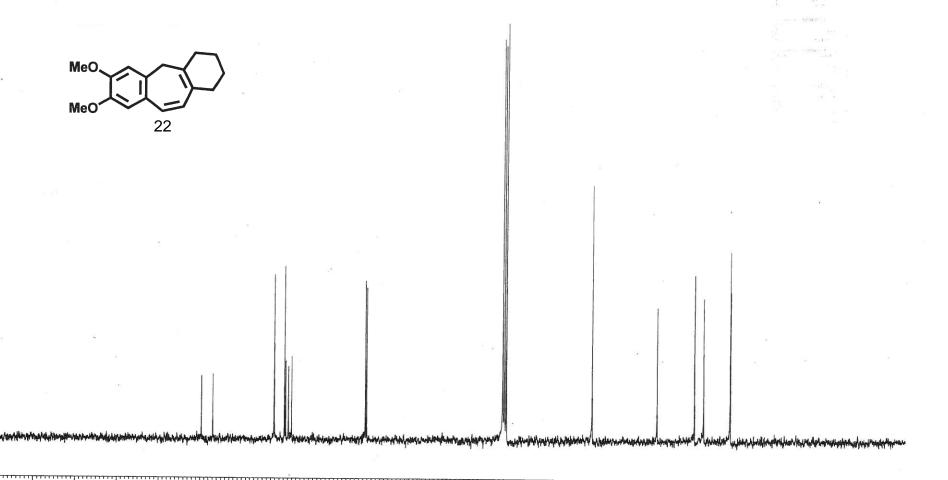


TricyclicFrameworkDecomplexed-SiEt3
Topspin 500 3.1
Thursday 05 July 2012
Experiment 1





190 180 170 160 150 140 130 120 110 100 90



60

50

ppm 242

