Supplementary Material (ESI) for Green Chemistry

Electronic Supplementary Information (ESI)

Organic reaction in Solkane®365mfc: Homocoupling reaction of terminal alkynes

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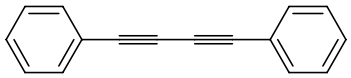
1. General Methods

All of the reactions were monitored by thin-layer chromatography (TLC) carried out on 0.25 mm Merck silica-gel (60-F254). The TLC plates were visualized with UV light and 7% phosphomolybdic acid or $KMnO_4$ in water/heat. Column chromatography was carried out on a column packed with silica-gel 60N spherical neutral size 63-210 μm. The ^{1}H-NMR (300 MHz), ^{1}H-NMR (200 MHz), ^{19}F-NMR (282.3 MHz), ^{19}F-NMR (188.2 MHz) and ^{13}C-NMR (150.9 MHz) spectra for solution in $CDCl_3$ or THF-d_8 were recorded on a Buruker Avance 600 and Varian Mercury 200 or 300. Chemical shifts (δ) are expressed in ppm downfield from internal TMS or $CHCl_3$ or THF-d_8. Mass spectra were recorded on a SHIMADZU GCMS-QP5050A. Infrared (IR), UV-vis and steady-state fluorescence spectra were recorded on a JASCO FT/IR-4100 Spectrometer, V-530 spectrometer and FP-6200 Fluorospectrometer, respectively. Fluorescence quantum yields were calculated following the procedure mentioned before. MALDI-TOF MS was measured by Shimadzu Axima CFR+. Reverse phase HPLC analyses were performed on a JASCO PU-2080 Plus using 4.6 x 250 mm Develosil ODS-HG-5 column and MD-2015 multiwavelength detector.

2. Homocoupling reaction of terminal alkynes in Solkane®365mfc

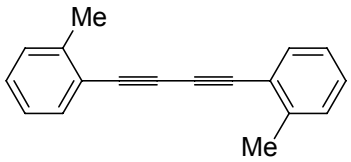
1,4-Diphenylbuta-1,3-diyne (2a)



A mixture of terminal alkyne **1a** (54.9 μL, 0.50 mmol), TMEDA (7.5 μL, 0.050 mmol), CuCl (5.0 mg, 0.050 mmol) in Solkane®365mfc (1.0 mL) was stirred under an atmosphere of air at rt. The resulting mixture was stirred at rt for 1.0 h. After dilution with sat. NH_4Cl aq, aqueous layer was extracted with CH_2Cl_2, and the combined organic layer was washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (*n*-hexane = 100) to give **2a** as a white solid (47.1 mg, 93%).

^{1}H NMR ($CDCl_3$, 200 MHz) δ 7.28-7.36 (m, 6H), 7.48-7.53 (m, 4H) ; ^{13}C NMR ($CDCl_3$, 150.9 MHz) δ 73.9, 81.5, 121.7, 128.4, 129.2, 132.5 ; IR (KBr) 3049, 2924, 2371, 2316, 2148, 1591, 1568, 1484, 1439, 754, 686 cm^{-1} ; MS (EI, *m/z*) 202 (M^+). These assignments are in good accord with those in the literature.[1]

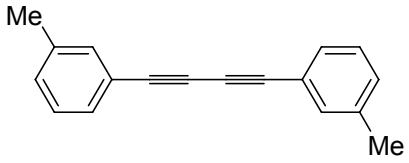
1,4-Di-*o*-tolylbuta-1,3-diyne (2b)



A mixture of terminal alkyne **1b** (63.0 μL, 0.50 mmol), TMEDA (7.5 μL, 0.050 mmol), DBU (74.7 μL, 0.50 mmol), CuCl (5.0 mg, 0.050 mmol) in Solkane®365mfc (1.0 mL) was stirred under an atmosphere of air at rt. The resulting mixture was stirred at rt for 2.5 h. After dilution with sat. NH_4Cl aq, aqueous layer was extracted with CH_2Cl_2, and the combined organic layer was washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (*n*-hexane = 100) to give **2b** as a wheat solid (51.6 mg, 90%).

^{1}H NMR ($CDCl_3$, 200 MHz) δ 2.49 (s, 6H), 7.10-7.30 (m, 6H), 7.49 (d, J = 7.6 Hz, 2H) ; ^{13}C NMR ($CDCl_3$, 150.9 MHz) δ 20.7, 77.5, 81.1, 121.7, 125.6, 129.1, 129.6, 132.9, 141.6 ; IR (KBr) 2950, 2925, 1728, 1595, 1455, 1288, 1276, 941, 750, 714 cm^{-1} ; MS (EI, *m/z*) 230 (M^+). These assignments are in good accord with those in the literature.[2]

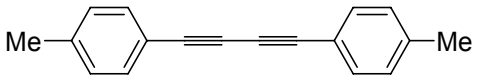
1,4-Di-*m*-tolylbuta-1,3-diyne (2c)



A mixture of terminal alkyne **1c** (64.5 μL, 0.50 mmol), TMEDA (7.5 μL, 0.050 mmol), DBU (74.7 μL, 0.50 mmol), CuCl (5.0 mg, 0.050 mmol) in Solkane®365mfc (1.0 mL) was stirred under an atmosphere of air at rt. The resulting mixture was stirred at rt for 3.0 h. After dilution with sat. NH_4Cl aq, aqueous layer was extracted with CH_2Cl_2, and the combined organic layer was washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (*n*-hexane = 100) to give **2c** as a lightyellow solid (50.0 mg, 87%).

^{1}H NMR ($CDCl_3$, 200 MHz) δ 2.33 (s, 6H), 7.14-7.25 (m, 4H), 7.30-7.34 (m, 4H) ; ^{13}C NMR ($CDCl_3$, 150.9 MHz) δ 21.2, 73.6, 81.6, 121.6, 128.3, 129.6, 130.1, 132.9, 138.1 ; IR (KBr) 2916, 2142, 1596, 1577, 1480, 1090, 904, 876, 786, 686 cm^{-1} ; MS (EI, *m/z*) 230 (M^+). These assignments are in good accord with those in the literature.[2]

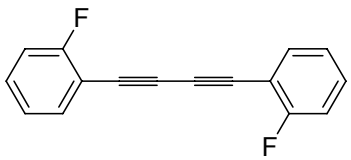
1,4-Di-*p*-tolylbuta-1,3-diyne (2d)



A mixture of terminal alkyne **1d** (63.4 μL, 0.50 mmol), TMEDA (7.5 μL, 0.050 mmol), CuCl (5.0 mg, 0.050 mmol) in Solkane®365mfc (1.0 mL) was stirred under an atmosphere of air at rt. The resulting mixture was stirred at rt for 1.0 h. After dilution with sat. NH_4Cl aq, aqueous layer was extracted with CH_2Cl_2, and the combined organic layer was washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (*n*-hexane = 100) to give **2d** as a white solid (51.0 mg, 89%).

^{1}H NMR ($CDCl_3$, 300 MHz) δ 2.36 (s, 6H), 7.14 (d, *J* = 8.1 Hz, 4H), 7.42 (d, *J* = 8.1 Hz, 4H) ; ^{13}C NMR ($CDCl_3$, 150.9 MHz) δ 21.6, 73.4, 81.5, 118.7, 129.2, 132.4, 139.5 ; IR (KBr) 2918, 2135, 1897, 1502, 1406, 1173, 1039, 1017, 808, 522 cm^{-1} ; MS (EI, *m/z*) 230 (M^+). These assignments are in good accord with those in the literature.[1]

1,4-Bis(2-fluorophenyl)buta-1,3-diyne (2e)

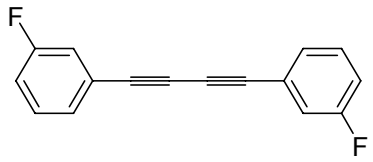


A mixture of terminal alkyne **1e** (56.7 μL, 0.50 mmol), TMEDA (7.5 μL, 0.050 mmol), CuCl (5.0 mg, 0.050 mmol) in Solkane®365mfc (1.0 mL) was stirred under an atmosphere of air at rt. The resulting mixture was stirred at rt for 3.0 h. After dilution with sat. NH_4Cl aq, aqueous layer was extracted with CH_2Cl_2, and the combined organic layer was washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (*n*-hexane = 100) to give **2e** as a wheat solid (54.2

mg, 91%).

^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.07-7.16 (m, 4H), 7.33-7.40 (m, 2H), 7.50-7.55 (m, 2H) ; ^{13}C NMR ($CDCl_3$, 150.9 MHz) δ 75.8, 78.3, 110.4 (d, *J* = 15.5 Hz), 115.7 (d, *J* = 20.5 Hz), 124.1 (d, *J* = 3.5 Hz), 131.1 (d, *J* = 8.0 Hz), 134.3, 163.7 (d, *J* = 253.7 Hz) ; ^{19}F NMR ($CDCl_3$, 282.3 MHz) δ -108.9 (dd, *J* = 14.4, 7.1 Hz, 2F) ; IR (KBr) 1570, 1483, 1445, 1269, 1231, 1203, 1100, 833, 750, 730 cm^{-1} ; MS (EI, *m/z*) 238 (M^+). These assignments are in good accord with those in the literature.[2]

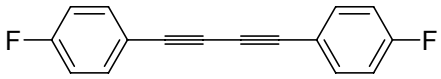
1,4-Bis(3-fluorophenyl)buta-1,3-diyne (2f)



A mixture of terminal alkyne **1f** (57.8 μL, 0.50 mmol), TMEDA (7.5 μL, 0.050 mmol), CuCl (5.0 mg, 0.050 mmol) in Solkane®365mfc (1.0 mL) was stirred under an atmosphere of air at rt. The resulting mixture was stirred at rt for 2.0 h. After dilution with sat. NH_4Cl aq, aqueous layer was extracted with CH_2Cl_2, and the combined organic layer was washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (*n*-hexane = 100) to give **2f** as a wheat solid (52.9 mg, 89%).

^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.08-7.13 (m, 2H), 7.21-7.26 (m, 2H), 7.32 (s, 4H) ; ^{13}C NMR ($CDCl_3$, 150.9 MHz) δ 74.4, 80.6 (d, *J* = 3.5 Hz), 116.9 (d, *J* = 21.3 Hz), 119.2 (d, *J* = 23.1 Hz), 123.3 (d, *J* = 9.5 Hz), 128.5 (d, *J* = 3.0 Hz), 130.1 (d, *J* = 8.6 Hz), 162.2 (d, *J* = 247.5 Hz) ; ^{19}F NMR ($CDCl_3$, 282.3 MHz) δ -112.7-112.8 (m, 2F) ; IR (KBr) 1604, 1582, 1482, 1463, 1429, 1265, 1131, 931, 869, 783 cm^{-1} ; MS (EI, *m/z*) 238 (M^+). These assignments are in good accord with those in the literature.[3]

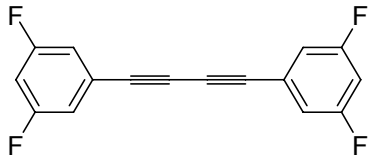
1,4-Bis(4-fluorophenyl)buta-1,3-diyne (2g)



A mixture of terminal alkyne **1g** (57.3 μL, 0.50 mmol), TMEDA (7.5 μL, 0.050 mmol), CuCl (5.0 mg, 0.050 mmol) in Solkane®365mfc (1.0 mL) was stirred under an atmosphere of air at rt. The resulting mixture was stirred at rt for 4.5 h. After dilution with sat. NH_4Cl aq, aqueous layer was extracted with CH_2Cl_2, and the combined organic layer was washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (*n*-hexane = 100) to give **2g** as a lightyellow solid (51.2 mg, 86%).

^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.04 (t, *J* = 9.0 Hz, 4H), 7.51 (dd, *J* = 8.7, 5.7 Hz, 4H), ^{13}C NMR ($CDCl_3$, 150.9 MHz) δ 73.5, 80.4, 115.9 (d, *J* = 22.3 Hz), 117.8, 134.5 (d, *J* = 8.6 Hz), 163.0 (d, *J* = 251.6 Hz) ; ^{19}F NMR ($CDCl_3$, 282.3 MHz) δ -109.0 (tt, *J* = 9.0, 4.8 Hz, 2F) ; IR (KBr) 2143, 1888, 1595, 1586, 1502, 1227, 1159, 1093, 828, 696 cm^{-1} ; MS (EI, *m/z*) 238 (M^+). These assignments are in good accord with those in the literature.[1]

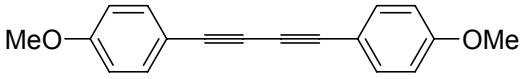
1,4-Bis(3,5-difluorophenyl)buta-1,3-diyne (2h)



A mixture of terminal alkyne **1h** (59.4 μL, 0.50 mmol), TMEDA (7.5 μL, 0.050 mmol), CuCl (5.0 mg, 0.050 mmol) in Solkane®365mfc (1.0 mL) was stirred under an atmosphere of air at rt. The resulting mixture was stirred at rt for 1.0 h. After dilution with sat. NH_4Cl aq, aqueous layer was extracted with CH_2Cl_2, and the combined organic layer was washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (*n*-hexane = 100) to give **2h** as a khaki solid (56.2 mg, 82%).

^{1}H NMR ($CDCl_3$, 200 MHz) δ 6.85 (tt, *J* = 8.8, 2.4 Hz, 2H), 7.00-7.05 (m, 4H) ; ^{13}C NMR ($CDCl_3$, 150.9 MHz) δ 75.0, 80.0 (t, *J* = 3.9 Hz), 106.0 (t, *J* = 25.4 Hz), 115.5 (dd, *J* = 21.2, 6.0 Hz), 123.9 (t, *J* = 11.8 Hz), 162.6 (dd, *J* = 250.2, 13.1 Hz) ; ^{19}F NMR ($CDCl_3$, 188.2 MHz) δ -108.2 (t, *J* = 7.9 Hz, 4F) ; IR (KBr) 3097, 3075, 1615, 1587, 1450, 1428, 1328, 1123, 993, 963, 859, 846, 665 cm^{-1} ; MS (EI, *m/z*) 274 (M^+). These assignments are in good accord with those in the literature.[4]

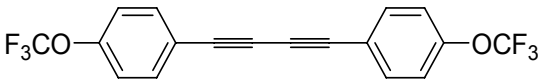
1,4-Bis(4-methoxyphenyl)buta-1,3-diyne (2i)



A mixture of terminal alkyne **1i** (64.8 μL, 0.50 mmol), TMEDA (7.5 μL, 0.050 mmol), DBU (74.7 μL, 0.50 mmol), CuCl (5.0 mg, 0.050 mmol) in Solkane®365mfc (1.0 mL) was stirred under an atmosphere of air at rt. The resulting mixture was stirred at rt for 5.0 h. After dilution with sat. NH_4Cl aq, aqueous layer was extracted with CH_2Cl_2, and the combined organic layer was washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (*n*-hexane/ethyl acetate = 90/10) to give **2i** as a lightyellow solid (55.7 mg, 85%).

^{1}H NMR ($CDCl_3$, 200 MHz) δ 3.82 (s, 6H), 6.84 (d, *J* = 9.0 Hz, 4H), 7.45 (d, *J* = 8.8 Hz, 4H) ; ^{13}C NMR ($CDCl_3$, 150.9 MHz) δ 55.3, 72.9, 81.2, 113.8, 114.1, 134.0, 160.2 ; IR (KBr) 2841, 2138, 1599, 1503, 1294, 1255, 1167, 1028, 841, 821 cm^{-1} ; MS (EI, *m/z*) 262 (M^+). These assignments are in good accord with those in the literature.[1]

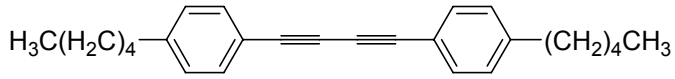
1,4-Bis(4-trifluoromethoxyphenyl)buta-1,3-diyne (2j)



A mixture of terminal alkyne **1j** (76.6 μL, 0.50 mmol), TMEDA (7.5 μL, 0.050 mmol), CuCl (5.0 mg, 0.050 mmol) in Solkane®365mfc (1.0 mL) was stirred under an atmosphere of air at rt. The resulting mixture was stirred at rt for 4.0 h. After dilution with sat. NH_4Cl aq, aqueous layer was extracted with CH_2Cl_2, and the combined organic layer was washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (*n*-hexane = 100) to give **2j** as a darkorange solid (77.6 mg, 84%).

^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.20 (d, *J* = 8.1 Hz, 4H), 7.56 (d, *J* = 8.7 Hz, 4H) ; ^{13}C NMR ($CDCl_3$, 150.9 MHz) δ 74.4, 80.4, 120.27, 120.31 (q, *J* = 258.8 Hz), 120.9, 134.1, 149.6 ; ^{19}F NMR ($CDCl_3$, 282.3 MHz) δ -58.2 (s, 6F) ; IR (KBr) 2962, 2144, 1900, 1577, 1501, 1254, 1215, 1176, 1016, 836 cm^{-1} ; mp = 111-114 ºC ; MS (EI, *m/z*) 370 (M^+) ; HRMS calcd. for $C_{18}H_8F_6O_2^+$: 370.0428 Found : 370.0400.

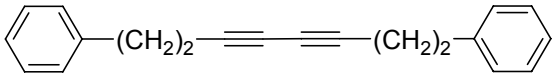
1,4-Bis(4-n-pentylphenyl)buta-1,3-diyne (2k)



A mixture of terminal alkyne **1k** (97.3 μL, 0.50 mmol), TMEDA (7.5 μL, 0.050 mmol), DBU (74.7 μL, 0.50 mmol), CuCl (5.0 mg, 0.050 mmol) in Solkane®365mfc (1.0 mL) was stirred under an atmosphere of air at rt. The resulting mixture was stirred at rt for 5.5 h. After dilution with sat. NH_4Cl aq, aqueous layer was extracted with CH_2Cl_2, and the combined organic layer was washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (*n*-hexane = 100) to give **2k** as a white solid (83.6 mg, 98%).

^{1}H NMR ($CDCl_3$, 200 MHz) δ 0.89 (t, *J* = 6.6 Hz, 6H), 1.27-1.38 (m, 8H), 1.53-1.67 (m, 4H), 2.60 (t, *J* = 8.0 Hz, 4H), 7.12 (d, *J* = 8.2 Hz, 4H), 7.42 (d, *J* = 8.0 Hz, 4H) ; ^{13}C NMR ($CDCl_3$, 150.9 MHz) δ 14.0, 22.5, 30.8, 31.4, 35.9, 73.5, 81.5, 118.9, 128.5, 132.4, 144.5 ; IR (KBr) 2927, 2857, 2140, 1602, 1504, 1466, 1455, 1175, 1018, 839 cm^{-1} ; MS (EI, *m/z*) 342 (M^+). These assignments are in good accord with those in the literature.[3]

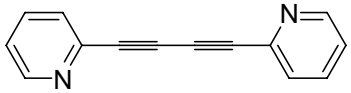
1,8-Di-phenylocta-3,5-diyne (2l)



A mixture of terminal alkyne **1l** (70.3 μL, 0.50 mmol), TMEDA (7.5 μL, 0.050 mmol), DBU (74.7 μL, 0.50 mmol), CuCl (5.0 mg, 0.050 mmol) in Solkane®365mfc (1.0 mL) was stirred under an atmosphere of air at rt. The resulting mixture was stirred at rt for 6.0 h. After dilution with sat. NH_4Cl aq, aqueous layer was extracted with CH_2Cl_2, and the combined organic layer was washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (*n*-hexane/ethyl acetate = 99/1) to give **2l** as a white solid (61.4 mg, 95%).

^{1}H NMR ($CDCl_3$, 200 MHz) δ 2.51 (t, *J* = 7.6 Hz, 4H), 2.81 (t, *J* = 7.6 Hz, 4H), 7.15-7.32 (m, 10H) ; ^{13}C NMR ($CDCl_3$, 150.9 MHz) δ 21.4, 34.6, 65.9, 76.8, 126.4, 128.3, 128.4, 140.1 ; IR (KBr) 2927, 1601, 1496, 1451, 1252, 1078, 1029, 751, 720, 700 cm^{-1} ; MS (EI, *m/z*) 258 (M^+). These assignments are in good accord with those in the literature.[5]

1,4-Di(pyridin-2-yl)buta-1,3-diyne (2m)

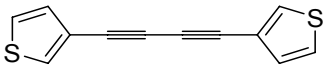


A mixture of terminal alkyne **1m** (50.5 μL, 0.50 mmol), TMEDA (7.5μL, 0.050 mmol), CuCl (5.0 mg, 0.050 mmol) in Solkane®365mfc (1.0 mL) was stirred under an atmosphere of air at rt. The resulting mixture was

stirred at rt for 6.0 h. After dilution with sat. NH_4Cl aq, aqueous layer was extracted with CH_2Cl_2, and the combined organic layer was washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (*n*-hexane/ethyl acetate = 70/30) to give **2m** as a darkkhaki solid (46.9 mg, 92%).

^{1}H NMR ($CDCl_3$, 200 MHz) δ 7.29 (ddd, *J* = 7.6, 4.9, 1.4 Hz, 2H), 7.53 (dt, *J* = 7.8, 1.2 Hz, 2H), 7.68 (dt, *J* = 7.8, 1.8 Hz, 2H), 8.61 (d, *J* = 4.6 Hz, 2H) ; ^{13}C NMR ($CDCl_3$, 150.9 MHz) δ 73.0, 80.8, 123.7, 128.3, 136.1, 141.8, 150.3 ; IR (KBr) 1575, 1560, 1459, 1425, 1240, 986, 774, 734 cm^{-1} ; MS (EI, *m/z*) 204 (M^+). These assignments are in good accord with those in the literature.[1]

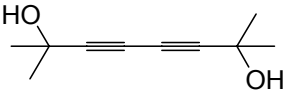
1,4-Di(thiophen-3-yl)buta-1,3-diyne (2n)



A mixture of terminal alkyne **1n** (49.3 μL, 0.50 mmol), TMEDA (7.5 μL, 0.050 mmol), CuCl (5.0 mg, 0.050 mmol) in Solkane®365mfc (1.0 mL) was stirred under an atmosphere of air at rt. The resulting mixture was stirred at rt for 3.0 h. After dilution with sat. NH_4Cl aq, aqueous layer was extracted with CH_2Cl_2, and the combined organic layer was washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (*n*-hexane = 100) to give **2n** as a darkkhaki solid (47.9 mg, 89%).

^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.17 (dd, *J* = 4.8, 0.9 Hz, 2H), 7.28 (dd, *J* = 8.1, 3.3 Hz, 2H), 7.59 (d, *J* = 2.1, 2H) ; ^{13}C NMR ($CDCl_3$, 150.9 MHz) δ 73.5, 76.5, 120.8, 125.6, 130.1, 131.2 ; IR (KBr) 3102, 2143, 1352, 1215, 1077, 928, 868, 781, 690, 622 cm^{-1} ; MS (EI, *m/z*) 214 (M^+). These assignments are in good accord with those in the literature.[6]

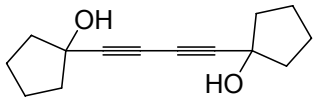
2,7-Dimethylocta-3,5-diyne-2,7-diol (2o)



A mixture of terminal alkyne **1o** (48.5 μL, 0.50 mmol), TMEDA (7.5 μL, 0.050 mmol), CuCl (5.0 mg, 0.050 mmol) in Solkane®365mfc (1.0 mL) was stirred under an atmosphere of air at rt. The resulting mixture was stirred at rt for 3.0 h. After dilution with sat. NH_4Cl aq, aqueous layer was extracted with CH_2Cl_2, and the combined organic layer was washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (*n*-hexane/ethyl acetate = 70/30) to give **2o** as a white solid (36.5 mg, 88%).

^{1}H NMR ($CDCl_3$, 200 MHz) δ 1.53 (s, 12H), 1.93 (brs, 2H) ; ^{13}C NMR ($CDCl_3$, 150.9 MHz) δ 31.0, 65.6, 66.3, 83.9 ; IR (KBr) 3217, 2981, 1448, 1382, 1364, 1171, 965, 954, 741, 710 cm^{-1} ; MS (EI, *m/z*) 166 (M^+). These assignments are in good accord with those in the literature.[2]

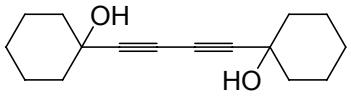
1,1'-(Buta-1,3-diyne-1,4-diyl)dicyclopentanol (2p)



A mixture of terminal alkyne **1p** (57.3 μL, 0.50 mmol), TMEDA (7.5μL, 0.050 mmol), CuCl (5.0 mg, 0.050 mmol) in Solkane®365mfc (1.0 mL) was stirred under an atmosphere of air at rt. The resulting mixture was stirred at rt for 2.5 h. After dilution with sat. NH_4Cl aq, aqueous layer was extracted with CH_2Cl_2, and the combined organic layer was washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (*n*-hexane/ethyl acetate = 80/20) to give **2p** as a white solid (52.3 mg, 96%).

^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.71-1.99 (m, 18H) ; ^{13}C NMR ($CDCl_3$, 150.9 MHz) δ 23.4, 42.4, 67.4, 74.8, 83.2 ; IR (KBr) 3279, 2978, 2962, 2948, 1385, 1206, 1092, 993, 906, 689 cm^{-1} ; MS (EI, *m/z*) 200 (M^+-H_2O). These assignments are in good accord with those in the literature.[1]

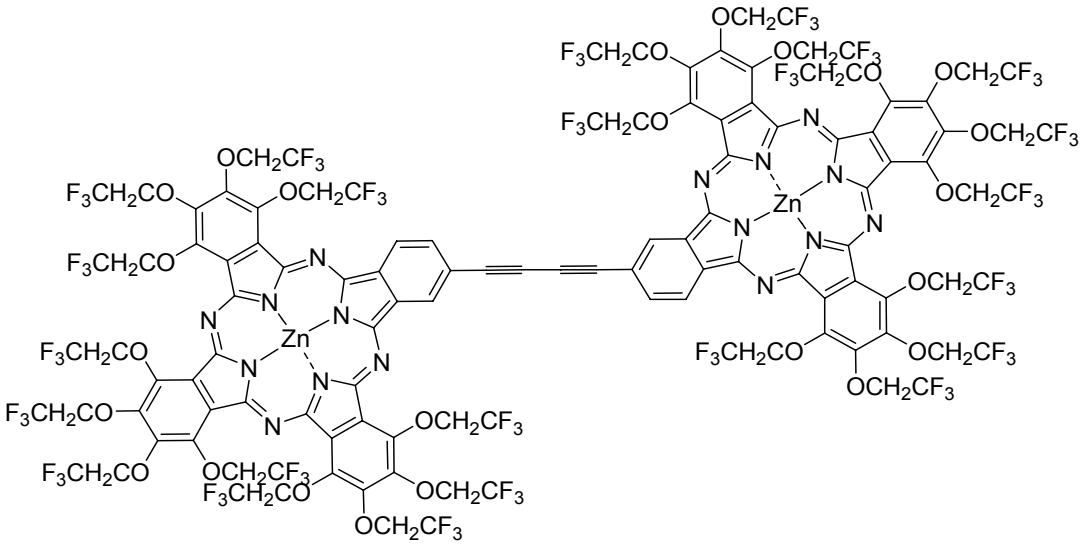
1,1'-(Buta-1,3-diyne-1,4-diyl)dicyclohexanol (2q)



A mixture of terminal alkyne **1q** (62.1 mg, 0.50 mmol), TMEDA (7.5 μL, 0.050 mmol), CuCl (5.0 mg, 0.050 mmol) in Solkane®365mfc (1.0 mL) was stirred under an atmosphere of air at rt. The resulting mixture was stirred at rt for 24.0 h. After dilution with sat. NH_4Cl aq, aqueous layer was extracted with CH_2Cl_2, and the combined organic layer was washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (*n*-hexane/ethyl acetate = 80/20) to give **2q** as a white solid (56.0 mg, 91%).

^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.25-1.27 (m, 4H), 1.52-1.72 (m, 12H), 1.91-1.95 (m, 4H), 2.03 (s, 2H) ; ^{13}C NMR ($CDCl_3$, 150.9 MHz) δ 23.1, 25.0, 39.6, 68.3, 69.2, 82.9 ; IR (KBr) 3253, 2935, 2855, 1453, 1396, 1340, 1188, 1160, 1061, 962 cm^{-1} ; MS (EI, *m/z*) 228 (M^+-H_2O). These assignments are in good accord with those in the literature.[1]

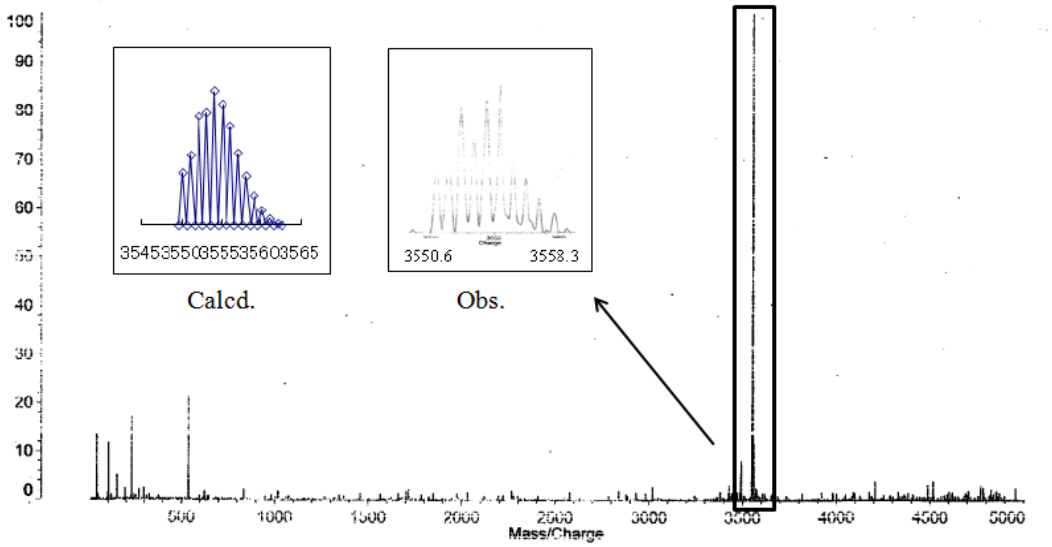
Bis[zinc(II)(23-ethynyl-1,2,3,4,8,9,10,11,15,16,17,18-dodecakis(2,2,2-trifluoroethoxy)phthalocyaninate)]butadiyne (4)



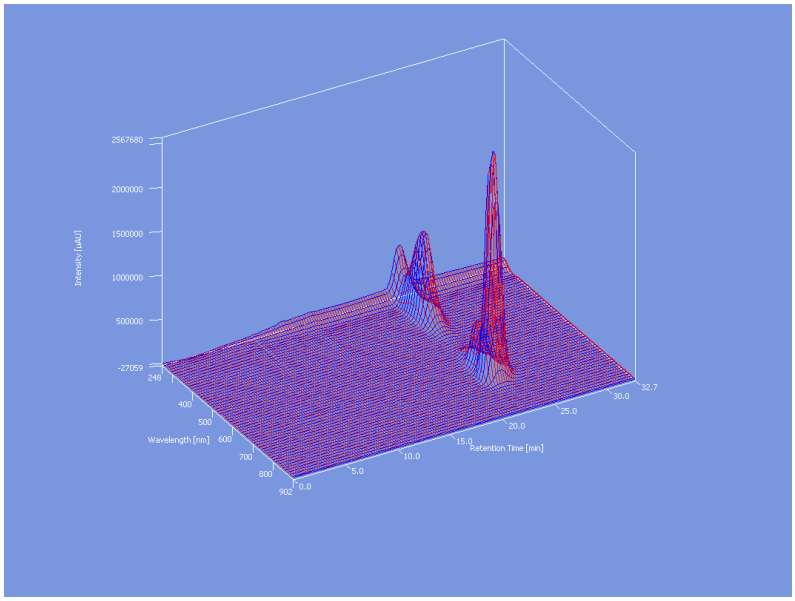
A mixture of terminal alkyne **3** (50.0 mg, 0.0281 mmol), TMEDA (4.2 μL, 0.0281 mmol), DBU 4.2 μL, 0.0281 mmol), CuCl (2.8 mg, 0.0281 mmol) in Solkane®365mfc (1.0 mL) was stirred under an atmosphere of air at rt. The resulting mixture was stirred at rt for 24.0 h. After dilution with sat. NH_4Cl aq, aqueous layer was extracted with ethyl acetate, and the combined organic layer was washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (*n*-hexane/ethyl acetate = 60/40) to give **4** as a green solid (32.9 mg, 66%).

^{1}H NMR (THF-d_8, 300 MHz) δ 5.02-5.15 (m, 24H), 5.65-5.90 (m, 24H), 8.59 (d, J = 7.8 Hz, 2H), 9.57 (d, J = 7.5 Hz, 2H), 9.79 (s, 2H) ; ^{19}F NMR (THF-d_8, 282.3 MHz) δ -73.4 - -73.5 (m, 12F), -74.0 - -74.1 (m, 24F), -74.5 - -74.7 (m, 36F) ; IR (KBr) 2968, 1487, 1439, 1274, 1158, 1120, 1069, 1022, 970, 924, 839, 762, 706, 663 cm^{-1} ; UV-vis (10^{-5} M in $PhCF_3$) λ_{max} (log ε) = 365 (5.08), 635 (4.86), 704 (5.55) nm, UV-vis (10^{-5} M in Dioxane) λ_{max} (log ε) = 364 (5.11), 636 (4.90), 708 (5.63) nm ; Fluorescence ($PhCF_3$) λ_{em} 719 nm, φ_f = 0.0352, Fluorescence (Dioxane) λ_{em} 724 nm, φ_f = 0.366.

MALDI-TOF MS (dithranol) : 3550.6-3558.5 ([M^+] isotopic pattern).



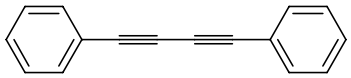
HPLC using an ODS columun (H_2O/MeCN/THF = 3.3/16.7/80, flow rate 0.3 mL/min, t_R = 27.9 min)



These assignments are in good accord with those in the literature.[7]

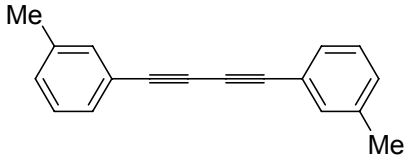
3. Homocoupling reaction of terminal alkynes in Solkane®365/227 (93/7)

1,4-Diphenylbuta-1,3-diyne (2a)



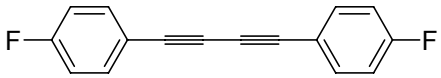
A mixture of terminal alkyne **1a** (54.9 μL, 0.50 mmol), TMEDA (7.5 μL, 0.050 mmol), CuCl (5.0 mg, 0.050 mmol) in Solkane®365/227 (93/7) (1.0 mL) was stirred under an atmosphere of air at rt. The resulting mixture was stirred at rt for 4.0 h. After dilution with sat. NH_4Cl aq, aqueous layer was extracted with CH_2Cl_2, and the combined organic layer was washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (*n*-hexane = 100) to give **2a** as a white solid (44.1 mg, 87%). ^{1}H NMR spectrum is completely much with homocoupling reaction of terminal alkynes 1a in Solkane®365mfc.

1,4-Di-*m*-tolylbuta-1,3-diyne (2c)



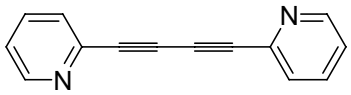
A mixture of terminal alkyne **1c** (64.5 μL, 0.50 mmol), TMEDA (7.5 μL, 0.050 mmol), DBU (74.7 μL, 0.50 mmol), CuCl (5.0 mg, 0.050 mmol) in Solkane®365/227 (93/7) (1.0 mL) was stirred under an atmosphere of air at rt. The resulting mixture was stirred at rt for 5.0 h. After dilution with sat. NH_4Cl aq, aqueous layer was extracted with CH_2Cl_2, and the combined organic layer was washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (*n*-hexane = 100) to give **2c** as a lightyellow solid (52.3 mg, 93%). ^{1}H NMR spectrum is completely much with homocoupling reaction of terminal alkynes 1c in Solkane®365mfc.

1,4-Bis(4-fluorophenyl)buta-1,3-diyne (2g)



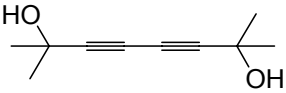
A mixture of terminal alkyne **1g** (57.3 μL, 0.50 mmol), TMEDA (7.5 μL, 0.050 mmol), CuCl (5.0 mg, 0.050 mmol) in Solkane®365/227 (93/7) (1.0 mL) was stirred under an atmosphere of air at rt. The resulting mixture was stirred at rt for 9.0 h. After dilution with sat. NH_4Cl aq, aqueous layer was extracted with CH_2Cl_2, and the combined organic layer was washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (*n*-hexane = 100) to give **2g** as a lightyellow solid (45.5 mg, 76%). ^{1}H NMR spectrum is completely much with homocoupling reaction of terminal alkynes 1g in Solkane®365mfc.

1,4-Di(pyridin-2-yl)buta-1,3-diyne (2m)



A mixture of terminal alkyne **1m** (50.5 μL, 0.50 mmol), TMEDA (7.5μL, 0.050 mmol), CuCl (5.0 mg, 0.050 mmol) in Solkane®365/227 (93/7) (1.0 mL) was stirred under an atmosphere of air at rt. The resulting mixture was stirred at rt for 9.0 h. After dilution with sat. NH_4Cl aq, aqueous layer was extracted with CH_2Cl_2, and the combined organic layer was washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (*n*-hexane/ethyl acetate = 70/30) to give **2m** as a darkkhaki solid (44.6 mg, 87%). ^{1}H NMR spectrum is completely much with homocoupling reaction of terminal alkynes 1m in Solkane®365mfc.

2,7-Dimethylocta-3,5-diyne-2,7-diol (2o)



A mixture of terminal alkyne **1o** (48.5 μL, 0.50 mmol), TMEDA (7.5 μL, 0.050 mmol), CuCl (5.0 mg, 0.050 mmol) in Solkane®365/227 (93/7) (1.0 mL) was stirred under an atmosphere of air at rt. The resulting mixture was stirred at rt for 3.0 h. After dilution with sat. NH_4Cl aq, aqueous layer was extracted with CH_2Cl_2, and the combined organic layer was washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (*n*-hexane/ethyl acetate = 70/30) to give **2o** as a white solid (38.6 mg, 93%). ^{1}H NMR spectrum is completely much with homocoupling reaction of terminal alkynes 1o in Solkane®365mfc.

4. References

1) S. Adimurthy, C. C. Malakar and U. Beifuss, *J. Org. Chem.*, 2009, **74**, 5648.

2) Z. W. Chen, H. F. Jiang, A. Z. Wang and S. R. Yang, *J. Org. Chem.*, 2010, **75**, 6700.

3) L. Zhou, H. Y. Zhan, H. L. Liu and H. F. Jiang, *Chin. J. Chem.*, 2007, **25**, 1413.

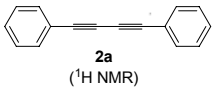
4) G. T. Crisp and B. L. Flynn, *J. Org. Chem.*, 1993, **58**, 6614.

5) A. Lei, M. Srivastava and X. Zhang, *J. Org. Chem.*, 2002, **67**, 1969.

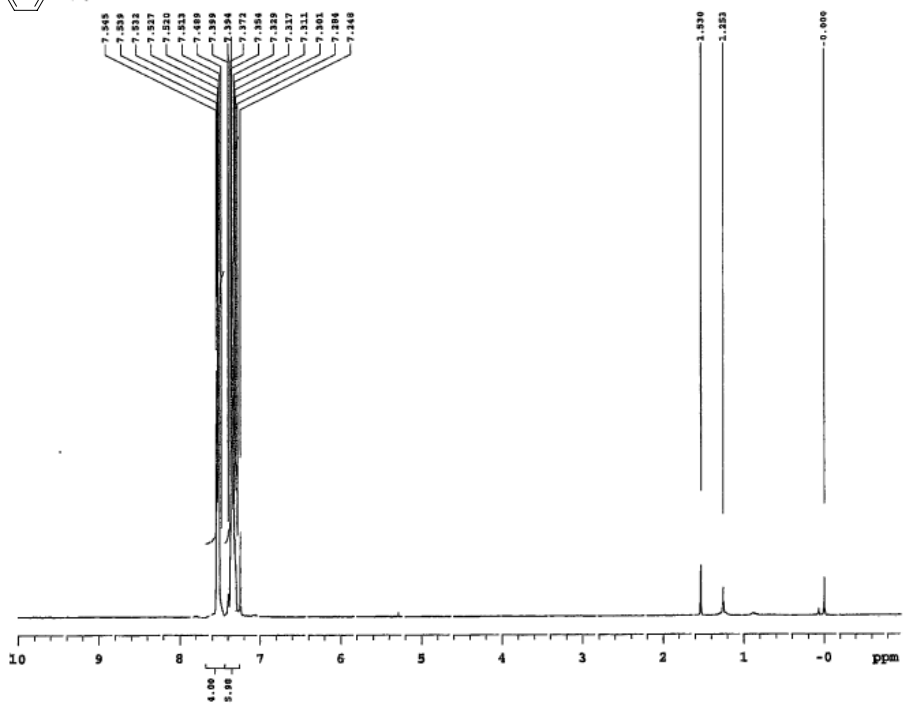
6) D. Wang, J. H. Li, N. Li, T. T. Gao, S. H. Hou and B. H. Chen, *Green Chem.*, 2010, **12**, 45.

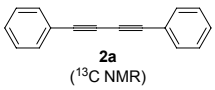
7) H. Yoshiyama, N. Shibata, T. Sato, S. Nakamura and T. Toru, *Chem. Commun.*, 2008, 1977.

5. NMR chart

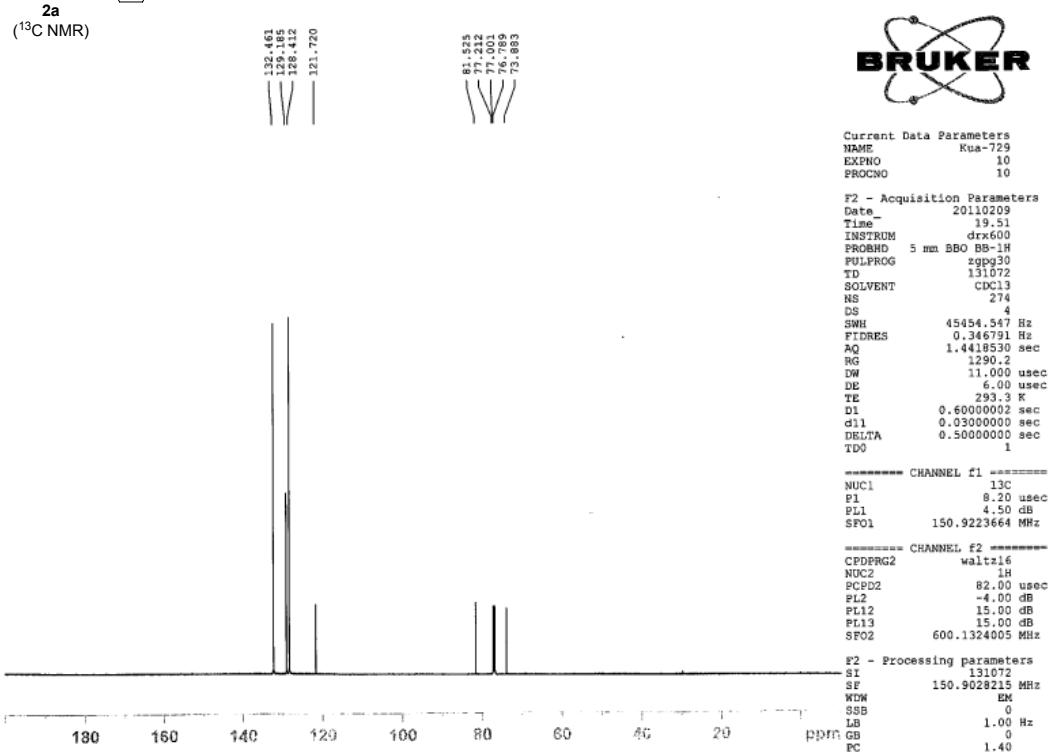


2a
(^{1}H NMR)





2a
(^{13}C NMR)



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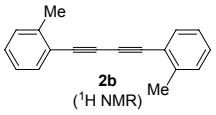
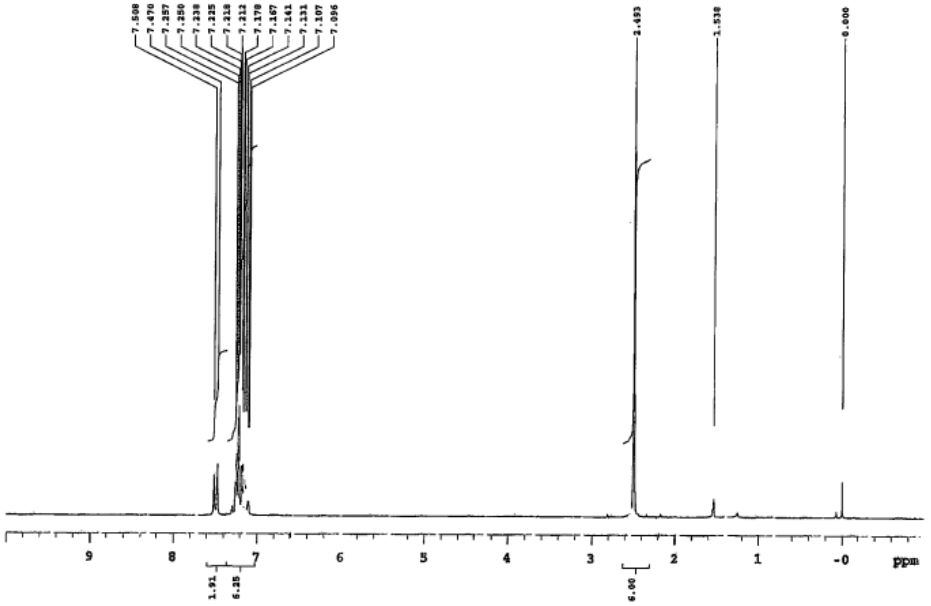
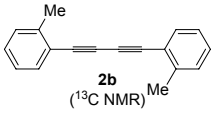
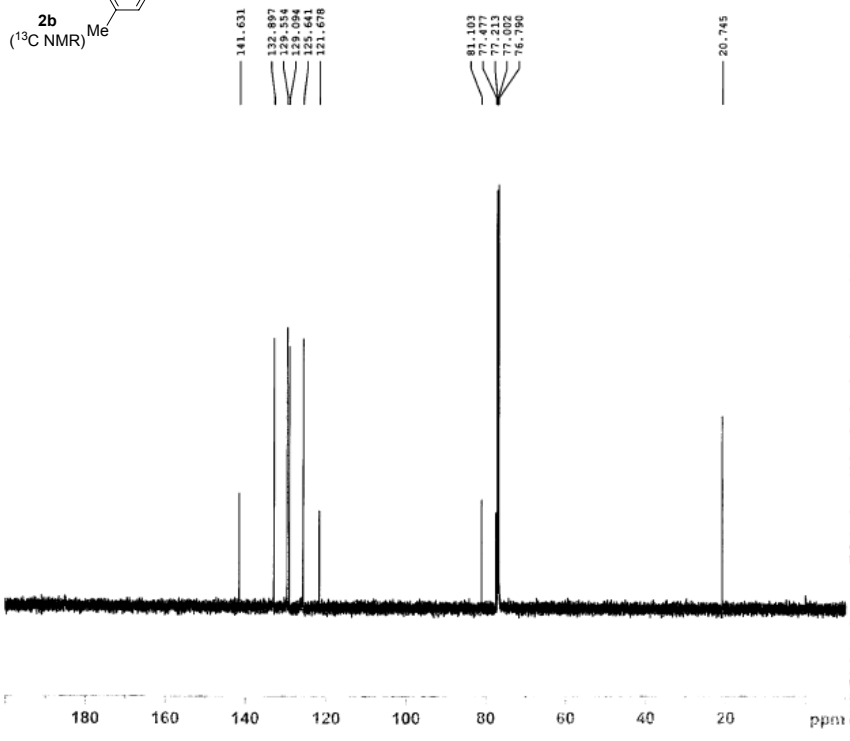


2b
(^{1}H NMR)





2b
(^{13}C NMR)

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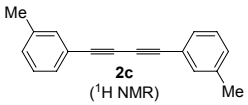
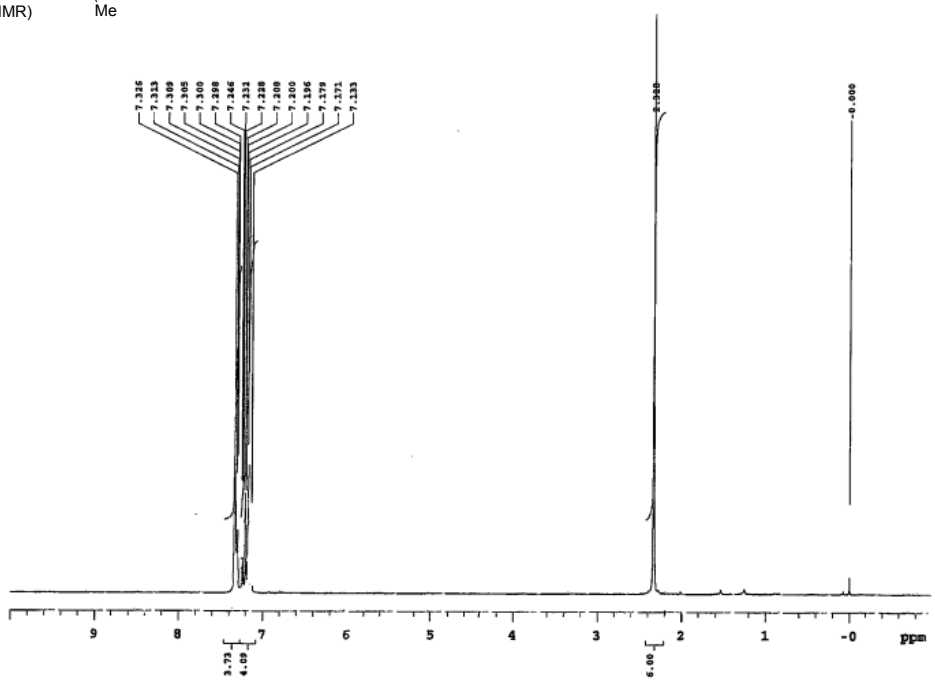
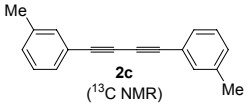
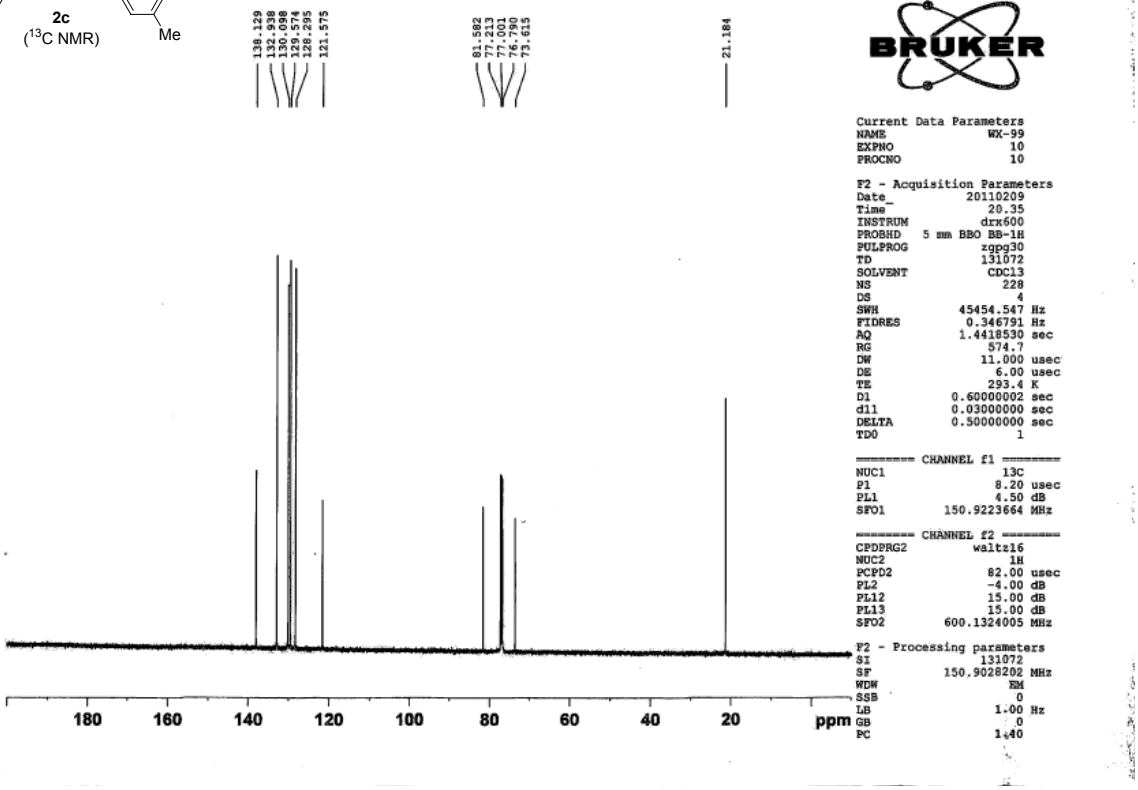


2c
(^{1}H NMR)





2c
(^{13}C NMR)



BRUKER

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FIDRES             0.346791 Hz
AQ                 1.4418530 sec
RG                     574.7
DW                  11.000 usec
DE                   6.00 usec
TE                   293.4 K
D1                0.60000002 sec
d11               0.03000000 sec
DELTA             0.50000000 sec
TD0                       1

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P1                   8.20 usec
PL1                  4.50 dB
SFO1           150.9223664 MHz

======== CHANNEL f2 ========
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NUC2                    1H
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PL12                15.00 dB
PL13                15.00 dB
SFO2           600.1324005 MHz

F2 - Processing parameters
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PC                      1.40
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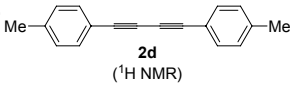
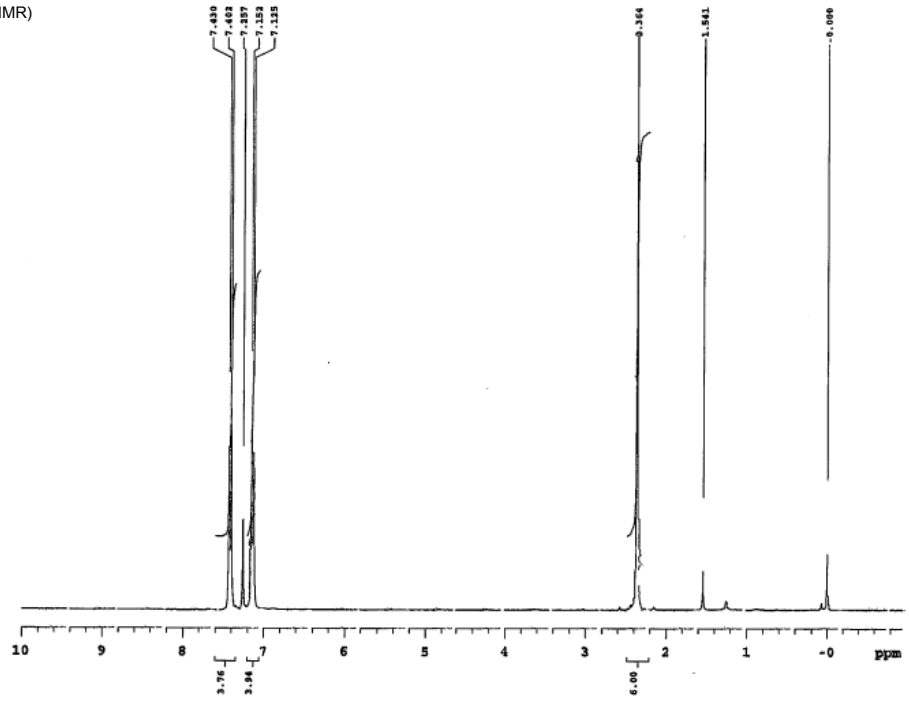
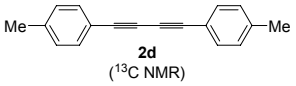
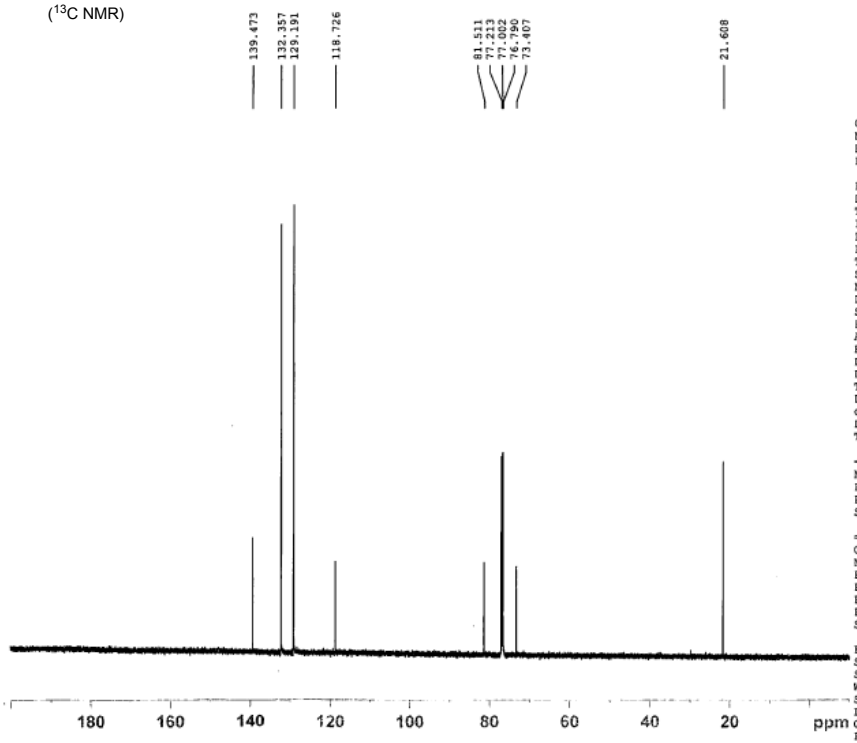


2d
(^{1}H NMR)





2d
(^{13}C NMR)



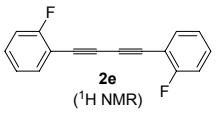

```
Current Data Parameters
NAME                  KUA-426
EXPNO                      10
PROCNO                     10

F2 - Acquisition Parameters
Date_               20110209
Time                   20.54
INSTRUM                drx600
PROBHD     5 mm BBO BB-1H
PULPROG              zgpg30
TD                     131072
SOLVENT                 CDCl3
NS                        145
DS                          4
SWH                 45454.547 Hz
FIDRES              0.346791 Hz
AQ                  1.4418530 sec
RG                     1149.4
DW                    11.000 usec
DE                     6.00 usec
TE                     293.4 K
D1                0.60000002 sec
d11               0.03000000 sec
DELTA             0.50000000 sec
TD0                         1

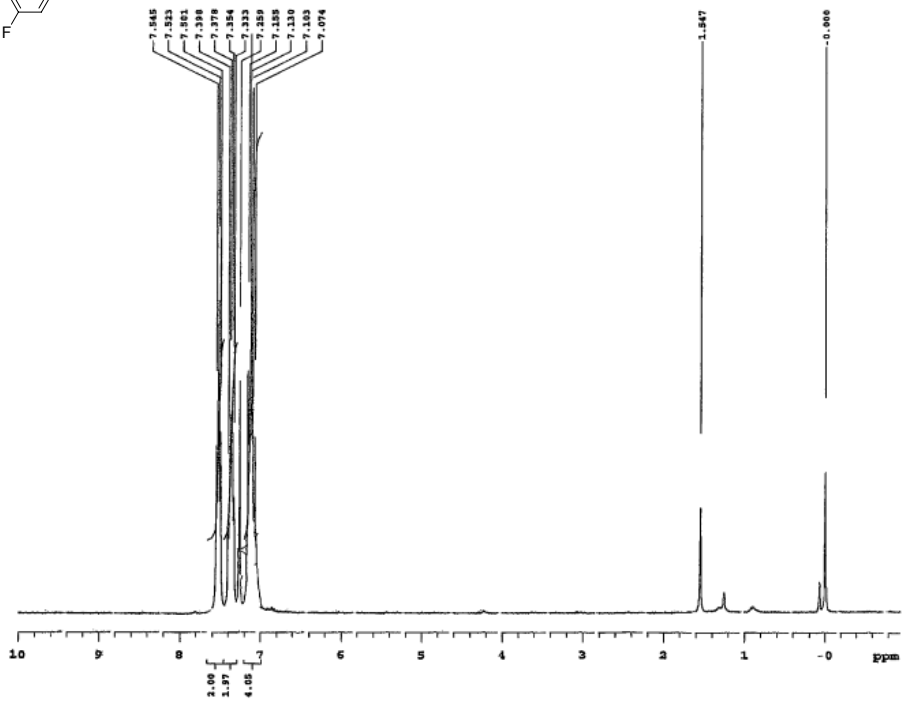
======== CHANNEL f1 ========
NUC1                      13C
P1                       8.20 usec
PL1                      4.50 dB
SFO1             150.9223664 MHz

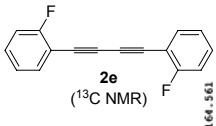
======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   82.00 usec
PL2                     -4.00 dB
PL12                    15.00 dB
PL13                    15.00 dB
SFO2             600.1324005 MHz

F2 - Processing parameters
SI                     131072
SF               150.9028173 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40
```

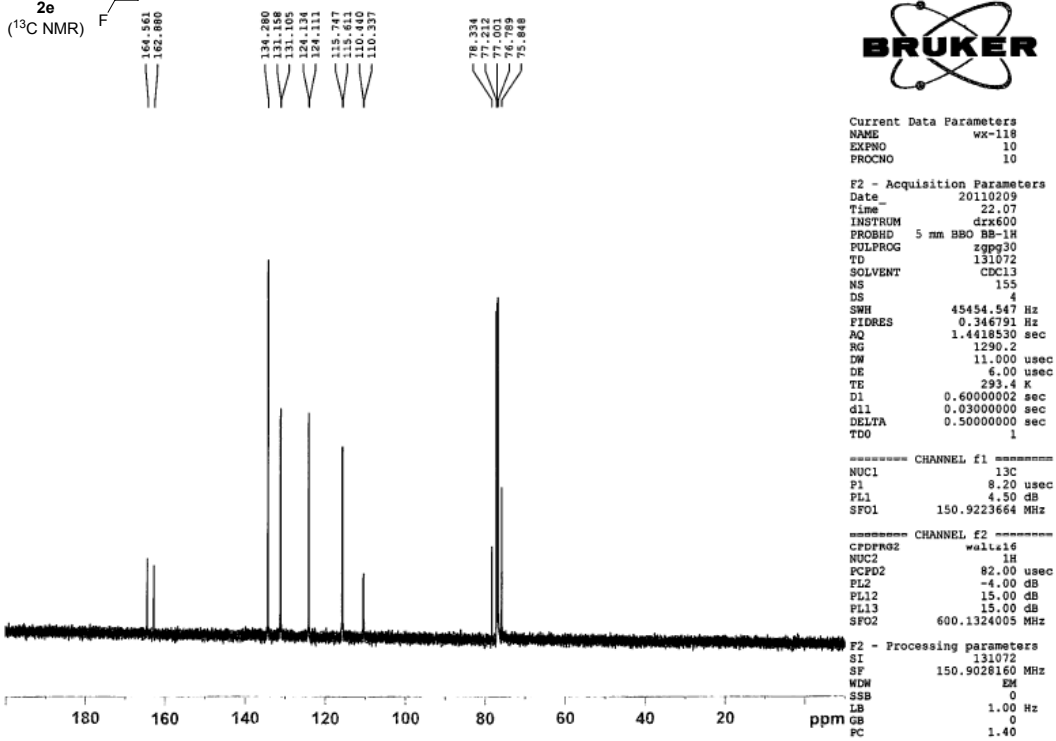


2e
(^{1}H NMR)





2e
(^{13}C NMR)



BRUKER

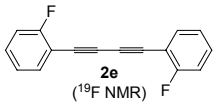
```
Current Data Parameters
NAME                  wx-118
EXPNO                     10
PROCNO                    10

F2 - Acquisition Parameters
Date_              20110209
Time                 22.07
INSTRUM              drx600
PROBHD   5 mm BBO BB-1H
PULPROG            zgpg30
TD                 131072
SOLVENT               CDCl3
NS                      155
DS                        4
SWH              45454.547 Hz
FIDRES           0.346791 Hz
AQ               1.4418530 sec
RG                  1290.2
DW                  11.000 usec
DE                   6.00 usec
TE                  293.4 K
D1              0.60000002 sec
d11             0.03000000 sec
DELTA           0.50000000 sec
TD0                       1

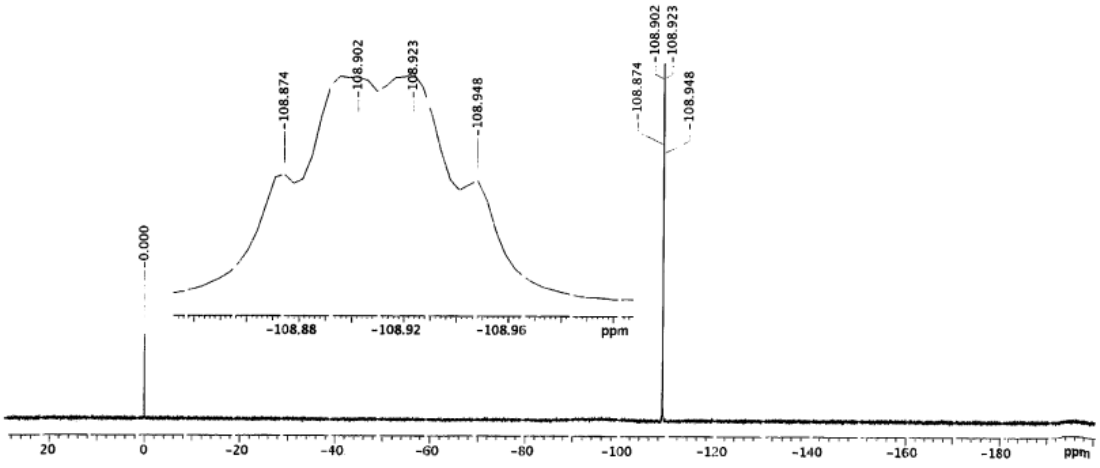
======== CHANNEL f1 ========
NUC1                    13C
P1                     8.20 usec
PL1                    4.50 dB
SFO1          150.9223664 MHz

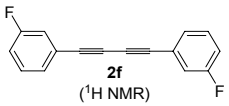
======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                    1H
PCPD2                 82.00 usec
PL2                   -4.00 dB
PL12                  15.00 dB
PL13                  15.00 dB
SFO2          600.1324005 MHz

F2 - Processing parameters
SI                    131072
SF            150.9028160 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40
```

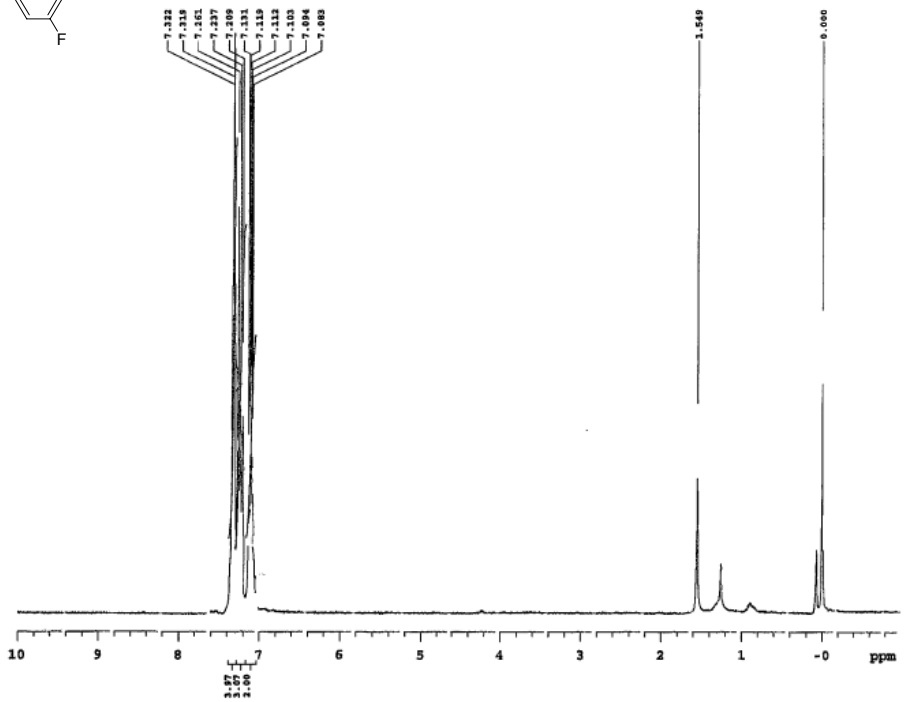


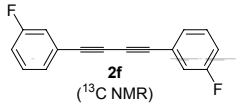
2e
(^{19}F NMR)



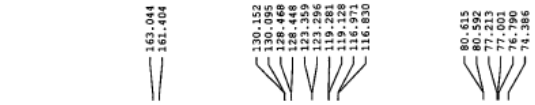


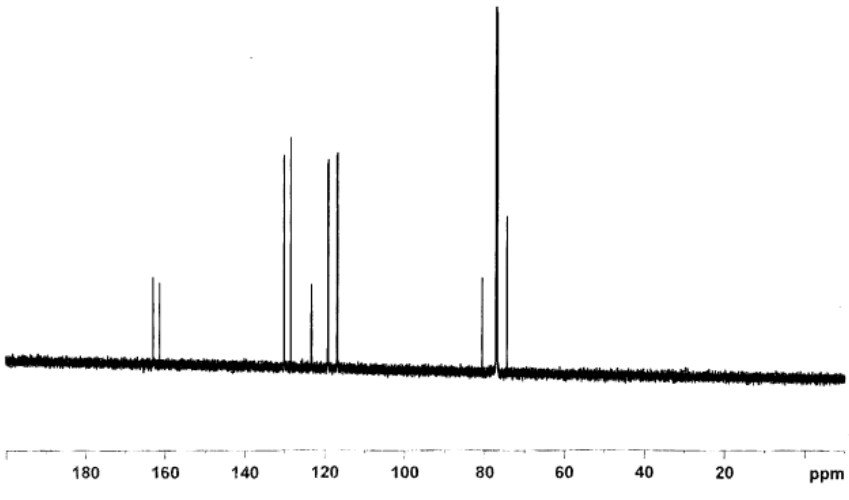
2f
(^{1}H NMR)





2f
(^{13}C NMR)






```
Current Data Parameters
NAME                  wx-113
EXPNO                     10
PROCNO                    10

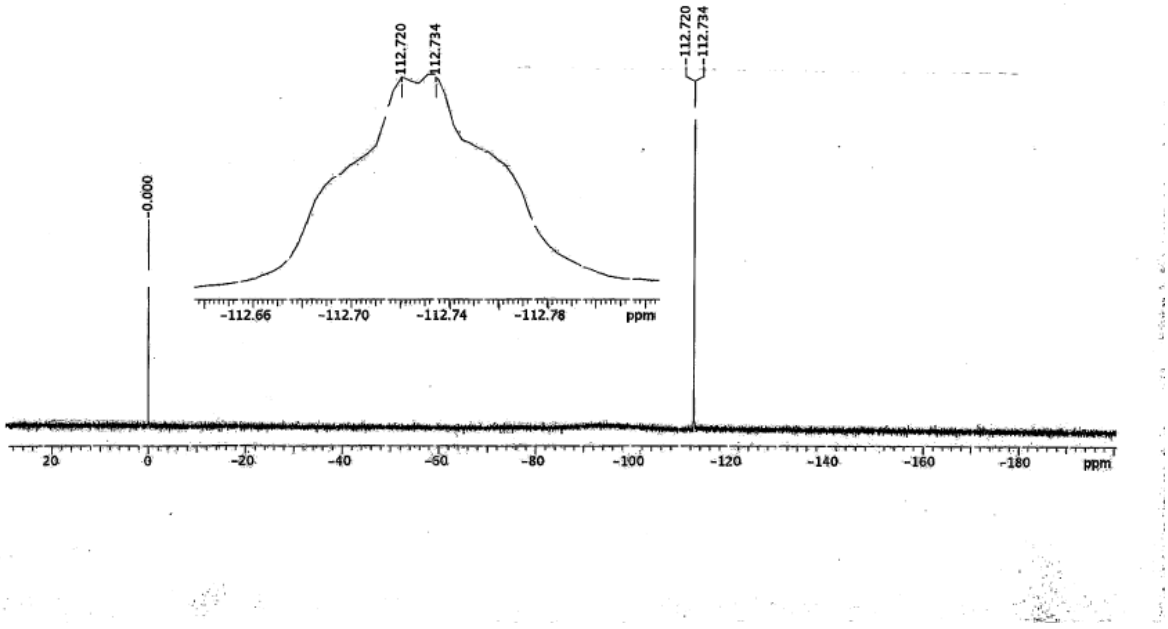
F2 - Acquisition Parameters
Date_              20110209
Time                  22.41
INSTRUM              drx600
PROBHD    5 mm BBO BB-1H
PULPROG            zgpg30
TD                 131072
SOLVENT               CDCl3
NS                      103
DS                        4
SWH               45454.547 Hz
FIDRES            0.346791 Hz
AQ                1.4418530 sec
RG                    574.7
DW                 11.000 usec
DE                  6.00 usec
TE                   293.4 K
D1              0.60000002 sec
d11             0.03000000 sec
DELTA           0.50000000 sec
TD0                       1

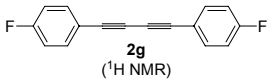
========== CHANNEL f1 ========
NUC1                    13C
P1                   8.20 usec
PL1                  4.50 dB
SFO1          150.9223664 MHz

========== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                    1H
PCPD2               82.00 usec
PL2                 -4.00 dB
PL12                15.00 dB
PL13                15.00 dB
SFO2          600.1324005 MHz

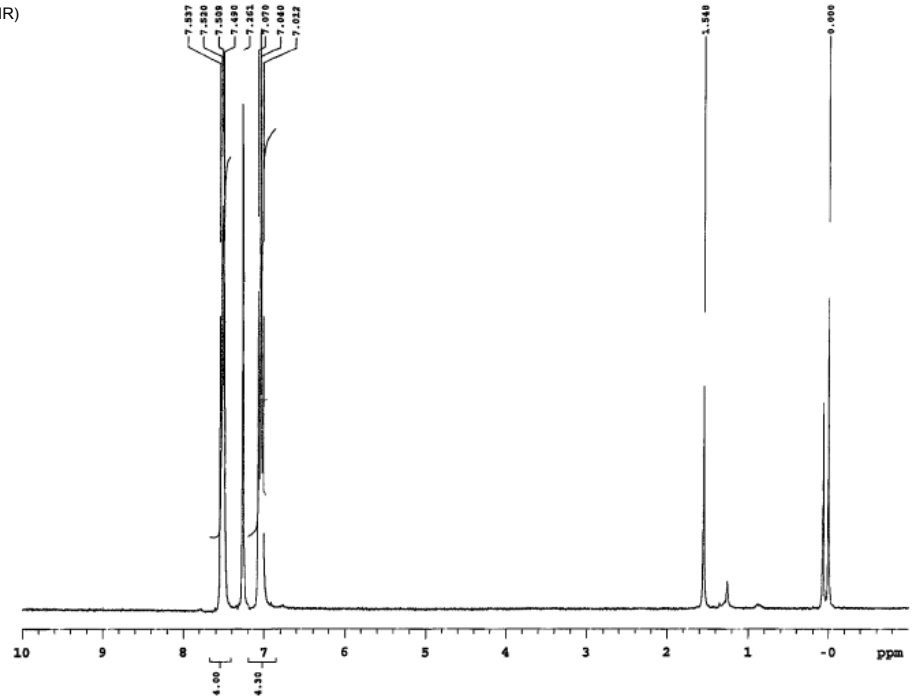
F2 - Processing parameters
SI                    131072
SF            150.9028152 MHz
WDW                       EM
SSB                        0
LB                    1.00 Hz
GB                         0
PC                      1.40
```

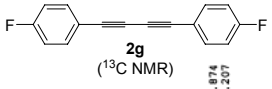
2f
(^{19}F NMR)



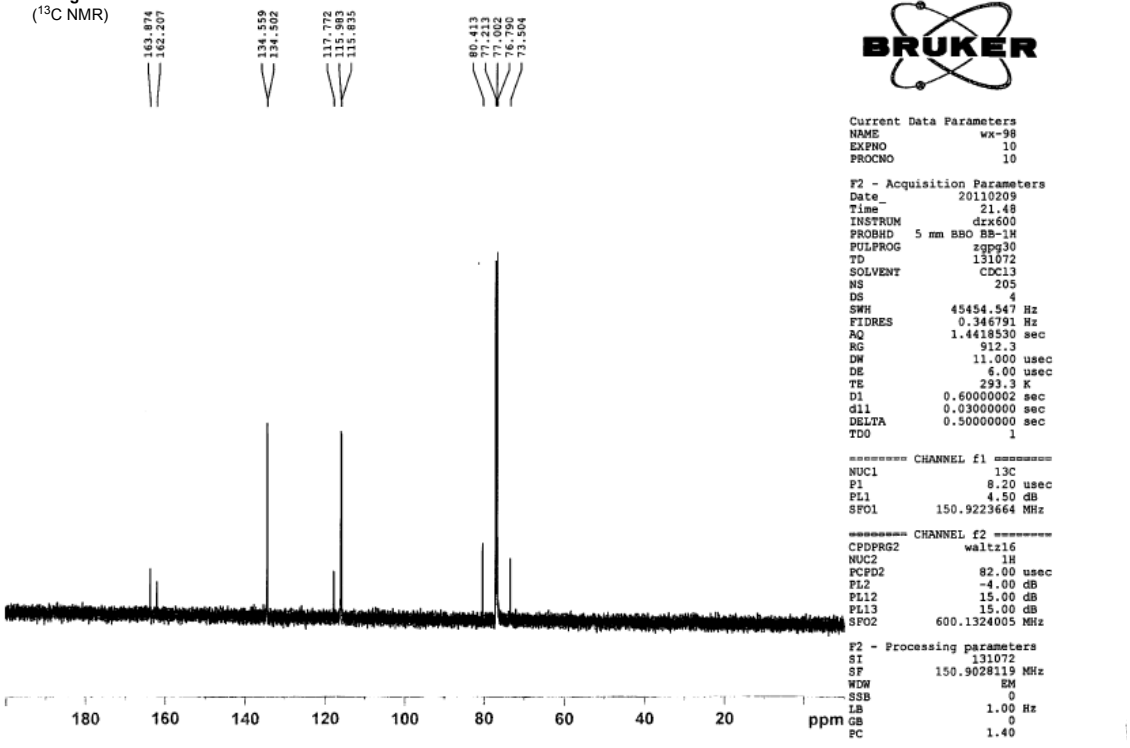


2g
(^{1}H NMR)





2g
(^{13}C NMR)



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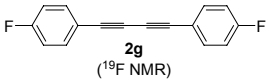
```
Current Data Parameters
NAME                  wx-98
EXPNO                    10
PROCNO                   10

F2 - Acquisition Parameters
Date_              20110209
Time                 21.48
INSTRUM             drx600
PROBHD   5 mm BBO BB-1H
PULPROG            zgpg30
TD                 131072
SOLVENT               CDCl3
NS                      205
DS                        4
SWH                45454.547 Hz
FIDRES             0.346791 Hz
AQ                 1.4418530 sec
RG                     912.3
DW                    11.000 usec
DE                     6.00 usec
TE                    293.3 K
D1                0.60000002 sec
d11               0.03000000 sec
DELTA             0.50000000 sec
TD0                       1

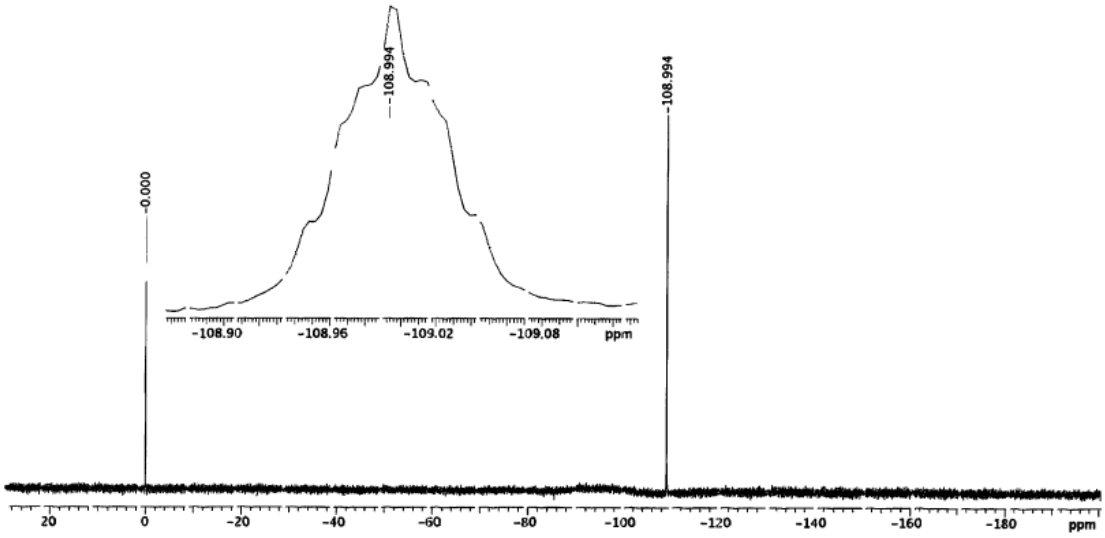
======== CHANNEL f1 ========
NUC1                    13C
P1                     8.20 usec
PL1                    4.50 dB
SFO1           150.9223664 MHz

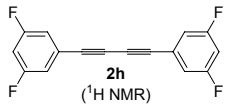
======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                    1H
PCPD2                 82.00 usec
PL2                   -4.00 dB
PL12                  15.00 dB
PL13                  15.00 dB
SFO2           600.1324005 MHz

F2 - Processing parameters
SI                    131072
SF             150.9028119 MHz
WDW                       EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```

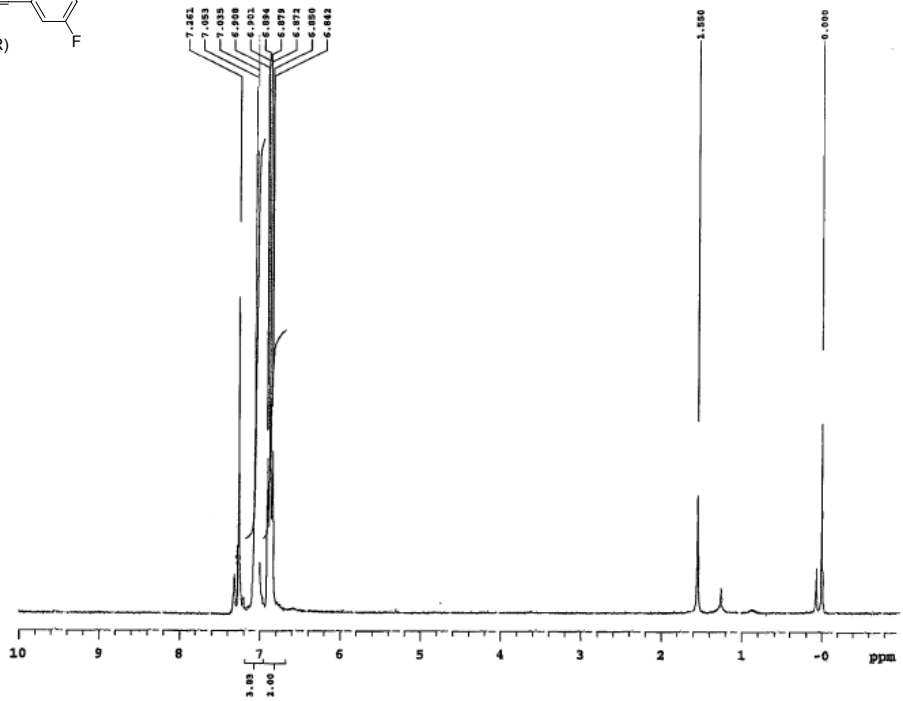


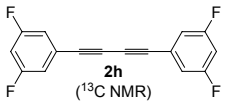
2g
(^{19}F NMR)



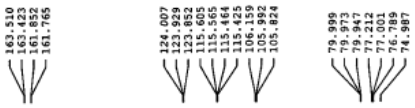


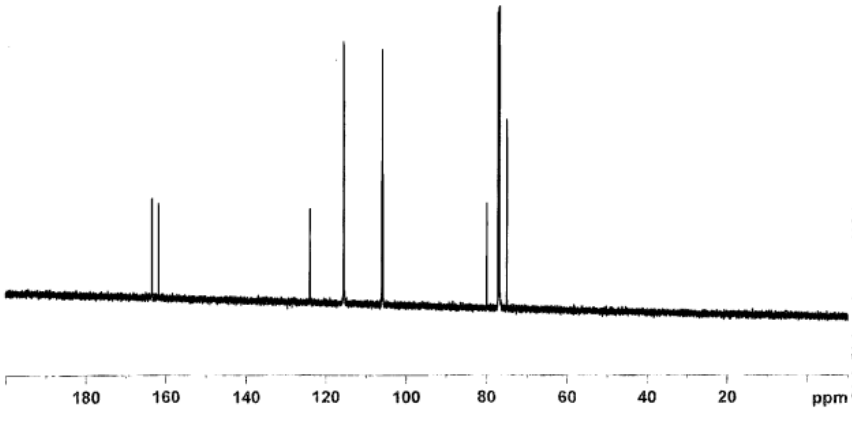
2h
(^{1}H NMR)





2h
(^{13}C NMR)





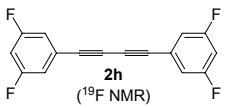

```
Current Data Parameters
NAME                  wx-105
EXPNO                     10
PROCNO                    10

F2 - Acquisition Parameters
Date_              20110209
Time                  22.23
INSTRUM             drx600
PROBHD    5 mm BBO BB-1H
PULPROG             zgpg30
TD                  131072
SOLVENT               CDCl3
NS                      194
DS                        4
SWH               45454.547 Hz
FIDRES             0.346791 Hz
AQ                 1.4418530 sec
RG                    574.7
DW                   11.000 usec
DE                    6.00 usec
TE                   293.4 K
D1                0.60000002 sec
d11               0.03000000 sec
DELTA             0.50000000 sec
TD0                       1

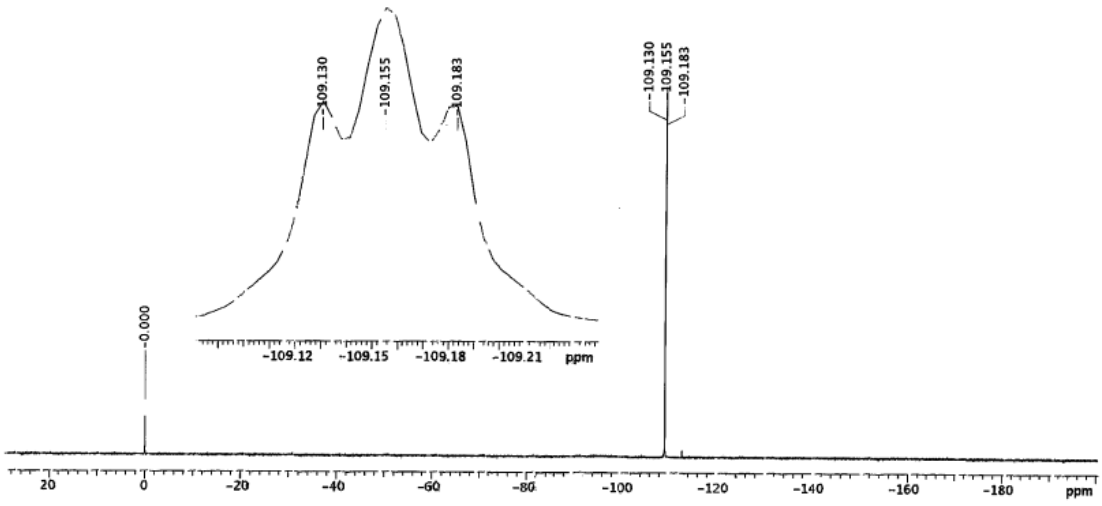
========== CHANNEL f1 ========
NUC1                    13C
P1                     8.20 usec
PL1                    4.50 dB
SFO1            150.9223664 MHz

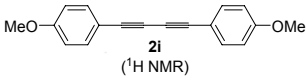
========== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                    1H
PCPD2                 82.00 usec
PL2                   -4.00 dB
PL12                  15.00 dB
PL13                  15.00 dB
SFO2            600.1324005 MHz

F2 - Processing parameters
SI                      131072
SF              150.9028125 MHz
WDW                        EM
SSB                         0
LB                      1.00 Hz
GB                          0
PC                      1.40
```

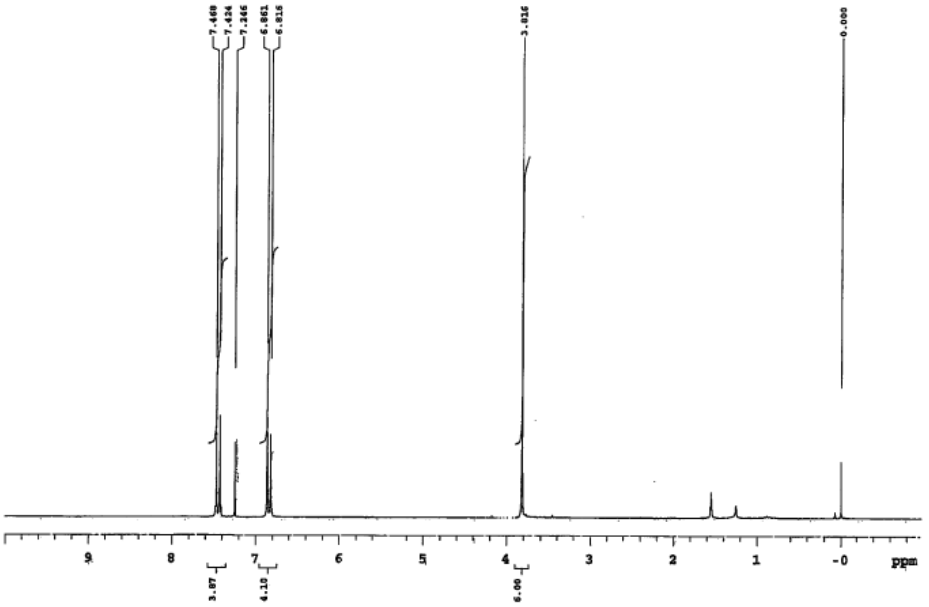


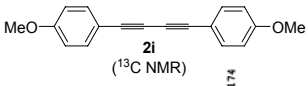
2h
(^{19}F NMR)



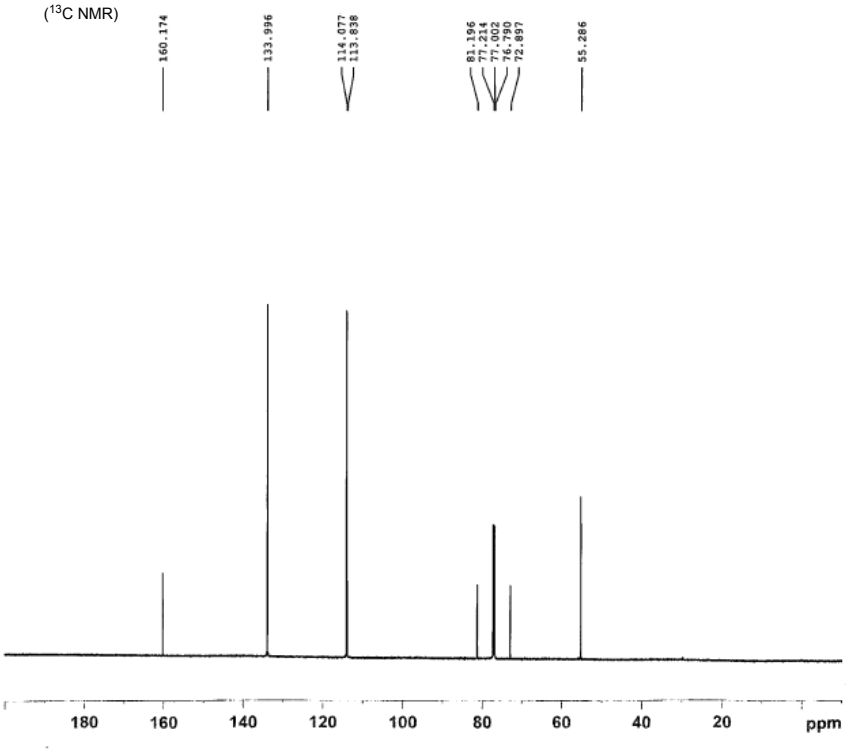


2i
(^{1}H NMR)





2i
(^{13}C NMR)



BRUKER

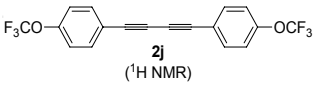
```
Current Data Parameters
NAME                  wx-103
EXPNO                     10
PROCNO                    10

F2 - Acquisition Parameters
Date_              20110209
Time                  19.26
INSTRUM              drx600
PROBHD   5 mm BBO BB-1H
PULPROG              zgpg30
TD                   131072
SOLVENT                CDCl3
NS                       332
DS                         4
SWH               45454.547 Hz
FIDRES             0.346791 Hz
AQ                1.4418530 sec
RG                    645.1
DW                 11.000 usec
DE                  6.00 usec
TE                  298.1 K
D1                0.60000002 sec
d11               0.03000000 sec
DELTA             0.50000000 sec
TD0                        1

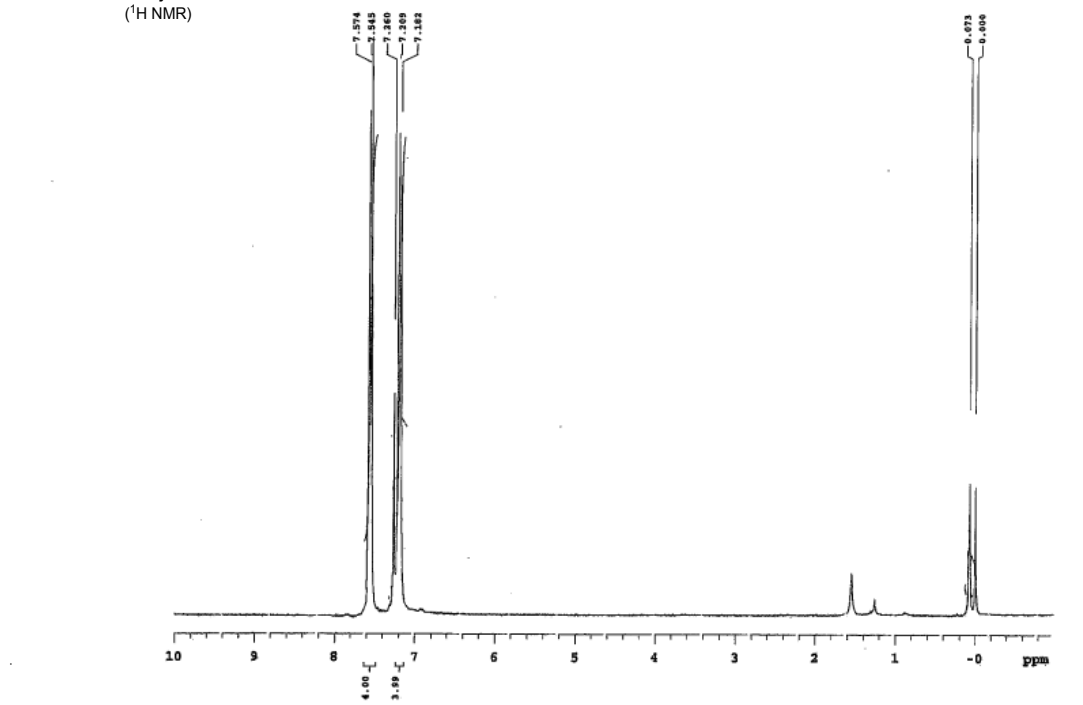
======== CHANNEL f1 ========
NUC1                     13C
P1                    8.20 usec
PL1                   4.50 dB
SFO1           150.9223664 MHz

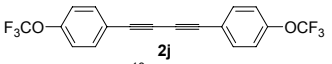
======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                      1H
PCPD2                82.00 usec
PL2                  -4.00 dB
PL12                 15.00 dB
PL13                 15.00 dB
SFO2           600.1324005 MHz

F2 - Processing parameters
SI                   131072
SF             150.9028193 MHz
WDW                      EM
SSB                       0
LB                 1.00 Hz
GB                        0
PC                     1.40
```

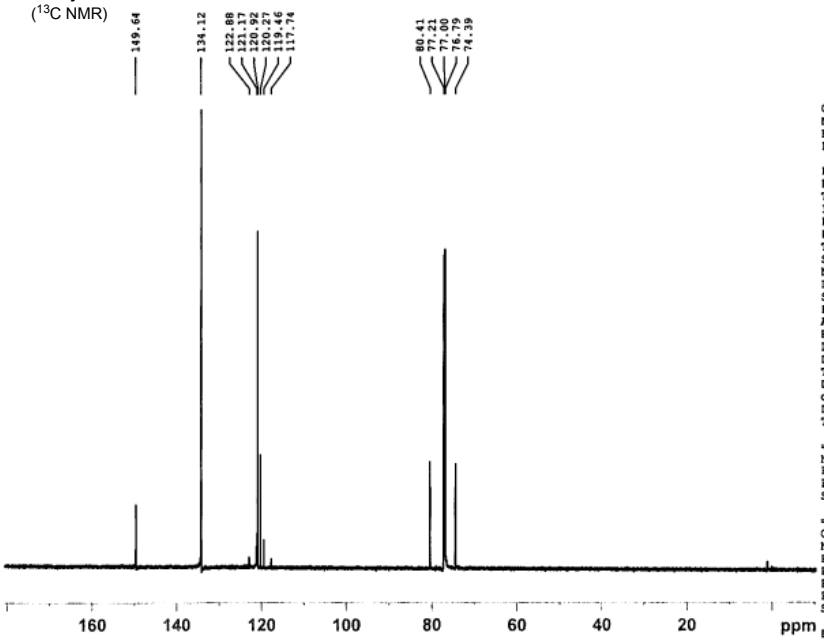


2j
(^{1}H NMR)





2j
(^{13}C NMR)



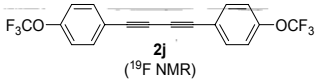

```
Current Data Parameters
NAME                  wx-114
EXPNO                     10
PROCNO                    10

F2 - Acquisition Parameters
Date_               20110106
Time                   17.40
INSTRUM                drx600
PROBHD   5 mm BBO BB-1H
PULPROG              zgpg30
TD                   131072
SOLVENT                CDCl3
NS                       757
DS                         4
SWH                45454.547 Hz
FIDRES                 0.346791 Hz
AQ                     1.4418530 sec
RG                       812.7
DW                      11.000 usec
DE                       6.00 usec
TE                      293.9 K
D1                  0.60000002 sec
d11                 0.03000000 sec
DELTA               0.50000000 sec
TD0                          1

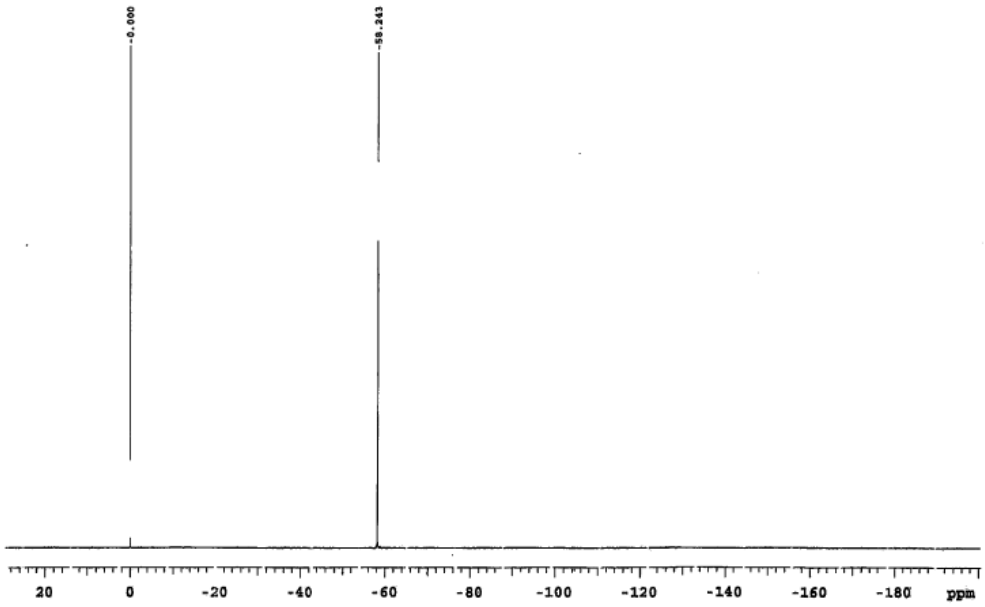
======== CHANNEL f1 ========
NUC1                       13C
P1                        8.20 usec
PL1                       4.50 dB
SFO1              150.9223664 MHz

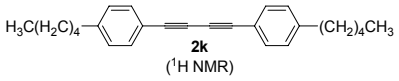
======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                    82.00 usec
PL2                      -4.00 dB
PL12                     15.00 dB
PL13                     15.00 dB
SFO2              600.1324005 MHz

F2 - Processing parameters
SI                       131072
SF                150.9028114 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40
```

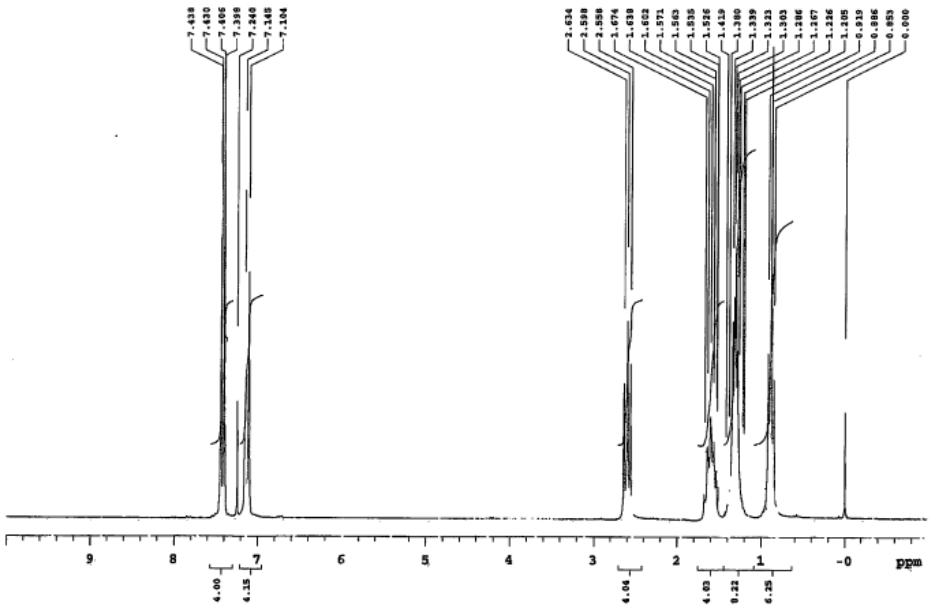


2j
(^{19}F NMR)



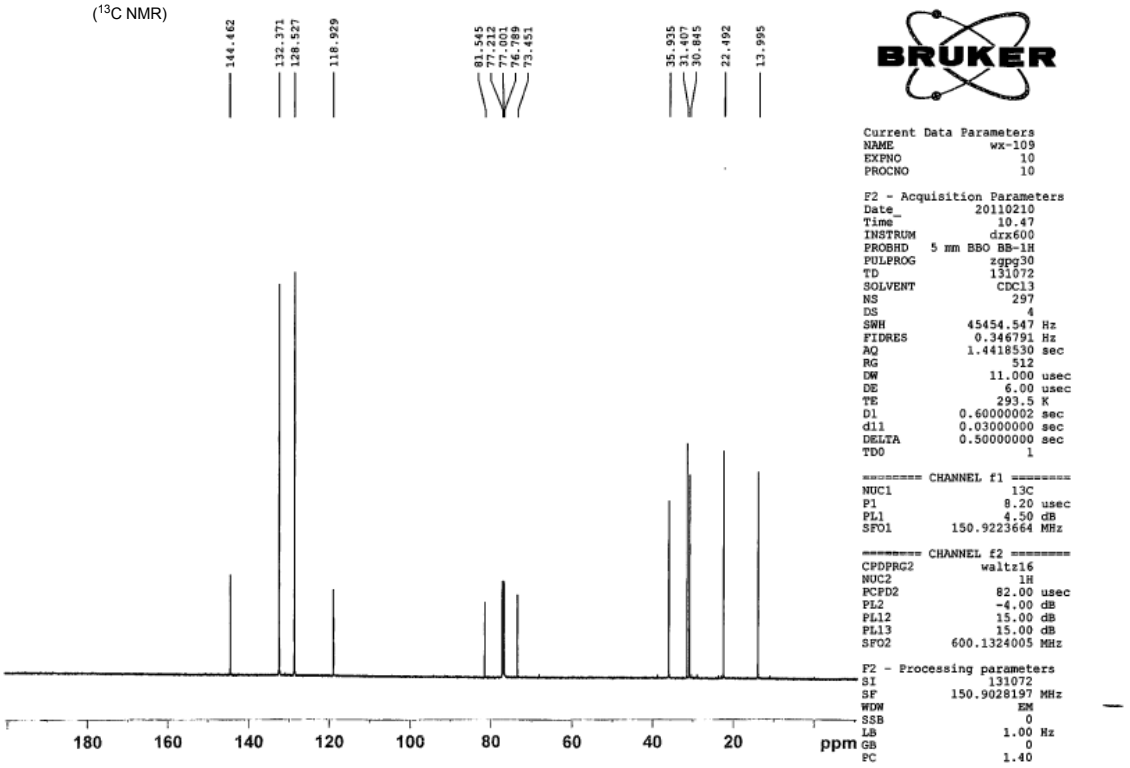


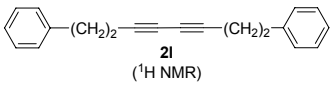
2k
(^{1}H NMR)



2k

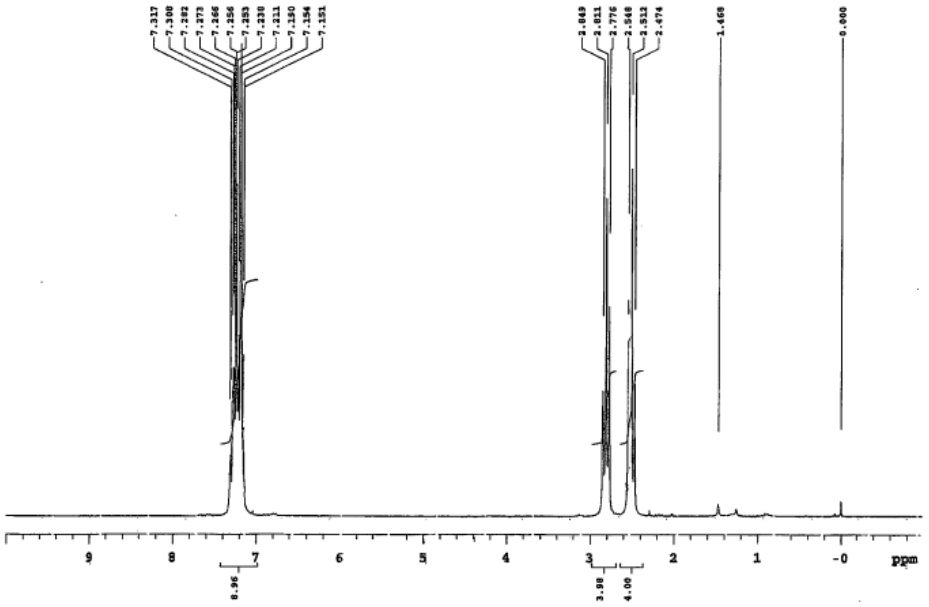
(^{13}C NMR)

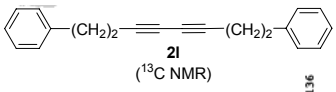




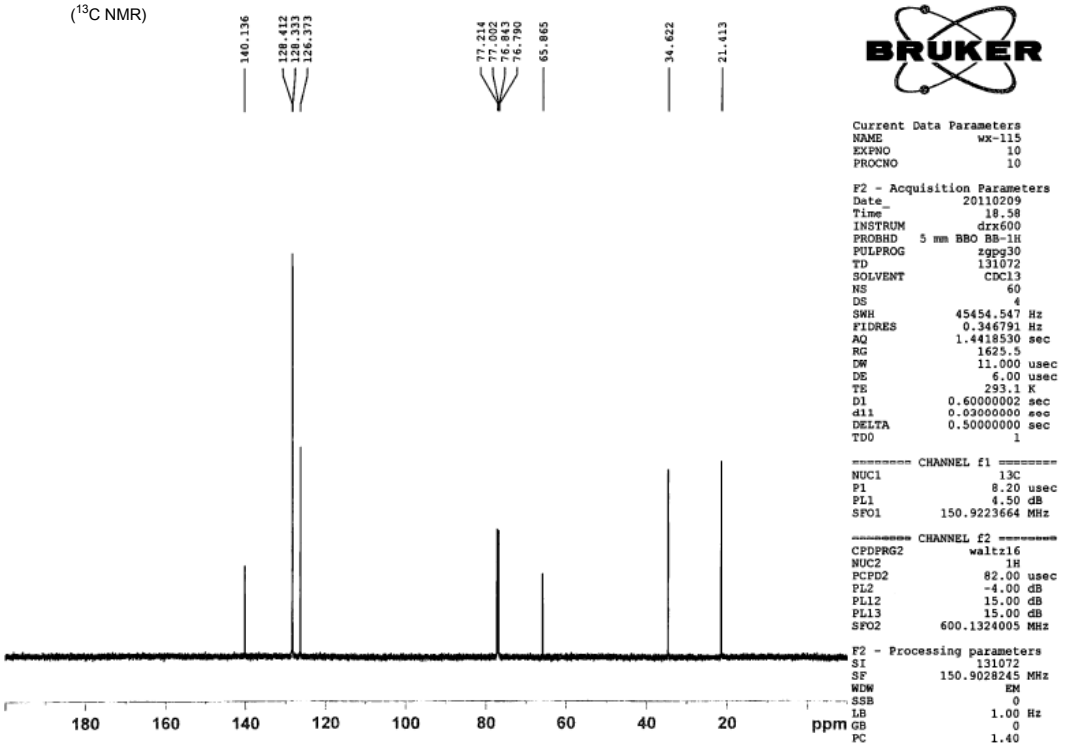
2l

(^{1}H NMR)





2l
(^{13}C NMR)

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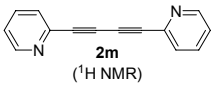
Current Data Parameters
NAME                  wx-115
EXPNO                     10
PROCNO                    10

F2 - Acquisition Parameters
Date_               20110209
Time                   18.58
INSTRUM                drx600
PROBHD    5 mm BBO BB-1H
PULPROG                zgpg30
TD                     131072
SOLVENT                CDCl3
NS                         60
DS                          4
SWH                45454.547 Hz
FIDRES             0.346791 Hz
AQ                 1.4418530 sec
RG                    1625.5
DW                    11.000 usec
DE                     6.00 usec
TE                    293.1 K
D1                 0.60000002 sec
d11                0.03000000 sec
DELTA              0.50000000 sec
TD0                         1

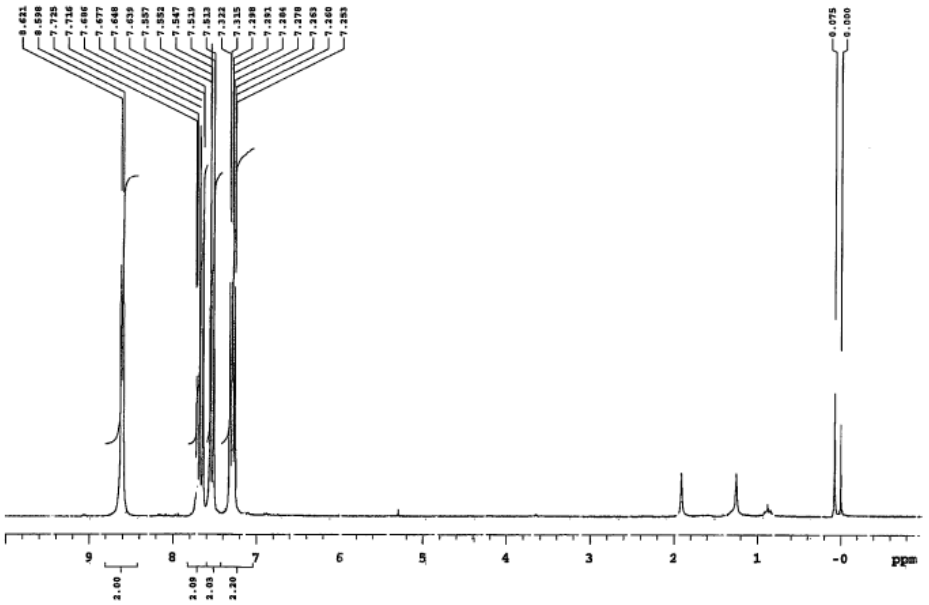
======== CHANNEL f1 ========
NUC1                      13C
P1                       8.20 usec
PL1                      4.50 dB
SFO1            150.9223664 MHz

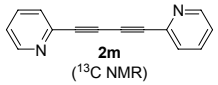
======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                      1H
PCPD2                   82.00 usec
PL2                     -4.00 dB
PL12                    15.00 dB
PL13                    15.00 dB
SFO2            600.1324005 MHz

F2 - Processing parameters
SI                        131072
SF              150.9028245 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40
```

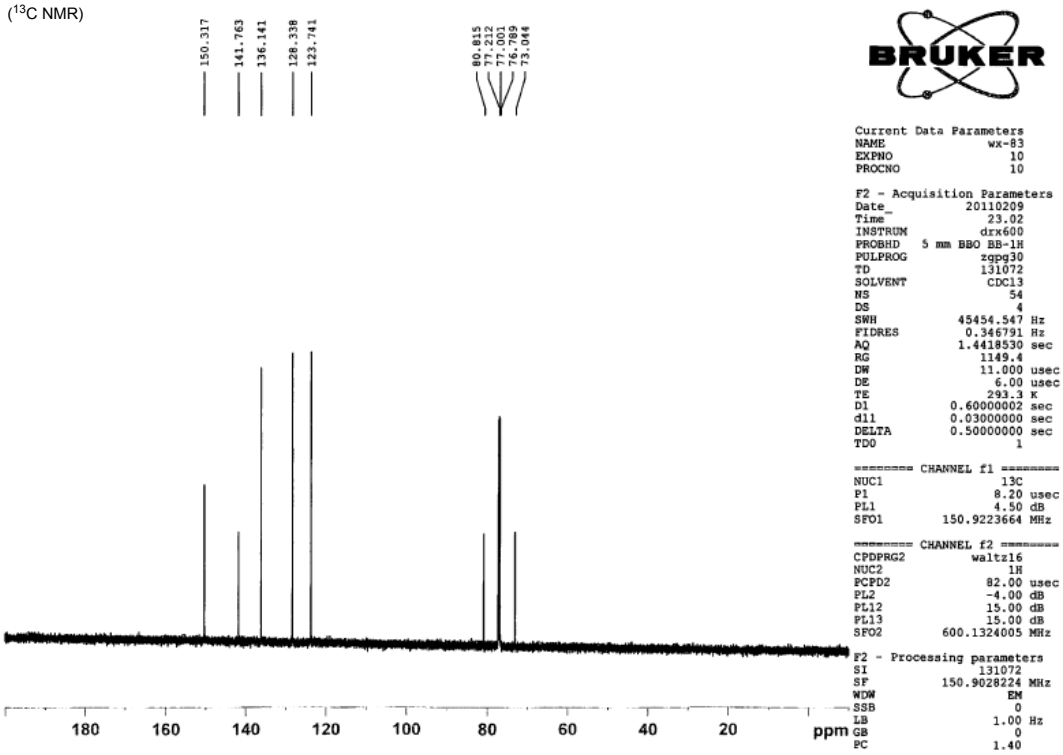


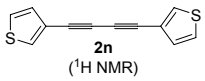
2m
(^{1}H NMR)



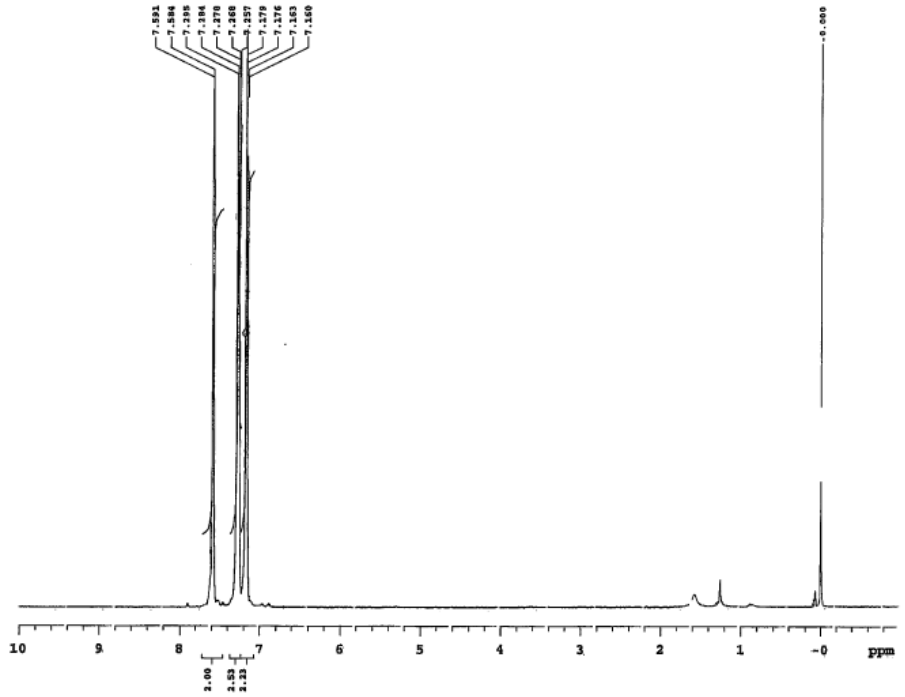


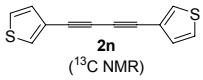
2m
(^{13}C NMR)



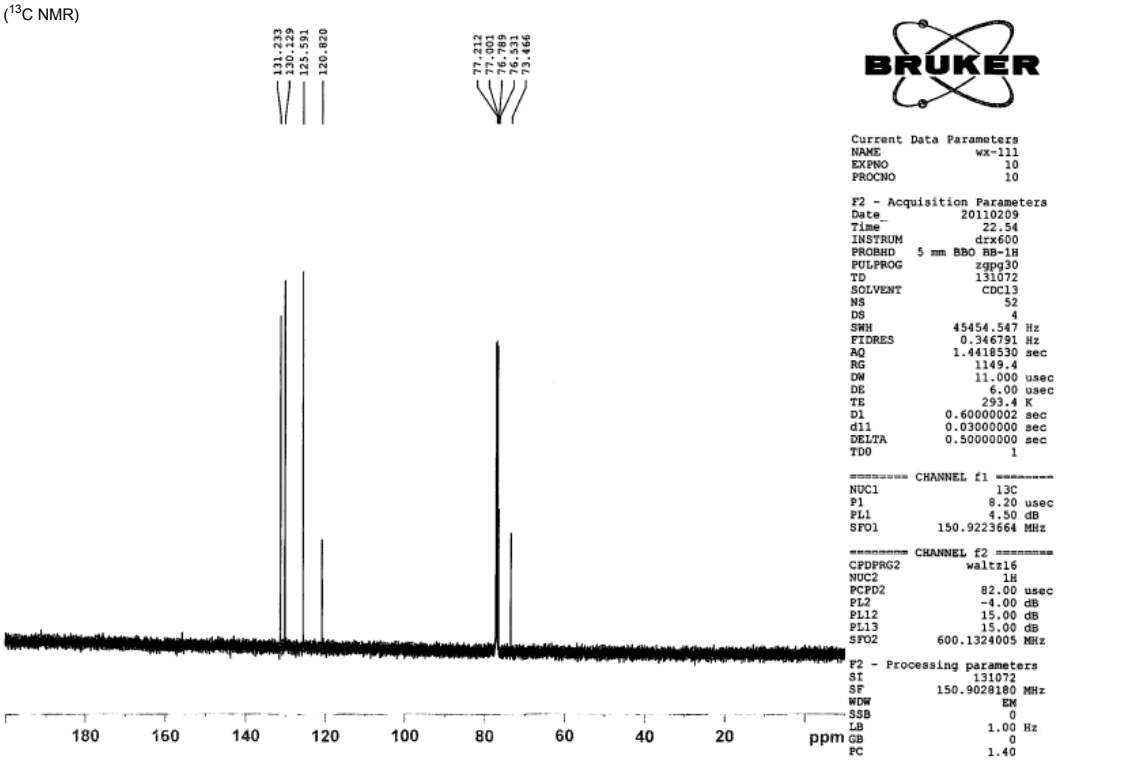


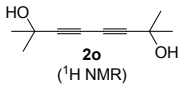
2n
(^{1}H NMR)



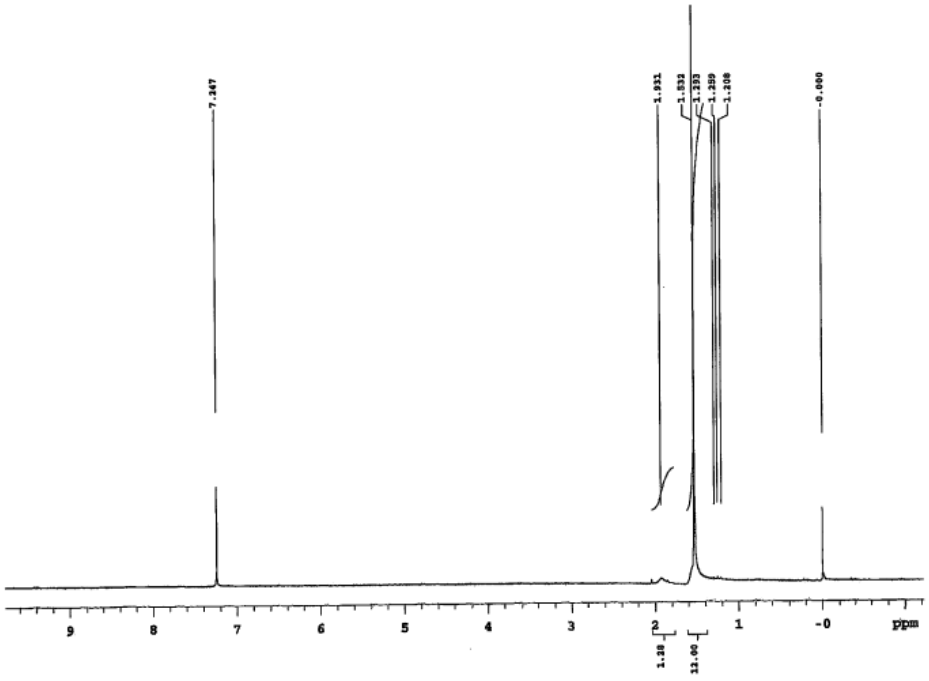


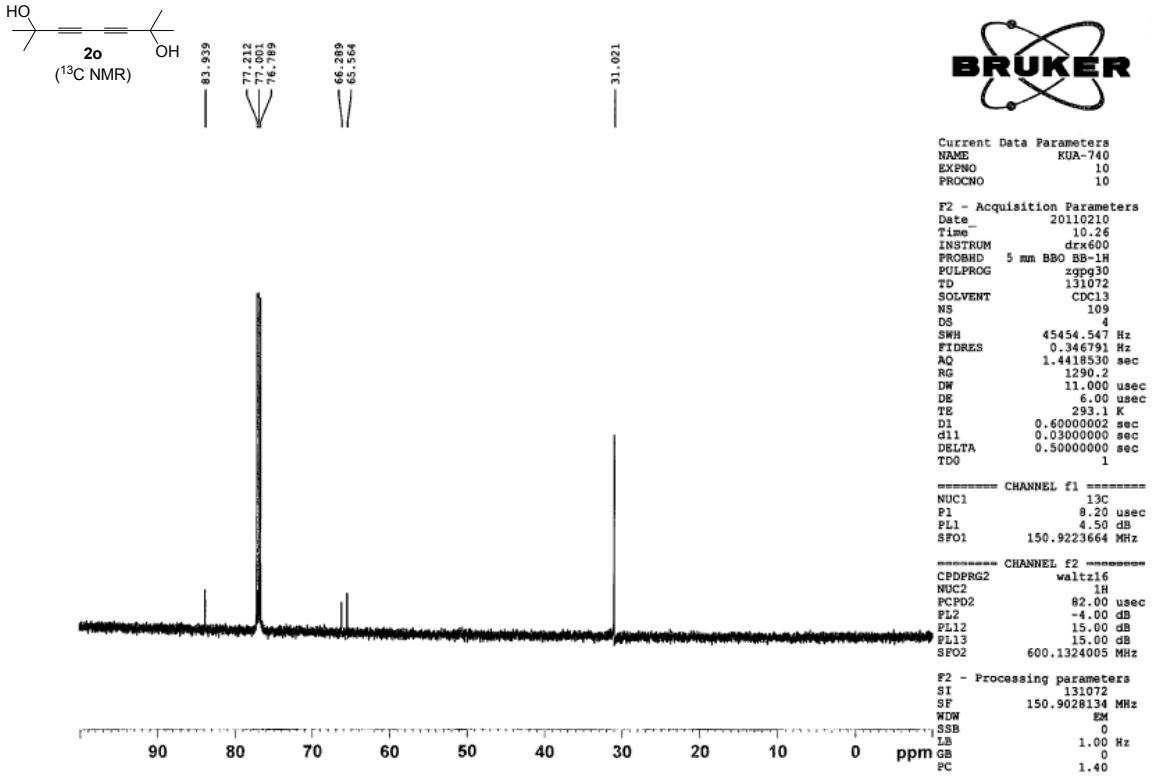
2n
(^{13}C NMR)

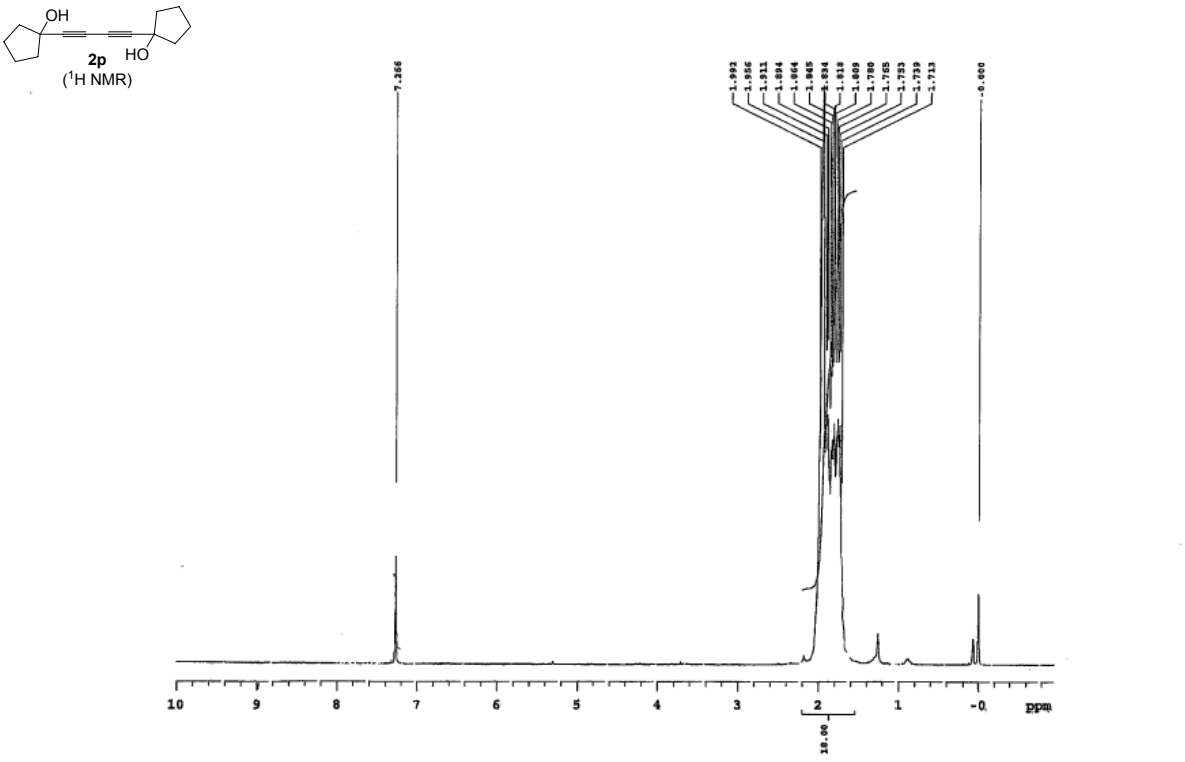


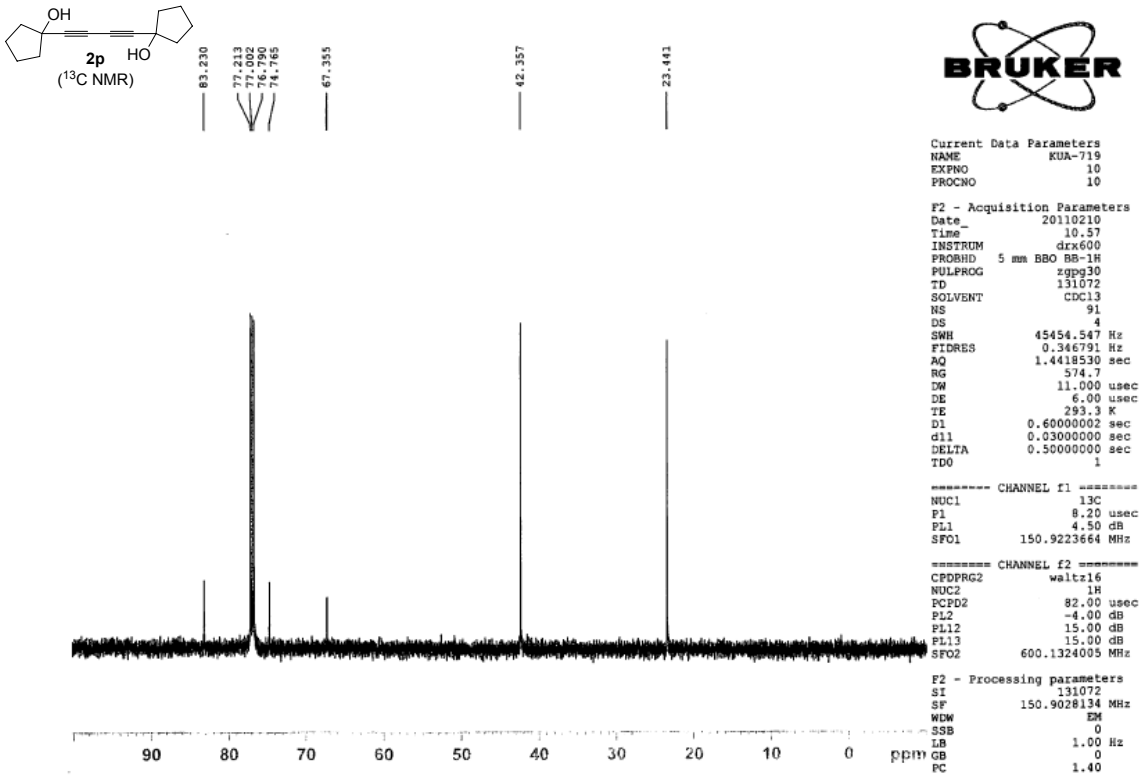


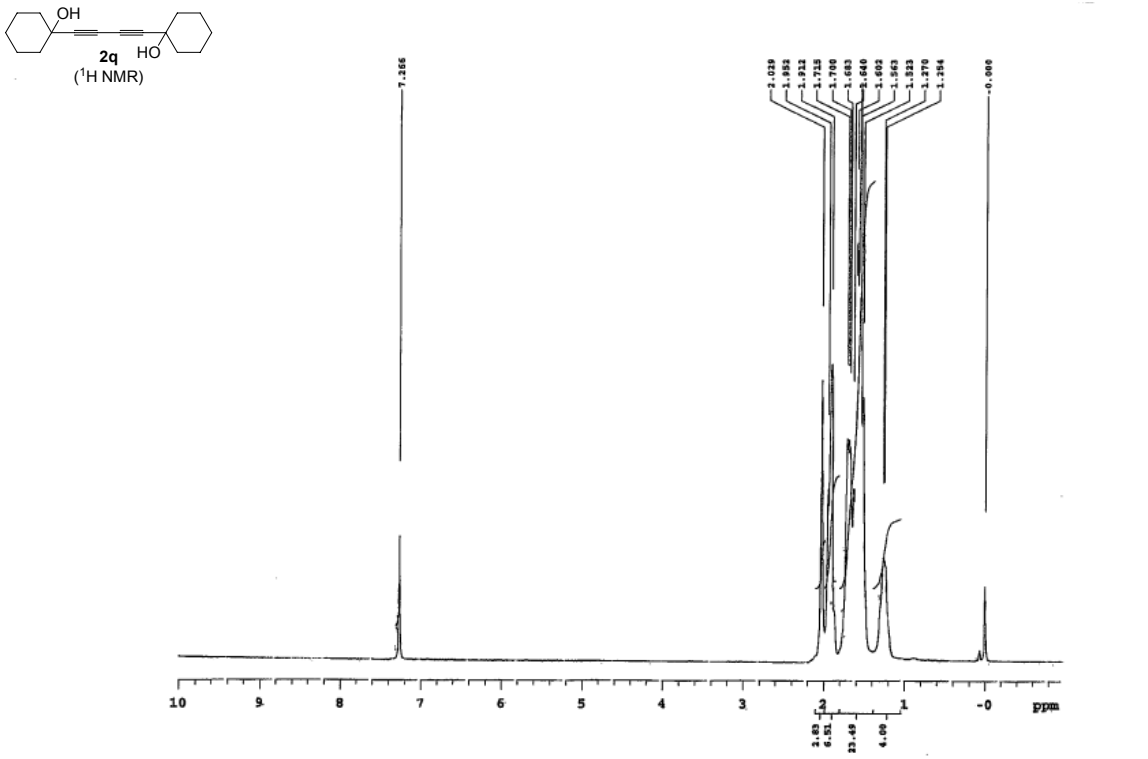
2o
(^{1}H NMR)

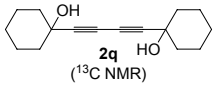



HO
OH
2o
(13C NMR)
83.939
77.212
77.001
76.789
66.289
65.564
31.021
BRUKER
Current Data Parameters
NAME KUA-740
EXPNO 10
PROCNO 10
F2 - Acquisition Parameters
Date_ 20110210
Time 10.26
INSTRUM drx600
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 131072
SOLVENT CDCl3
NS 109
DS 4
SWH 45454.547 Hz
FIDRES 0.346791 Hz
AQ 1.4418530 sec
RG 1290.2
DW 11.000 usec
DE 6.00 usec
TE 293.1 K
D1 0.60000002 sec
d11 0.03000000 sec
DELTA 0.50000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.20 usec
PL1 4.50 dB
SFO1 150.9223664 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 82.00 usec
PL2 -4.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 600.1324005 MHz
F2 - Processing parameters
SI 131072
SF 150.9028134 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
90
80
70
60
50
40
30
20
10
0
ppm

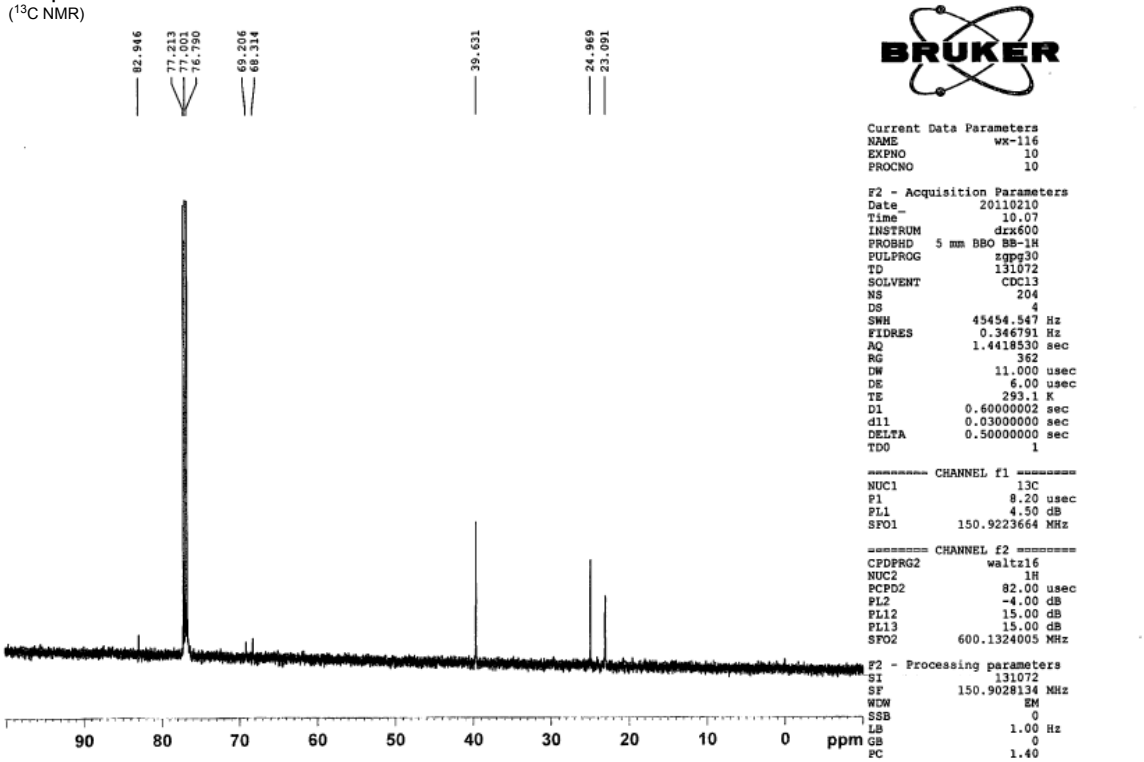

OH
HO
2p
(1H NMR)
7.266
1.992
1.956
1.911
1.894
1.864
1.845
1.834
1.813
1.809
1.780
1.765
1.753
1.739
1.713
-0.000
10
9
8
7
6
5
4
3
2
1
-0
ppm
18.00


OH
HO
2p
(13C NMR)
83.230
77.213
77.002
76.790
74.765
67.355
42.357
23.441
BRUKER
Current Data Parameters
NAME KUA-719
EXPNO 10
PROCNO 10
F2 - Acquisition Parameters
Date_ 20110210
Time 10.57
INSTRUM drx600
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 131072
SOLVENT CDCl3
NS 91
DS 4
SWH 45454.547 Hz
FIDRES 0.346791 Hz
AQ 1.4418530 sec
RG 574.7
DW 11.000 usec
DE 6.00 usec
TE 293.3 K
D1 0.60000002 sec
d11 0.03000000 sec
DELTA 0.50000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.20 usec
PL1 4.50 dB
SFO1 150.9223664 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 82.00 usec
PL2 -4.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 600.1324005 MHz
F2 - Processing parameters
SI 131072
SF 150.9028134 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
90
80
70
60
50
40
30
20
10
0
ppm


OH
HO
2q
(1H NMR)
7.266
2.029
1.952
1.912
1.725
1.700
1.683
1.640
1.602
1.563
1.523
1.270
1.254
-0.000
10
9
8
7
6
5
4
3
2
1
-0
ppm
2.83
6.51
23.48
4.00



2q
(^{13}C NMR)



BRUKER

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Current Data Parameters
NAME                  wx-116
EXPNO                     10
PROCNO                    10

F2 - Acquisition Parameters
Date_              20110210
Time                  10.07
INSTRUM             drx600
PROBHD   5 mm BBO BB-1H
PULPROG             zgpg30
TD                  131072
SOLVENT               CDCl3
NS                      204
DS                        4
SWH                45454.547 Hz
FIDRES             0.346791 Hz
AQ                 1.4418530 sec
RG                      362
DW                   11.000 usec
DE                    6.00 usec
TE                   293.1 K
D1                0.60000002 sec
d11               0.03000000 sec
DELTA             0.50000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                    8.20 usec
PL1                   4.50 dB
SFO1           150.9223664 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2                82.00 usec
PL2                  -4.00 dB
PL12                 15.00 dB
PL13                 15.00 dB
SFO2           600.1324005 MHz

F2 - Processing parameters
SI                    131072
SF             150.9028134 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
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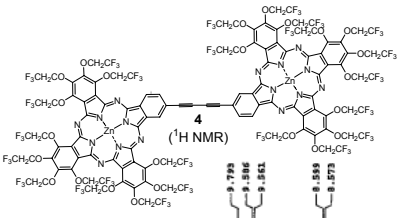
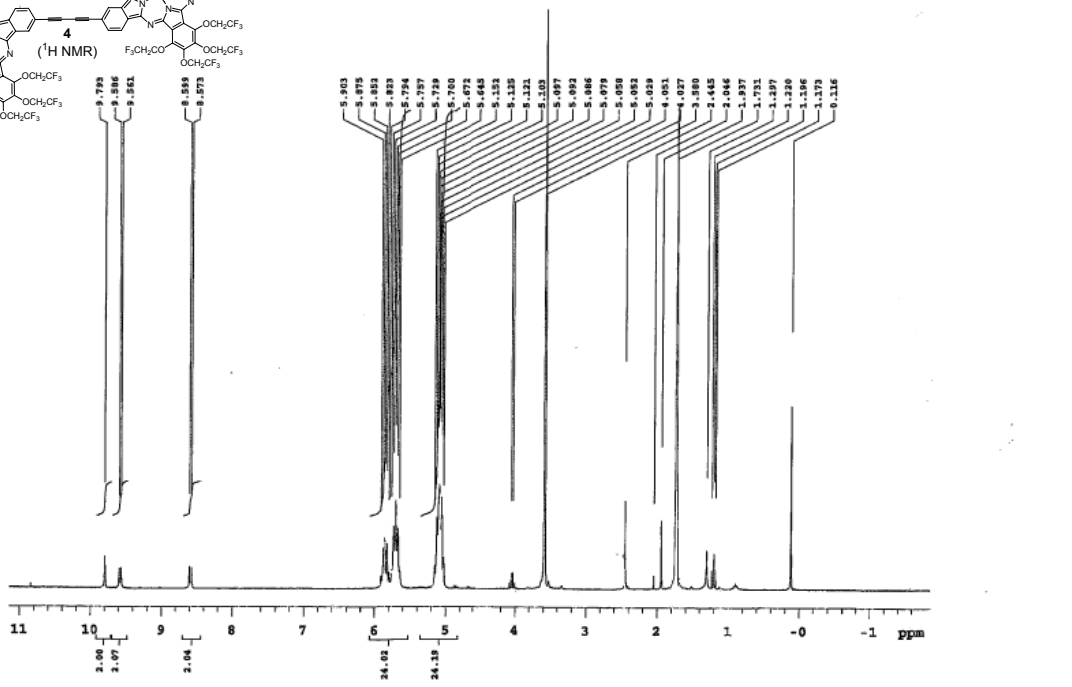
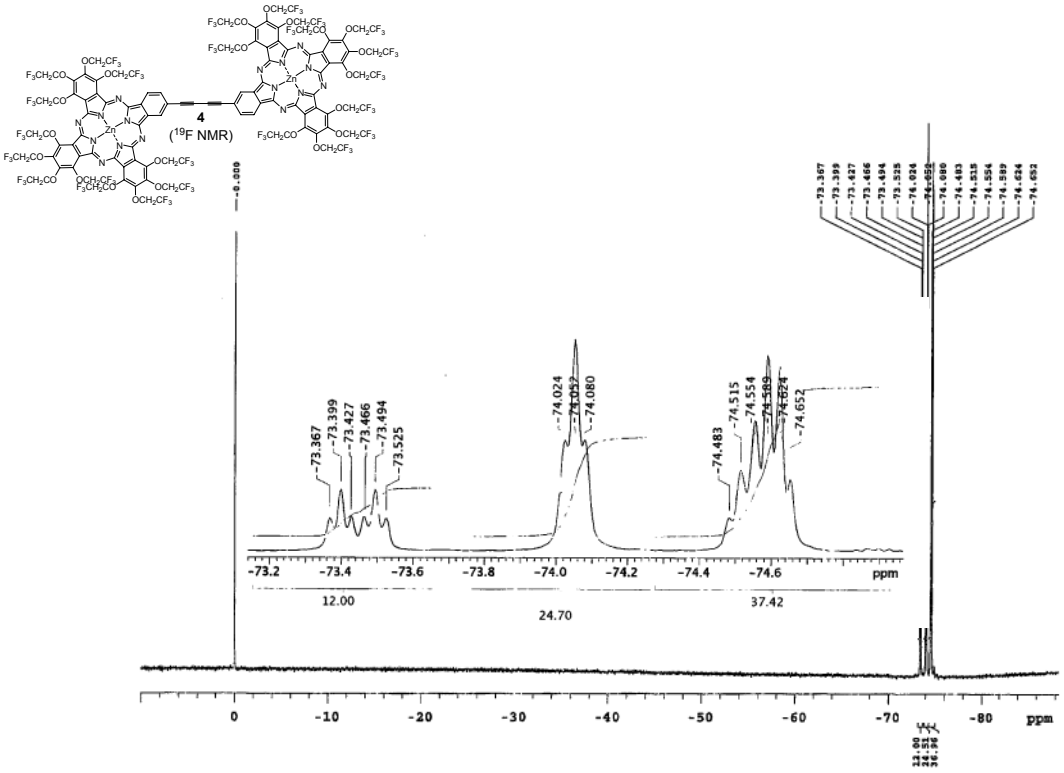


4
(^{1}H NMR)




4
(^{19}F NMR)
-73.367
-73.399
-73.427
-73.466
-73.494
-73.525
-74.024
-74.052
-74.080
-74.483
-74.515
-74.554
-74.589
-74.624
-74.652
-73.2
-73.4
-73.6
-73.8
-74.0
-74.2
-74.4
-74.6
ppm
12.00
24.70
37.42
0
-10
-20
-30
-40
-50
-60
-70
-80
ppm