Supporting information

For

Alder-Ene Reactions of Arynes to Form Medium-Sized and Macrocyclic Frameworks of Sizes up to a 46-Membered Ring

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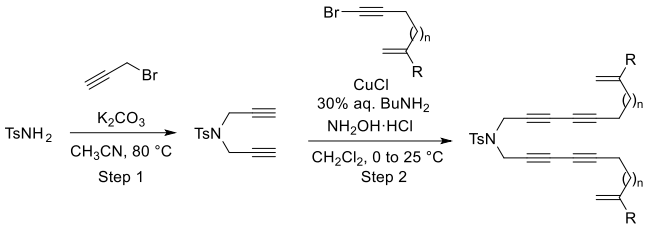
General Information

Reactions were carried out in oven-dried glassware unless otherwise noted. Compounds were purchased from Aldrich or Acros or TCI America or Oakwood Chemicals unless otherwise noted. Toluene, acetonitrile, dichloromethane and α,α,α-trifluorotoluene were distilled over calcium hydride (CaH_2) under nitrogen atmosphere. THF was distilled over sodium-benzophenone ketyl under nitrogen atmosphere. Column chromatography was performed using silica gel 60 Å (32−63 mesh) purchased from Silicycle Inc. Analytical thin layer chromatography (TLC) was performed on 0.25 mm E. Merck precoated silica gel 60 (particle size 0.040−0.063 mm). Yields refer to chromatographically and spectroscopically pure compounds unless otherwise stated. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker AV-500 spectrometer. ^{19}F NMR spectrum was recorded in Varian Mercury-Vx-300 spectrometer. ^{1}H NMR chemical shifts (δ) are reported in parts per million (ppm) downfield of TMS and are referenced relative to the residual proteated solvent peak ($CDCl_3$ (7.26 ppm)). ^{13}C chemical shifts (δ) are reported in parts per million downfield of TMS and are referenced to the carbon

resonance of the solvent ($CDCl_3$ (77.2 ppm)). Multiplicities are indicated by s (singlet), d (doublet), t (triplet), q (quartet), quin (quintet), sext (sextet) or m (multiplet). ^{1}H NMR signals that fall within a ca. 0.3 ppm range are generally reported as a multiplet, with a range of chemical shift values corresponding to the peak or center of the peak. Coupling constants, *J*, are reported in Hz (Hertz). Electrospray ionization (ESI) mass spectra were recorded on a Waters Micromass Q-Tof Ultima in the University of Illinois at Urbana-Champaign. Electron impact (EI) mass spectra and Chemical Ionization (CI) mass spectra were obtained using a Micromass 70-VSE in the University of Illinois at Urbana–Champaign.

Experimental Procedures

General scheme for the synthesis of symmetrical bis-1,3-diyne



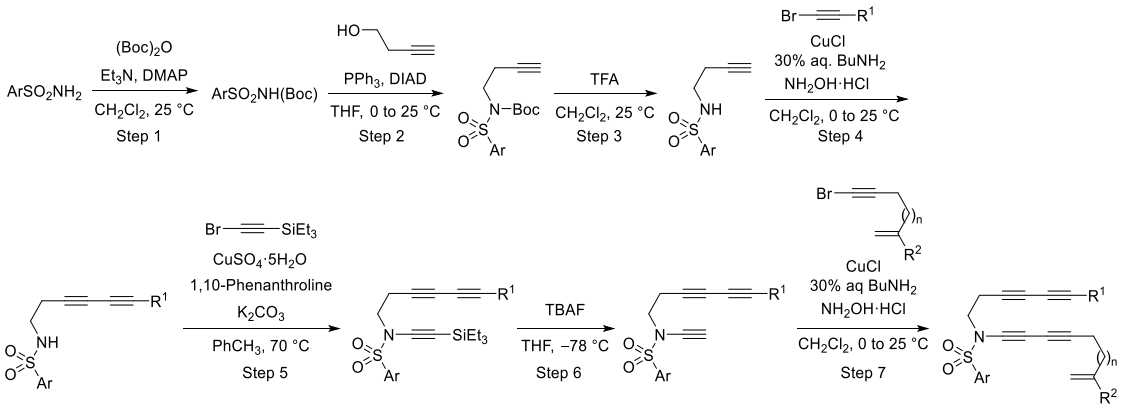
General procedure for bis-propargylation of p-toluenesulfonamide (Step 1):

To a solution of arylsulfonamide (1.0 equiv) in acetonitrile was added K2CO3 (3.0 equiv) followed by propargyl bromide (2.5 equiv), and the reaction mixture was stirred at 80 C. After complete consumption of the starting material, the reaction mixture was concentrated under reduced pressure and partitioned between water and ethyl acetate. The organic phase was washed with brine, dried over anhydrous Na2SO4, filtered and concentrated under reduced pressure. Purification on a silica gel column afforded the desired product.

General procedure for the Cadiot–Chodkiewicz reaction (Step 2):

In a two-neck round–bottom flask containing CuCl (0.6 equiv) was added 30% aqueous $BuNH_2$ solution under nitrogen flow. A pinch of $NH_2OH.HCl$ was added until a blue color disappeared. A solution of terminal alkyne (1.0 equiv) in dichloromethane was added at 0 °C to the flask and the solution became yellow. A dilute solution of bromoalkyne (2.5 equiv) in dichloromethane was added drop wise at 0 °C. The reaction mixture was stirred for 5 minutes at room temperature. The progress of the reaction was monitored by TLC. The nitrogen flow was removed and the biphasic reaction mixture was transferred to a separatory funnel. Dichloromethane layer was separated and dried over anhydrous Na_2SO_4. After filtration, the organic layer was concentrated and the crude material was purified by column chromatography to get pure product.

General scheme for the synthesis of unsymmetrical bis-1,3-diyne



General procedure for Boc protection of arylsulfonamide (Step 1):

To a solution of arylsulfonamide (1.0 equiv) in dichloromethane were added trimethylamine (1.5 equiv) and DMAP (0.1 equiv). A solution of $(Boc)_2O$ (1.05 equiv) in dichloromethane was added slowly to the reaction mixture at room temperature and stirred until complete consumption of starting material (monitored by TLC). After completion, the reaction mixture was transferred to a separatory funnel diluted with dichloromethane and washed with water (×2) and brine (×1). The organic layer was dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure to get the crude which was used for the next reaction without further purification.

General procedure for Mitsunobu reaction (Step 2):

DIAD (1.2 equiv) was added dropwise to a solution of $ArSO_2NH(Boc)$ (1.0 equiv), PPh_3 (1.2 equiv), and alcohol (1.0 equiv) in dry THF at 0 °C under nitrogen balloon. The reaction mixture was warmed up to room temperature and further stirred overnight. After completion, the reaction mixture was concentrated and loaded on a silica gel column to get pure product.

General procedure for N–Boc removal (Step 3):

TFA (5.0 equiv) was added to a solution of Boc-protected sulfonamide (1.0 equiv) in dichloromethane at room temperature and stirred until complete consumption of starting material (monitored by TLC). After completion, the reaction mixture was transferred to a separatory funnel diluted with dichloromethane and washed with water (×2) and brine (×1). The organic layer was dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure to get the crude which was further purified by column chromatography.

General procedure for the Cadiot–Chodkiewicz reaction (Step 4 and Step 7):

In a two-neck round–bottom flask containing CuCl (0.3 equiv) was added 30% aqueous $BuNH_2$ solution under nitrogen flow. A pinch of $NH_2OH.HCl$ was added until a blue color disappeared. A solution of terminal alkyne (1.0 equiv) in dichloromethane was added at 0 °C to the flask and the

solution became yellow. A dilute solution of bromoalkyne (1.2 equiv) in dichloromethane was added drop wise at 0 °C. The reaction mixture was stirred for 5 minutes at room temperature. The progress of the reaction was monitored by TLC. The nitrogen flow was removed and the biphasic reaction mixture was transferred to a separatory funnel. Dichloromethane layer was separated and dried over anhydrous Na_2SO_4. After filtration, the organic layer was concentrated and the crude material was purified by column chromatography to get pure product.

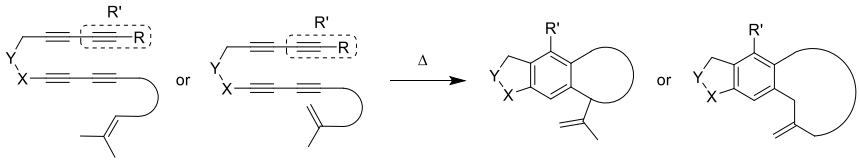
General procedure for the Coupling reaction between a sulfonamide and a bromoalkyne (Step 5):

$CuSO_4 \bullet 5H_2O$ (10 mol %), 1,10-phenanthroline (20 mol %), K_2CO_3 (2.0 equiv) and bromoalkyne (2.0 equiv) were added to a solution of sulfonamide (1.0 equiv) in dry toluene. The reaction mixture was stirred at 70 °C for overnight. After complete consumption of starting material, the reaction mixture was filtered through a small pad of celite. The filtrate was concentrated to get the pure product which was further purified by silica gel column chromatography to isolate pure product.

General procedure for the removal of trialkylsilyl group from alkynyltrialkylsilanes (Step 6):

Tetrabutylammonium fluoride (1.1 equiv) was added to a solution of alkynyltrialkylsilane (1.0 equiv) in dry THF at –78 °C under nitrogen and progress of the reaction was checked by TLC. Upon completion, the reaction mixture was transferred to a separatory funnel, diluted with ethyl acetate, washed with water (×2) and then brine (×1). The organic layer was separated, drier over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The crude product was used for the next reaction without further purification.

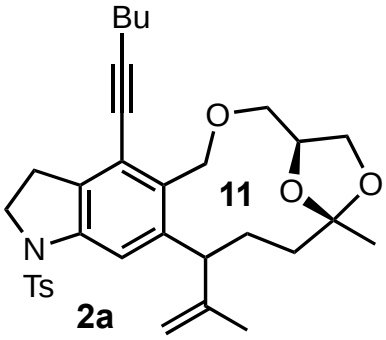
General procedure for Alder-ene reaction:



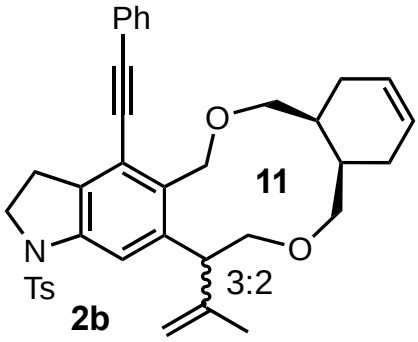
Method A: A solution of the substrate in toluene (0.03–0.05 M) was heated to 90 °C. After completion of the reaction (progress was monitored by TLC), toluene was evaporated under reduced pressure. The crude product was purified by column chromatography (Hexanes/EtOAc, 10:1 to 5:1). This method was used for the synthesis of macrocycles up to 20-membered ring.

Method B: A solution of the substrate in toluene (4 mM) was heated to 90 °C. After completion of the reaction (progress was monitored by TLC), toluene was evaporated under reduced pressure. The crude product was purified by column chromatography (Hexanes/EtOAc, 10:1 to 5:1). This procedure was used for the construction of macrocycles with larger than 20-membered rings.

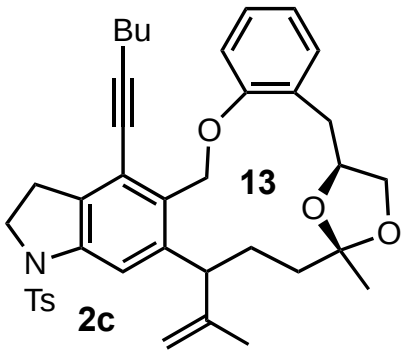
Characterization Data



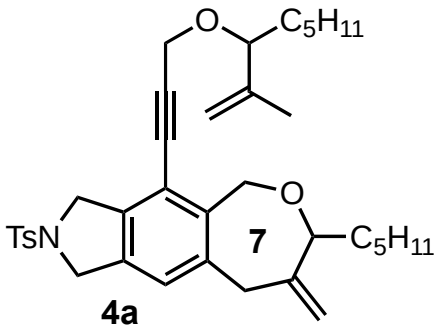
Method A. Yield: 47%. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.62–7.60 (m, 2H), 7.40 (s, 1H), 7.19–7.17 (m, 2H), 5.13 (d, 1H, *J* = 9.4 Hz), 5.09 (s, 1H), 5.06 (s, 1H), 4.50 (d, 1H, *J* = 9.4 Hz), 4.26 (t, 1H, *J* = 7.3 Hz), 4.02 (t, 1H, *J* = 7.5 Hz), 3.97 (t, 1H, *J* = 7.5 Hz), 3.96–3.90 (m, 2H), 3.86–3.78 (m, 2H), 3.72 (dd, 1H, *J* = 12.8, 2.0 Hz), 3.00–2.85 (m, 2H), 2.42 (t, 2H, *J* = 6.9 Hz), 2.35 (s, 3H), 2.01–1.74 (m, 4H), 1.60–1.52 (m, 2H), 1.52 (s, 3H), 1.52–1.41 (m, 2H), 1.35 (s, 3H), 0.92 (t, 3H, *J* = 7.3 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 144.1, 141.7, 133.3, 132.2, 129.5, 127.5, 121.4, 112.7, 111.9, 110.0, 97.8, 75.6, 70.9, 69.1, 65.9, 50.0, 35.2, 30.8, 28.3, 27.9, 25.6, 23.6, 21.9, 21.5, 19.3, 13.6. HRMS (EEI) calcd for $C_{33}H_{42}NO_5S$ $[M+H]^+$ 564.2778, found 564.2777.



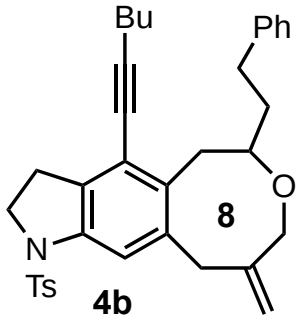
Method A. Total yield: 64%. Two isomers isolated in 3:2 ratio. Major diastereomer: ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.65–7.63 (m, 2H), 7.52 (s, 1H), 7.44–7.42 (m, 2H), 7.34–7.33 (m, 3H), 7.19–7.18 (m, 2H), 5.62–5.55 (m, 2H), 5.47 (t, 1H, *J* = 7.0 Hz), 5.07 (s, 2H), 4.82 (d, 1H, *J* = 6.0 Hz), 4.71 (d, 1H, *J* = 6.5 Hz), 4.03 (dt, 1H, *J* = 10.0, 6.5 Hz), 3.88 (dt, 1H, *J* = 10.0, 7.5 Hz), 3.81–3.77 (m, 2H), 3.60 (dd, 1H, *J* = 8.5, 7.5 Hz), 3.48–3.42 (m, 2H), 3.32–3.24 (m, 2H), 3.15–3.02 (m, 2H), 2.36 (s, 3H), 2.32–2.19 (m, 4H), 2.10–1.92 (m, 4H), 1.81 (s, 3H), 1.75–1.59 (m, 7H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 147.6, 145.2, 144.2, 143.8, 141.7, 133.3, 132.4, 132.3, 131.4, 129.6, 128.6, 128.4, 127.4, 125.8, 125.6, 123.0, 121.7, 121.0, 113.5, 110.4, 96.8, 85.8, 71.1, 70.2, 67.9, 66.1, 50.0, 46.5, 34.2, 33.6, 32.4, 29.8, 27.9, 27.4, 26.3, 23.1, 23.0, 21.5. HRMS (EEI) calcd for $C_{42}H_{48}NO_4S$ $[M+H]^+$ 662.3299, found 662.3295. Minor diastereomer: ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.67–7.65 (m, 2H), 7.55 (s, 1H), 7.44–7.43 (m, 2H), 7.35–7.33 (m, 3H), 7.21–7.19 (m, 2H), 5.64–5.57 (m, 2H), 5.45 (t, 1H, *J* = 7.0 Hz), 5.14 (s, 1H), 5.12 (s, 1H), 4.81 (d, 1H, *J* = 10.0 Hz), 4.60 (d, 1H, *J* = 10.0 Hz), 4.00–3.84 (m, 4H), 3.69 (dd, 1H, *J* = 8.5, 6.5 Hz), 3.62–3.55 (m, 1H), 3.47–3.38 (m, 3H), 3.12–3.01 (m, 2H), 2.37 (s, 3H), 2.30–2.28 (m, 1H), 2.22 –1.91 (m, 8H), 1.85–1.75 (m, 5H), 1.63 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 146.4, 144.3, 144.3, 144.2, 141.8, 133.5, 132.7, 132.6, 131.4, 129.6, 128.5, 128.4,127.4, 125.9, 125.8, 123.1, 121.7, 120.6, 113.4, 111.2, 96.4, 85.7, 72.0, 71.7, 67.9, 66.8, 50.1, 49.0, 35.1, 33.6, 33.1, 31.2, 28.5, 27.9, 26.2, 23.3, 23.0, 21.5. HRMS (EEI) calcd for $C_{42}H_{48}NO_4S$ $[M+H]^+$ 662.3299, found 662.3297.



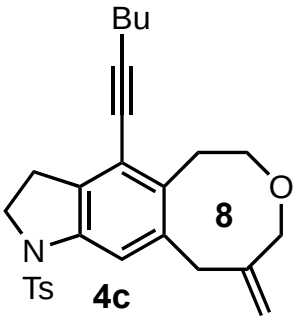
Method A. Yield: 44%. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.69–7.68 (m, 2H), 7.54 (s, 1H), 7.24–7.22 (m, 3H), 7.20–7.19 (m, 1H), 7.03–7.02 (m, 1H), 6.96–6.94 (m, 1H), 5.46 (d, 1H, *J* = 9.5 Hz), 4.93 (s, 1H), 4.91 (d, 1H, *J* = 9.5 Hz), 4.84 (s, 1H), 4.37–4.32 (m, 1H), 4.16 (t, 2H, *J* = 7.2 Hz), 3.99–3.86 (m, 3H), 3.59 (dd, 1H, *J* = 7.5, 6.0 Hz), 3.22 (dd, 1H, *J* = 7.0, 6.5 Hz), 3.03–2.92 (m, 2H), 2.38 –2.35 (m, 6H), 2.04–1.98 (m, 1H), 1.95–1.87 (m, 2H), 1.71–1.61 (m, 2H), 1.56 (s, 3H), 1.52–1.46 (m, 2H), 1.42–1.35 (m, 2H), 1.20 (s, 3H), 0.84 (t, 3H, *J* = 7.2 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 157.6, 146.6, 145.6, 144.2, 141.9, 133.6, 132.1, 130.5, 129.8, 129.6, 127.5, 127.3, 122.2, 120.4, 112.8, 112.1, 111.1, 110.7, 98.4, 77.6, 76.5, 69.7, 65.1, 50.0, 48.3, 35.4, 33.6, 30.7, 28.0, 26.5, 23.7, 21.9, 21.5, 21.3, 19.3, 14.1, 13.5. LRMS (ES+) calcd for $C_{39}H_{45}NO_5S$ $[M+H]^+$ 640.3, found 640.3.



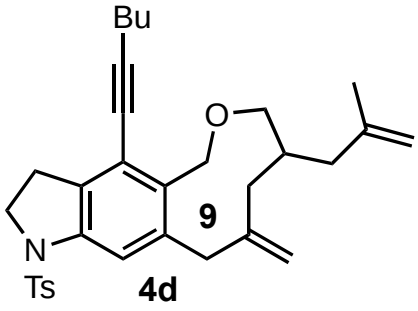
Method A. Yield: 76%. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.76–7.74 (m, 2H), 7.31–7.29 (m, 2H), 6.88 (s, 1H), 7.32 (d, 1H, *J* = 16.5 Hz), 5.00 (s, 1H), 4.96 (s, 1H), 4.94 (s, 1H), 4.87 (s, 1H), 4.62–4.51 (m, 5H), 4.35 (d, 1H, *J* = 16.0 Hz), 4.18 (d, 1H, *J* = 16.0 Hz), 4.05 (t, 1H, *J* = 6.2 Hz), 3.88 (dt, 1H, *J* = 7.0, 2.5 Hz), 3.81 (d, 1H, *J* = 14.5 Hz), 3.18 (d, 1H, *J* = 14.5 Hz), 2.40 (s, 3H), 1.71–1.58 (m, 5H), 1.53–1.23 (m, 16H), 0.91–0.87 (m, 6H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 148.8, 143.7, 143.7, 139.3, 138.9, 136.9, 134.4, 133.8, 129.8, 127.5, 123.1, 115.5, 114.6, 112.9, 95.8, 85.0, 83.2, 83.1, 79.9, 69.7, 55.7, 54.1, 53.9, 38.2, 34.9, 33.4, 31.8, 31.7, 25.5, 25.5, 22.6, 21.5, 16.5, 14.1.



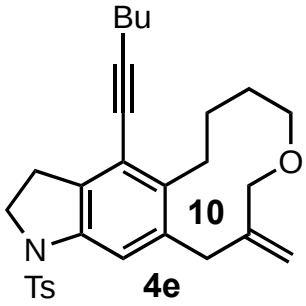
Method A. Yield: 56%. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.65–7.63 (m, 2H), 7.40 (s, 1H), 7.29–7.26 (m, 2H), 7.21–7.16 (m, 5H), 5.07 (s, 1H), 4.86 (s, 1H), 4.20 (d, 1H, *J* = 13.5 Hz), 3.90–3.84 (m, 3H), 3.45–3.35 (m, 3H), 3.29 (d, 1H, *J* = 14.0 Hz), 2.92–2.82 (m, 4H), 2.75–2.69 (m, 1H), 2.39–2.37 (m, 5H), 2.04–1.96 (m, 1H), 1.89–1.82 (m, 1H), 1.54–1.49 (m, 2H), 1.46–1.39 (m, 2H), 0.91 (t, 3H, *J* = 7.2 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 148.9, 144.1, 142.2, 140.2, 138.7, 135.0, 133.6, 132.9, 129.6, 128.5, 128.3, 127.4, 125.7, 121.1, 115.2, 111.3, 98.5, 82.8, 77.2, 73.0, 49.8, 40.0, 39.0, 38.6, 32.3, 30.9, 28.1, 22,0, 21.5, 19.2, 13.6. HRMS (EEI) calcd for $C_{35}H_{40}NO_3S$ $[M+H]^+$ 554.2723, found 554.2720.



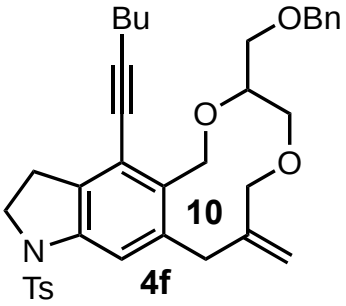
Method A. Yield: 60%. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.65–7.64 (m, 2H), 7.40 (s, 1H), 7.20–7.18 (m, 2H), 4.97 (s, 1H), 4.89 (s, 2H), 4.74 (s, 1H), 3.88 (t, 2, *J* = 8.5 Hz), 3.73–3.71 (m, 4H), 2.90 (t, 2H, *J* = 8.5 Hz), 2.42 (t, 2H, *J* = 7.0 Hz), 2.36 (s, 3H), 2.28–2.26 (m, 2H), 1.58–1.52 (m, 2H), 1.47–1.40 (m, 2H), 0.91 (t, 3H, *J* = 7.2 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 148.0, 144.2, 141.2, 139.1, 133.5, 133.4, 133.1, 129.6, 127.4, 120.6, 115.9, 111.7, 99.6, 76.0, 70.1, 69.1, 49.9, 42.0, 37.0, 30.8, 27.8, 22.0, 21.5, 19.3, 13.6. HRMS (EEI) calcd for $C_{27}H_{32}NO_3S$ $[M+H]^+$ 450.2103, found 450.2110.



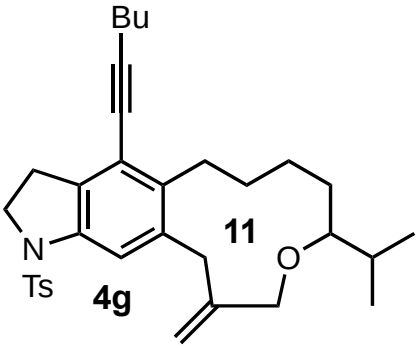
Method A. Yield: 47%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.68–7.66 (m, 2H), 7.42 (s, 1H), 7.21–7.19 (m, 2H), 4.90 (d, 1H, *J* = 12.8 Hz), 4.89 (s, 1H), 4.84 (s, 1H), 4.77 (s, 1H), 4.74 (d, 1H, *J* = 12.8 Hz), 4.65 (s, 1H), 3.90 (t, 2H, *J* = 8.6 Hz), 3.67–3.62 (m, 2H), 3.46 (d, 1H, *J* = 14.4 Hz), 2.92 (t, 2H, *J* = 8.4 Hz), 2.40 (t, 2H, *J* = 7.0 Hz), 2.36 (s, 3H), 2.17 (dd, 1H, *J* = 13.4, 2.6 Hz), 1.87–1.82 (m, 1H), 1.67 (s, 3H), 1.58–1.50 (m, 2H), 1.48–1.39 (m, 2H), 0.91 (t, 3H, *J* = 7.4 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 148.6, 144.1, 143.6, 141.1, 133.5, 133.2, 132.1, 129.6, 127.4, 121.1, 116.5, 112.6, 112.0, 98.8, 76.3, 74.1, 69.6, 49.9, 40.6, 40.3, 38.0, 30.8, 27.9, 22.1, 21.9, 21.5, 19.2, 13.5. HRMS (EEI) calcd for $C_{32}H_{40}NO_3S$ $[M+H]^+$ 518.2723, found 518.2726.



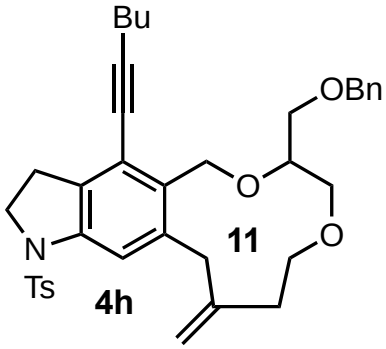
Method A. Yield: 50% (in toluene), 52% (in acetonitrile). ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.65–7.63 (m, 2H), 7.40 (s, 1H), 7.20–7.18 (m, 2H), 5.10 (s, 1H), 5.04 (s, 1H), 3.88 (t, 2H, *J* = 8.2 Hz), 3.71 (s, 2H), 3.59 (t, 2H, *J* = 4.8 Hz), 3.49 (s, 2H), 3.08 (t, 2H, *J* = 6.5 Hz), 2.91 (t, 2H, *J* = 8.5 Hz), 2.40 (t, 2H, *J* = 7.0 Hz), 2.37 (s, 3H), 2.06 (m, 2H), 1.57–1.52 (m, 2H), 1.48–1.41 (m, 2H), 1.29–1.25 (m, 2H), 0.92 (t, 3H, *J* = 7.2 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 147.2, 144.0, 139.2, 138.3, 137.6, 133.6, 133.4, 129.5, 127.5, 121.0, 115.7, 112.9, 98.0, 77.7, 72.8, 71.5, 49.9, 37.2, 30.8, 28.8, 28.1, 27.7, 24.3, 22.0, 21.5, 19.3, 13.6. HRMS (EEI) calcd for $C_{29}H_{36}NO_3S$ $[M+H]^+$ 478.2410, found 478.2400.



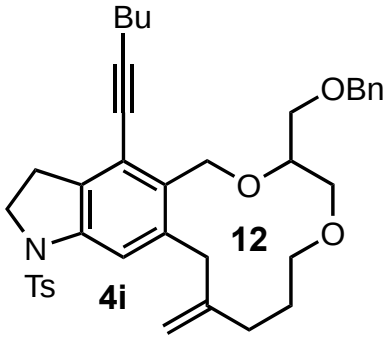
Method A. Yield: 50%. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.66–7.64 (m, 2H), 7.48 (s, 1H), 7.33–7.32 (m, 4H), 7.26 (m, 1H), 7.19–7.18 (m, 2H), 5.30 (d, 1H, *J* = 10.5 Hz), 5.00 (s, 1H), 4.94 (s, 1H), 4.58–4.54 (m, 3H), 4.10 (d, 1H, *J* = 11.0 Hz), 3.97 (dd, 1H, *J* = 17.5, 8.5 Hz), 3.92 (d, 1H, *J* = 11.0 Hz), 3.83–3.80 (m, 3H), 3.65 (d, 1H, *J* = 14.0 Hz), 3.49 (dd, 1H, *J* = 10.0, 5.5 Hz), 3.43–3.40 (m, 2H), 3.27 (dd, 1H, *J* = 12.8, 9.8 Hz), 2.93 (t, 2H, *J* = 8.5 Hz), 2.36 (s, 3H), 2.31 (t, 2H, *J* = 6.8 Hz), 1.51–1.45 (m, 2H), 1.43–1.36 (m, 2H), 0.88 (t, 3H, *J* = 7.2 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 147.4, 144.2, 141.2, 140.3, 138.2, 133.5, 132.5, 132.5, 129.6, 128.4, 127.6, 127.5, 127.4, 121.7, 116.5, 114.5, 98.2, 80.7, 76.5, 75.8, 73.5, 71.3, 70.3, 70.0, 50.0, 36.3, 30.8, 27.9, 21.9, 21.5, 19.2, 13.6. HRMS (EEI) calcd for $C_{36}H_{42}NO_5S$ $[M+H]^+$ 600.2784, found 600.2782.



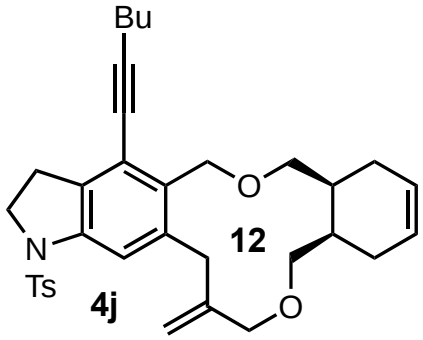
Method A. Yield: 43%. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.66–7.65 (m, 2H), 7.38 (s, 1H), 7.22–7.20 (m, 2H), 5.07 (s, 1H), 4.87 (s, 1H), 4.11 (d, 1H, *J* = 12.0 Hz), 3.97–3.92 (m, 2H), 3.86 (dd, 1H, *J* = 19.2, 8.8 Hz), 3.68 (d, 1H, *J* = 11.5 Hz), 3.19 (d, 1H, *J* = 15.5 Hz), 2.94–2.90 (m, 2H), 2.88–2.80 (m, 3H), 2.41 (t, 2H, *J* = 6.8 Hz), 2.37 (s, 3H), 1.85–1.78 (m, 2H), 1.56–1.38 (m, 10H), 0.93–0.89 (m, 6H), 0.86 (d, 3H, *J* = 6.5 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 147.2, 144.0, 139.2, 139.0, 137.3, 133.8, 132.6, 129.6, 127.5, 121.3, 116.0, 115.2, 97.5, 83.7, 77.4, 72.9, 49.9, 36.0, 31.3, 30.9, 29.4, 28.1, 28.0, 26.9, 25.8, 21.9, 21.5, 19.2, 19.0, 17.3, 13.6. HRMS (EEI) calcd for $C_{33}H_{44}NO_3S$ $[M+H]^+$ 534.3036, found 534.3027.



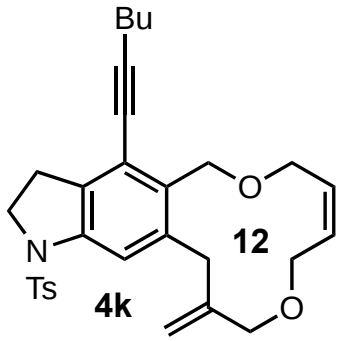
Method A. Yield: 52%. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.67–7.65 (m, 2H), 7.46 (s, 1H), 7.33–7.32 (m, 4H), 7.28–7.26 (m, 1H), 7.20–7.18 (m, 2H), 5.07 (d, 1H, *J* = 10.5 Hz), 4.93 (s, 1H), 4.74 (m, 1H), 4.69 (d, 1H, *J* = 10.5 Hz), 4.55 (s, 2H), 3.97–3.73 (m, 6H), 3.62–3.58 (m, 1H), 3.54–3.51 (m, 1H), 3.49–3.46 (m, 2H), 3.31 (dd, 1H, *J* = 11.2, 9.2 Hz), 2.92 (t, 2H, *J* = 8.5 Hz), 2.40–2.35 (m, 4H), 2.32 (t, 2H, *J* = 7.0 Hz), 2.22–2.18 (m, 1H), 1.52–1.46 (m, 2H), 1.44–1.36 (m, 2H), 0.89 (t, 3H, *J* = 7.2 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 148.6, 144.1, 141.3, 141.0, 138.2, 133.6, 132.4, 132.1, 129.6, 128.4, 127.6, 127.5, 122.1, 116.1, 113.2, 97.9, 78.3, 76.6, 74.0, 73.6, 73.5, 70.1, 68.8, 50.0, 39.3, 36.0, 30.8, 28.0, 21.9, 21.5, 19.2, 13.6. HRMS (EEI) calcd for $C_{37}H_{44}NO_5S$ $[M+H]^+$ 614.2940, found 614.2949.



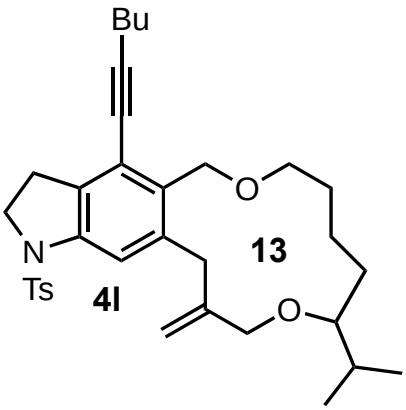
Method A. Yield: 34%. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.66–7.64 (m, 2H), 7.43 (s, 1H), 7.35–7.27 (m, 5H), 7.19–7.18 (m, 2H), 4.84–4.83 (m, 2H), 4.80 (d, 1H, *J* = 9.5 Hz), 4.52 (dd, 2H, *J* = 18.2, 12.2 Hz), 4.22–4.21 (m, 1H), 3.93–3.71 (m, 6H), 3.52 (dd, 1H, *J* = 10.0, 5.0 Hz), 3.45–3.34 (m, 4H), 2.92 (t, 2H, *J* = 8.5 Hz), 2.39–2.33 (m, 4H), 2.31 (t, 2H, *J* = 6.8 Hz), 2.15–2.10 (m, 1H), 1.93–1.89 (m, 1H), 1.79–1.77 (m, 1H), 1.51–1.45 (m, 2H), 1.44–1.36 (m, 2H), 0.89 (t, 3H, *J* = 7.0 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 149.9, 144.1, 141.4, 140.8, 138.2, 133.4, 132.9, 132.4, 129.6, 128.3, 127.6, 127.5, 122.3, 116.7, 110.9, 97.5, 76.7, 73.6, 73.4, 71.8, 70.0, 68.4, 49.9, 39.8, 36.1, 30.8, 29.2, 27.9, 21.9, 21.5, 19.2, 13.6. LRMS (ES+) calcd for $C_{38}H_{46}NO_5S$ $[M+H]^+$ 628.3, found 628.3.



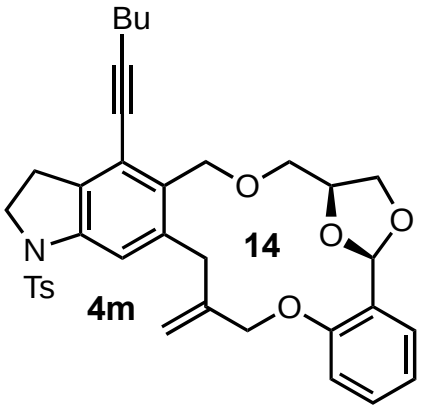
Method A. Yield: 70%. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.66–7.64 (m, 2H), 7.50 (s, 1H), 7.47–7.43 (m, 2H), 7.34–7.32 (m, 3H), 7.20–7.18 (m, 2H), 5.63–5.56 (m, 2H), 5.12 (m, 1H), 5.03 (m, 1H), 4.95 (d, 1H, *J* = 9.5 Hz), 4.52 (d, 1H, *J* = 9.0 Hz), 4.04–3.93 (m, 4H), 3.90–3.84 (m, 1H), 3.59 (dd, 1H, *J* = 11.0, 9.0 Hz), 3.51 (d, 1H, *J* = 10.5 Hz), 3.34 (dd, 1H, *J* = 11.5, 1.5 Hz), 3.29 (d, 1H, *J* = 14.5 Hz), 3.24 (dd, 1H, *J* = 9.0, 4.0 Hz), 3.06 (t, 2H, *J* = 8.5 Hz), 2.68–2.65 (m, 1H), 2.37 (s, 3H), 2.32–2.28 (m, 2H), 2.02–1.98 (m, 1H), 1.88–1.78 (m, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 145.9, 144.3, 141.5, 139.3, 133.4, 133.1, 132.3, 131.5, 129.6, 128.5, 128.4, 127.4, 125.6, 125.4, 123.1, 121.8, 116.3, 115.5, 96.6, 85.4, 73.5, 73.0, 72.5, 67.5, 50.1, 36.1, 34.9, 30.3, 29.0, 27.9, 25.2, 21.5; HRMS (EEI) calcd for $C_{36}H_{38}NO_4S$ $[M+H]^+$ 580.2516, found 580.2516.



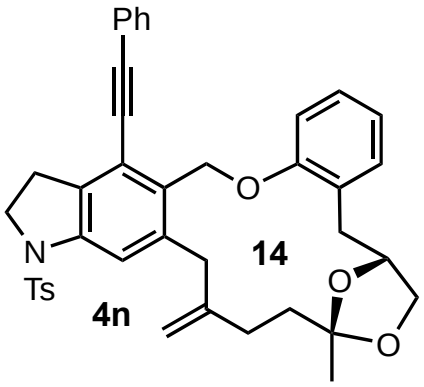
Method A. Yield: 67%. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.67–7.65 (m, 2H), 7.42 (s, 1H), 7.20–7.19 (m, 2H), 5.93–5.88 (m, 1H), 5.78 (dt, 1H, *J* = 11.0, 5.5 Hz), 5.10 (s, 1H), 4.88–4.87 (m, 1H), 4.65 (s, 2H), 4.12 (d, 2H, *J* = 5.0 Hz), 4.02 (d, 2H, *J* = 7.0 Hz), 3.93–3.90 (m, 4H), 3.55 (s, 2H), 2.96 (t, 2H, *J* = 8.5 Hz), 2.42 (t, 2H, *J* = 7.0 Hz), 2.37 (s, 3H), 1.58–1.53 (m, 2H), 1.49–1.42 (m, 2H), 0.92 (t, 3H, *J* = 7.2 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 145.9, 144.2, 141.3, 139.3, 133.5, 132.8, 132.3, 130.6, 129.7, 129.6, 127.4, 122.9, 115.9, 115.7, 98.3, 76.6, 73.3, 67.4, 66.6, 64.5, 50.0, 35.6, 30.9, 27.9, 22.0, 21.5, 19.2, 13.6; HRMS (EEI) calcd for $C_{30}H_{36}NO_4S$ $[M+H]^+$ 506.2360, found 506.2360.



Method A. Yield: 62%. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.67–7.66 (m, 2H), 7.44–7.42 (m, 3H), 7.34–7.32 (m, 3H), 7.21–7.20 (m, 2H), 4.99 (s, 1H), 4.37 (m, 1H), 4.20 (d, 1H, *J* = 12.5 Hz), 4.05 (d, 1H, *J* = 12.5 Hz), 3.98–3.88 (m, 2H), 3.80 (d, 1H, *J* = 17.5 Hz), 3.34–3.27 (m, 2H), 3.02 (t, 2H, *J* = 8.5 Hz), 2.69–2.58 (m, 2H), 2.37 (s, 3H), 2.18–2.12 (m, 1H), 1.74–1.25 (m, 11H), 0.89 (d, 3H, 6.5 Hz), 0.88 (d, 3H, *J* = 6.0 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 146.2, 144.1, 139.7, 139.2, 136.9, 133.6, 132.8, 131.4, 129.6, 128.4, 128.4, 127.4, 123.3, 120.3, 117.9, 114.0, 96.0, 85.9, 79.9, 71.6, 49.9, 37.6, 28.7, 28.1, 27.7, 27.5, 26.8, 26.4, 25.2, 21.7, 21.5, 19.0, 15.2; HRMS (EEI) calcd for $C_{37}H_{44}NO_3S$ $[M+H]^+$ 582.3036, found 582.3035.

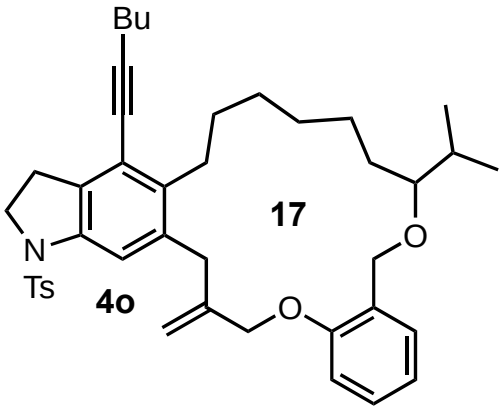


Method A. Yield: 34%. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.70–7.69 (m, 2H), 7.51 (s, 1H), 7.36–7.33 (m, 1H), 7.24 (dd, 1H, *J* = 7.0, 1.5 Hz), 7.22–7.20 (m, 2H), 6.96 (d, 1H, *J* = 8.0 Hz), 6.92 (t, 1H, *J* = 7.5 Hz), 5.54 (s, 1H), 5.12 (s, 1H), 4.87 (s, 2H), 4.57 (s, 2H), 4.46–4.44 (m, 3H), 4.03 (s, 2H), 3.98 (d, 2H, *J* = 12.0 Hz), 3.92 (t, 2H, *J* = 8.5 Hz), 3.60 (s, 1H), 2.97 (t, 2H, *J* = 8.5 Hz), 2.42 (t, 2H, *J* = 6.8 Hz), 2.37 (s, 3H), 1.57–1.52 (m, 2H), 1.50–1.42 (m, 2H), 0.92 (t, 3H, *J* = 7.2 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 157.3, 145.8, 144.2, 141.3, 140.1, 133.6, 132.8, 132.5, 130.9, 130.2, 129.7, 127.4, 125.6, 122.1, 119.9, 117.4, 114.0, 112.3, 104.6, 98.0, 76.6, 71.8, 69.4, 69.1, 64.1, 50.0, 35.7, 30.8, 28.0, 21.9, 21.5, 19.2, 13.5; HRMS (EEI) calcd for $C_{36}H_{40}NO_6S$ $[M+H]^+$ 614.2571, found 614.2569.

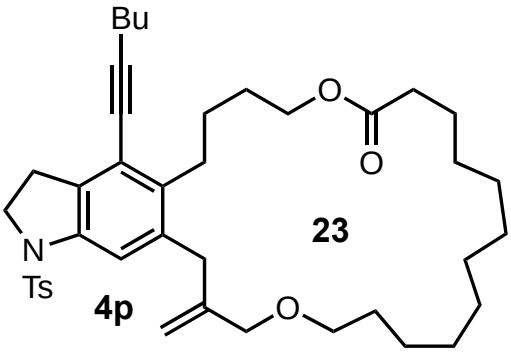


Method A. Yield: 53%. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.69–7.68 (m, 2H), 7.61 (s, 1H), 7.29–7.23 (m, 7H), 7.21–7.20 (m, 2H), 7.16 (d, 1H, *J* = 8.0 Hz), 6.96 (t, 1H, *J* = 7.0 Hz), 5.34 (d, 1H, *J* = 9.5 Hz), 5.12 (d, 1H, *J* = 10.0 Hz), 4.95 (s, 1H), 4.93 (s, 1H), 4.47–4.42 (m, 1H), 4.13–4.04 (m, 2H), 3.85 (q, 1H, *J* = 9.5 Hz), 3.64 (dd, 1H, *J* = 7.5, 4.5 Hz), 3.56 (d, 1H, *J* = 14.0 Hz), 14.5 Hz), 3.21 (dd, 1H, *J* = 14.0, 11.5 Hz), 3.09 (t, 2H, *J* = 8.5 Hz), 2.39 (s, 3H), 2.35 (dd, 1H, *J* = 14.0, 2.0 Hz), 2.31–2.25 (m, 1H), 2.07–2.02 (m, 1H), 1.94–1.88 (m, 1H), 1.83–1.77 (m, 1H), 1.26 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 157.5, 149.5, 144.4, 142.0, 140.7, 133.2, 132.9, 131.5, 130.9, 129.7, 129.5, 128.6, 128.3, 127.6,

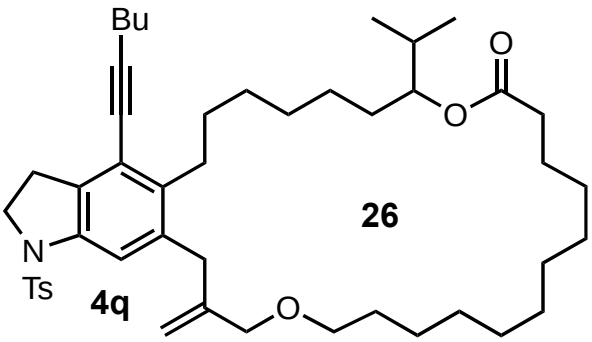
127.5, 127.4, 122.7, 121.7, 120.9, 116.0, 112.2, 111.6, 110.5, 96.9, 85.3, 76.8, 69.4, 65.7, 50.0, 40.6, 37.4, 32.6, 29.0, 27.9, 24.7, 21.5; HRMS (EEI) calcd for $C_{40}H_{40}NO_5S$ $[M+H]^+$ 646.2627, found 646.2631.



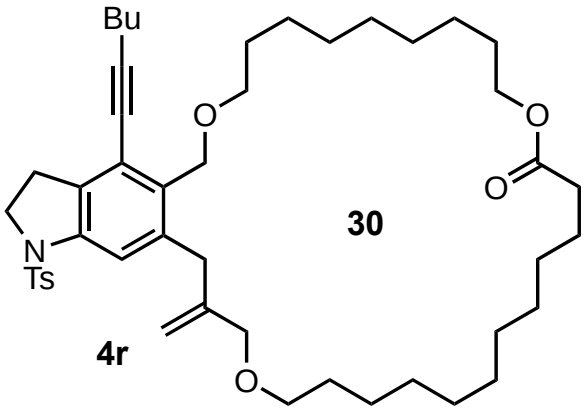
Method A. Yield: 39%. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.65–7.64 (m, 2H), 7.44–7.43 (m, 1H), 7.38 (s, 1H), 7.25–7.24 (m, 1H), 7.21–7.19 (m, 2H), 6.98 (t, 1H, *J* = 7.5 Hz), 6.89 (d, 1H, *J* = 8.0 Hz), 5.32 (s, 1H), 4.85 (d, 1H, *J* = 11.5 Hz), 4.70 (s, 1H), 4.54 (d, 1H, *J* = 11.5 Hz), 4.47 (d, 1H, *J* = 11.5 Hz), 4.40 (d, 1H, *J* = 11.5 Hz), 3.88 (dt, 2H, *J* = 8.5, 2.0 Hz), 3.60 (d, 1H, *J* = 17.0 Hz), 3.50 (d, 1H, *J* = 16.5 Hz), 3.17–3.14 (m, 1H), 2.91 (t, 2H, *J* = 8.5 Hz), 2.41 (t, 2H, *J* = 7.0 Hz), 2.37 (s, 3H), 1.93 –1.85 (m, 1H), 1.56–1.26 (m, 17H), 0.99 (d, 3H, *J* = 6.5 Hz), 0.94–0.90 (m, 6H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 156.2, 144.3, 144.0, 139.4, 139.3, 135.8, 133.7, 133.0, 129.8, 129.6, 128.5, 127.6, 127.4, 121.1, 120.7, 116.3, 114.6, 111.2, 97.6, 84.5, 71.2, 66.3, 49.9, 37.9, 31.7, 31.1, 30.9, 30.3, 29.3, 29.1, 28.1, 28.0, 24.6, 21.9, 21.5, 19.2, 18.7, 18.6, 13.6; HRMS (EEI) calcd for $C_{42}H_{54}NO_4S$ $[M+H]^+$ 668.3774, found 668.3774.



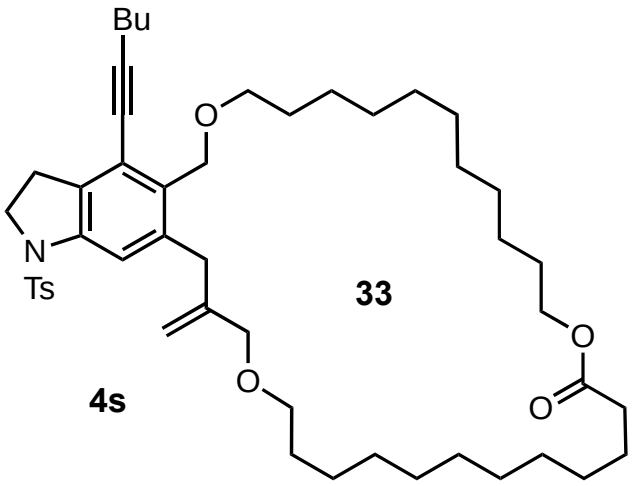
Method B. Yield: 42%. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.54–7.63 (m, 2H), 7.37 (s, 1H), 7.20–7.18 (m, 2H), 5.08 (s, 1H), 4.60 (s, 1H), 4.07 (t, 2H, *J* = 7.0 Hz), 3.90 (s, 2H), 3.88 (t, 2H, *J* = 8.5 Hz), 3.43–3.40 (m, 4H), 2.91 (t, 2H, *J* = 8.5 Hz), 2.73–2.70 (m, 2H), 2.42 (t, 2H, *J* = 6.8 Hz), 2.36 (s, 3H), 2.31 (t, 2H, *J* = 6.8 Hz), 1.71–1.25 (m, 28H), 0.92 (t, 3H, *J* = 7.2 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 174.0, 146.2, 144.0, 139.6, 137.8, 136.6, 133.6, 132.8, 129.5, 127.5, 121.1, 116.1, 114.0, 97.8, 77.2, 74.1, 70.1, 64.2, 49.9, 36.8, 34.2, 30.9, 30.3, 29.7, 28.8, 28.8, 28.3, 28.1, 27.9, 27.9, 27.5, 26.8, 26.0, 24.6, 21.9, 21.5, 19.2, 13.6; HRMS (EEI) calcd for $C_{41}H_{58}NO_5S$ $[M+H]^+$ 676.4036, found 676.4037.



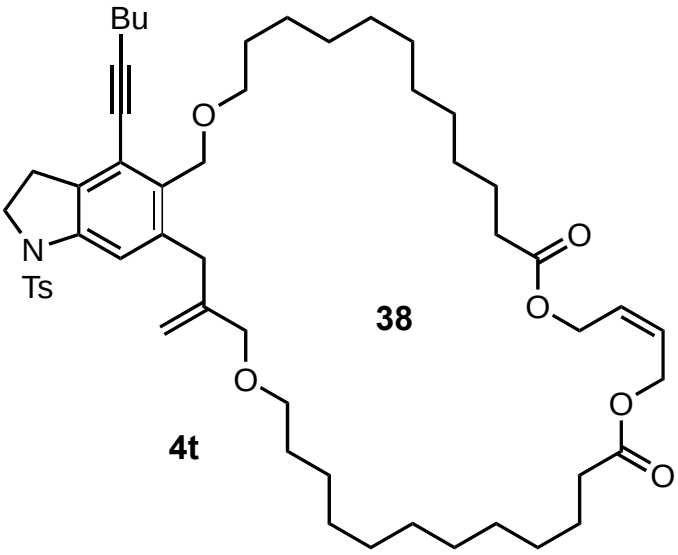
Method B. Yield: 41%. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.64–7.62 (m, 2H), 7.34 (s, 1H), 7.19–7.18 (m, 2H), 5.10 (s, 1H), 4.75–4.72 (m, 1H), 4.67–4.66 (m, 1H), 3.89–3.85 (m, 4H), 3.40–3.37 (m, 4H), 2.91 (t, 2H, *J* = 8.5 Hz), 2.68–2.64 (m, 2H), 2.42 (t, 2H, *J* = 6.8 Hz), 2.36 (s, 3H), 2.36–2.26 (m, 2H), 1.84–1.78 (m, 1H) 1.69–1.64 (m, 1H), 1.62–1.25 (m, 31H), 0.92 (t, 3H, *J* = 7.2 Hz), 0.87 (d, 6H, *J* = 6.5 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 173.8, 146.1, 144.0, 139.3, 138.7, 136.5, 133.6, 132.7, 129.5, 127.5, 120.9, 115.8, 114.3, 97.4, 78.3, 74.0, 70.1, 49.9, 36.6, 34.6, 31.5, 31.2, 31.0, 30.9, 30.9, 30.4, 30.0, 29.7, 29.0, 28.7, 28.6, 28.5, 28.1, 26.0, 25.9, 24.9, 21.9, 21.5, 19.2, 18.4, 18.0, 13.6; HRMS (EEI) calcd for $C_{47}H_{70}NO_5S$ $[M+H]^+$ 760.4975, found 760.4981.



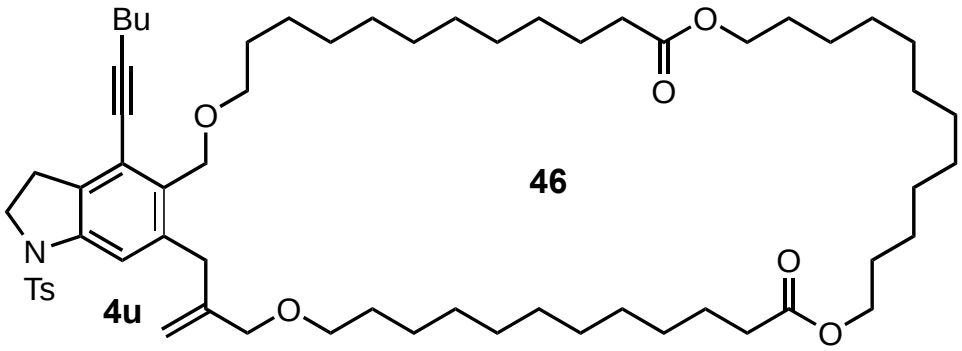
Method B. Yield: 34%. ^{1}H NMR ($CDCl_3$, 500 MHz): 7.65–7.63 (m, 2H), 7.40 (s, 1H), 7.20–7.18 (m, 2H), 5.07 (s, 1H), 4.57 (s, 1H), 4.51 (s, 2H), 4.09 (t, 2H, *J* = 5.5 Hz), 3.90–3.86 (m, 4H), 3.50 (s, 2H), 3.45 (t, 2H, *J* = 6.8 Hz), 3.42 (t, 2H, *J* = 6.5 Hz), 2.93 (t, 2H, *J* = 8.2 Hz), 2.41 (t, 2H, *J* = 6.8 Hz), 2.36 (s, 3H), 2.31 (t, 2H, *J* = 6.8 Hz), 1.61–1.28 (m, 36H), 0.91 (t, 3H, *J* = 7.0 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 174.0, 146.3, 144.1, 141.4, 139.4, 133.5, 132.8, 132.6, 129.6, 127.4, 122.4, 116.0, 113.5, 97.9, 76.7, 74.3, 71.0, 70.4, 67.8, 64.2, 49.9, 36.5, 34.6, 30.8, 30.0, 29.8, 29.6, 29.5, 29.5, 29.4, 29.3, 29.1, 28.8, 28.7, 27.9, 26.5, 26.3, 26.2, 25.2, 21.9, 21.5, 19.2, 13.6; HRMS (EEI) calcd for $C_{47}H_{70}NO_6S$ $[M+H]^+$ 776.4918, found 776.4918.



Method B. Yield: 37%. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.66–7.64 (m, 2H), 7.41 (s, 1H), 7.20–7.19 (m, 2H), 5.10 (s, 1H), 4.60 (s, 1H), 4.52 (s, 2H), 4.10 (t, 2H, *J* = 6.0 Hz), 3.90–3.86 (m, 4H), 3.49 (s, 2H), 3.46–3.41 (m, 4H), 2.93 (t, 2H, *J* = 8.2 Hz), 2.41 (t, 2H, *J* = 7.0 Hz), 2.37 (s, 3H), 2.31 (t, 2H, *J* = 6.8 Hz), 1.65–1.26 (m, 42H), 0,92 (t, 3H, *J* = 7.2 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 174.0, 146.2, 144.1, 141.4, 139.4, 133.5, 132.8, 132.5, 129.6, 127.4, 122.4, 116.0, 113.1, 97.9, 74.1, 70.9, 70.5, 67.8, 64.2, 49.9, 36.6, 34.6, 30.8, 30.0, 29.8, 29.6, 29.6, 29.5, 29.5, 29.2, 29.1, 28.9, 28.6, 27.9, 26.4, 26.3, 26.1, 25.2, 21.9, 21.5, 19.2, 13.6; HRMS (EEI) calcd for $C_{50}H_{76}NO_6S$ $[M+H]^+$ 818.5388, found 818.5388.

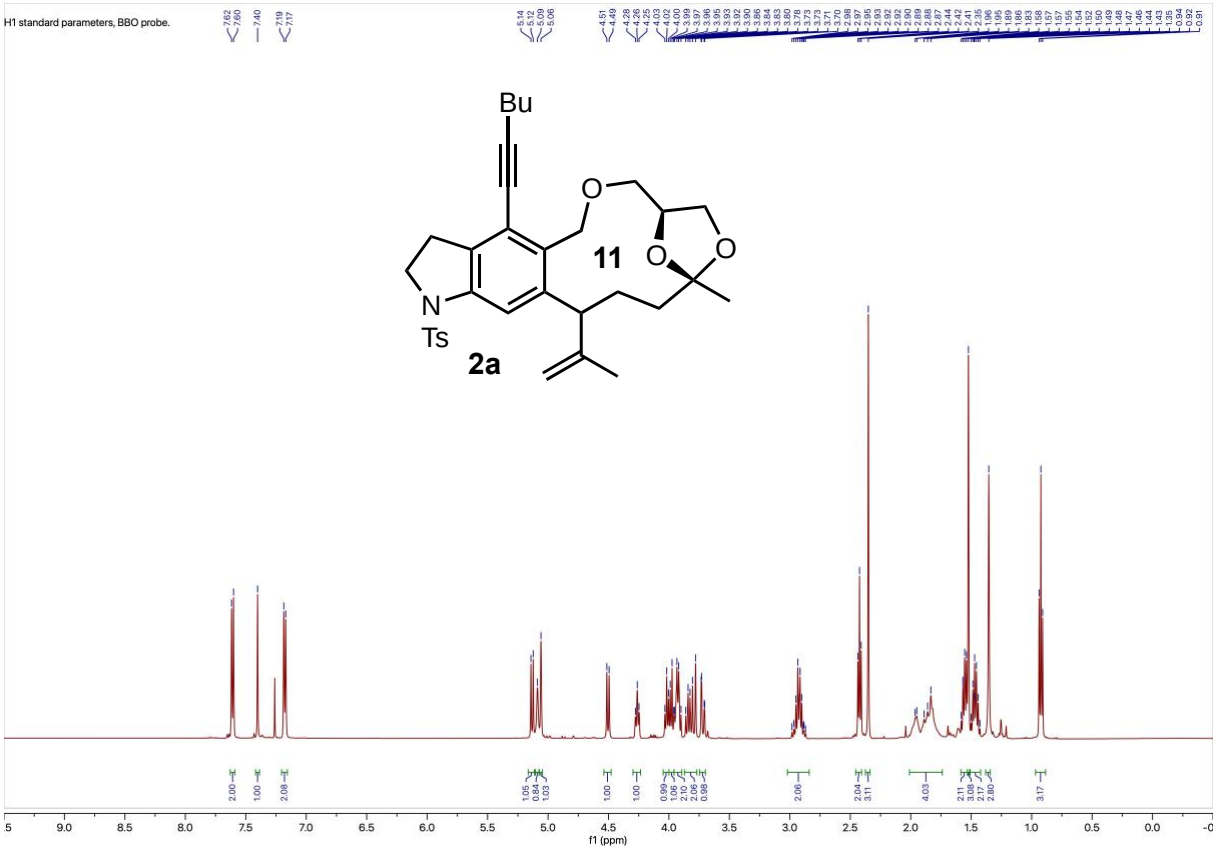


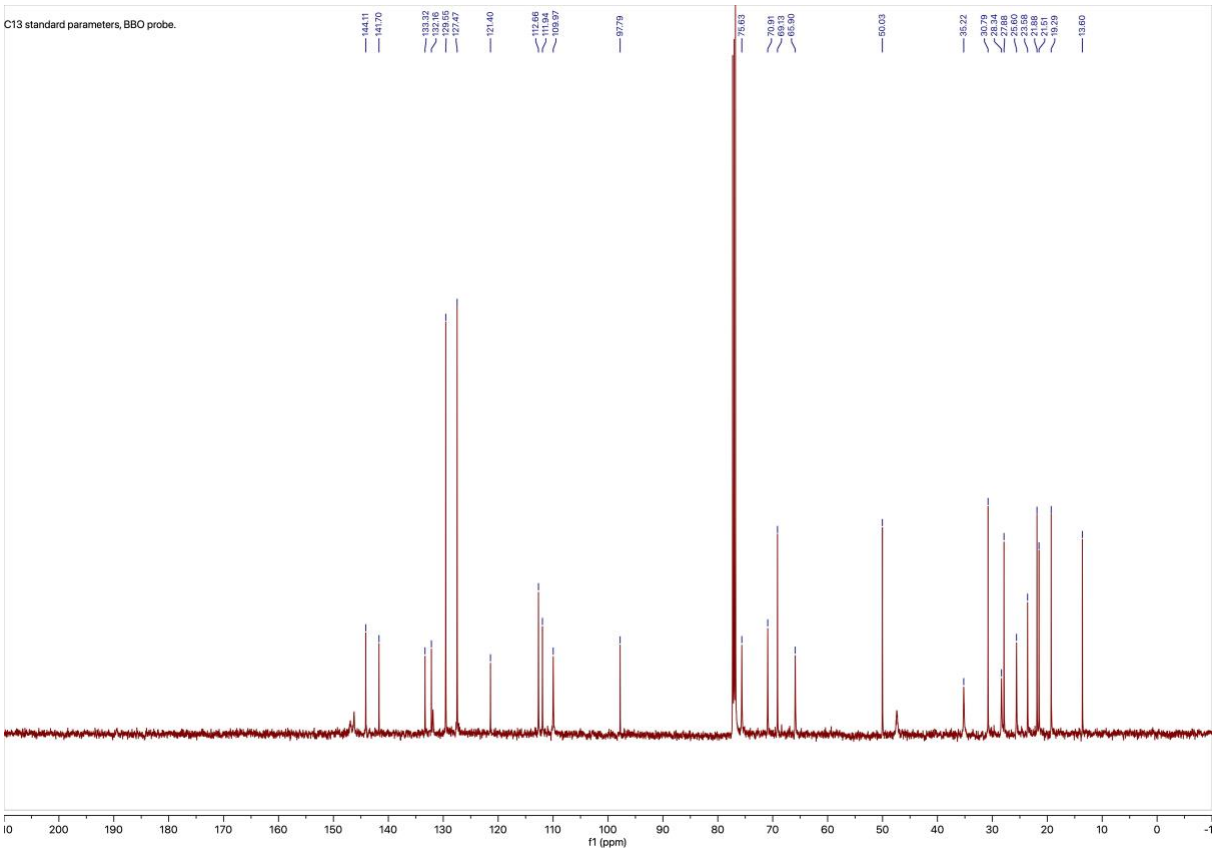
Method B. Yield: 25%. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.66–7.64 (m, 2H), 7.41 (s, 1H), 7.20–7.19 (d, *J* = 8.13 Hz, 2H), 5.79 (t, *J* = 4.09 Hz, 2H), 5.10 (s, 1H), 4.68 (m, 4H), 4.62 (s, 1H), 4.52 (s, 2H), 3.88 (m, 4H), 3.49 (s, 2H), 3.43 (m, 4H), 2.93 (t, *J* = 8.48 Hz, 2H), 2.41 (t, *J* = 6.90 Hz, 2H), 2.37 (s, 3H), 2.31 (t, *J* = 7.35 Hz, 4H), 1.67–1.19 (m, 40H), 0.92 (t, *J* = 7.26 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 173.4, 146.1, 144.1, 141.3, 139.4, 133.5, 132.8, 132.5, 129.6, 128.4, 128.3, 127.4, 122.4, 115.9, 113.0, 97.9, 73.9, 70.8, 70.4, 67.8, 59.6, 49.9, 36.6, 34.2, 30.8, 29.9, 29.8, 29.6, 29.6, 29.5, 29.5, 29.4, 29.3, 29.2, 29.1, 29.0, 28.9, 27.9, 26.3, 26.3, 24.9, 21.9, 21.5, 19.2, 13.6; HRMS (EEI) calcd for $C_{54}H_{80}NO_8S$ $[M+H]^+$ 902.5605, found 902.5610.

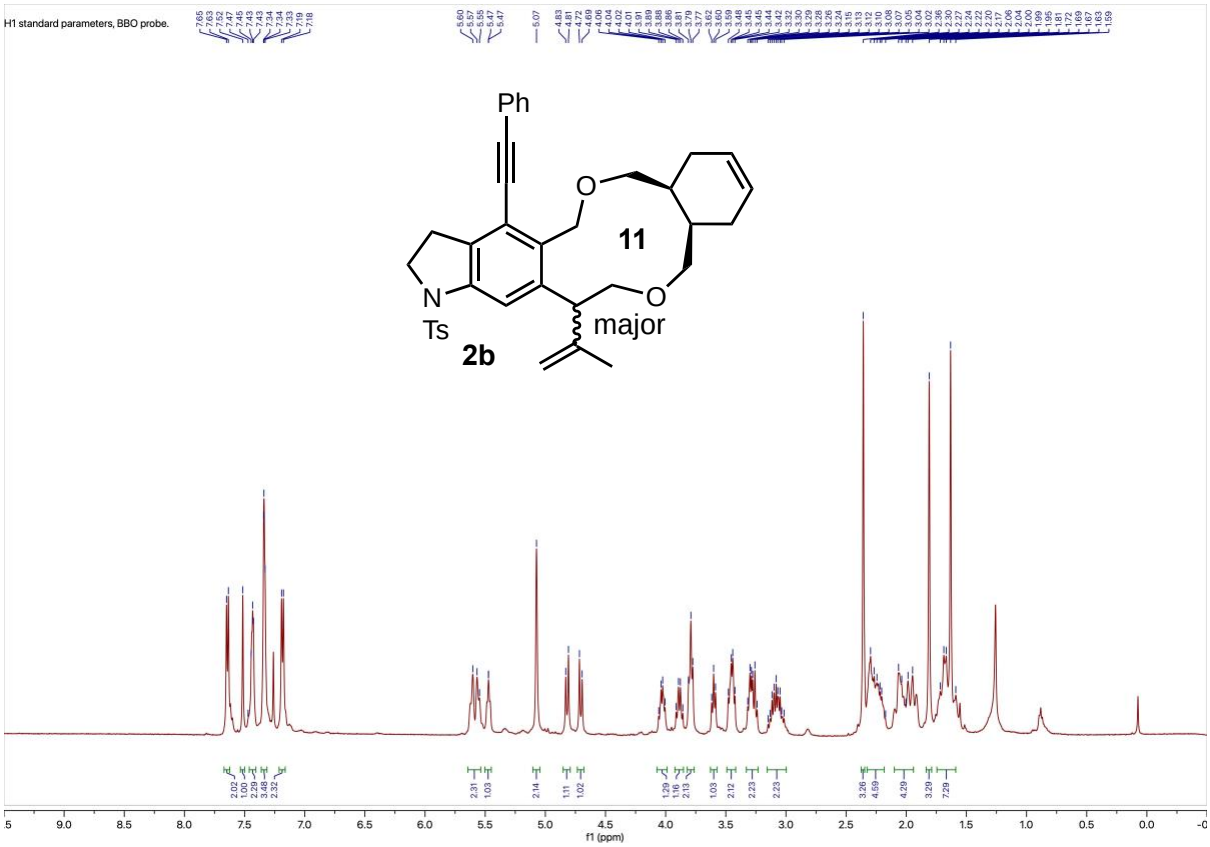


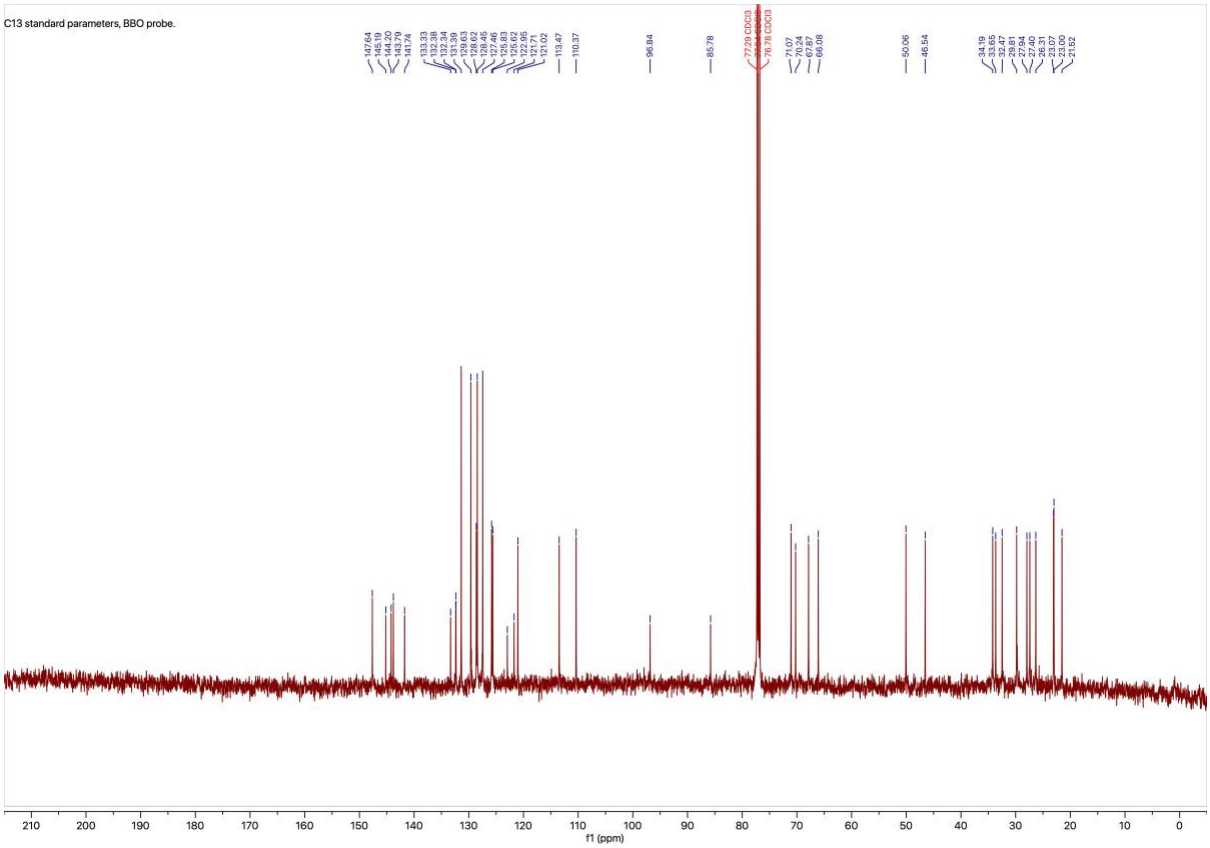
Method B. Yield: 12%. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.66–7.64 (m, 2H), 7.41 (s, 1H), 7.21–7.19 (m, 2H), 5.11 (s, 1H), 4.62 (s, 1H), 4.52 (s, 2H), 4.07 (t, 4H, *J* = 6.2 Hz), 3.89 (s, 2H), 3.86 (s, 2H), 3.49 (s, 2H), 3.45–3.41 (m, 4H), 2.93 (t, 2H, *J* = 8.5 Hz), 2.41 (t, 2H, *J* = 6.8 Hz), 2.37 (s, 3H), 2.29 (t, 4H, *J* = 7.2 Hz), 1.63–1.52 (m, 10H), 1.49–1.27 (m, 50H), 0.92 (t, 3H, *J* = 7.2 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 174.0, 154.2, 146.0, 144.1, 142.6, 141.4, 139.4, 133.6, 132.8, 132.5, 129.6, 127.4, 122.4, 115.9, 112.9, 111.7, 97.9, 74.8, 73.8, 70.8, 70.5, 70.2, 67.8, 64.4, 64.3, 49.9, 36.7, 36.0, 34.5, 32.8, 30.9, 30.8, 29.9, 29.8, 29.6, 29.5, 29.4, 29.2, 29.2, 29.1, 29.0, 28.7, 28.6, 27.9, 26.4, 26.3, 26.3, 26.2, 25.9, 25.5, 25.5, 25.3, 25.1, 24.7, 21.9, 21.5, 19.4, 19.2, 13.6. HRMS (EEI) calcd for $C_{62}H_{98}NO_8S$ $[M+H]^+$ 1016.7013, found 1016.6995.

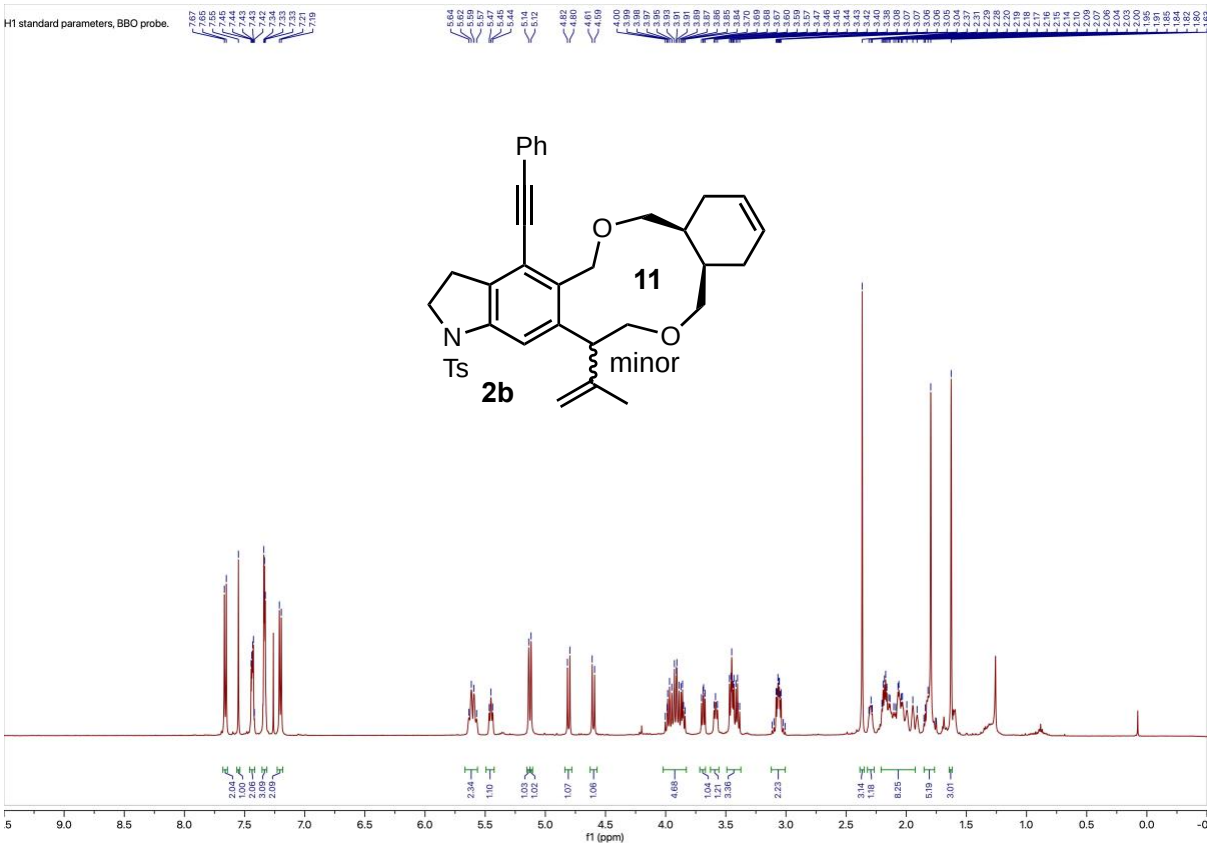
^{1}H and ^{13}C NMR Spectra

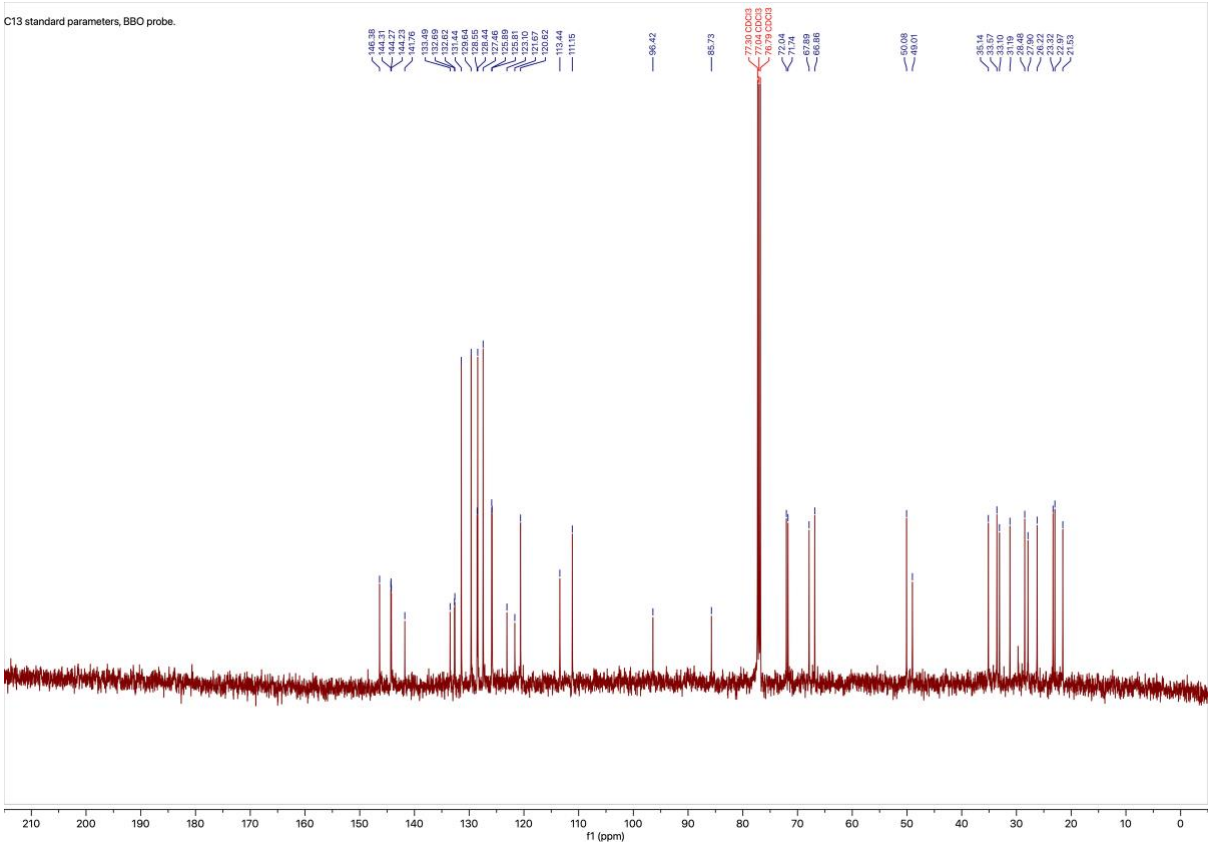


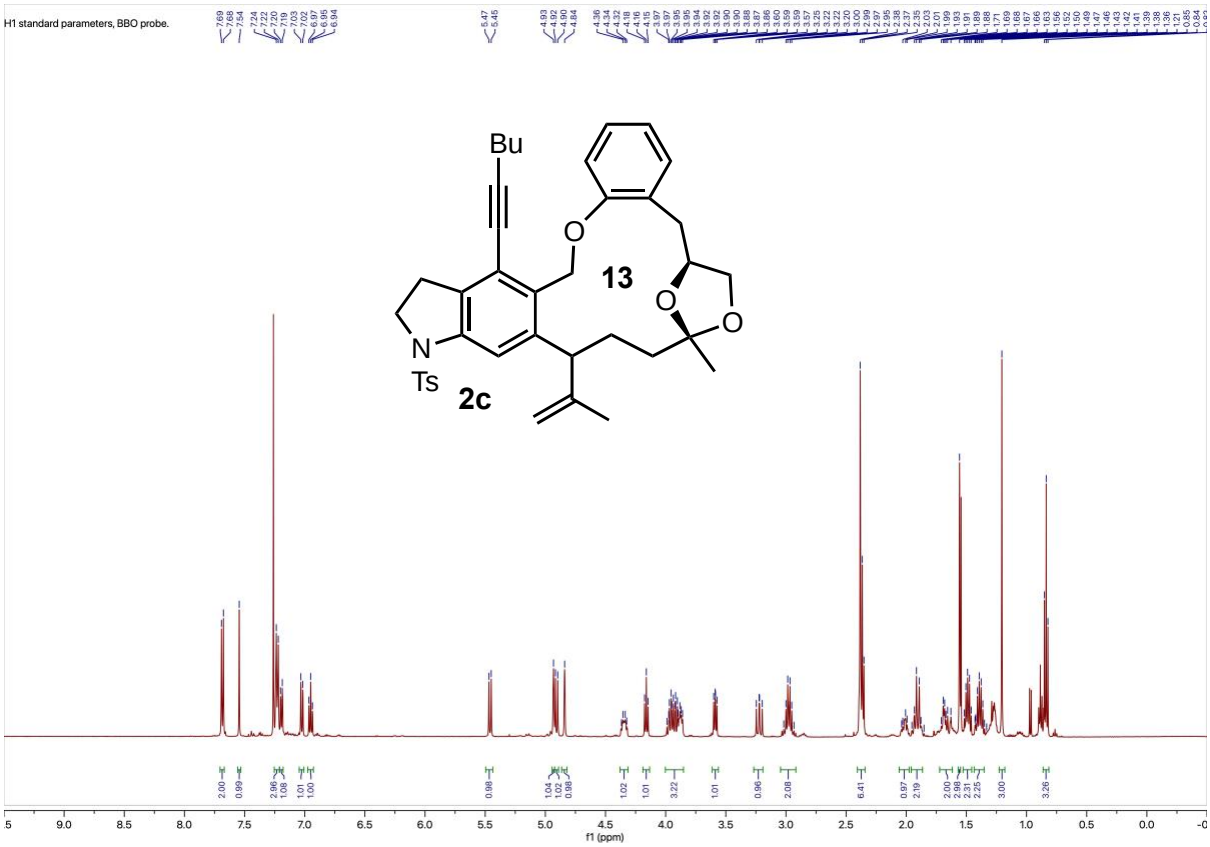


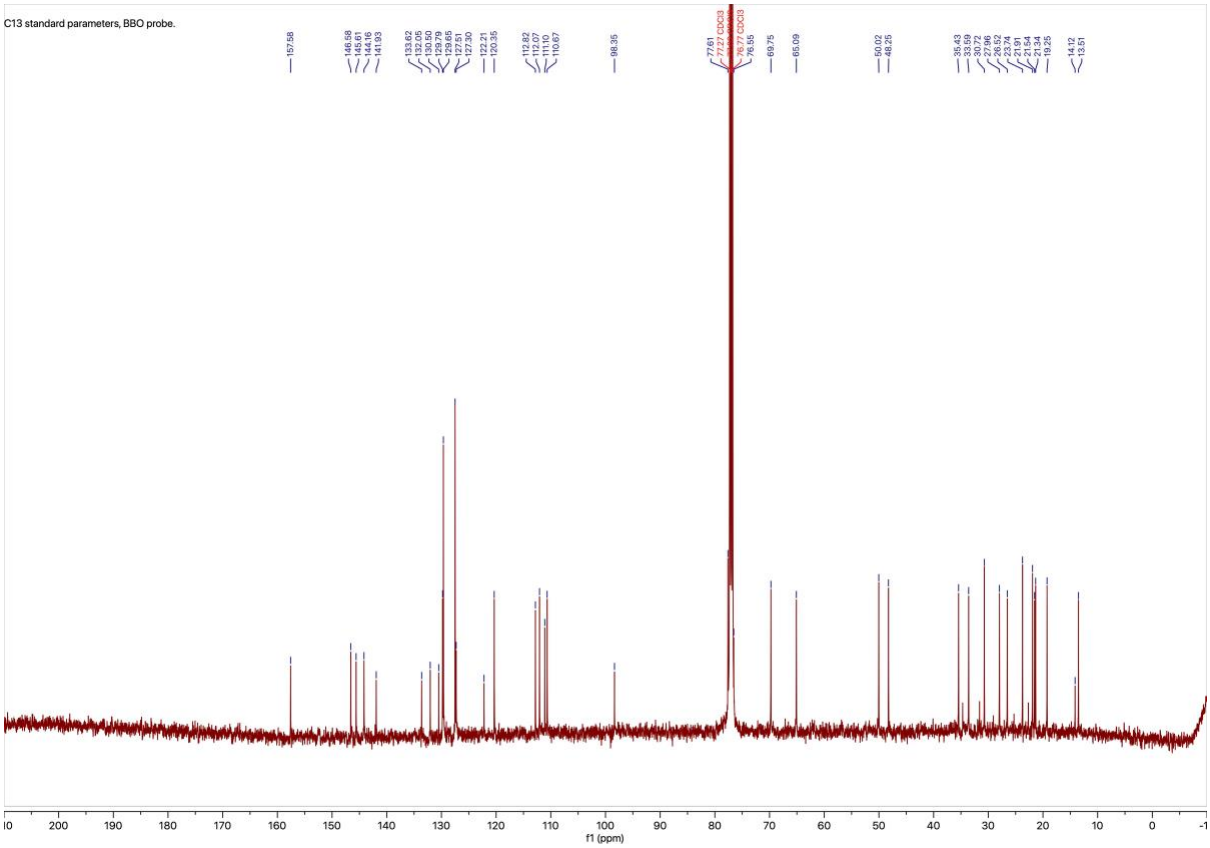

H1 standard parameters, BBO probe.
Ph
O
11
N
Ts
O
major
2b
f1 (ppm)

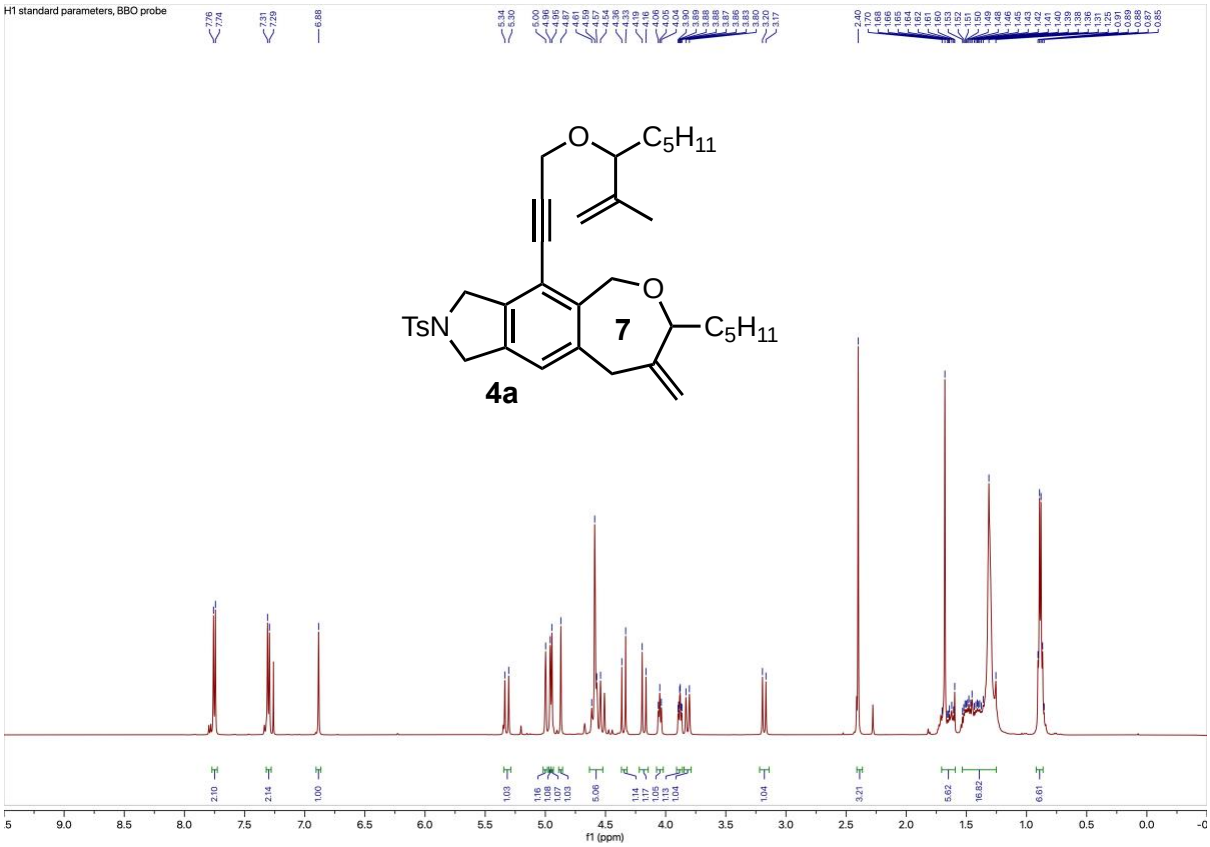

C13 standard parameters, BBO probe.
f1 (ppm)

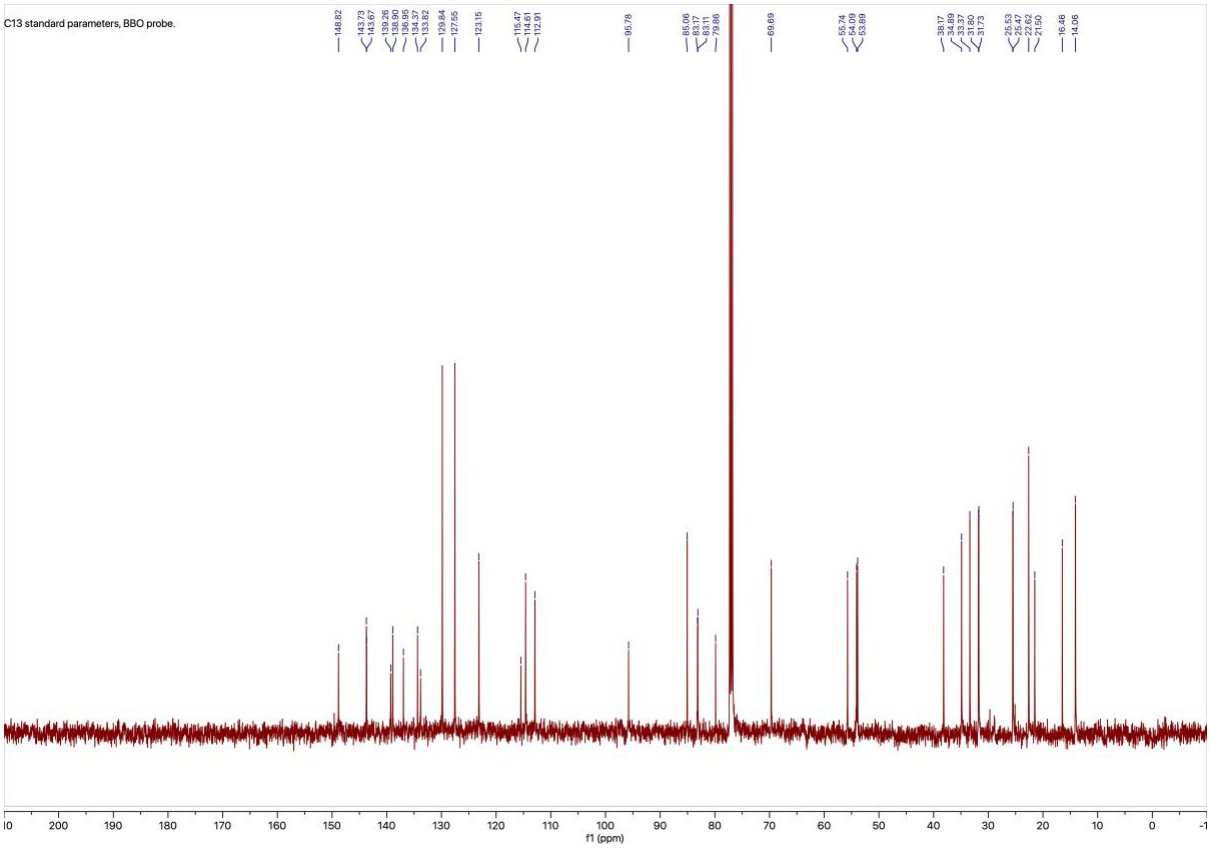

H1 standard parameters, BBO probe.
Ph
O
11
N
Ts
O
minor
2b
f1 (ppm)

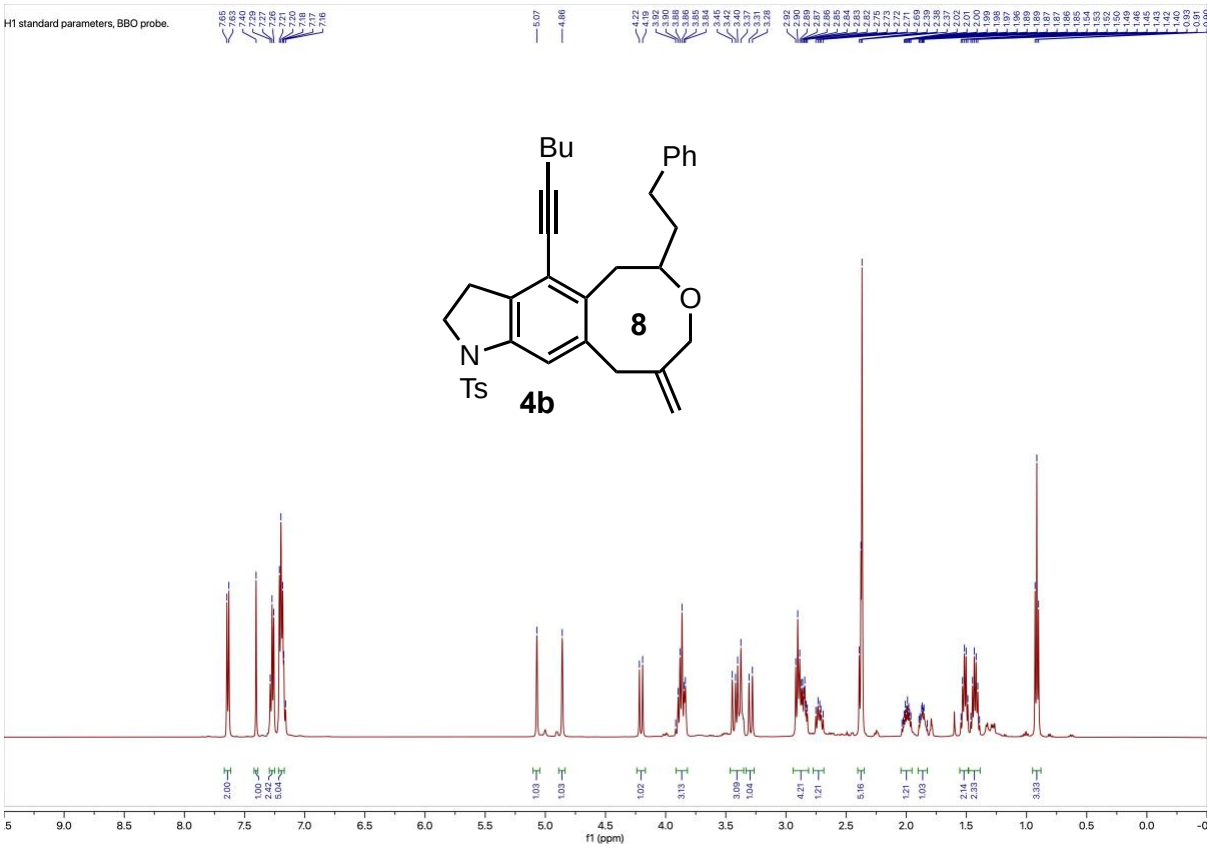

C13 standard parameters, BBO probe.
f1 (ppm)

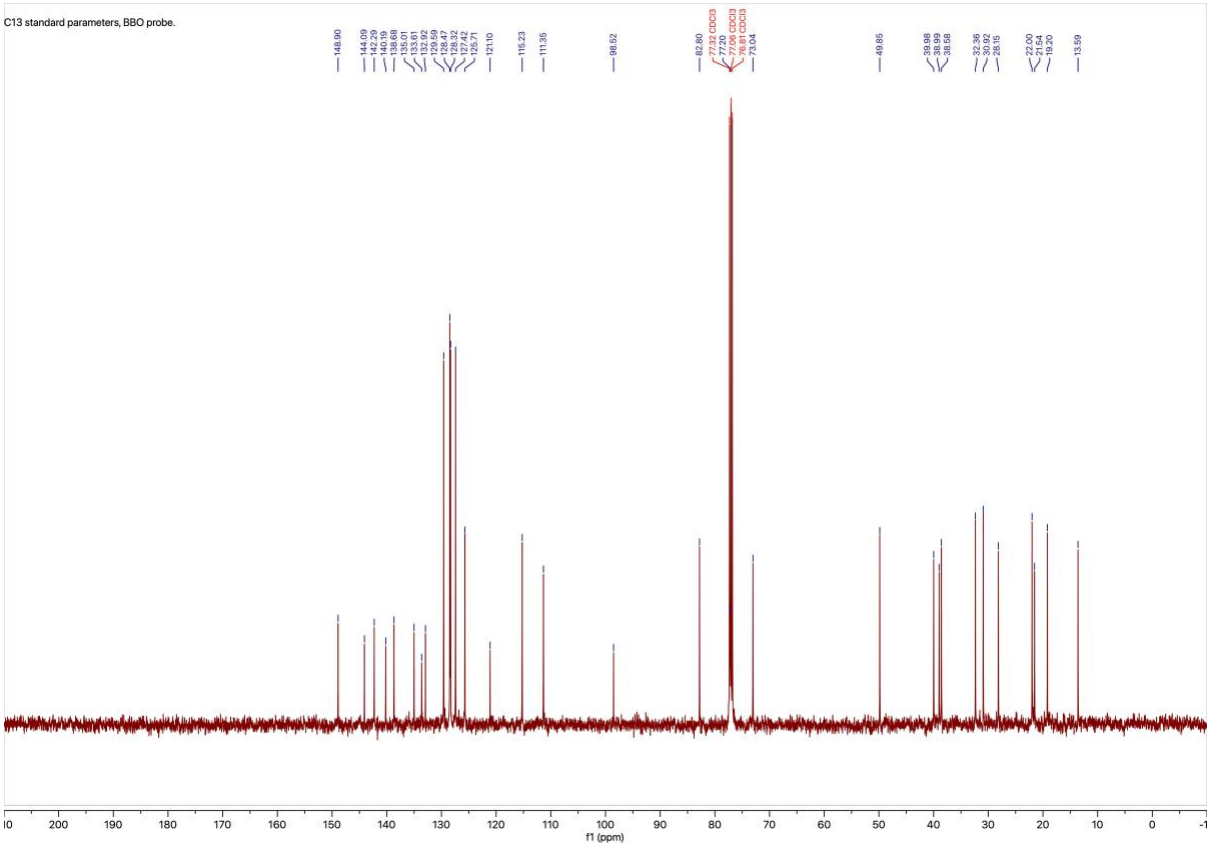

H1 standard parameters, BBO probe.
Bu
O
13
O
O
N
Ts
2c
f1 (ppm)

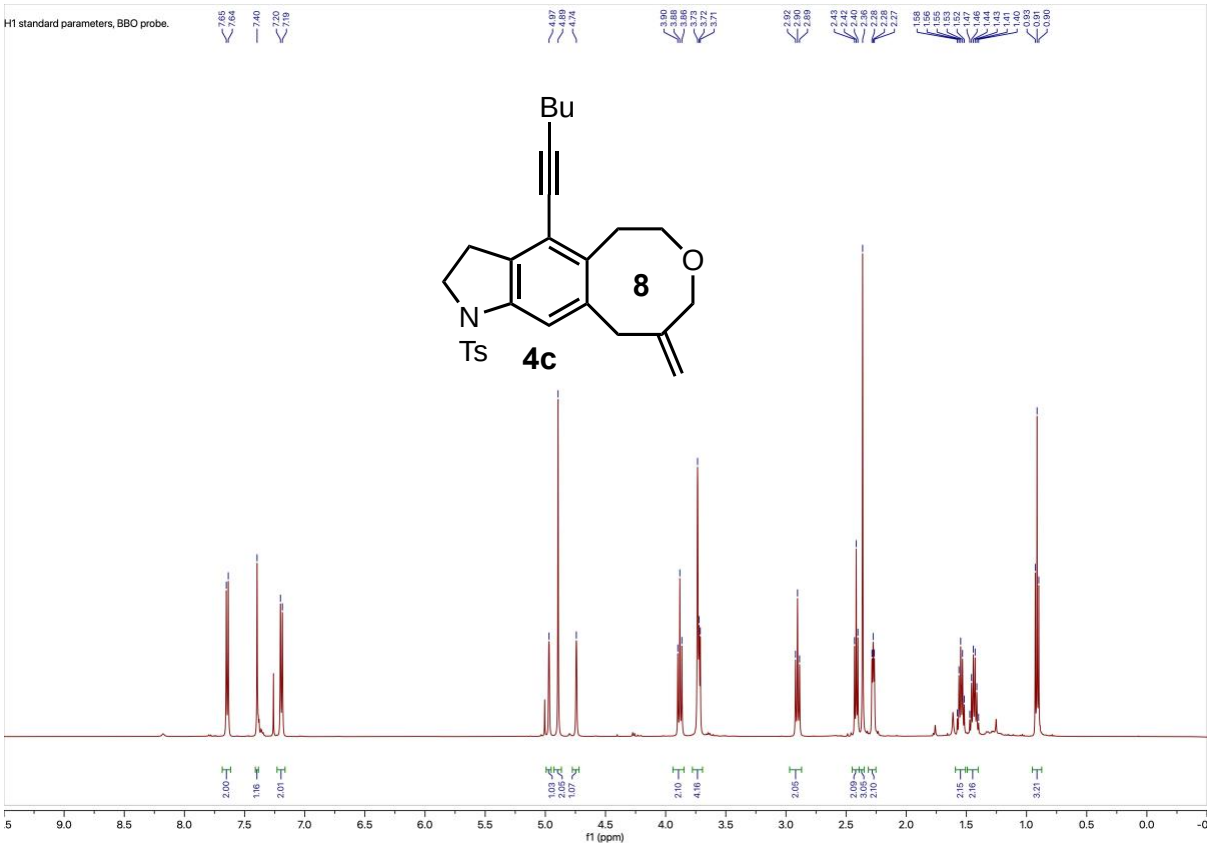

C13 standard parameters, BBO probe.
f1 (ppm)

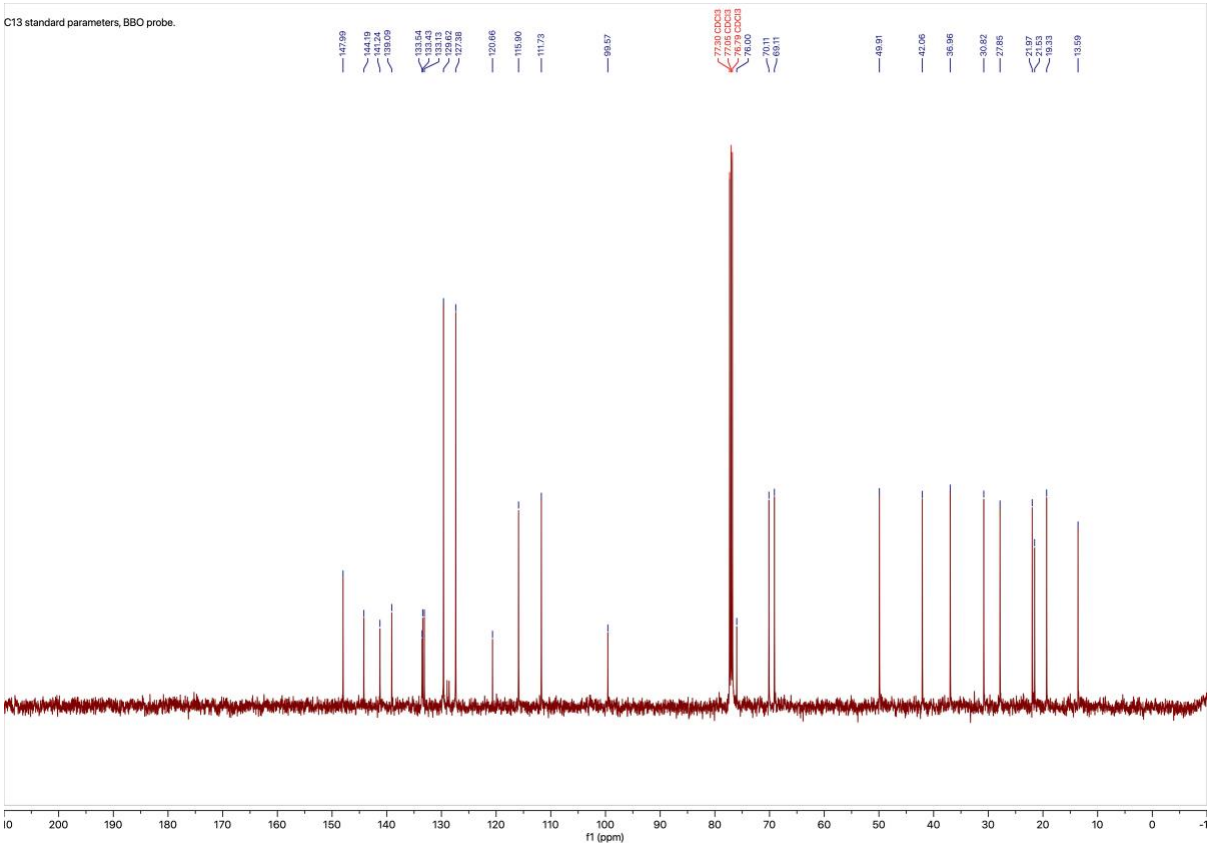

H1 standard parameters, BBO probe
O
C_5H_{11}
TsN
7
O
C_5H_{11}
4a
f1 (ppm)

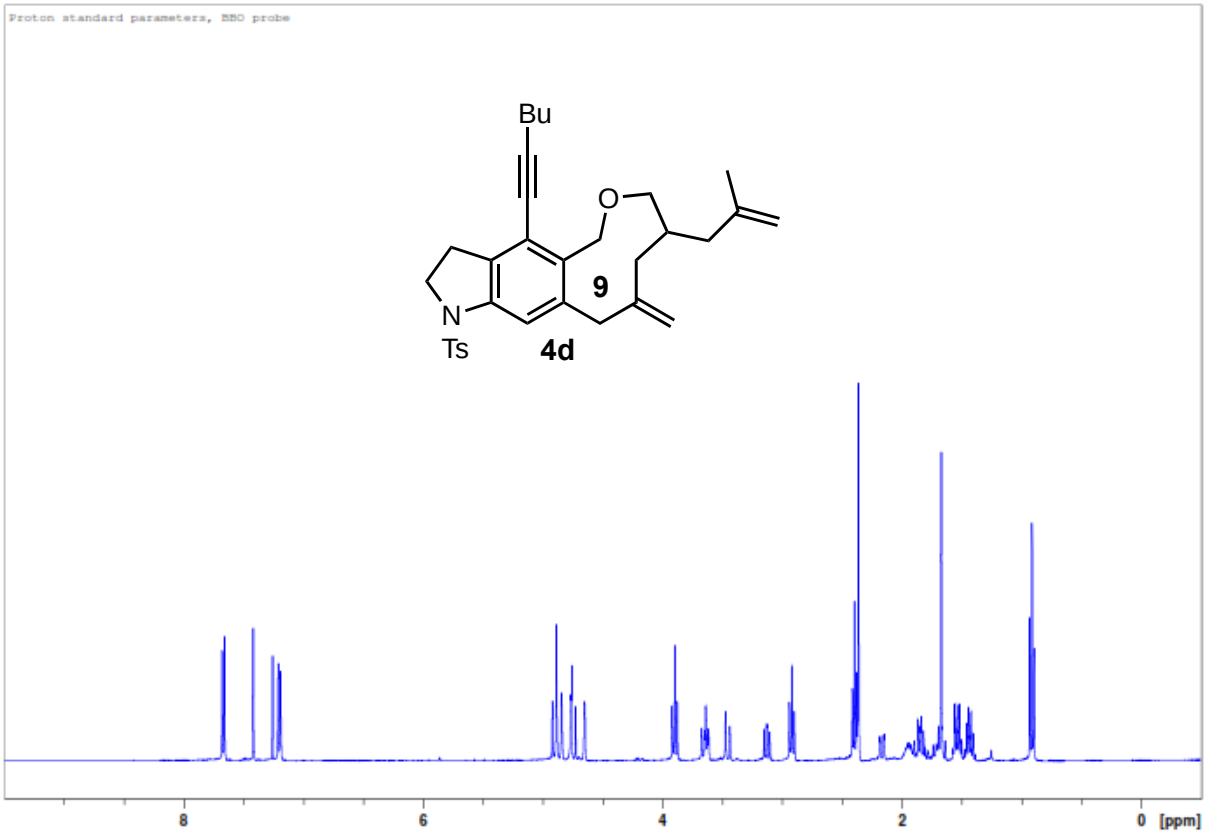

C13 standard parameters, BBO probe.
f1 (ppm)

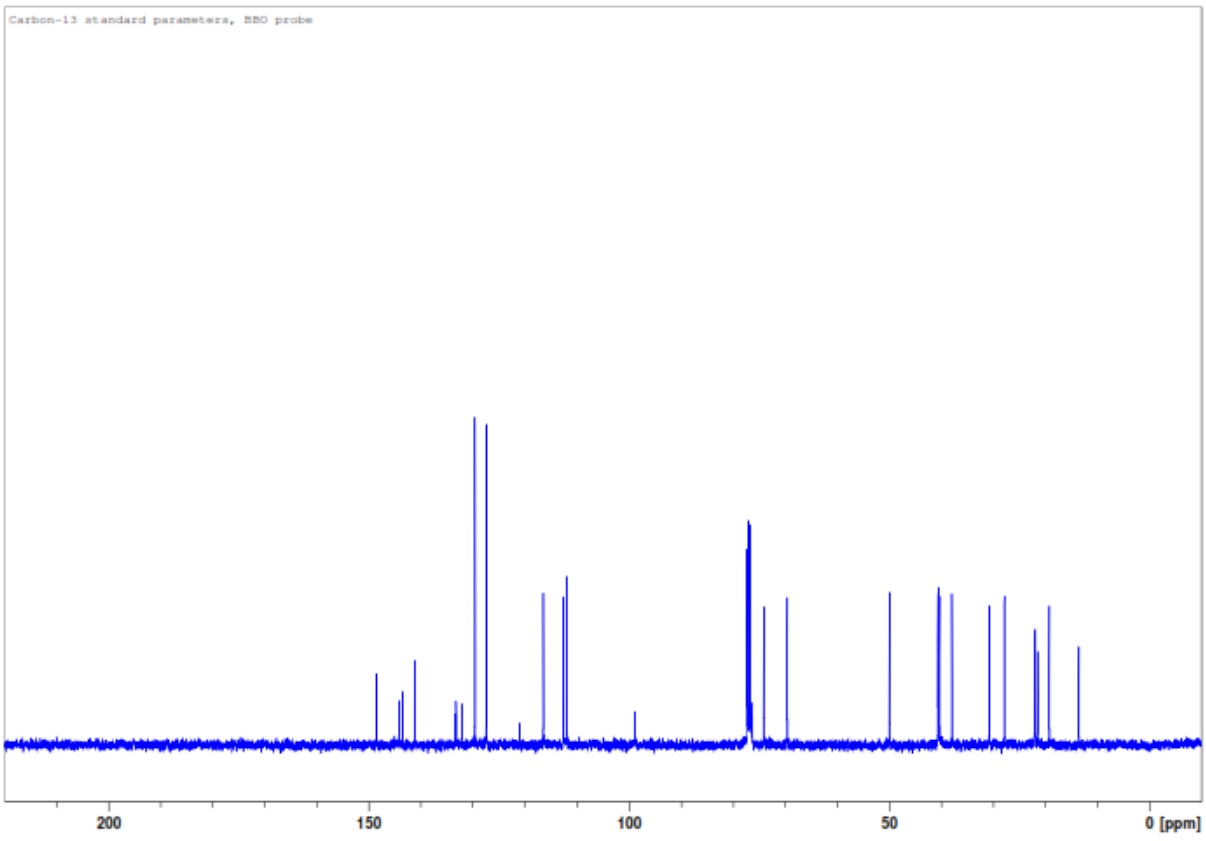

H1 standard parameters, BBO probe.
Bu
Ph
O
8
N
Ts
4b
f1 (ppm)

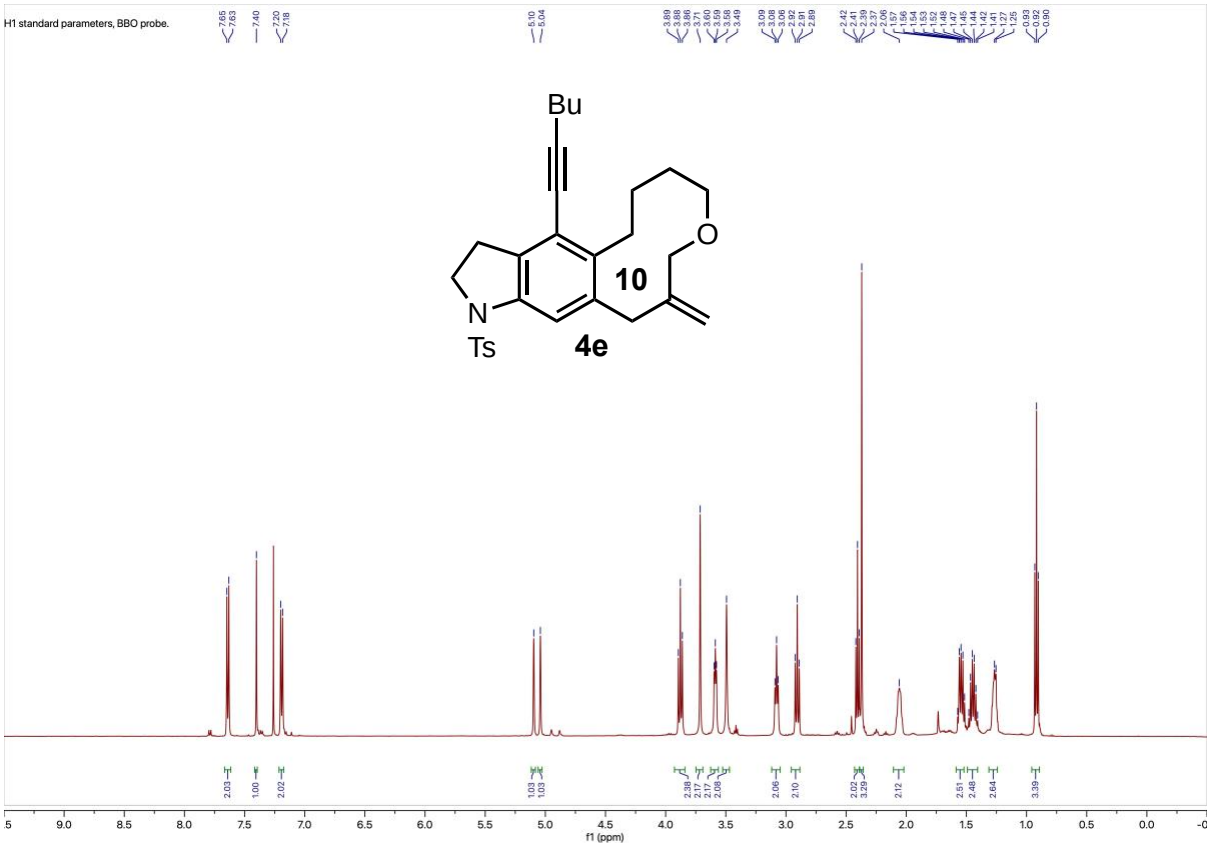

C13 standard parameters, BBO probe.
f1 (ppm)

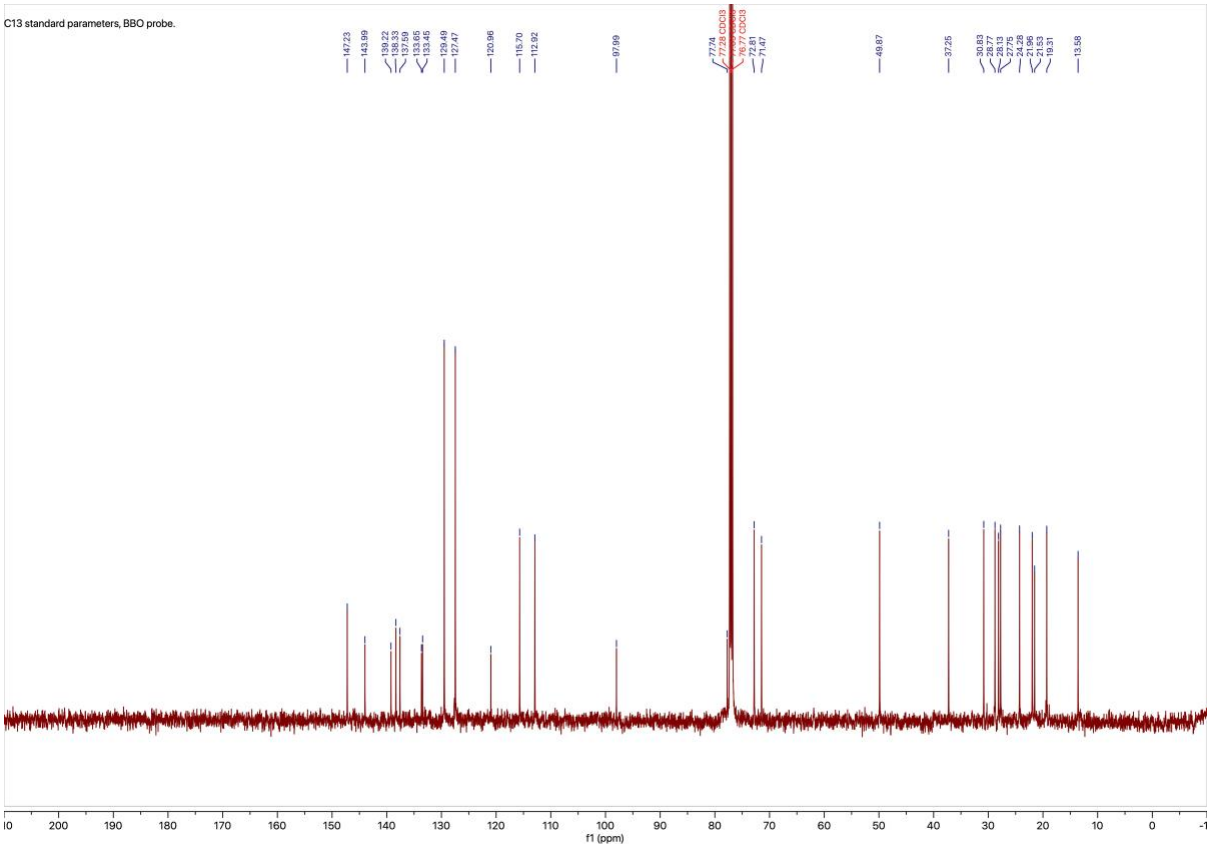

H1 standard parameters, BBO probe.
Bu
O
8
N
Ts
4c
f1 (ppm)

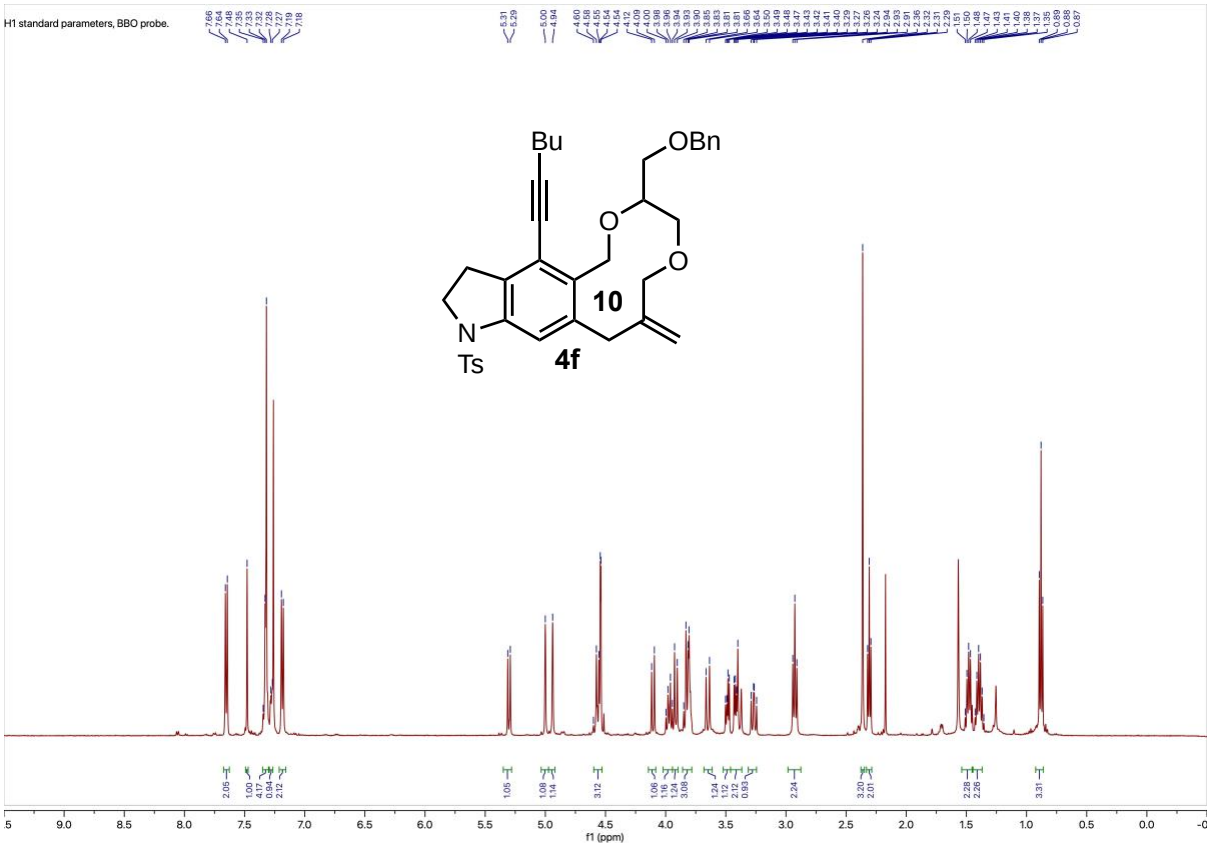

C13 standard parameters, BBO probe.
f1 (ppm)

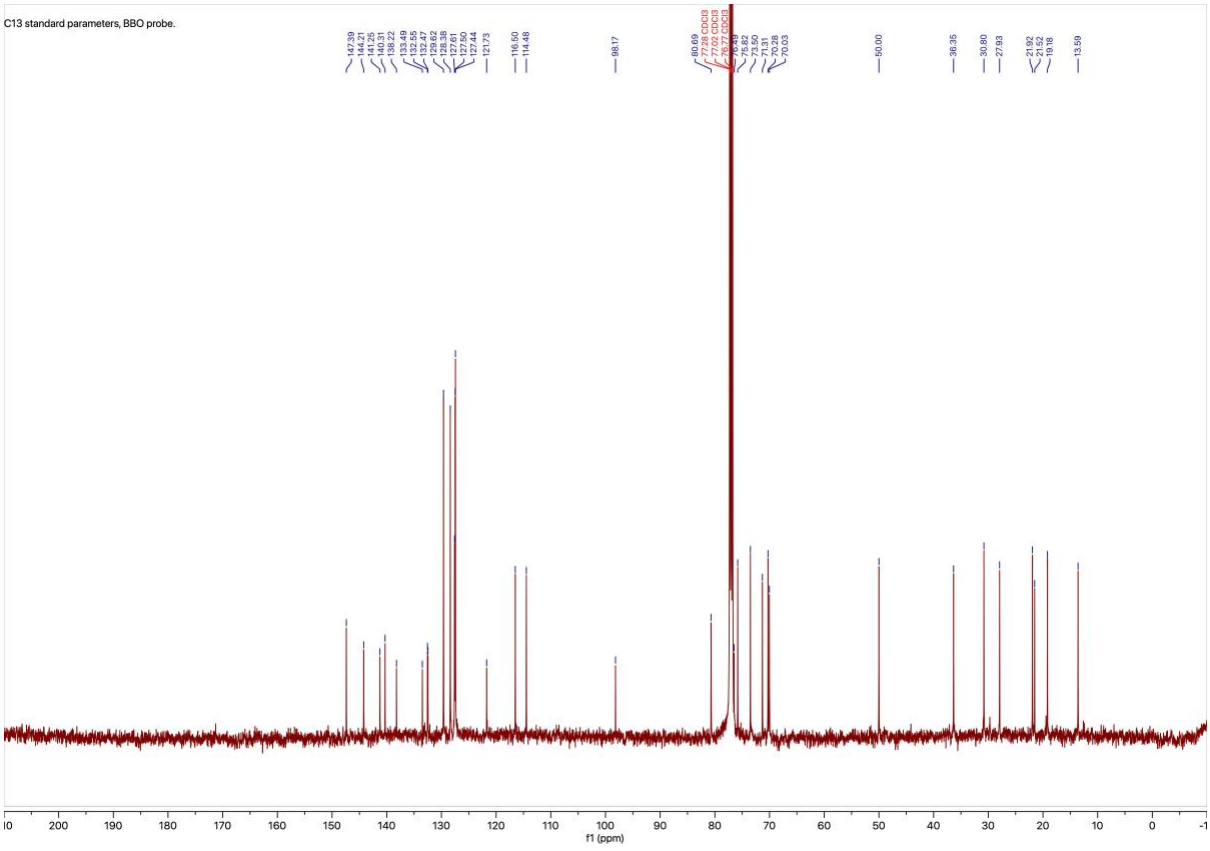

Proton standard parameters, BBO probe
Bu
O
9
N
Ts
4d
8
6
4
2
0 [ppm]

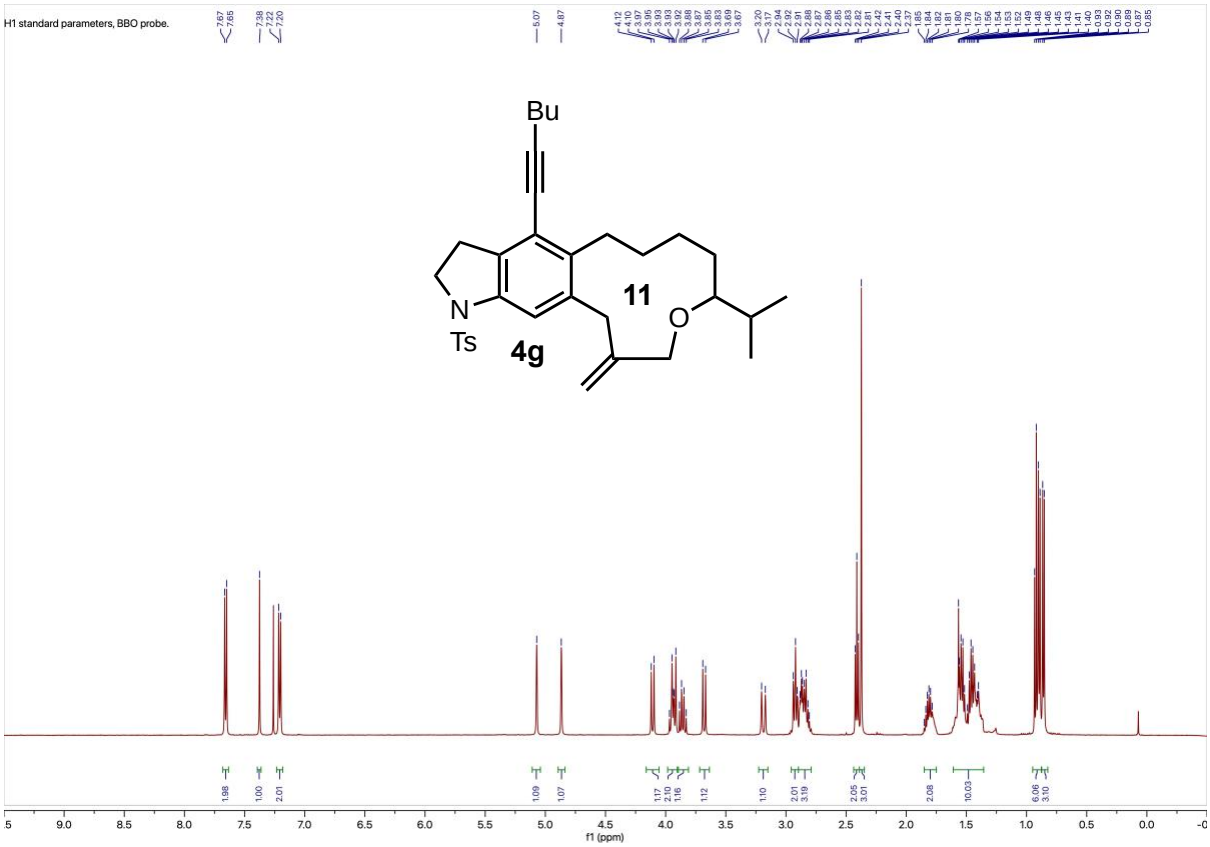

Carbon-13 standard parameters, BBO probe
200
150
100
50
0 [ppm]

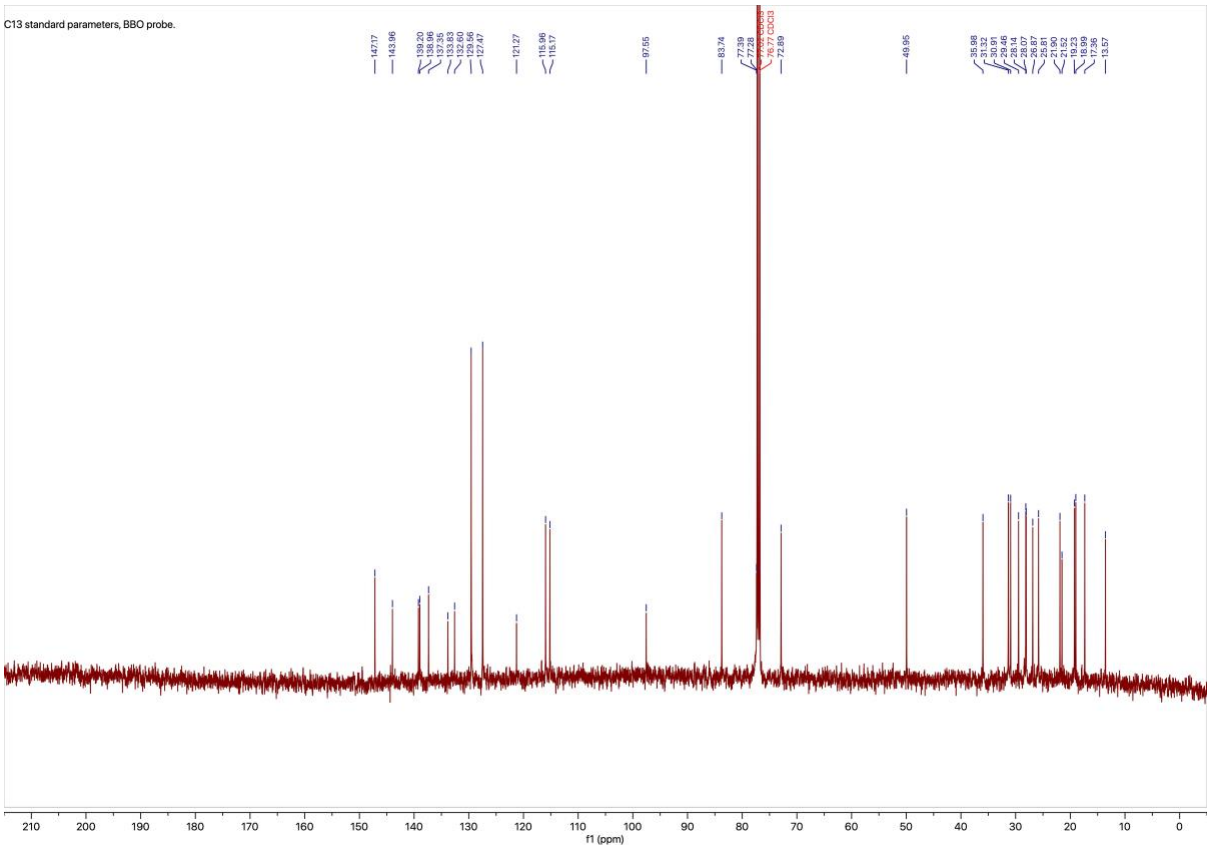

H1 standard parameters, BBO probe.
Bu
O
10
N
Ts
4e
f1 (ppm)

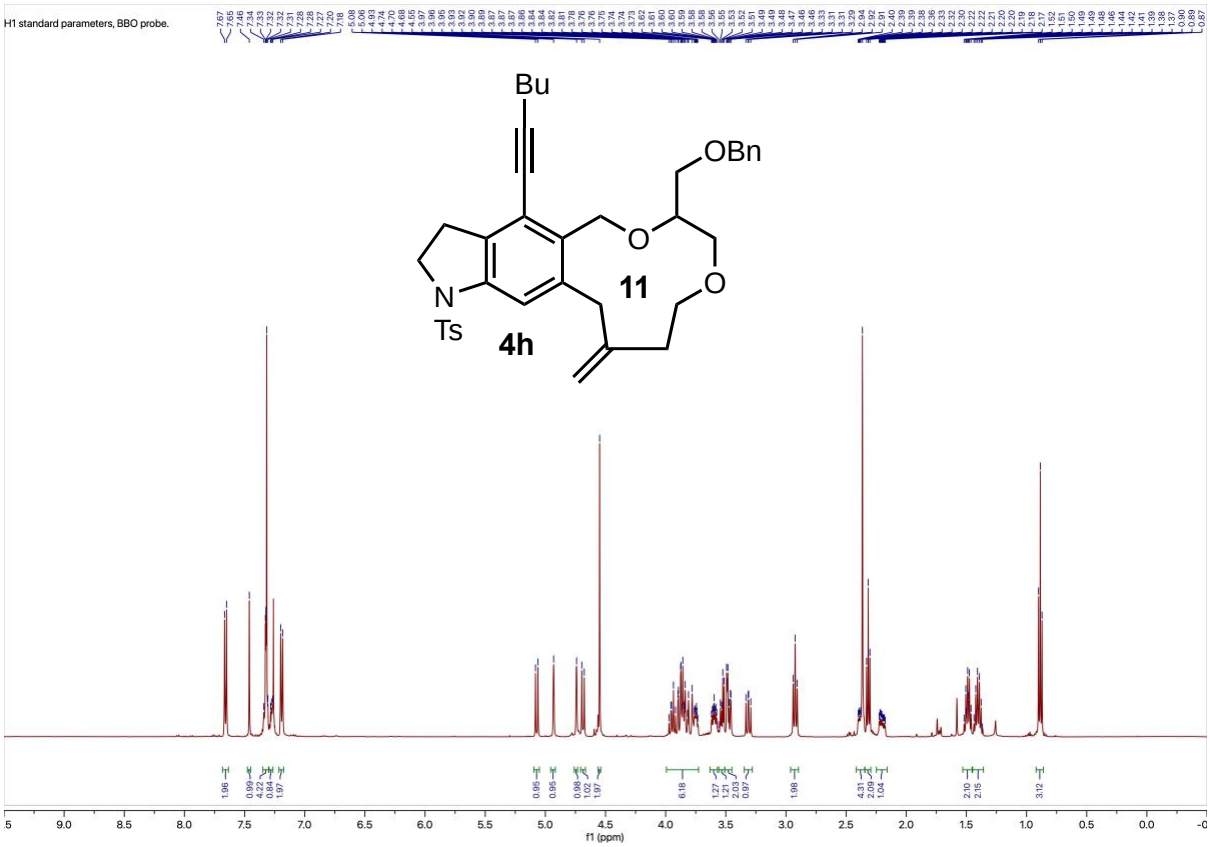

C13 standard parameters, BBO probe.
f1 (ppm)

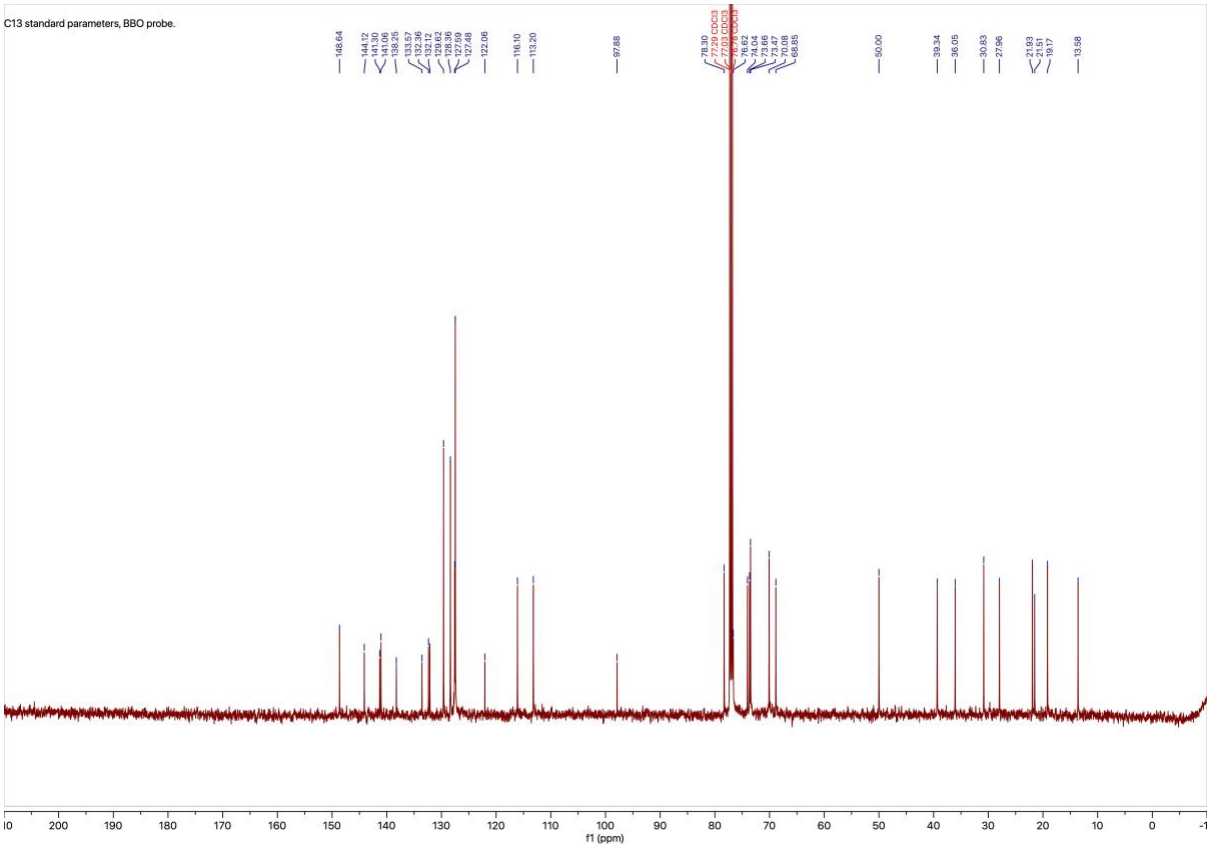

H1 standard parameters, BBO probe.
Bu
OBn
O
O
10
N
Ts
4f
f1 (ppm)

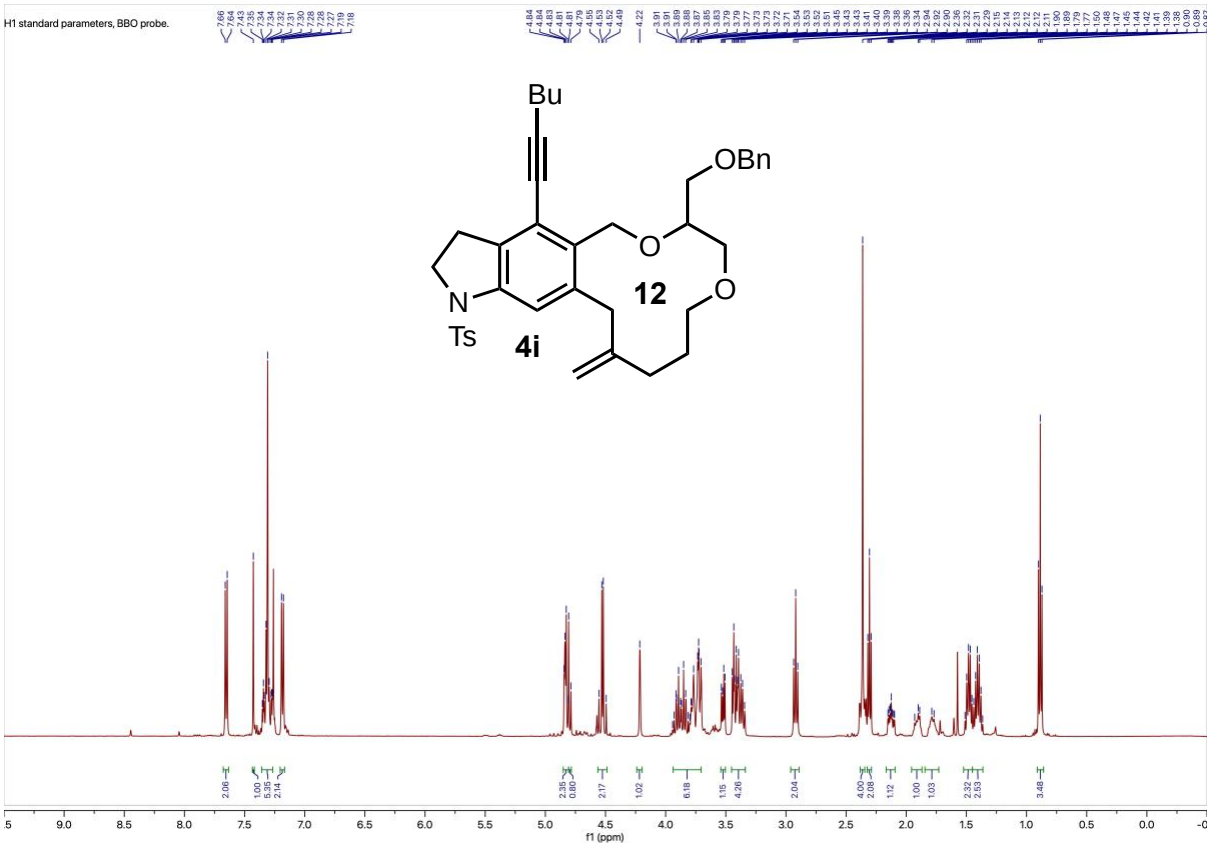

C13 standard parameters, BBO probe.
f1 (ppm)

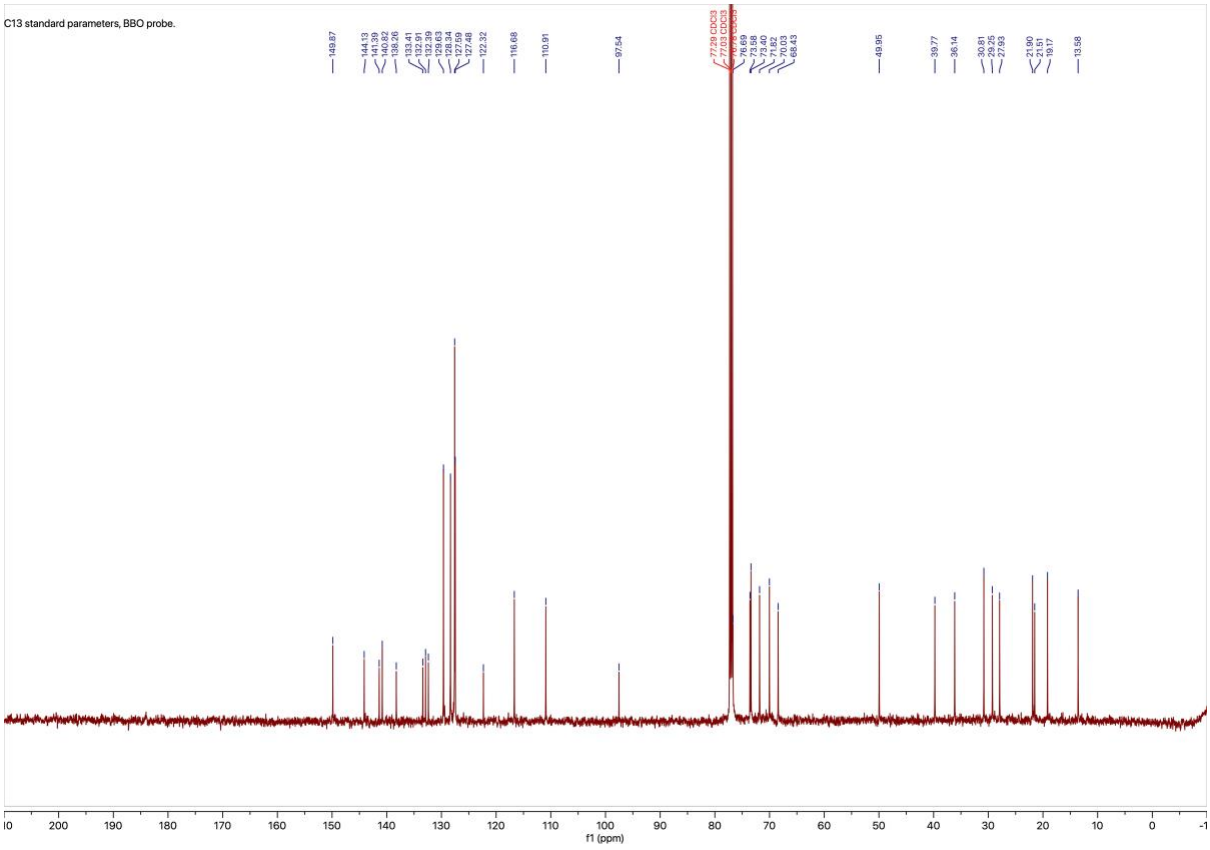

H1 standard parameters, BBO probe.
Bu
11
N
Ts
4g
O
f1 (ppm)


C13 standard parameters, BBO probe.
f1 (ppm)


H1 standard parameters, BBO probe.
Bu
OBn
O
O
N
Ts
11
4h
f1 (ppm)


C13 standard parameters, BBO probe.
f1 (ppm)


H1 standard parameters, BBO probe.
Bu
OBn
O
12
O
N
Ts
4i
f1 (ppm)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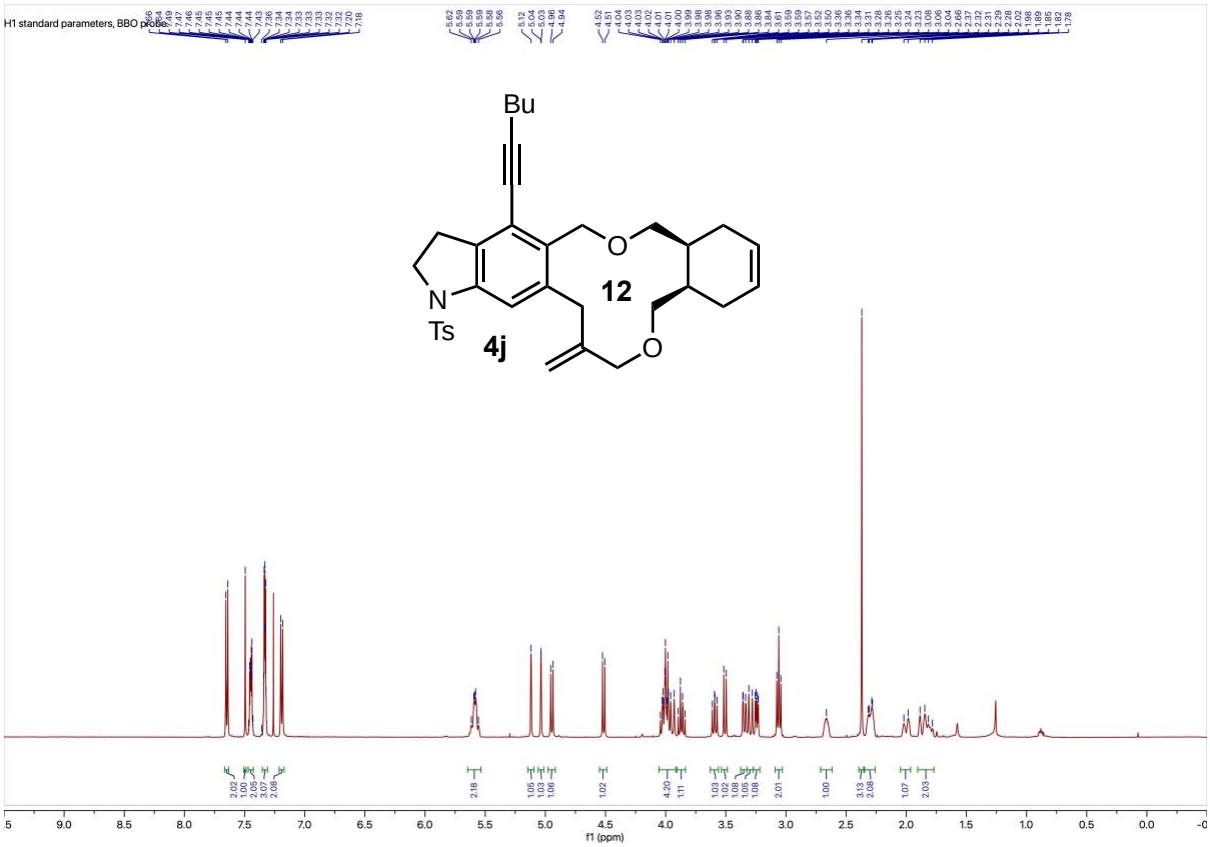
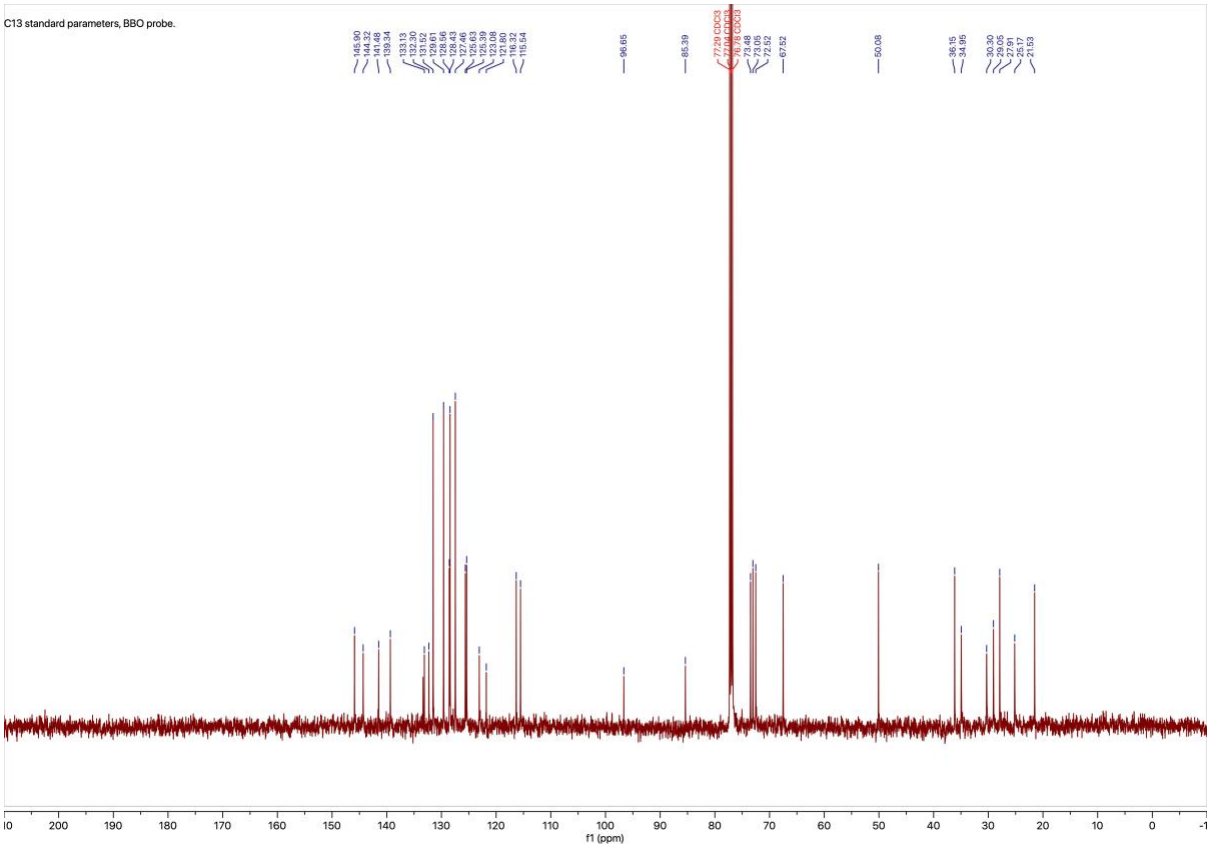
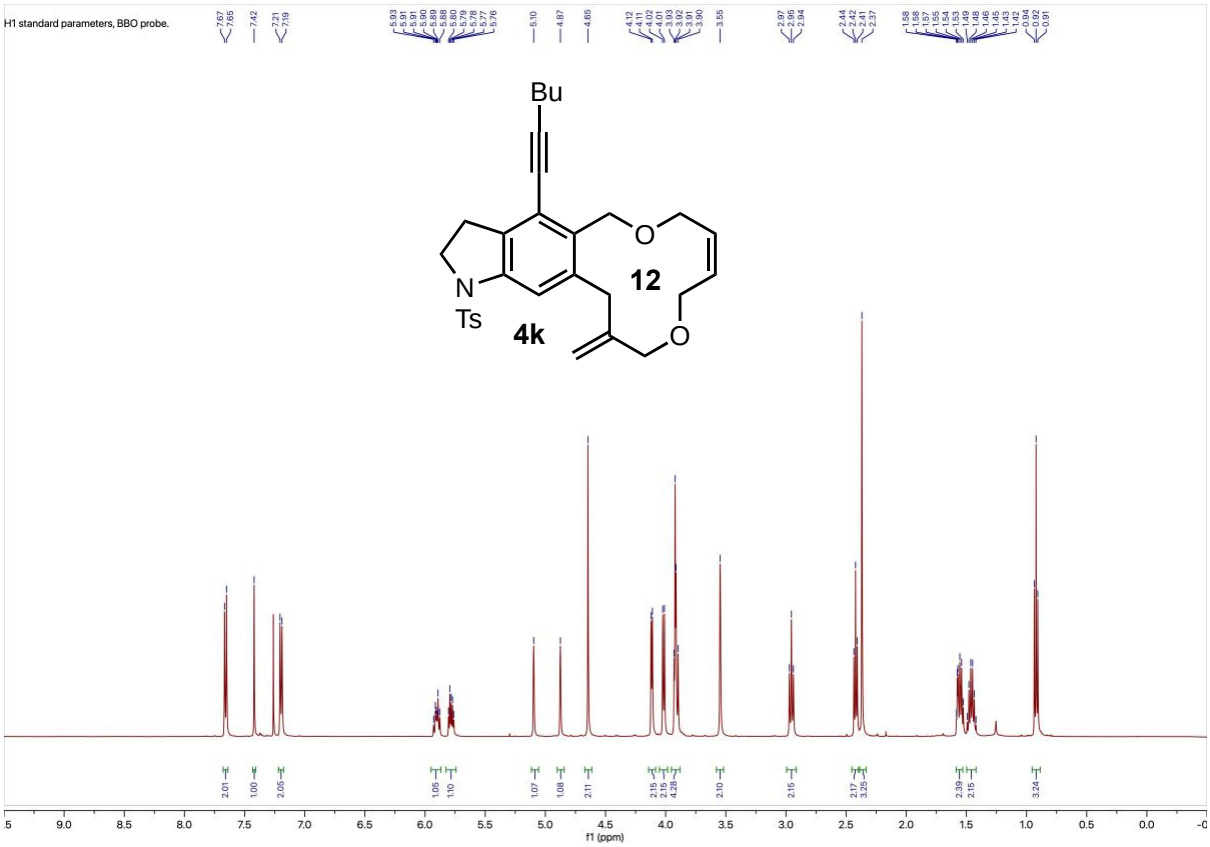
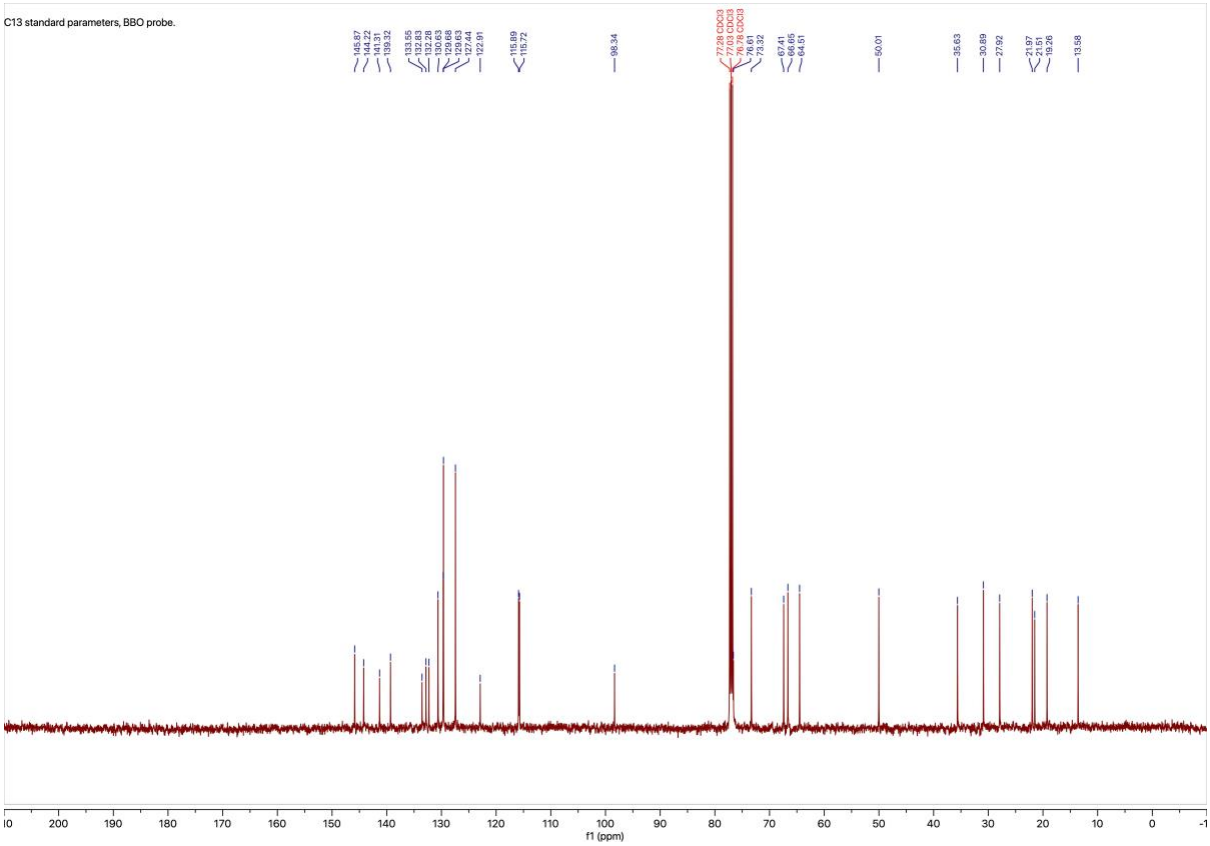
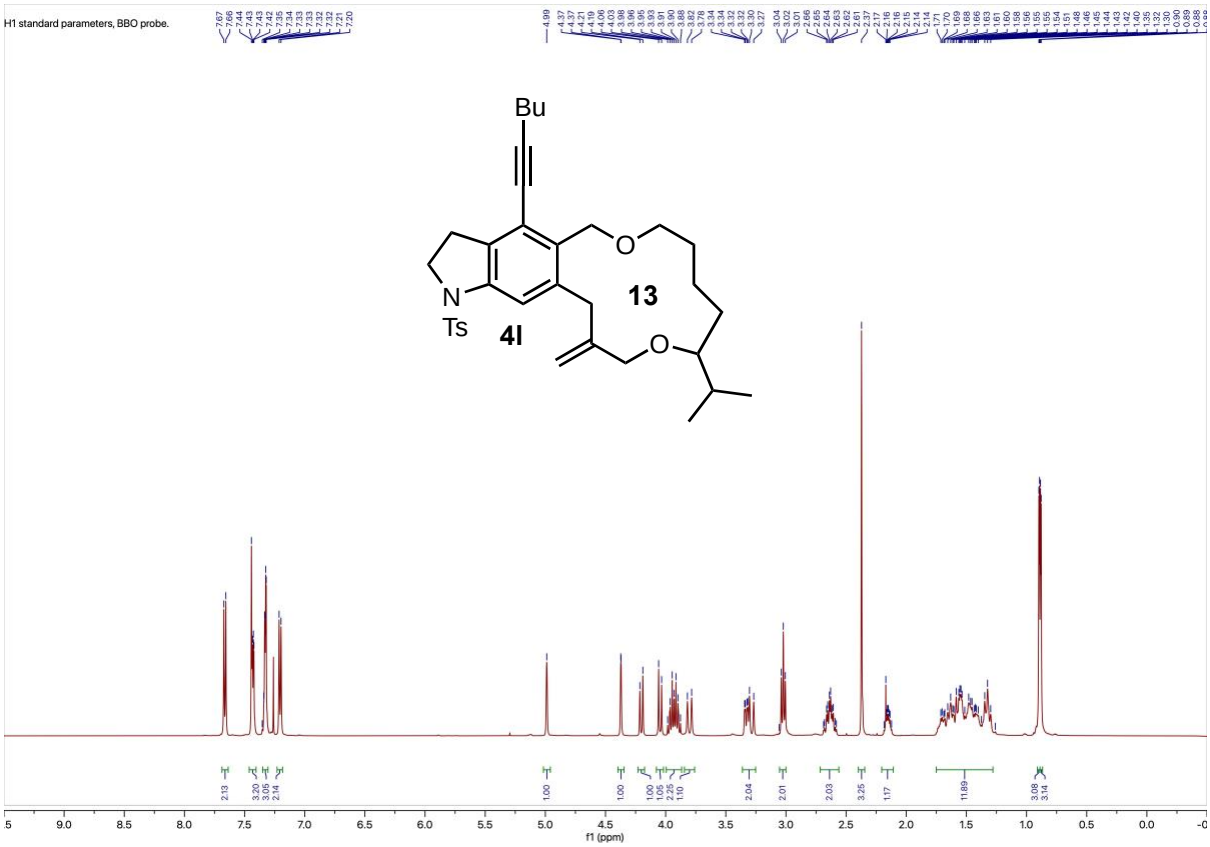
f1 (ppm)

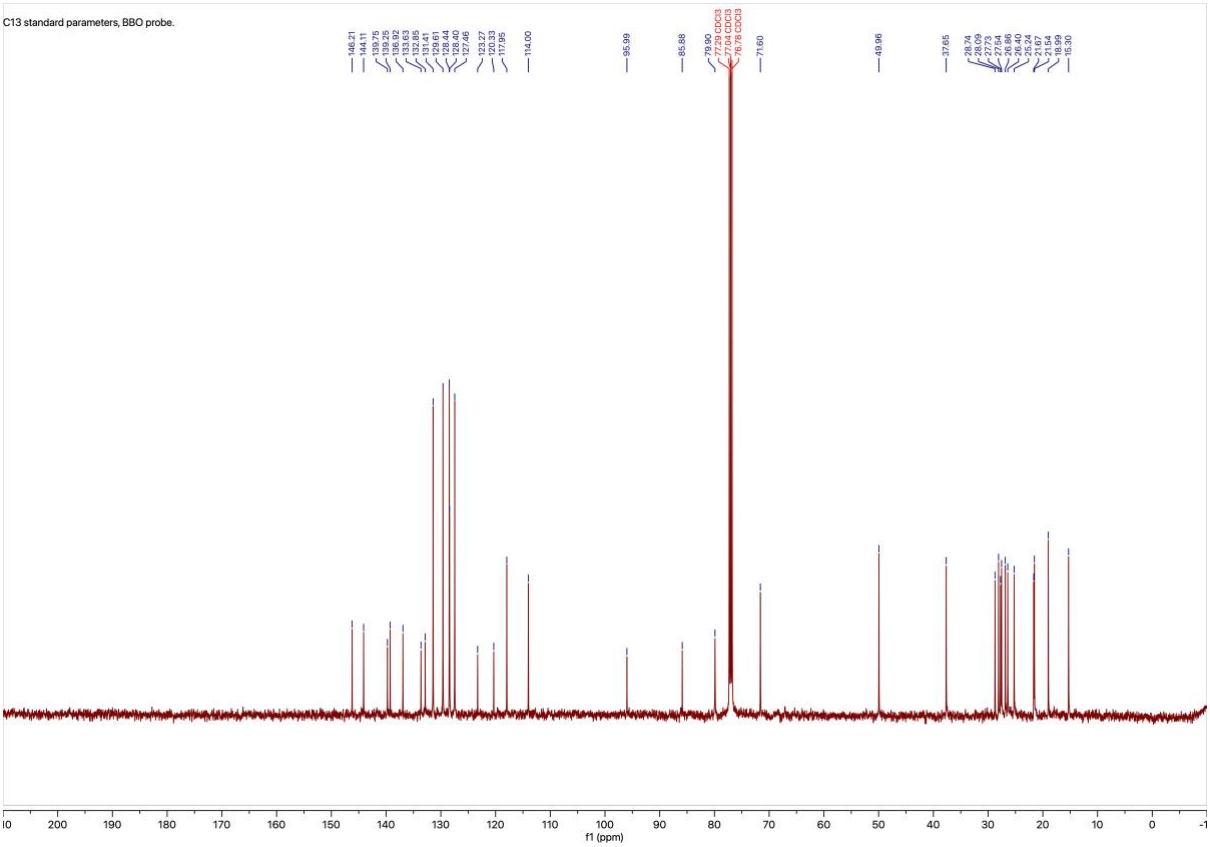

H1 standard parameters, BBO probe.
Bu
O
12
N
Ts
4j
O
f1 (ppm)

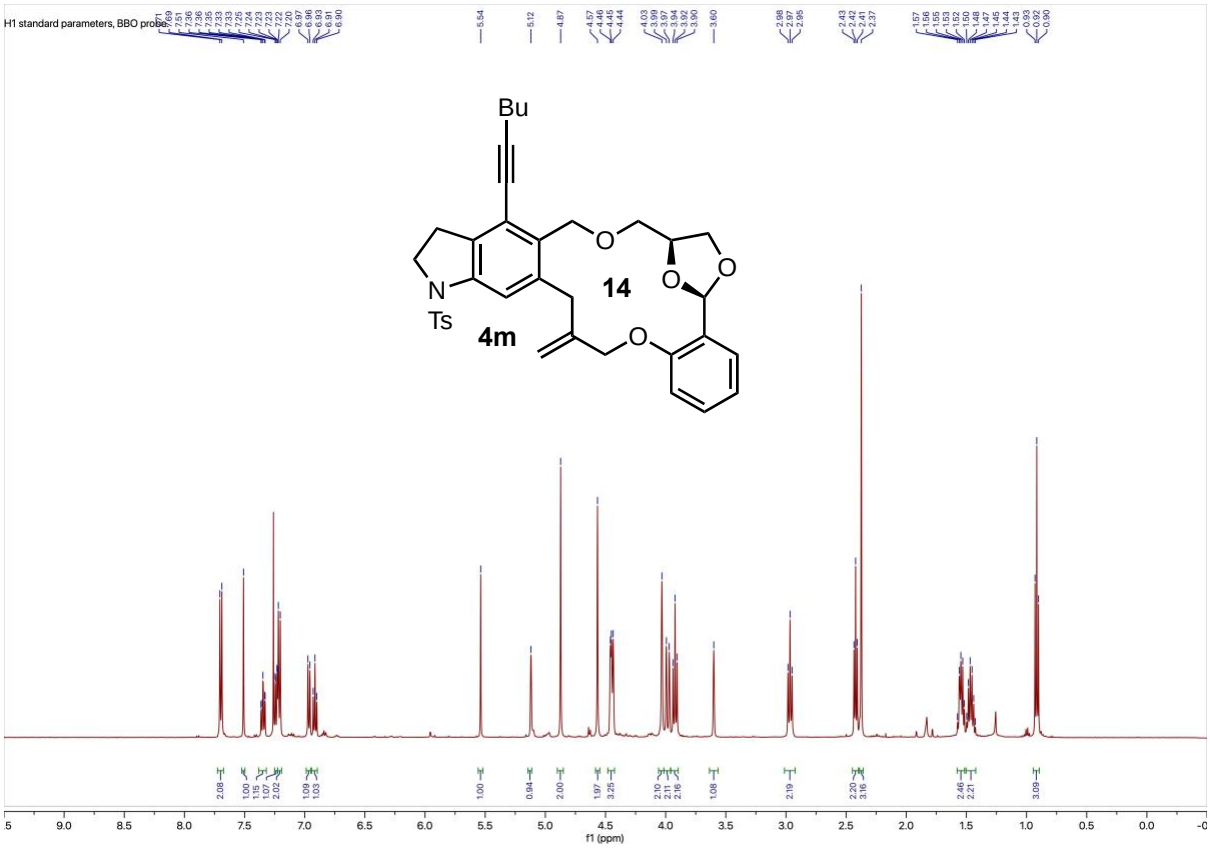

C13 standard parameters, BBO probe.
f1 (ppm)

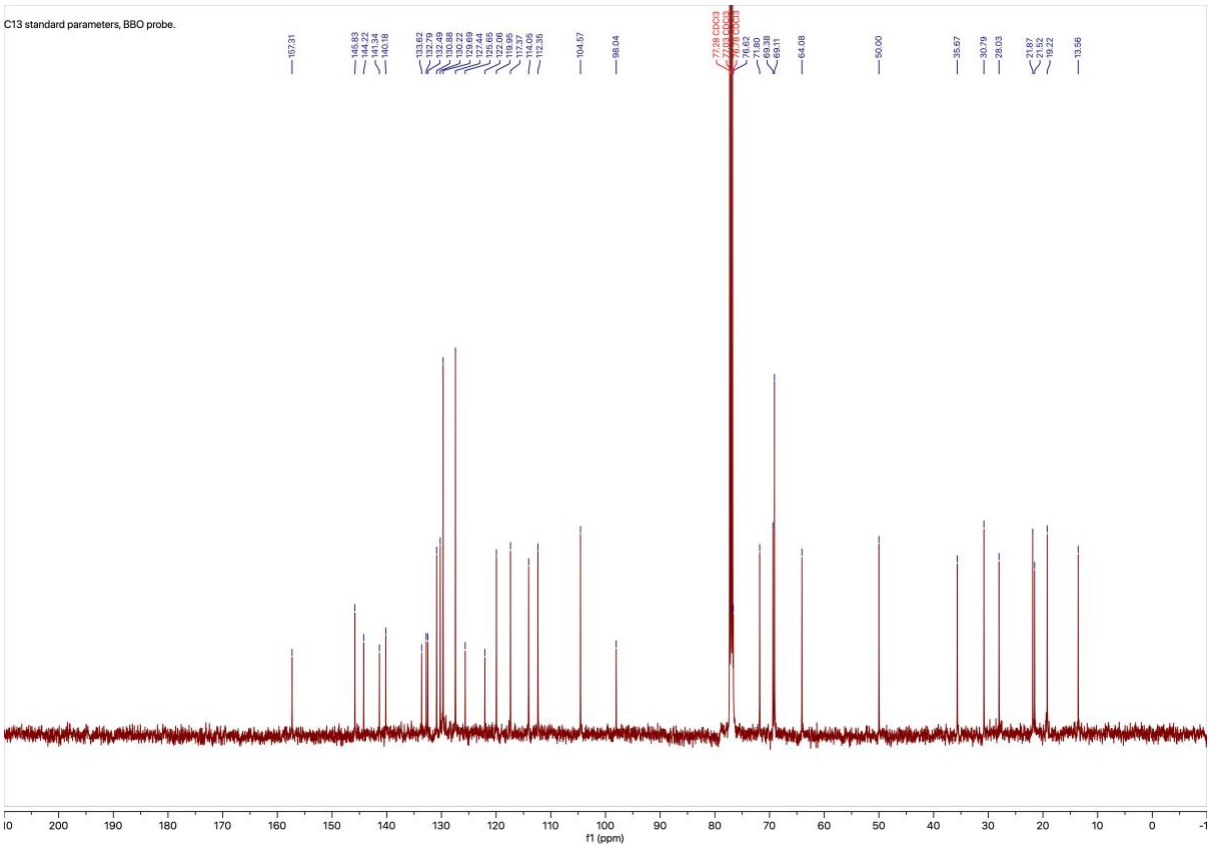

H1 standard parameters, BBO probe.
Bu
O
N
Ts
12
4k
O
f1 (ppm)

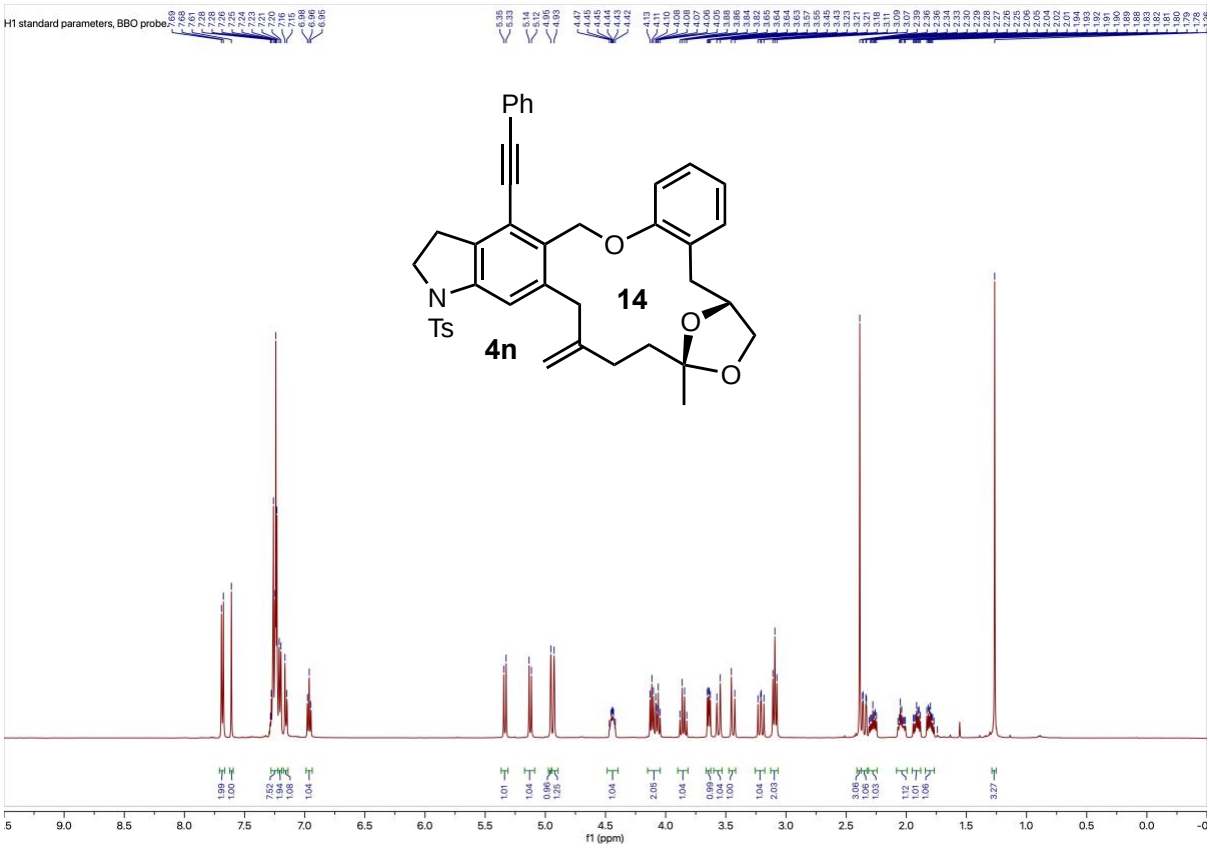

C13 standard parameters, BBO probe.
f1 (ppm)

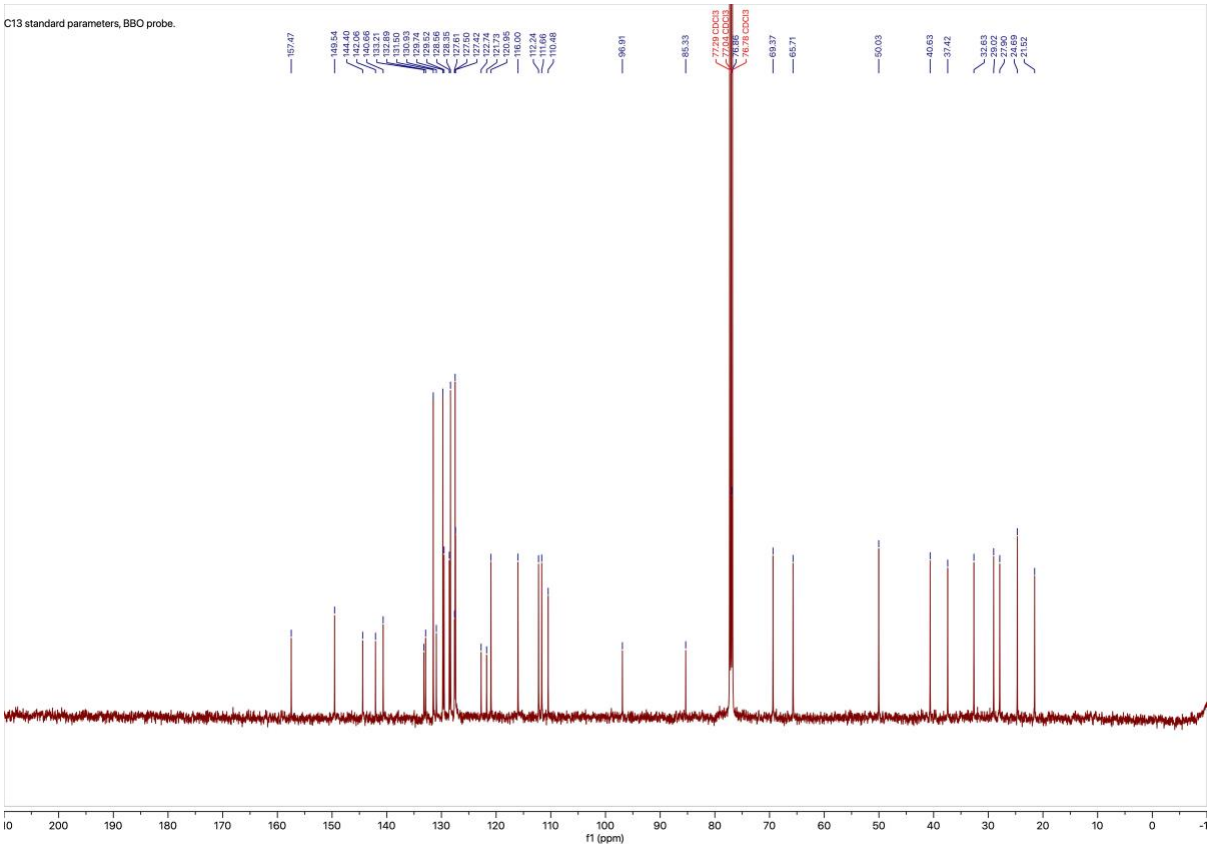

H1 standard parameters, BBO probe.
Bu
O
N
Ts
4l
13
O
f1 (ppm)

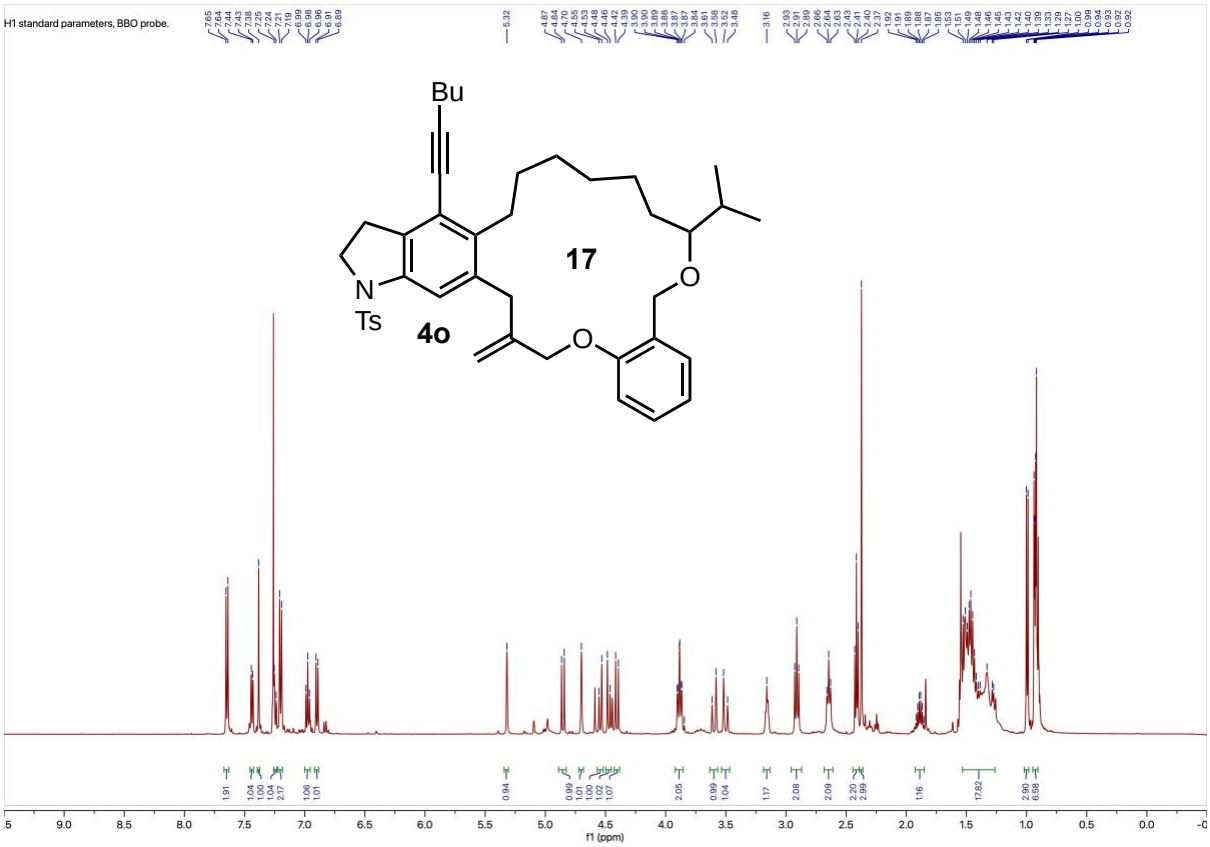

C13 standard parameters, BBO probe.
f1 (ppm)

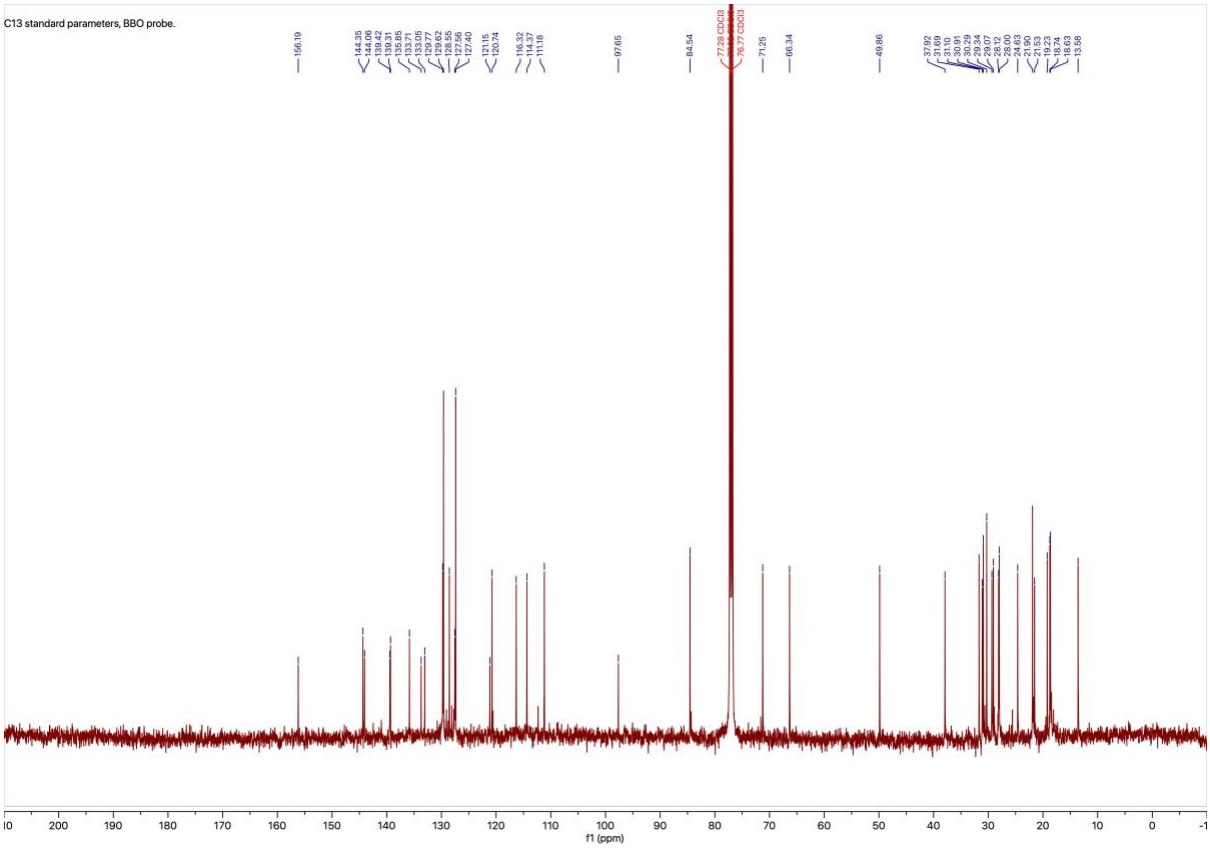

H1 standard parameters, BBO probe
Bu
N
Ts
4m
14
O
O
O
O
O
f1 (ppm)

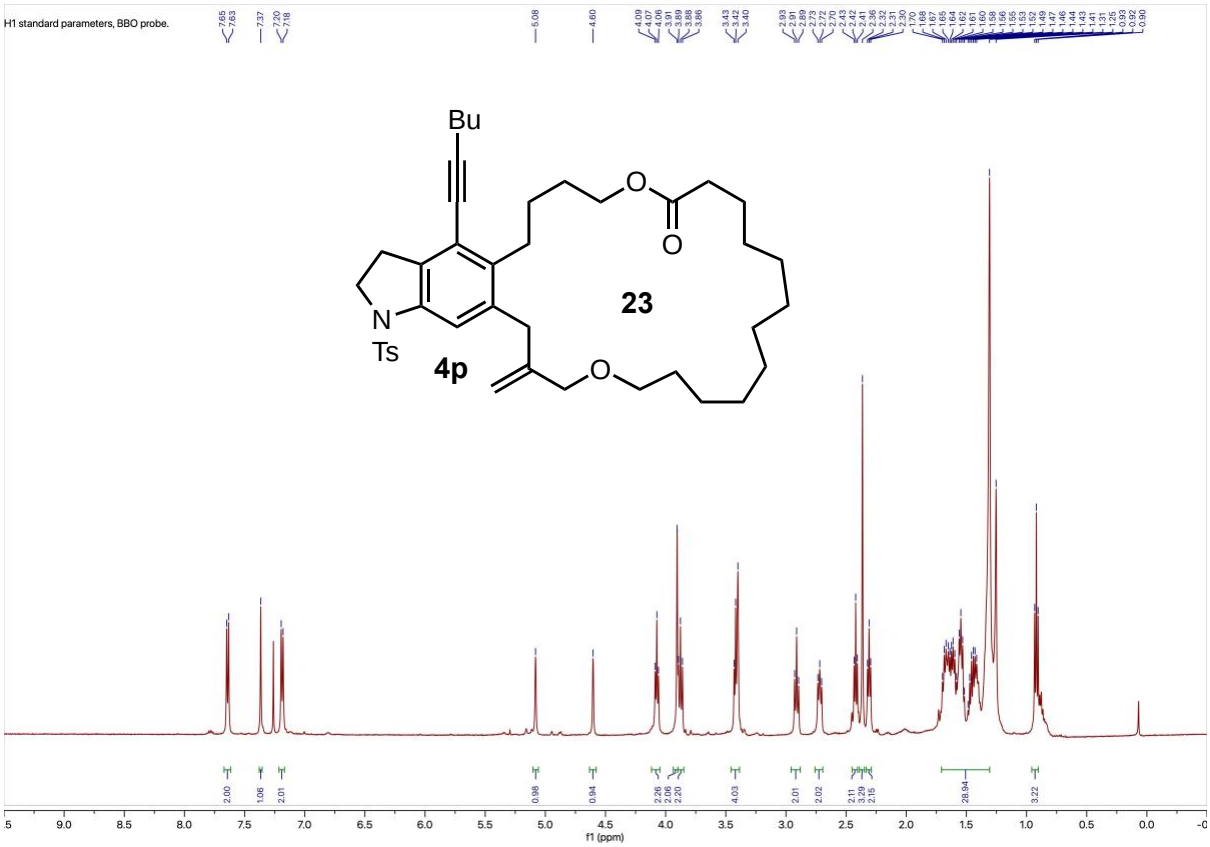

C13 standard parameters, BBO probe.
f1 (ppm)

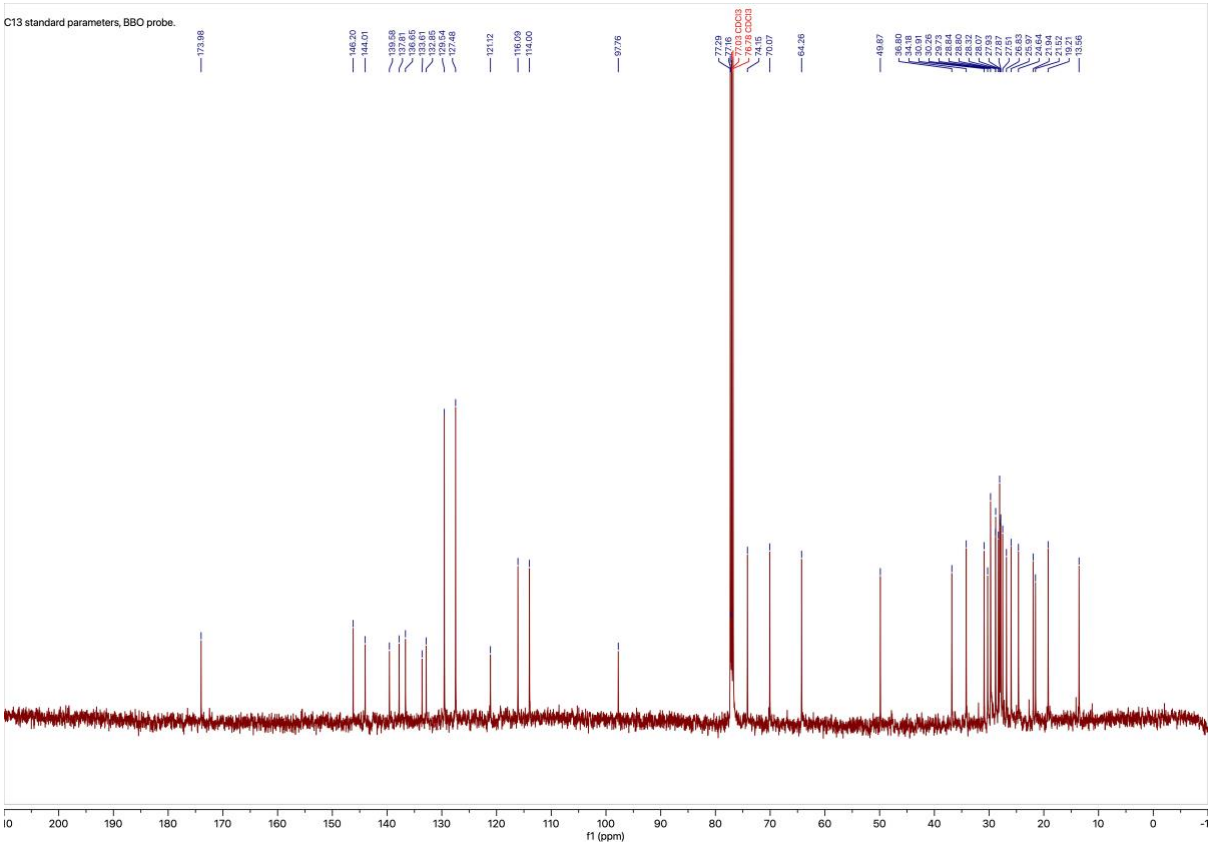

H1 standard parameters, BBO probe
Ph
O
N
Ts
14
4n
O
O
f1 (ppm)

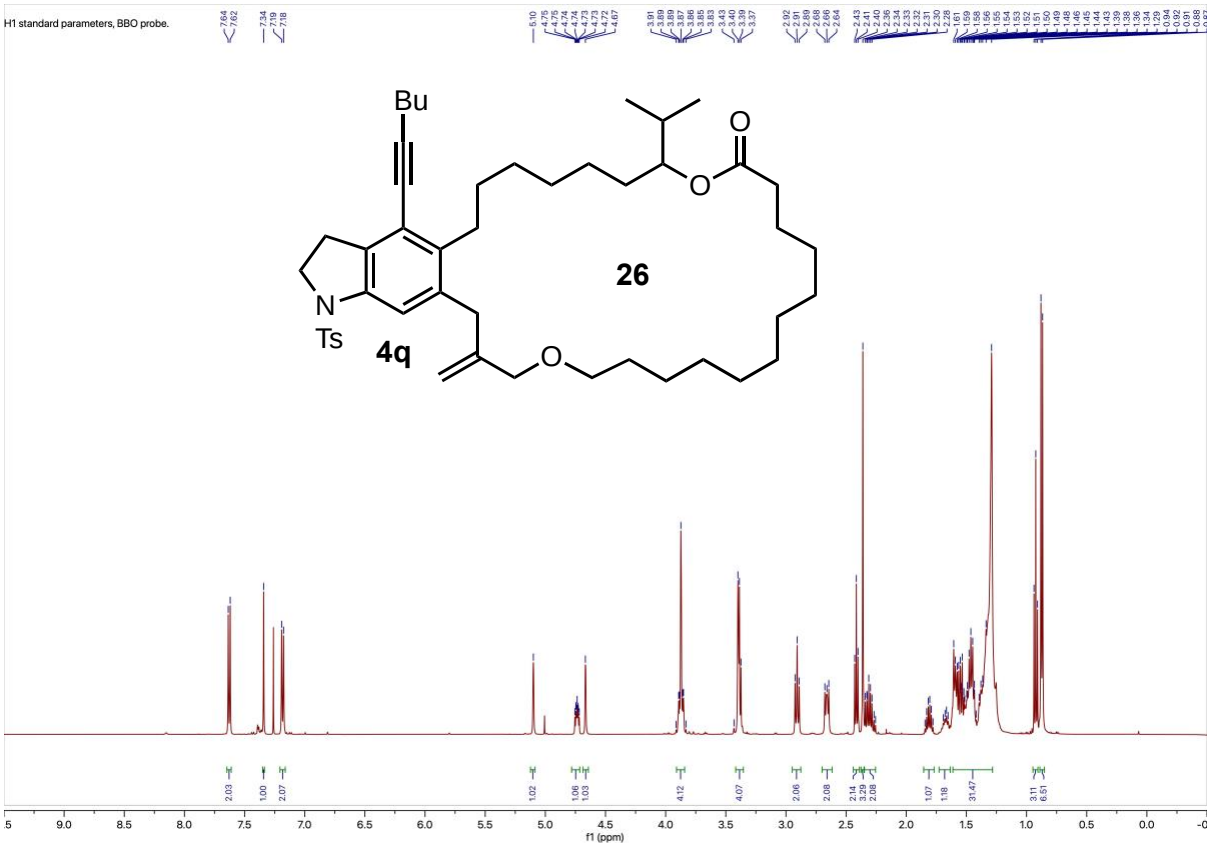

C13 standard parameters, BBO probe.
f1 (ppm)

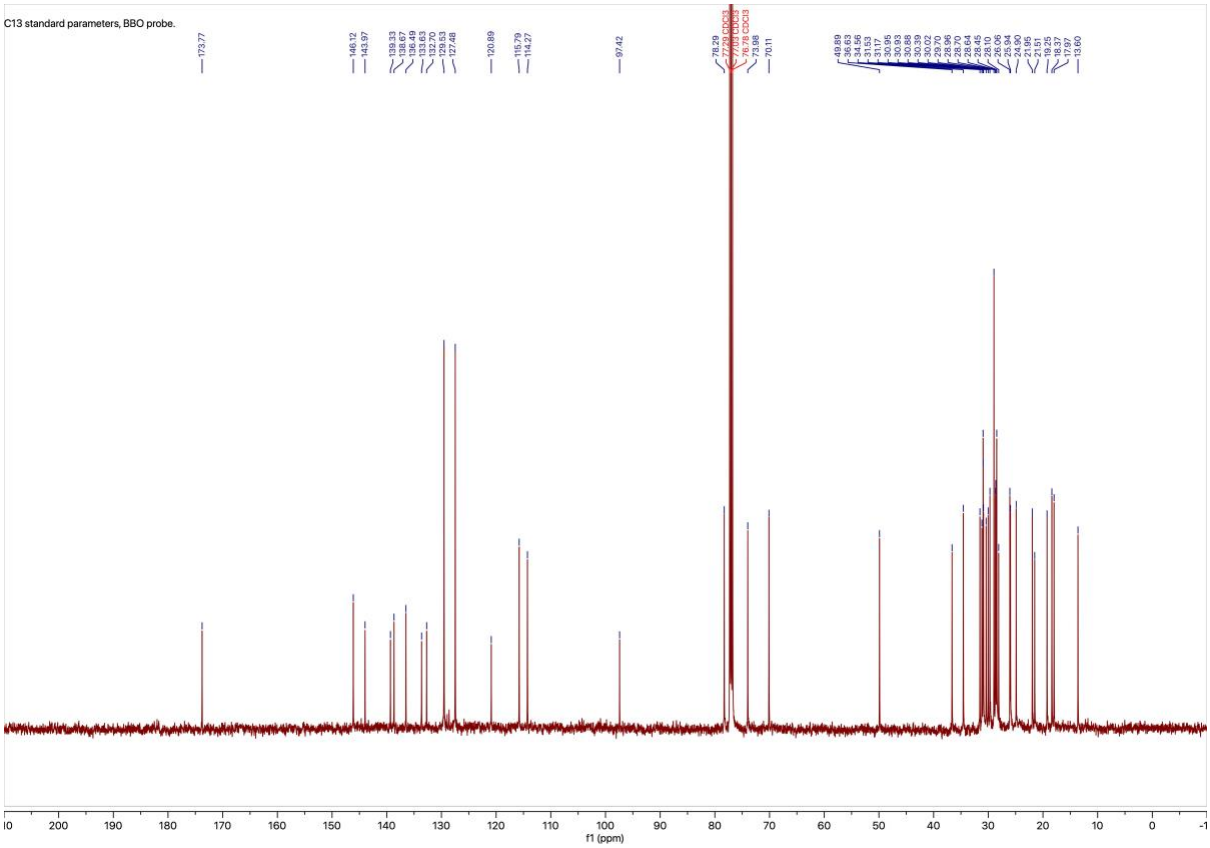

H1 standard parameters, BBO probe.
Bu
17
N
Ts
4o
O
O
f1 (ppm)

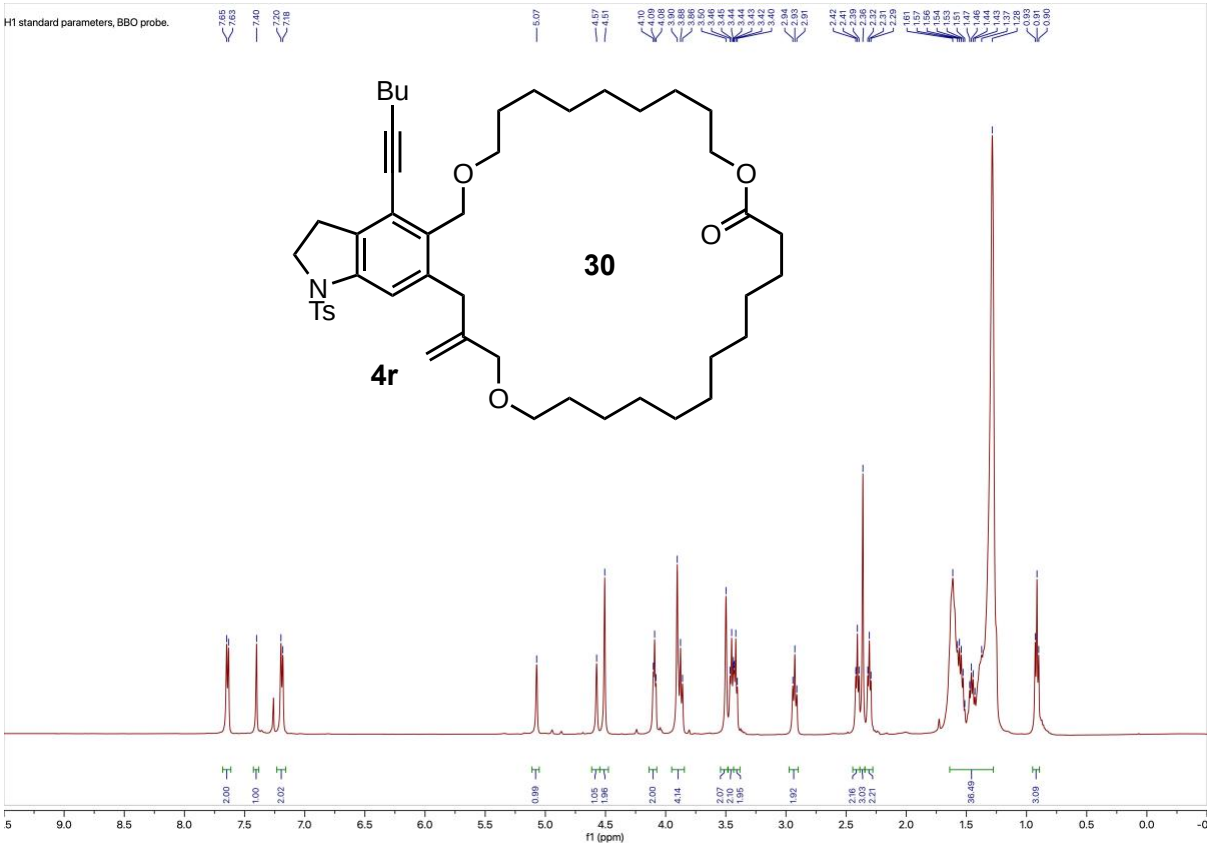

C13 standard parameters, BBO probe.
f1 (ppm)

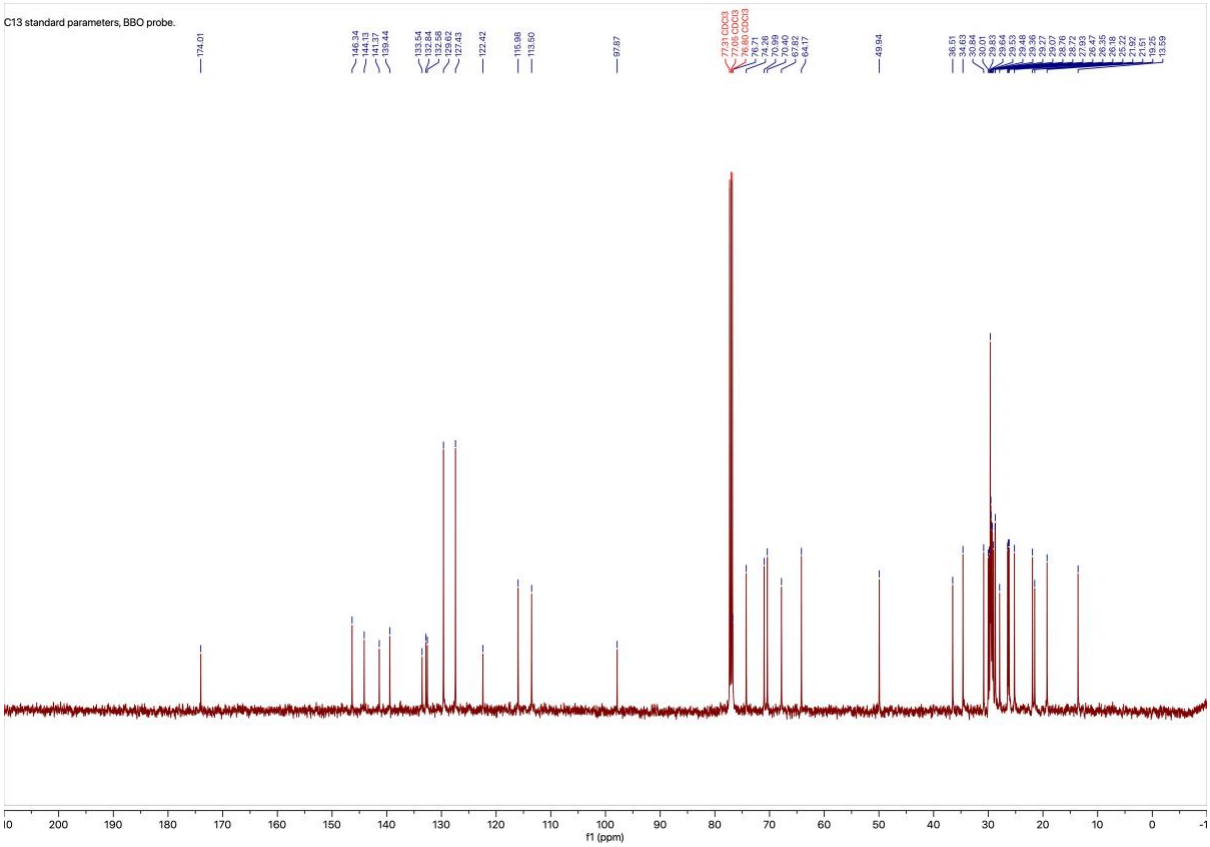

H1 standard parameters, BBO probe.
Bu
O
O
23
N
Ts
4p
O
f1 (ppm)

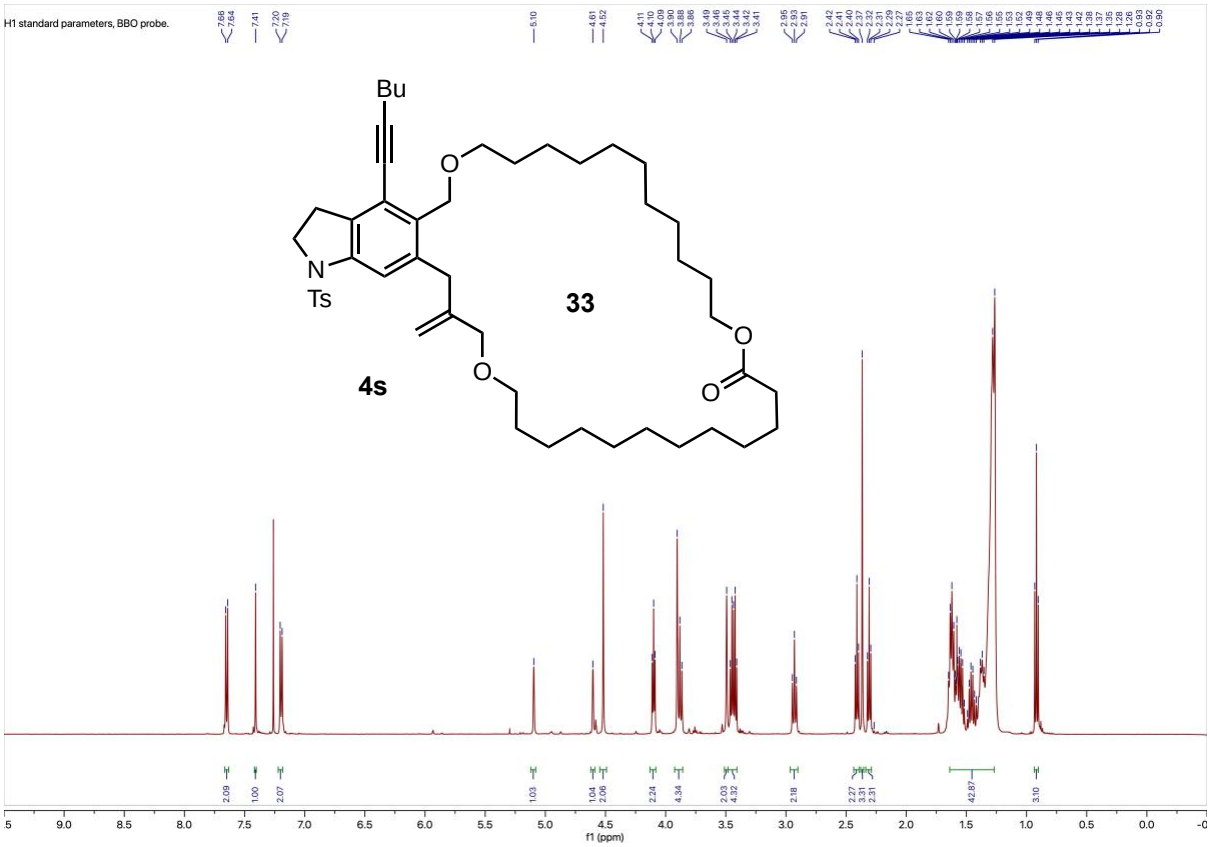

C13 standard parameters, BBO probe.
f1 (ppm)

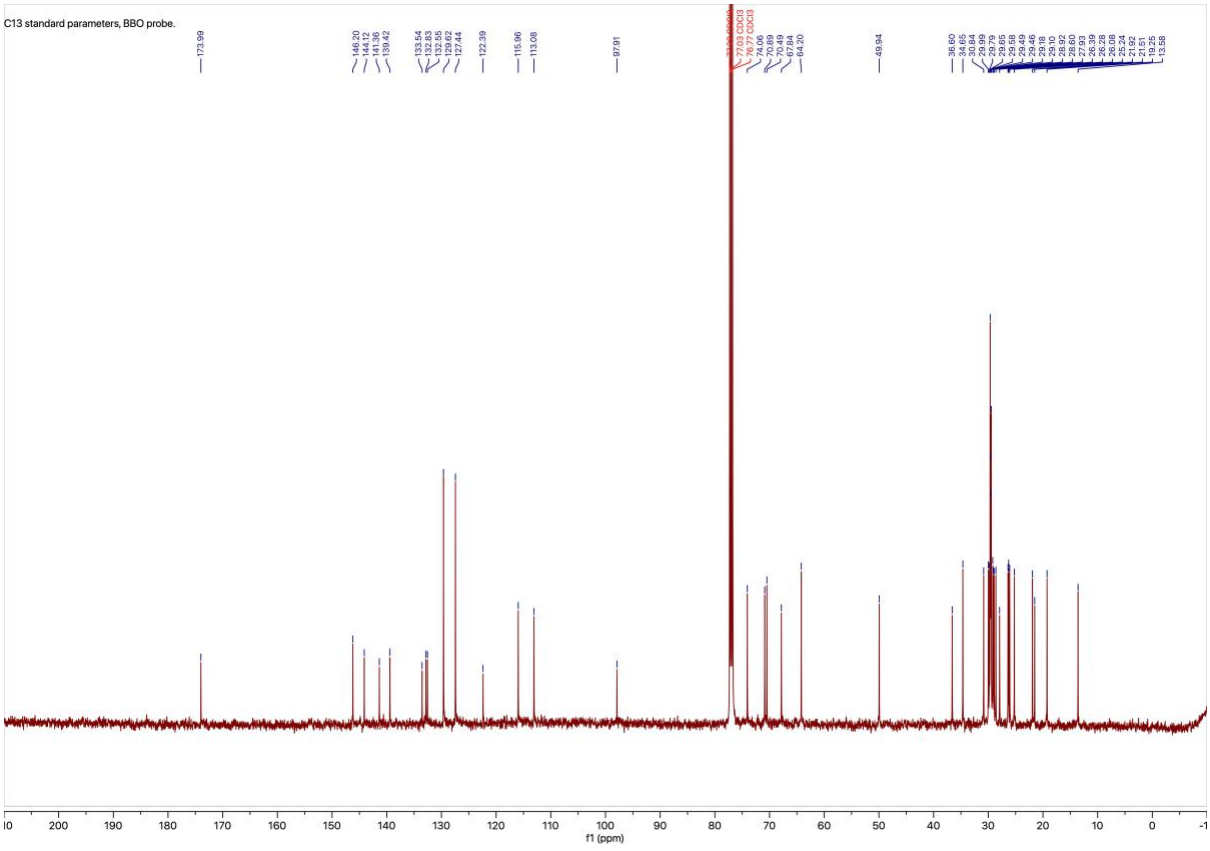

H1 standard parameters, BBO probe.
Bu
O
O
26
N
Ts
4q
O
f1 (ppm)

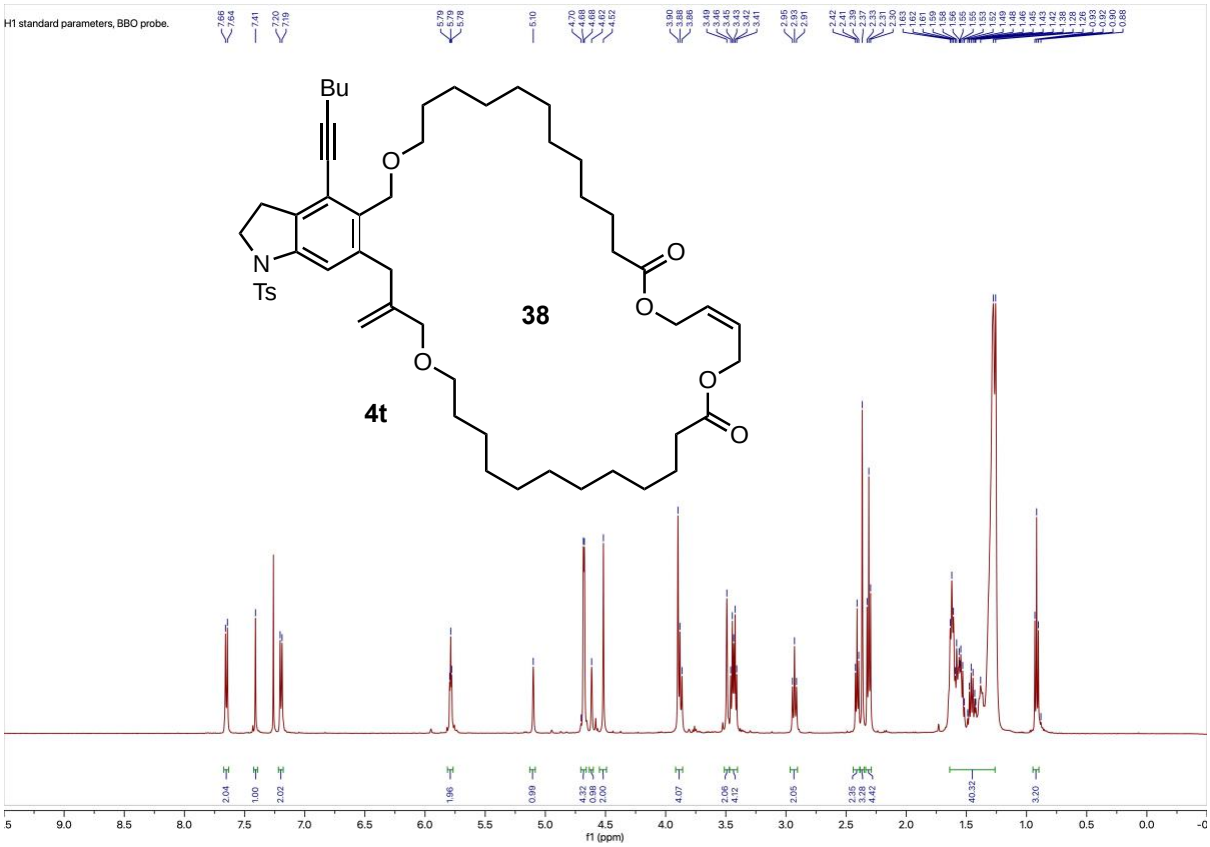

C13 standard parameters, BBO probe.
f1 (ppm)

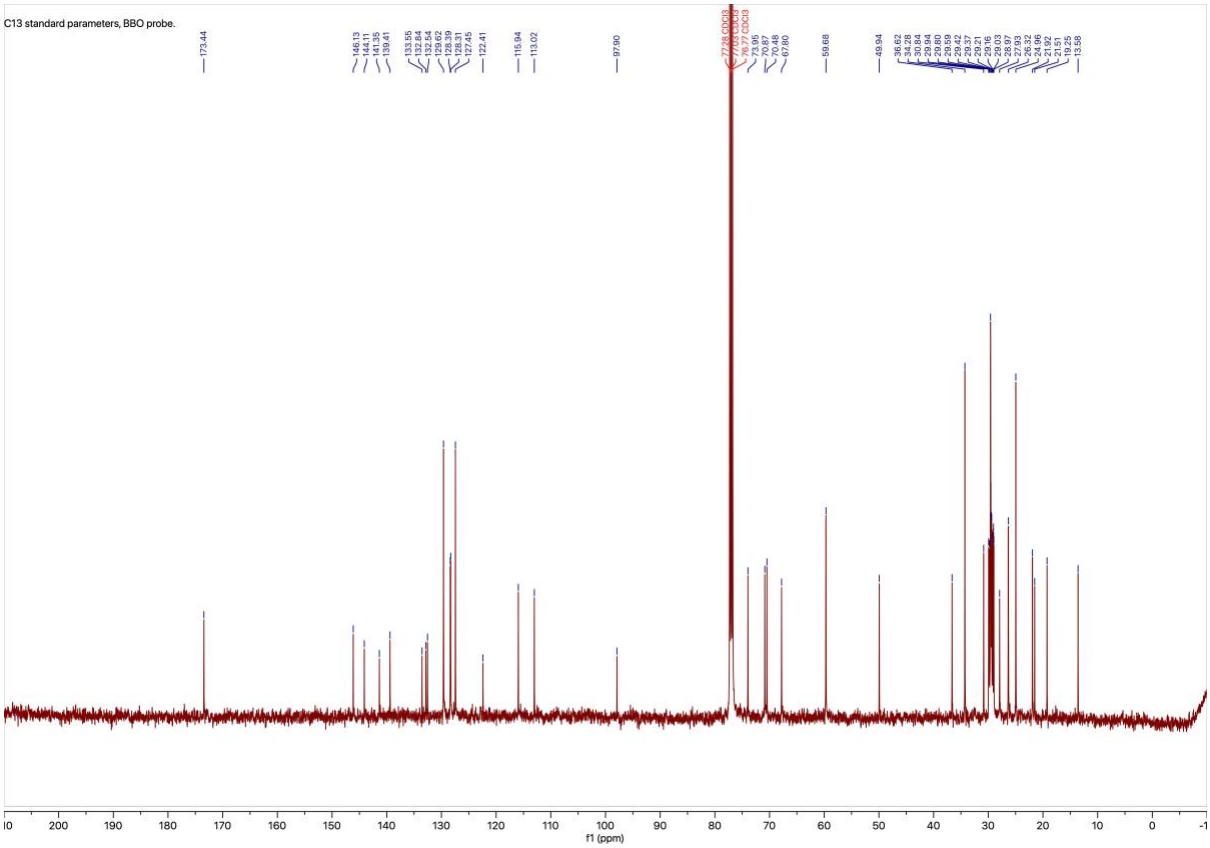

H1 standard parameters, BBO probe.
Bu
O
O
O
O
N
Ts
30
4r
f1 (ppm)

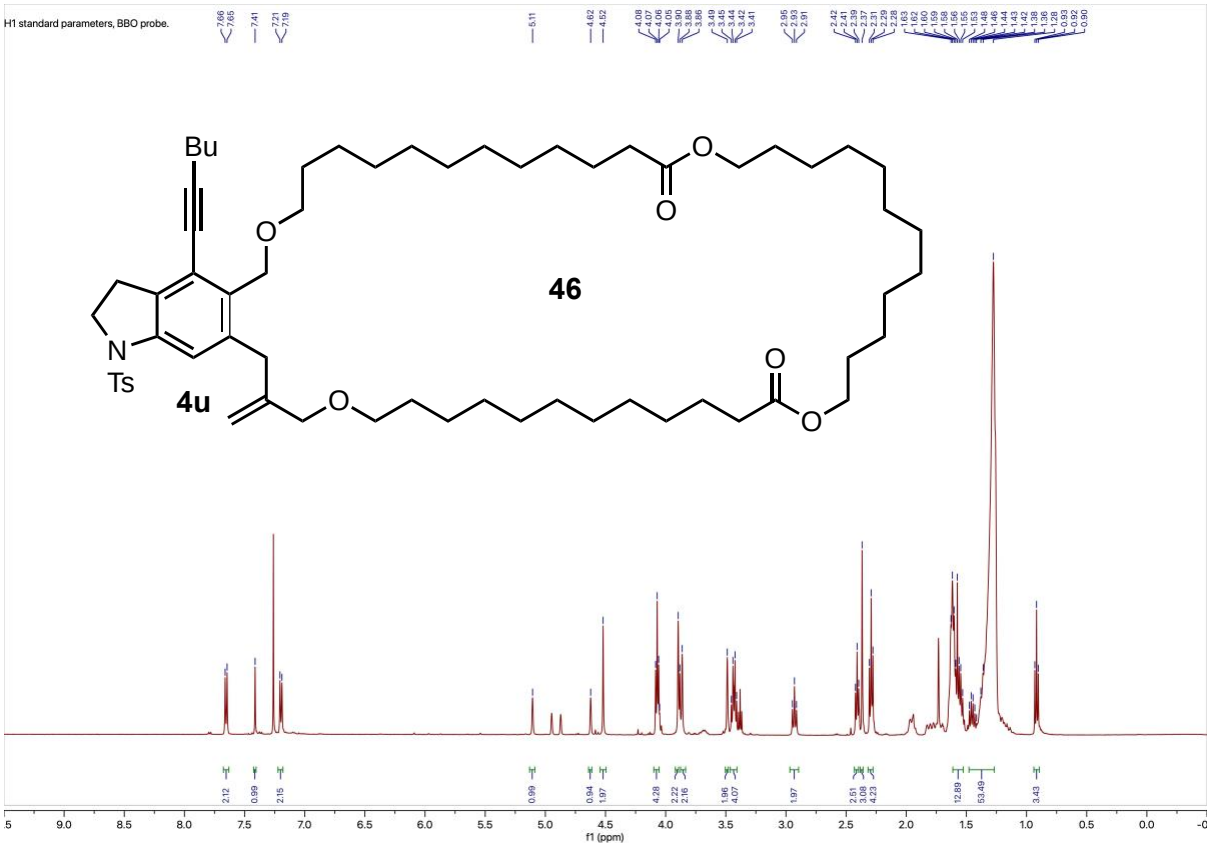

C13 standard parameters, BBO probe.
f1 (ppm)

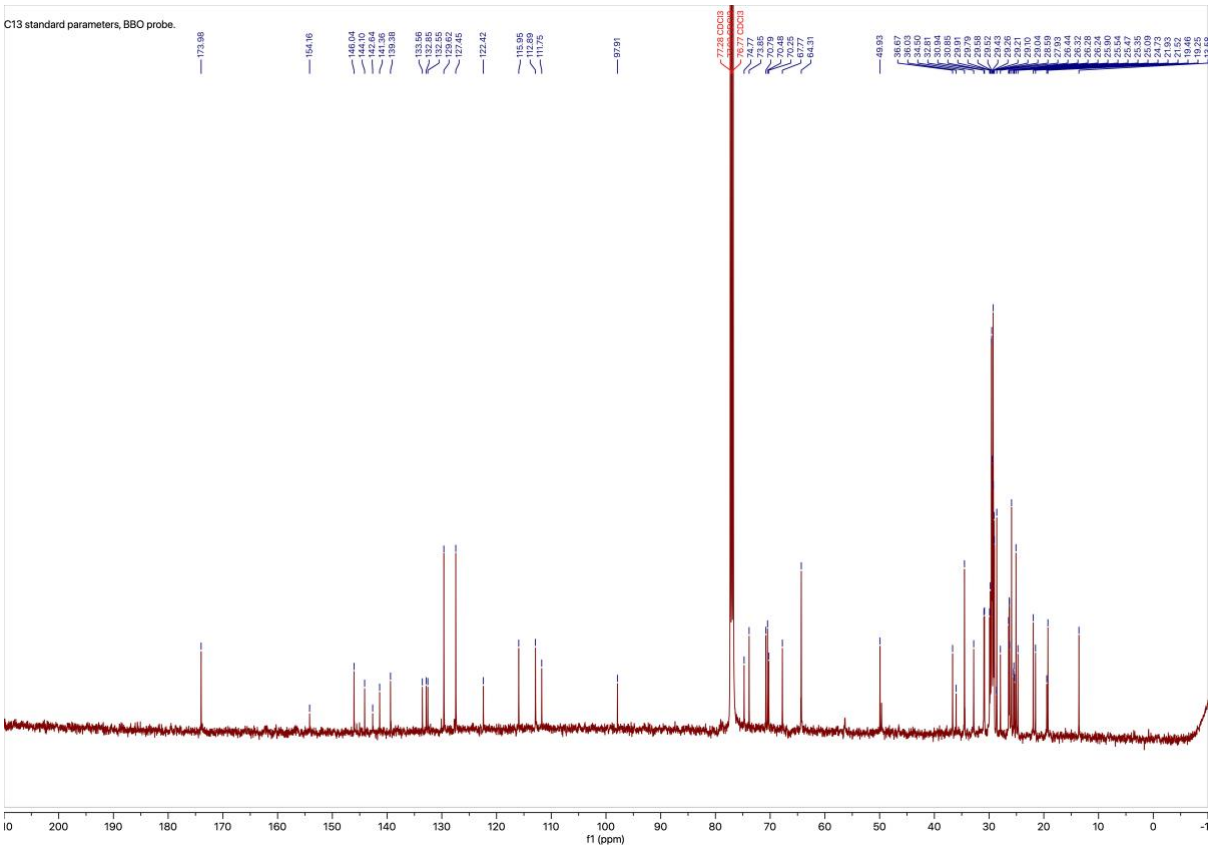

H1 standard parameters, BBO probe.
Bu
O
N
Ts
33
O
O
O
4s
f1 (ppm)


C13 standard parameters, BBO probe.
f1 (ppm)


H1 standard parameters, BBO probe.
Bu
O
N
Ts
38
O
O
O
O
O
4t
f1 (ppm)


C13 standard parameters, BBO probe.
f1 (ppm)


H1 standard parameters, BBO probe.
Bu
O
O
O
O
46
N
Ts
4u
O
O
f1 (ppm)


C13 standard parameters, BBO probe.
f1 (ppm)