

Supporting Information for Synthesis and properties of spiro-type heterasumanenes containing group 14 elements as bridging atoms

Shunsuke Furukawa,* Keisuke Hayashi, Ken Yamagishi, Masaichi Saito*

Table of contents

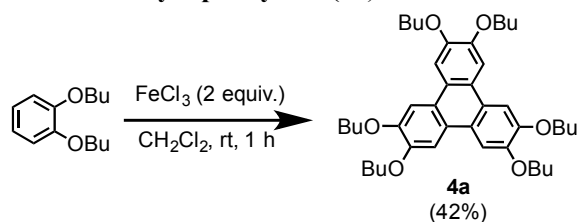
1. General consideration for synthesis and characterization	S2
2. Synthetic procedures for all compounds	S3
3. X-ray crystallographic analysis	S18
4. Electrochemical Study.....	S19
5. Chemical oxidation of spirobistannasumanene 3b	S20
6. Reference.....	S20
7. NMR charts	S21

1. General consideration for synthesis and characterization

NMR spectra were recorded on Bruker AVANCE500 spectrometer (^1H NMR, 500 MHz; ^{13}C NMR, 125 MHz), Bruker AVANCE400 spectrometer (^1H NMR, 400 MHz; ^{13}C NMR, 100 MHz) and Bruker AVANCE500T spectrometer (^{29}Si NMR, 99 MHz; ^{119}Sn NMR, 186 Hz). Chemical shifts for ^1H NMR spectra are reported in parts per million (ppm, δ scale) downfield from tetramethylsilane, and referenced internally to the residual proton in the solvent (CDCl_3 : δ 7.26). Chemical shifts for ^{13}C NMR spectra are reported in parts per million (ppm, δ scale) downfield from tetramethylsilane, and are referenced to the ^{13}C resonance of the NMR solvent (CDCl_3 : δ 77.16). Chemical shifts for ^{29}Si NMR spectra are reported in parts per million (ppm, δ scale) downfield and are referenced to an external tetramethylsilane as the standard. Chemical shifts for ^{119}Sn NMR spectra are reported in parts per million (ppm, δ scale) downfield and are referenced to an external tetramethylstannane as the standard. The data are presented as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet and/or multiple resonances), coupling constant in hertz (Hz), and integration. Mass spectra were obtained with Bruker AutoflexIII (MALDI-TOF) mass spectrometer. Cyclic voltammetry measurements were performed with Gamry Interface 1000 using a glassy carbon working electrode, a Pt counter electrode and an Ag/AgNO_3 reference electrode at room temperature in CH_2Cl_2 containing 0.1 M $n\text{-Bu}_4\text{NClO}_4$ as the supporting electrolyte. Melting points were measured by Yanaco MP-S3 instrument. The intensity data for X-ray crystallographic analyses were collected at $-173\text{ }^\circ\text{C}$ on Bruker SMART APEX II equipped with a CCD area detector with graphite-monochromated $\text{MoK}\alpha$ radiation ($\lambda = 0.71073\text{ \AA}$). Column chromatography was performed using Kanto silica gel 60N. Thin layer chromatography was performed using Merck Silica Gel 60 F254, TLC Plates. Medium pressure liquid chromatography was performed with Yamazen Corporation EPCLC AI-580 instrument using Universal columns premium. UV-vis absorption spectra were obtained with JASCO V-770 spectrometer. Electron spin resonance (ESR) spectra were recorded on Bruker EMX6/1 spectrometer.

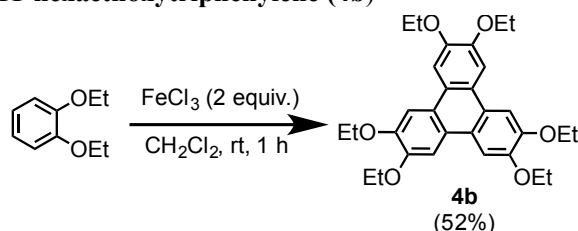
2. Synthetic procedures for all compounds

Synthesis of 2,3,6,7,10,11-hexabutoxytriphenylene (**4a**)¹



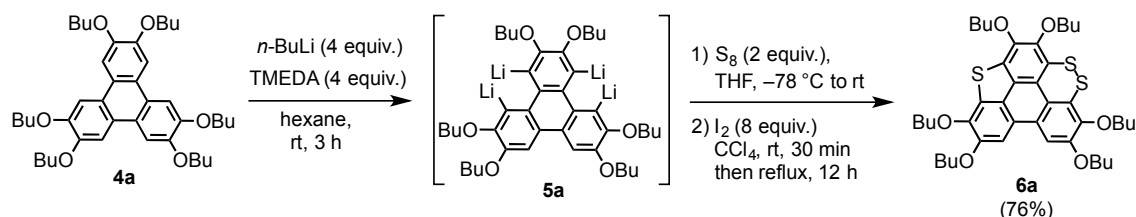
Anhydrous iron(III) chloride (39.1 g, 241 mmol) was added to a dichloromethane (400 mL) solution of 1,2-dibutoxybenzene (26.7 g, 120 mmol) for 40 min at room temperature. After stirring for 20 min at the same temperature, ethanol (320 mL) was added to the reaction mixture. The mixture was cooled in an ice-bath and then the resulting solid was corrected by suction filtration. The products were purified by silica gel column chromatography (eluent: hexane : CHCl₃ = 2 : 3, v/v) to give **4a** (11.2 g, 16.9 mmol, 42%) as a colorless solid.

Synthesis of 2,3,6,7,10,11-hexaethoxytriphenylene (**4b**)¹



A dichloromethane (140 mL) solution of 1,2-diethoxybenzene (36.5 g, 220 mmol) was added to a suspension of anhydrous iron(III) chloride (71.5 g, 441 mmol) in dichloromethane (360 mL) dropwise for 40 min at room temperature. After stirring for 45 min at the same temperature, ethanol (350 mL) was added to the reaction mixture. The mixture was cooled in an ice-bath and then the resulting solid was corrected by suction filtration. The products were purified by silica gel column chromatography (CHCl₃) to give **4b** (19.0 g, 38.6 mmol, 52%) as a colorless solid.

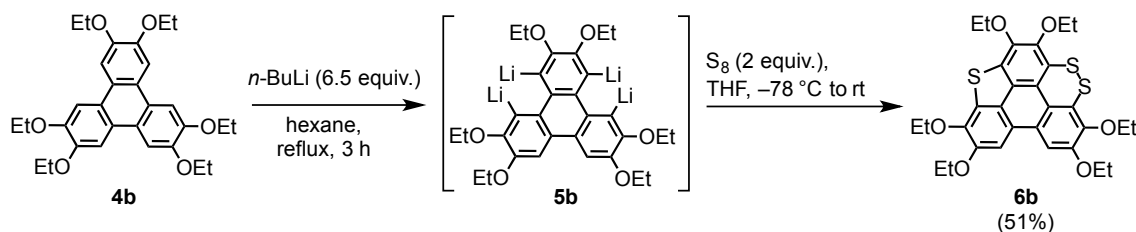
Synthesis of 2,3,6,7,10,11-hexabutoxy[4,5-*b,c,d*]thienotriphenylene[8,9-*c',d',e'*]1',2'-dithiine (**6a**)



Anhydrous hexane (28 mL) and TMEDA (6.4 mL, 43.0 mmol) were added to **4a** (7.06 g, 10.7

mmol) in a dried three-necked-flask under argon atmosphere. *n*-BuLi in hexane (2.66 M, 16.1 mL, 42.8 mmol) was added dropwise to the suspension for 20 min. The suspension was stirred at room temperature for 3 hours. Volatiles were removed under reduced pressure. THF (210 mL) was added to the resulting solid and then cooled to under $-70\text{ }^{\circ}\text{C}$. Sulfur powder (5.50 g, 21.4 mmol) was added to the suspension. After stirring for 30 min at $-70\text{ }^{\circ}\text{C}$, the mixture was allowed to warm to room temperature and stirred for 12 hours. After stirring, aqueous NH_4Cl was added to the mixture, and insoluble materials were removed by suction filtration. The reaction mixture was extracted with chloroform and the extract was dried over anhydrous magnesium sulfate. The extract was concentrated under reduced pressure to give a brown viscous solid (9.54 g). Carbon tetrachloride (180 mL) and iodine (19.0 g, 75.0 mmol) were added to the viscous solid under argon atmosphere, and the mixture was stirred at room temperature for 50 min. After heating under reflux conditions for 14 hours, the mixture was cooled to room temperature, then aqueous sodium sulfite was added. The reaction mixture was extracted with chloroform and the extract was dried over anhydrous magnesium sulfate. The extract was concentrated under reduced pressure to give a dark yellow solid. The products were purified by silica gel column chromatography (eluent, hexane: CHCl_3 , 2:3, *v/v*) to give **6a** (6.16 g, 8.18 mmol, 76%) as a yellow solid. mp. $105\text{--}107\text{ }^{\circ}\text{C}$; ^1H NMR (500 MHz, CDCl_3) δ 1.01–1.08 (m, 18H), 1.58–1.67 (m, 12H), 1.83–1.90 (m, 8H), 1.91–1.97 (m, 4H), 4.20–4.28 (m, 8H), 4.34–4.39 (m, 4H), 7.74 (s, 1H), 7.76 (s, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 14.07–14.14 (m, CH_3), 19.42 (CH_2), 19.46 (CH_2), 19.51 (CH_2), 19.59 (CH_2), 31.57 (CH_2), 31.85 (CH_2), 32.57, 32.59 (CH_2), 32.60 (CH_2), 32.62 (CH_2), 68.84 (CH_2), 70.32 (CH_2), 73.22 (CH_2), 73.29 (CH_2), 73.86 (CH_2), 74.74 (CH_2), 105.08 (CH), 105.83 (CH), 120.53 (C), 121.30 (C), 122.56 (C), 123.43 (C), 123.91 (C), 126.49 (C), 127.11 (C), 128.30 (C), 128.34 (C), 130.71 (C), 143.29 (C), 145.01 (C), 146.28 (C), 146.47 (C), 152.36 (C), 152.82 (C); MS (MALDI) *m/z* calcd for $\text{C}_{42}\text{H}_{56}\text{O}_6\text{S}_3$ ($[\text{M}]^+$) 752.3, found: 752.2; elemental analysis calcd for $\text{C}_{42}\text{H}_{56}\text{O}_6\text{S}_3$: C, 66.98; H, 7.50, found: C, 66.99; H, 7.55.

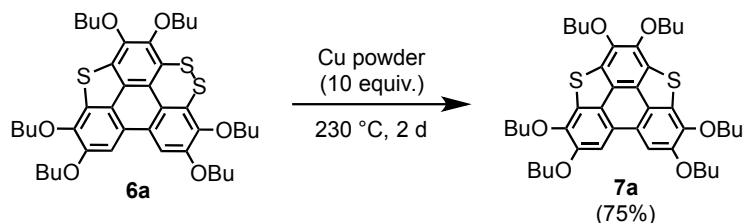
Synthesis of 2,3,6,7,10,11-hexaethoxy[4,5-*b,c,d*]thienotriphenyleno[8,9-*c',d',e'*]1',2'-dithiine (**6b**)



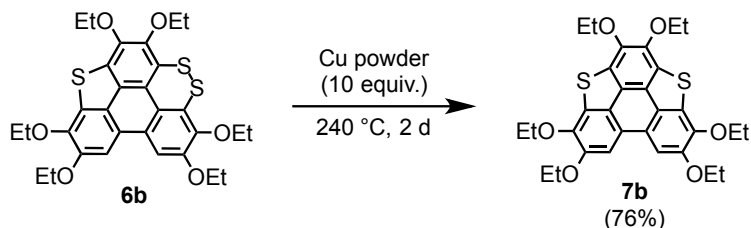
Anhydrous hexane (250 mL) was added to **4b** (5.01 g, 10.2 mmol) in a dried three-necked-flask under argon atmosphere. *n*-BuLi in hexane (2.66 M, 24.0 mL, 63.8 mmol) was added dropwise to the suspension. The suspension was refluxed for 3 hours. Volatiles were removed under reduced pressure. THF (200 mL) was added to the resulting solid and then the mixture was cooled to under

–70 °C. Sulfur powder (5.32 g, 20.7 mmol) was added to the suspension, and stirred for 30 min at –70 °C. The mixture was allowed to warm to room temperature and stirred for 12 hours. After stirring, aqueous NH₄Cl was added to the mixture, and insoluble materials were removed by suction filtration. The reaction mixture was extracted with chloroform and the extract was dried over anhydrous magnesium sulfate. The extract was concentrated under reduced pressure to give a brown solid. The products were purified by silica gel column chromatography (eluent, hexane: CH₂Cl₂ = 2 : 3, v/v) to give **6b** (3.04 g, 5.20 mmol, 51%) as a yellow solid. mp. 169-170 °C; ¹H NMR (500 MHz, CDCl₃) δ 1.48-1.53 (m, 12H), 1.57 (t, *J* = 7.0 Hz, 6H), 4.27–4.34 (m, 8H), 4.43 (q, *J* = 7.0 Hz, 4H), 4.47 (q, *J* = 7.0 Hz, 4H), 7.71 (s, 1H), 7.72 (s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 15.02 (CH₃), 15.27 (CH₃), 15.92 (CH₃), 15.96 (CH₃), 16.12 (CH₃), 64.72 (CH₂), 66.07 (CH₂), 68.94 (CH₂), 68.98 (CH₂), 69.76 (CH₂), 70.49 (CH₂), 105.03 (CH), 105.83 (CH), 120.43 (C), 121.18 (C), 122.39 (C), 123.31 (C), 123.77 (C), 126.30 (C), 127.01 (C), 128.24 (C), 128.26 (C), 130.65 (C), 142.87 (C), 144.75 (C), 145.98 (C), 146.13 (C), 152.01 (C), 152.53 (C); MS (MALDI) *m/z* calcd for C₃₀H₃₂O₆S₃ ([M]⁺) 584.1, found: 584.4; elemental analysis calcd for C₃₀H₃₂O₆S₃: C, 61.62; H, 5.52, found: C, 61.29; H, 5.44.

Synthesis of 2,3,6,7,10,11-hexabutoxy[4,5-*b,c,d:8,9-b',c',d'*]dithiophene (**7a**)

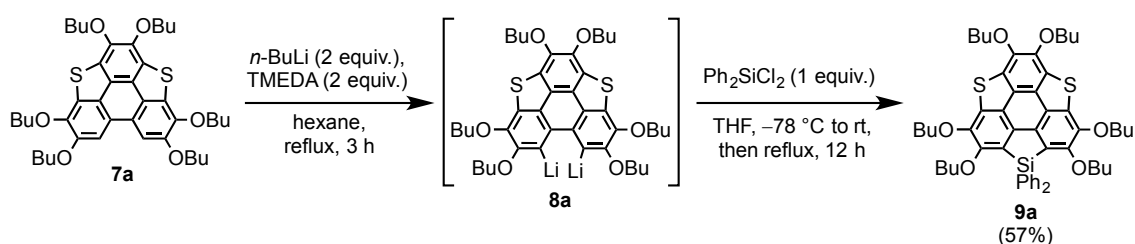


Synthesis of 2,3,6,7,10,11-hexaethoxy[4,5-*b,c,d*:8,9-*b',c',d'*]dithiophene (**7b**)



Compound **6b** (2.30 g, 3.94 mmol) and copper powder (2.49 g, 3.92 mmol) were mixed in a flask, and the mixture was heated at 240 °C for 2 days under argon atmosphere. After cooled to room temperature, dichloromethane was added to the reaction mixture, and the resulting insoluble solid was removed by suction filtration. The filtrate was concentrated and further purified by column chromatography on silica (eluent, hexane : CH₂Cl₂ = 2 : 5, v/v) to give **7b** as a pale yellow solid (1.676 g, 3.03 mmol, 75%). mp. 174 °C; ¹H NMR (500 MHz, CDCl₃) δ 1.52 (t, *J* = 8.5 Hz, 6H), 1.54 (t, *J* = 8.5 Hz, 6H), 1.58 (t, *J* = 8.5 Hz, 6H), 4.35 (q, *J* = 8.5 Hz, 4H), 4.49 (q, *J* = 8.5 Hz, 4H), 4.52 (q, *J* = 8.5 Hz, 4H), 7.85 (s, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 15.36 (CH₃), 16.02 (CH₃), 16.08 (CH₃), 66.47 (CH₂), 68.68 (CH₂), 68.85 (CH₂), 106.92 (CH), 123.19 (C), 123.58 (C), 125.11 (C), 127.57 (C), 132.52 (C), 143.48 (C), 145.70 (C), 151.97 (C); MS (MALDI) *m/z* calcd for C₃₀H₃₂O₆S₂ ([M]⁺) 552.2, found: 552.2; elemental analysis calcd for C₃₀H₃₂O₆S₂: C, 65.19; H, 5.84, found: C, 64.97; H, 5.75.

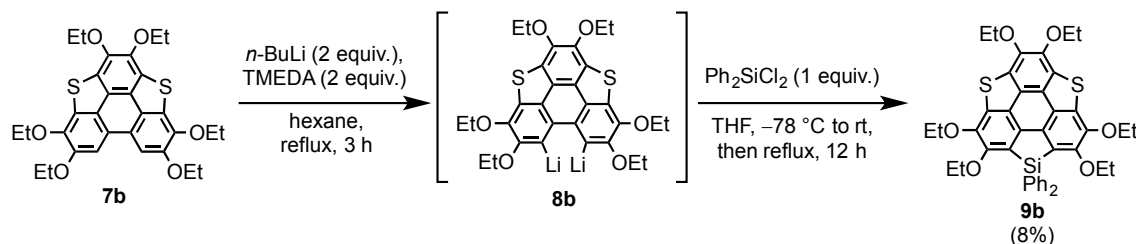
Synthesis of 2,3,6,7,10,11-hexabutoxytriphenylene[4,5-2',3',4',5']1',1'-diphenyl-1*H*-silolo[8,9-*b,c,d*:12,1-*b',c',d'*]dithiophene (**9a**)



Anhydrous hexane (4.4 mL) and TMEDA (0.40 mL, 2.68 mmol) were added to **7a** (948 mg, 1.32 mmol) in a dried three-necked-flask under argon atmosphere. *n*-BuLi in hexane (2.66 M, 1.00 mL, 2.66 mmol) was added dropwise to the suspension. After the suspension was refluxed for 3 hours, volatiles were removed under reduced pressure. THF (26 mL) was added to the resulting solid and then the mixture was cooled to under −70 °C. Dichlorodiphenylsilane (0.28 mL, 1.33 mmol) was added slowly to the solution and stirred for 30 min at −70 °C. The mixture was allowed to warm to room temperature and stirred for an hour then was refluxed for 12 hours. After cooling to room temperature, aqueous NH₄Cl was added to the mixture. The reaction mixture was extracted with

chloroform and the extract was dried over anhydrous magnesium sulfate. The extract was concentrated under reduced pressure to give an orange solid. The products were purified by silica gel column chromatography (eluent, hexane : CH₂Cl₂ = 1:1, v/v) to give **9a** (681 mg, 0.755 mmol, 57%) as a yellow solid. mp. 128-129 °C; ¹H NMR (400 MHz, CDCl₃) δ 0.74 (t, *J* = 9.0 Hz, 6 H), 1.02 (t, *J* = 9.5 Hz, 6H), 1.04 (t, *J* = 9.0 Hz, 6H), 1.19 (sext, *J* = 9.5 Hz, 4H), 1.51-1.68 (m, 12H), 1.85-1.93 (m, 8H), 3.92 (t, *J* = 9.0 Hz, 4H), 4.40 (t, *J* = 8.0 Hz, 4H), 4.43 (t, *J* = 8.0 Hz, 4H), 7.32-7.43 (m, 6H), 7.81-7.83 (m, 4H); ¹³C NMR (100 MHz, CDCl₃) δ 13.84 (CH₃), 14.02 (CH₃), 19.03 (CH₂), 19.41 (CH₂), 32.03 (CH₂), 32.39 (CH₂), 32.43 (CH₂), 72.43 (CH₂), 72.93 (CH₂), 73.53 (CH₂), 120.07 (C), 125.78 (C), 126.05 (C), 126.97 (C), 128.28 (CH), 130.42 (CH), 133.09 (C), 133.36 (C), 135.05 (C), 135.82 (CH), 146.40 (C), 146.49 (C), 156.25 (C); ²⁹Si NMR (99 MHz, CDCl₃) δ -0.04; MS (MALDI) *m/z* calcd for C₅₄H₆₄O₆S₂Si ([M]⁺) 900.4, found: 900.5; elemental analysis calcd for C₅₄H₆₄O₆S₂Si: C, 71.97; H, 7.16, found: C, 71.88; H, 7.21.

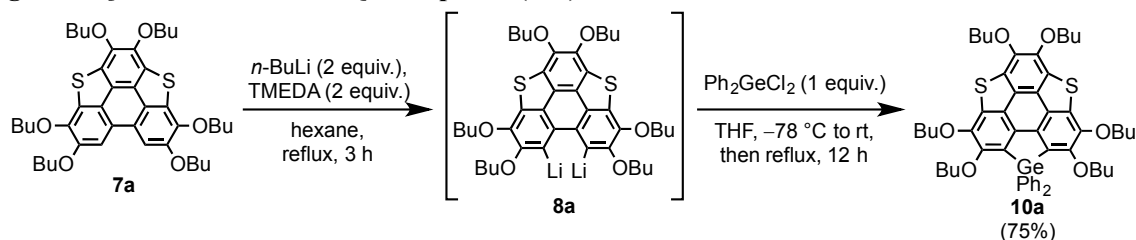
Synthesis of 2,3,6,7,10,11-hexaethoxytriphenyleno[4,5-2',3',4',5']1',1'-diphenyl-1*H*-silolo[8,9-*b,c,d*:12,1-*b'*,*c'*,*d'*]dithiophene (9b**)**



Anhydrous hexane (15 mL) and TMEDA (0.30 mL, 2.01 mmol) were added to **7b** (510 mg, 0.92 mmol) in a dried three-necked-flask under argon atmosphere. *n*-BuLi in hexane (2.66 M, 0.72 mL, 1.91 mmol) was added dropwise to the suspension. After the suspension was refluxed for 3 hours, volatiles were removed under reduced pressure. THF (15 mL) was added to the resulting solid and then cooled to under -70 °C. A THF (5 mL) solution of dichlorodiphenylsilane (0.20 mL, 0.96 mmol) was transferred to the reaction mixture via a PTFE tube over 10 min. After stirring for 30 min at -70 °C, the mixture was allowed to warm to room temperature and stirred for an hour then was refluxed for 12 hours. After cooling to room temperature, aqueous NH₄Cl was added to the mixture. The reaction mixture was extracted with chloroform and the extract was dried over anhydrous magnesium sulfate. The extract was concentrated under reduced pressure to give a brown solid. The products were purified by silica gel column chromatography (eluent, hexane : CH₂Cl₂ = 2 : 3, v/v) to give **9b** (53 mg, 0.073 mmol, 8%) as a yellow solid. mp. 216-217 °C; ¹H NMR (500 MHz, CDCl₃) δ 1.16 (t, *J* = 7.0 Hz, 6 H), 1.52 (t, *J* = 7.0 Hz, 6H), 1.54 (t, *J* = 7.0 Hz, 6H), 4.02 (q, *J* = 7.0 Hz, 4H), 4.48 (q, *J* = 7.0 Hz, 4H), 4.51 (q, *J* = 7.0 Hz, 4H), 7.34-7.37 (m, 4H), 7.40-7.44 (m, 2H), 7.82-7.84

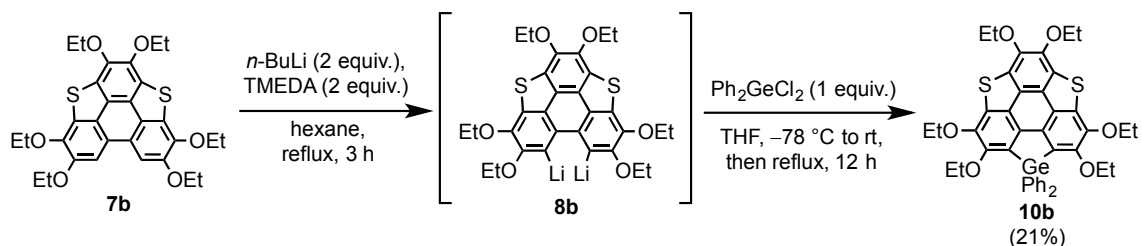
(m, 4H); ^{13}C NMR (125 MHz, CDCl_3) δ 15.38 (CH_3), 15.95 (CH_3), 15.97 (CH_3), 68.33 (CH_2), 68.85 (CH_2), 69.38 (CH_2), 120.78 (C), 126.01 (C), 126.19 (C), 127.19 (C), 128.33 (CH), 130.50 (CH), 133.11 (C), 133.14 (C), 135.14 (C), 135.78 (CH), 146.30 (C), 146.44 (C), 155.93 (C); ^{29}Si NMR (99 MHz, CDCl_3) δ 0.037; MS (MALDI) m/z calcd for $\text{C}_{42}\text{H}_{40}\text{O}_6\text{S}_2\text{Si}$ ($[\text{M}]^+$) 732.2, found: 732.2; elemental analysis calcd for $\text{C}_{42}\text{H}_{40}\text{O}_6\text{S}_2\text{Si}$: C, 68.82; H, 5.50, found: C, 68.68; H, 5.46.

Synthesis of 2,3,6,7,10,11-hexabutoxytriphenyleno[4,5-2',3',4',5']1',1'-diphenyl-1H-germolo[8,9-b,c,d:12,1-b',c',d']dithiophene (10a)



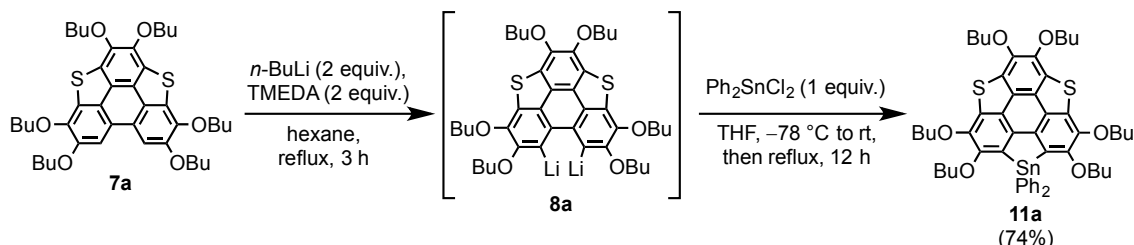
Anhydrous hexane (4.5 mL) and TMEDA (0.35 mL, 2.35 mmol) were added to **7a** (827 mg, 1.15 mmol) in a dried three-necked flask under argon atmosphere. *n*-BuLi in hexane (2.66 M, 0.87 mL, 2.31 mmol) was added dropwise to the suspension. After the suspension was refluxed for 3 hours, volatiles were removed under reduced pressure. THF (22 mL) was added to the resulting solid and then cooled to under $-70\text{ }^\circ\text{C}$. Dichlorodiphenylgermane (0.25 mL, 1.20 mmol) was added slowly to the solution. After stirring for 30 min at $-70\text{ }^\circ\text{C}$, the mixture was allowed to warm to room temperature and stirred for an hour. Then the mixture was refluxed for 12 hours. After cooling to room temperature, aqueous NH_4Cl was added to the mixture. The reaction mixture was extracted with chloroform and the extract was dried over anhydrous magnesium sulfate. The extract was concentrated under reduced pressure to give an orange solid. The products were purified by silica gel column chromatography (eluent, hexane : CH_2Cl_2 = 2 : 3, v/v) to give **10a** (813 mg, 0.859 mmol, 75%) as a pale yellow solid. mp. $126\text{ }^\circ\text{C}$; ^1H NMR (500 MHz, CDCl_3) δ 0.74 (t, J = 7.5 Hz, 6 H), 1.02 (t, J = 7.5 Hz, 6H), 1.04 (t, J = 7.5 Hz, 6H), 1.21 (sext, J = 7.5 Hz, 4H), 1.56-1.68 (m, 12H), 1.85-1.93 (m, 8H), 3.97 (t, J = 7.5 Hz, 4H), 4.40 (t, J = 7.5 Hz, 4H), 4.44 (t, J = 7.5 Hz, 4H), 7.35-7.42 (m, 6H), 7.72-7.73 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3) δ 13.84 (CH_3), 14.08 (CH_3), 19.07 (CH_2), 19.44 (CH_2), 32.04 (CH_2), 32.43 (CH_2), 32.48 (CH_2), 72.54 (CH_2), 73.00 (CH_2), 73.07 (CH_2), 121.13 (C), 125.40 (C), 125.81 (C), 126.92 (C), 128.78 (CH), 129.97 (CH), 132.44 (C), 134.10 (C), 134.95 (CH), 136.16 (C), 146.05 (C), 146.48 (C), 155.95 (C); MS (MALDI) m/z calcd for $\text{C}_{54}\text{H}_{65}\text{O}_6\text{S}_2\text{Ge}$ ($[\text{M}]^+$) 946.3, found: 992.3; elemental analysis calcd for $\text{C}_{54}\text{H}_{64}\text{O}_6\text{S}_2\text{Ge}$: C, 68.57; H, 6.82, found: C, 68.47; H, 6.82.

Synthesis of 2,3,6,7,10,11-hexaethoxytriphenyleno[4,5-2',3',4',5']1',1'-diphenyl-1H-germolo[8,9-b,c,d:12,1-b',c',d']dithiophene (10b)



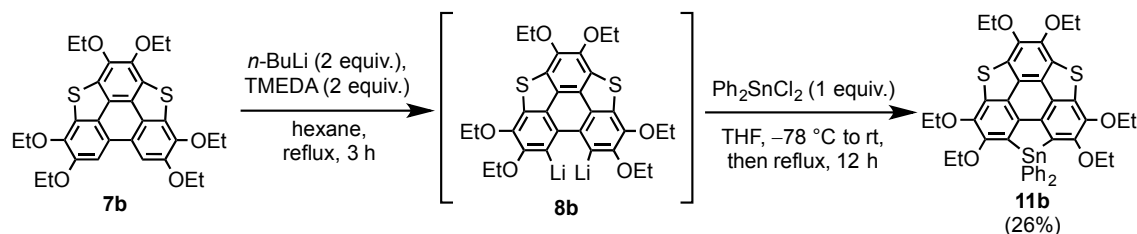
Anhydrous hexane (15 mL) and TMEDA (0.40 mL, 2.60 mmol) were added to **7b** (678 mg, 1.23 mmol) in a dried three-necked-flask under argon atmosphere. *n*-BuLi in hexane (2.65 M, 0.87 mL, 2.31 mmol) was added dropwise to the suspension. After the suspension was refluxed for 3 hours, volatiles were removed under reduced pressure. THF (15 mL) was added to the resulting solid and the mixture was cooled to under -78°C . Dichlorodiphenylgermane (0.26 mL, 1.25 mmol) was added slowly to the solution at -78°C and stirred for 30 min at the same temperature. The mixture was allowed to warm to room temperature and stirred for an hour then was refluxed for 12 hours. After cooling to room temperature, aqueous NH_4Cl was added to the mixture. The reaction mixture was extracted with chloroform and the extract was dried over anhydrous magnesium sulfate. The extract was concentrated under reduced pressure to give a brown solid. The products were purified by silica gel column chromatography (eluent, hexane : $\text{CH}_2\text{Cl}_2 = 1 : 1$, v/v) to give **10b** (198 mg, 0.255 mmol, 21%) as a pale yellow solid. mp. $224\text{--}225^\circ\text{C}$; ^1H NMR (500 MHz, CDCl_3) δ 1.20 (t, $J = 7.0$ Hz, 6 H), 1.53 (t, $J = 7.0$ Hz, 6H), 1.55 (t, $J = 7.0$ Hz, 6H), 4.07 (q, $J = 7.0$ Hz, 4H), 4.49 (q, $J = 7.0$ Hz, 4H), 4.53 (q, $J = 7.0$ Hz, 4H), 7.35–7.42 (m, 6H), 7.73–7.75 (m, 4H); ^{13}C NMR (125 MHz, CDCl_3) δ 15.41 (CH_3), 15.96 (CH_3), 16.00 (CH_3), 68.38 (CH_2), 68.82 (CH_2), 69.02 (CH_2), 122.04 (C), 125.61 (C), 125.94 (C), 127.18 (C), 128.78 (CH), 130.00 (CH), 132.48 (C), 134.17 (C), 134.90 (CH), 135.95 (C), 146.25 (C), 146.27 (C), 155.62 (C); MS (MALDI) m/z calcd for $\text{C}_{42}\text{H}_{40}\text{O}_6\text{S}_2\text{Ge}$ ($[\text{M}]^+$) 778.1, found: 778.2; elemental analysis calcd for $\text{C}_{42}\text{H}_{40}\text{O}_6\text{S}_2\text{Ge}$: C, 64.88; H, 5.19, found: C, 65.04; H, 5.12.

Synthesis of 2,3,6,7,10,11-hexabutoxytriphenyleno[4,5-2',3',4',5']1',1'-diphenyl-1*H*-stannolo[8,9-*b,c,d*:12,1-*b',c',d'*]dithiophene (11a**)**



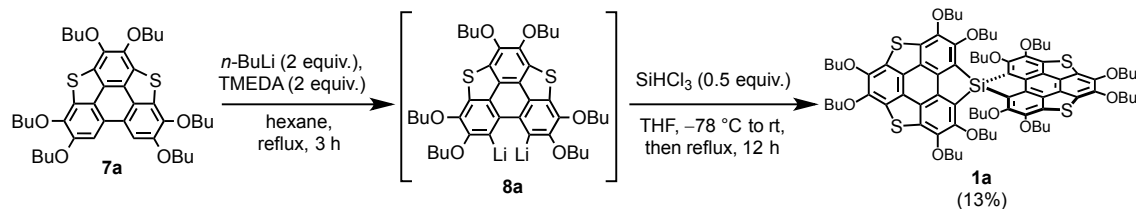
Anhydrous hexane (4.0 mL) and TMEDA (0.32 mL, 2.15 mmol) were added to **7a** (764 mg, 1.32 mmol) in a dried three-necked-flask under argon atmosphere. *n*-BuLi in hexane (2.66 M, 0.80 mL, 2.13 mmol) was added dropwise to the suspension. After the suspension was refluxed for 3 hours, volatiles were removed under reduced pressure. THF (16 mL) was added to the resulting solid and the mixture was cooled to under $-70\text{ }^{\circ}\text{C}$. A THF (7 mL) solution of dichlorodiphenylstannane (384 mg, 1.12 mmol) was transferred to the reaction mixture via a PTFE tube over 10 min. After stirring for 30 min at $-70\text{ }^{\circ}\text{C}$, the mixture was allowed to warm to room temperature and stirred for an hour. The solution was refluxed for 12 hours. After cooling to room temperature, aqueous NH_4Cl was added to the mixture. The reaction mixture was extracted with chloroform and the extract was dried over anhydrous magnesium sulfate. The extract was concentrated under reduced pressure to give an orange solid. The products were purified by silica gel column chromatography (eluent, hexane : $\text{CH}_2\text{Cl}_2 = 2 : 3$, v/v) to give **11a** (782 mg, 0.788 mmol, 74%) as a pale yellow solid. mp. $125\text{--}126\text{ }^{\circ}\text{C}$ (dec.); ^1H NMR (500 MHz, CDCl_3) δ 0.75 (t, $J = 7.5$ Hz, 6 H), 1.02 (t, $J = 7.5$ Hz, 6H), 1.04 (t, $J = 7.0$ Hz, 6H), 1.27 (sext, $J = 7.5$ Hz, 4H), 1.58-1.70 (m, 12H), 1.85-1.93 (m, 8H), 4.08 (t, $J = 7.0$ Hz, 4H), 4.40 (t, $J = 6.5$ Hz, 4H), 4.46 (t, $J = 6.5$ Hz, 4H), 7.37-7.42 (m, 6H), 7.67-7.69 (m, 4H); ^{13}C NMR (125 MHz, CDCl_3) δ 13.78 (CH_3), 14.09 (CH_3), 19.19 (CH_2), 19.45 (CH_2), 30.06 (CH_2), 32.03 (CH_2), 32.45 (CH_2), 32.56 (CH_2), 71.56 (CH_2), 72.65 (CH_2), 72.97 (CH_2), 118.71 (C), 124.22 (C), 125.23 (C), 127.05 (C), 129.25 (CH), 129.98 (CH), 133.07 (C), 134.94 (C), 137.05 (CH), 138.87 (C), 145.37 (C), 146.35 (C), 157.46 (C); ^{119}Sn NMR (186 MHz, CDCl_3) δ -62.96 ; MS (MALDI) m/z calcd for $\text{C}_{54}\text{H}_{64}\text{O}_6\text{S}_2\text{Sn}$ ($[\text{M}]^+$) 992.3, found: 992.3; elemental analysis calcd for $\text{C}_{54}\text{H}_{64}\text{O}_6\text{S}_2\text{Sn}$: C, 65.39; H, 6.50, found: C, 65.25; H, 6.53.

Synthesis of 2,3,6,7,10,11-hexaethoxytriphenyleno[4,5-2',3',4',5']1',1'-diphenyl-1*H*-stannolo[8,9-*b,c,d*:12,1-*b'*,*c'*,*d'*]dithiophene (11b**)**



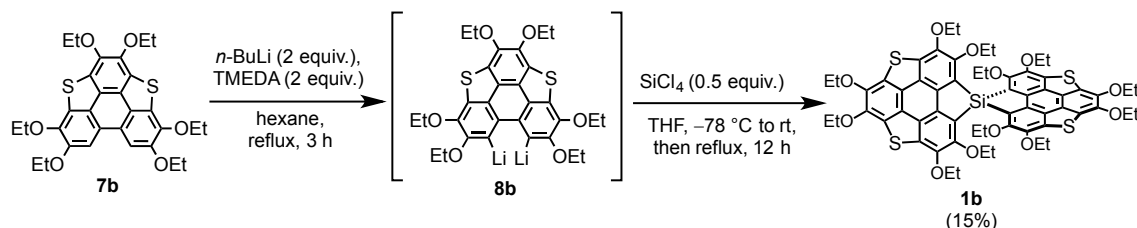
Anhydrous hexane (50 mL) and TMEDA (0.60 mL, 4.03 mmol) were added to **7b** (1.00 g, 1.82 mmol) in a dried three-necked-flask under argon atmosphere. *n*-BuLi in hexane (2.66 M, 1.40 mL, 3.72 mmol) was added dropwise to the suspension. After the suspension was refluxed for 3 hours, volatiles were removed under reduced pressure. THF (50 mL) was added to the resulting solid and the mixture was cooled to under -70°C . A THF (10 mL) solution of dichlorodiphenylstannane (635 mg, 1.85 mmol) was transferred to the reaction mixture via a PTFE tube over 5 min. After stirring for 30 min at -70°C , the mixture was allowed to warm to room temperature and stirred for an hour then was refluxed for 12 hours. After cooling to room temperature, aqueous NH_4Cl was added to the mixture. The reaction mixture was extracted with chloroform and the extract was dried over anhydrous magnesium sulfate. The extract was concentrated under reduced pressure to give a brown solid. The products were purified by silica gel column chromatography (eluent, hexane : CH_2Cl_2 = 1 : 1, v/v) to give **11b** (386 mg, 0.47 mmol, 26%) as a pale yellow solid. mp. $237\text{--}238^{\circ}\text{C}$; ^1H NMR (500 MHz, CDCl_3) δ 1.16 (t, $J = 7.0$ Hz, 6 H), 1.52 (t, $J = 7.0$ Hz, 6H), 1.54 (t, $J = 7.0$ Hz, 6H), 4.02 (q, $J = 7.0$ Hz, 4H), 4.48 (q, $J = 7.0$ Hz, 4H), 4.51 (q, $J = 7.0$ Hz, 4H), 7.34–7.37 (m, 4H), 7.40–7.43 (m, 2H), 7.82–7.84 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3) δ 15.52 (CH_3), 15.98 (CH_3), 16.07 (CH_3), 67.64 (CH_2), 68.48 (CH_2), 68.77 (CH_2), 119.70 (C), 124.38 (C), 125.31 (C), 127.26 (C), 129.28 (CH), 130.02 (CH), 133.07 (C), 134.99 (C), 137.02 (CH), 138.67 (C), 145.38 (C), 146.09 (C), 157.17 (C); ^{119}Sn NMR (186 MHz, CDCl_3) δ -62.13 ; MS (MALDI) m/z calcd for $\text{C}_{42}\text{H}_{40}\text{O}_6\text{S}_2\text{Sn}$ ($[\text{M}]^+$) 824.1, found: 824.1; elemental analysis calcd for $\text{C}_{42}\text{H}_{40}\text{O}_6\text{S}_2\text{Sn}$: C, 61.25; H, 4.90, found: C, 61.25; H, 4.73.

Synthesis of spirobi{2,3,6,7,10,11-hexabutoxytriphenyleno[4,5-2',3',4',5']1',1'-diphenyl -1*H*-silolo[8,9-*b,c,d*:12,1-*b',c',d'*]dithiophene} (1a)



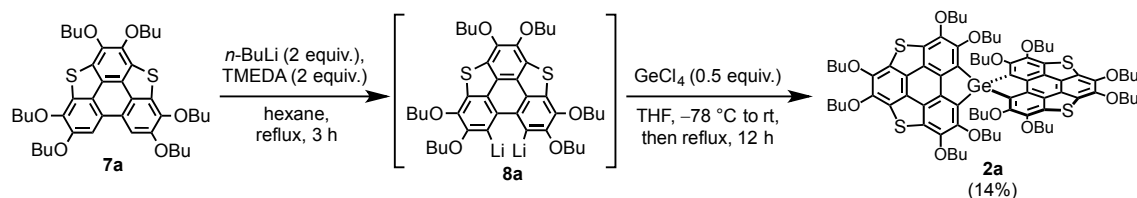
Anhydrous hexane (4.0 mL) and TMEDA (0.32 mL, 2.15 mmol) were added to **7a** (764 mg, 1.32 mmol) in dried a three-necked-flask under argon atmosphere. *n*-BuLi in hexane (2.66 M, 0.80 mL, 2.13 mmol) was added dropwise to the suspension. After the suspension was refluxed for 3 hours, volatiles were removed under reduced pressure. THF (16 mL) was added to the resulting solid and the mixture was cooled to under $-70\text{ }^{\circ}\text{C}$. A THF (7 mL) solution of dichlorodiphenylstannane (384 mg, 1.12 mmol) was transferred to the reaction mixture via a PTFE tube over 10 min. After stirring for 30 min at $-70\text{ }^{\circ}\text{C}$, the mixture was allowed to warm to room temperature and stirred for an hour then was refluxed for 12 hours. After cooling to room temperature, aqueous NH_4Cl was added to the mixture. The reaction mixture was extracted with chloroform and the extract was dried over anhydrous magnesium sulfate. The extract was concentrated under reduced pressure to give an orange solid. The products were purified by silica gel column chromatography (eluent, hexane : $\text{CH}_2\text{Cl}_2 = 2 : 3$, v/v) to give **1a** (782 mg, 0.788 mmol, 74%) as a pale yellow solid. mp. $84\text{--}85\text{ }^{\circ}\text{C}$ (dec.); ^1H NMR (500 MHz, CDCl_3) δ -0.06 (t, $J = 7.5$ Hz, 12 H), $-0.01\text{--}0.05$ (m, 8H), 0.97 (t, $J = 7.5$ Hz, 12H), $1.03\text{--}1.11$ (m, 8H), 1.06 (t, $J = 7.5$ Hz, 12H), $1.51\text{--}1.59$ (m, 8H), 1.66 (sext, $J = 7.5$ Hz, 8H), 1.79 (quin, $J = 7.5$ Hz, 8H), 1.92 (quint, $J = 7.5$ Hz, 8H), 3.60 (t, $J = 7.0$ Hz, 12H), 4.29 (t, $J = 6.5$ Hz, 12H), 4.47 (t, $J = 6.5$ Hz, 12H); ^{13}C NMR (125 MHz, CDCl_3) δ 12.88 (CH_3), 14.02 (CH_3), 14.07 (CH_3), 17.71 (CH_2), 19.35 (CH_2), 19.45 (CH_2), 30.96 (CH_2), 32.38 (CH_2), 32.40 (CH_2), 70.21 (CH_2), 72.76 (CH_2), 73.09 (CH_2), 114.81 (C), 125.31 (C), 125.68 (C), 126.27 (C), 134.57 (C), 134.95 (C), 144.68 (C), 146.69 (C), 156.94 (C); ^{29}Si NMR (99 MHz, CDCl_3) δ 3.39 ; MS (MALDI) m/z calcd for $\text{C}_{84}\text{H}_{109}\text{O}_{12}\text{S}_4\text{Si}$ ($[\text{M}+\text{H}]^+$) 1465.6 , found: 1465.3 ; elemental analysis calcd for $\text{C}_{84}\text{H}_{108}\text{O}_{12}\text{S}_4\text{Si}$: C, 68.82; H, 7.42, found: C, 68.79; H, 7.46.

Synthesis of spirobi{2,3,6,7,10,11-hexaethoxytriphenyleno[4,5-2',3',4',5']1',1'-diphenyl-1*H*-silolo[8,9-*b,c,d*:12,1-*b',c',d'*]dithiophene} (1b)



Anhydrous hexane (34 mL) and TMEDA (0.58 mL, 3.89 mmol) were added to **7b** (1.03 g, 1.87 mmol) in dried a three-necked-flask under argon atmosphere. *n*-BuLi in hexane (2.66 M, 1.40 mL, 3.72 mmol) was added dropwise to the suspension. After the suspension was refluxed for 3 hours, volatiles were removed under reduced pressure. THF (34 mL) was added to the resulting solid and the mixture was cooled to under $-70\text{ }^{\circ}\text{C}$. A toluene (21 mL) solution of tetrachlorosilane (0.12 mL, 1.05 mmol) was transferred to the reaction mixture via a PTFE tube over 15 min. After stirring for 30 min at $-70\text{ }^{\circ}\text{C}$, the mixture was allowed to warm to room temperature and stirred for an hour then was refluxed for 12 hours. After cooling to room temperature, aqueous NH_4Cl was added to the mixture. The reaction mixture was extracted with chloroform and the extract was dried over anhydrous magnesium sulfate. The extract was concentrated under reduced pressure to give a brown solid. The products were purified by silica gel column chromatography (eluent, hexane : CH_2Cl_2 = 1 : 4, *v/v*) to give **1b** (158 mg, 0.140 mmol, 15%) as a yellow solid. mp. $226\text{ }^{\circ}\text{C}$; ^1H NMR (500 MHz, CDCl_3) δ 0.61 (t, $J = 7.0$ Hz, 12 H), 1.44 (t, $J = 7.0$ Hz, 12H), 1.59 (t, $J = 7.0$ Hz, 12H), 3.71 (q, $J = 7.0$ Hz, 8H), 4.40 (q, $J = 7.0$ Hz, 8H), 4.57 (q, $J = 7.0$ Hz, 8H); ^{13}C NMR (125 MHz, CDCl_3) δ 14.92 (CH_3), 15.89 (CH_3), 15.99 (CH_3), 66.41 (CH_2), 68.48 (CH_2), 68.89 (CH_2), 114.64 (C), 125.05 (C), 125.14 (C), 125.78 (C), 134.00 (C), 134.47 (C), 144.91 (C), 146.54 (C), 156.72 (C); ^{29}Si NMR (99 MHz, CDCl_3) δ 2.72; MS (MALDI) m/z calcd for $\text{C}_{60}\text{H}_{61}\text{O}_{12}\text{S}_4\text{Si}$ ($[\text{M}+\text{H}]^+$) 1129.3, found: 1129.1; elemental analysis calcd for $\text{C}_{60}\text{H}_{60}\text{O}_{12}\text{S}_4\text{Si}$: C, 63.80; H, 5.35, found: C, 63.44; H, 5.29.

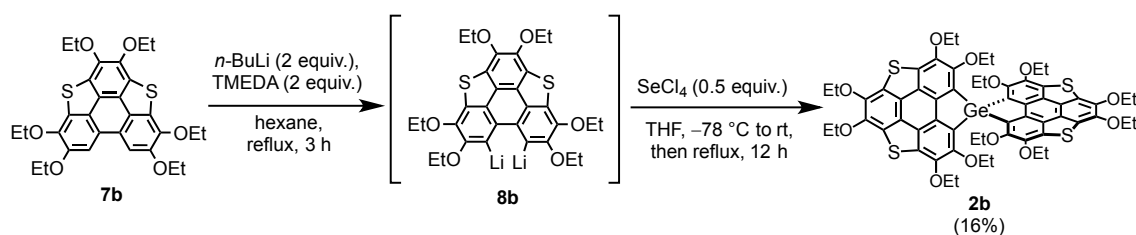
Synthesis of spirobi{2,3,6,7,10,11-hexabutoxytriphenyleno[4,5-2',3',4',5']1',1'-diphenyl-1*H*-germolo[8,9-*b,c,d*:12,1-*b',c',d'*]dithiophene} (2a)



Anhydrous hexane (7.0 mL) and TMEDA (0.57 mL, 3.83 mmol) were added to **7a** (1.37 g, 1.90 mmol) in dried a three-necked-flask under argon atmosphere. *n*-BuLi in hexane (2.66 M, 1.45 mL,

3.86 mmol) was added dropwise to the suspension. After the suspension was refluxed for 3 hours, volatiles were removed under reduced pressure. THF (25 mL) was added to the resulting solid and the mixture was cooled to under $-70\text{ }^{\circ}\text{C}$. A THF (10 mL) solution of tetrachlorogermane (0.11 mL, 0.964 mmol) was transferred to the reaction mixture via a PTFE tube over 15 min. After stirring for 30 min at $-70\text{ }^{\circ}\text{C}$, the mixture was allowed to warm to room temperature and stirred for an hour then was refluxed for 12 hours. After cooling to room temperature, aqueous NH_4Cl was added to the mixture. The reaction mixture was extracted with chloroform and the extract was dried over anhydrous magnesium sulfate. The extract was concentrated under reduced pressure to give an orange solid. The products were purified by silica gel column chromatography (eluent, hexane : $\text{CH}_2\text{Cl}_2 = 2 : 3$, v/v) to give **2a** (209 mg, 0.138 mmol, 14%) as a yellow solid. mp. $113\text{--}115\text{ }^{\circ}\text{C}$ (dec.) $^{\circ}\text{C}$; ^1H NMR (500 MHz, CDCl_3) δ -0.07 (t, $J = 7.5$ Hz, 12H), 0.09 (sext, $J = 7.5$ Hz, 8H), 0.97 (t, $J = 7.5$ Hz, 12H), 1.06 (t, $J = 7.5$ Hz, 12H), 1.13 (quint, $J = 7.5$ Hz, 8H), $1.55\text{--}1.60$ (m, 8H), 1.66 (sext, $J = 7.5$ Hz, 12H), 1.80 (quint, $J = 8.0$ Hz, 8H), 1.93 (quint, $J = 7.0$ Hz, 8H), 3.67 (t, $J = 7.5$ Hz, 12H), 4.31 (t, $J = 6.5$ Hz, 12H), 4.48 (t, $J = 6.5$ Hz, 12H); ^{13}C NMR (125 MHz, CDCl_3) δ 12.82 (CH_3), 14.03 (CH_3), 14.09 (CH_3), 17.79 (CH_2), 19.37 (CH_2), 19.46 (CH_2), 30.97 (CH_2), 32.41 (CH_2), 70.15 (CH_2), 72.81 (CH_2), 73.06 (CH_2), 117.06 (C), 124.95 (C), 125.36 (C), 126.22 (C), 132.75 (C), 134.23 (C), 144.85 (C), 146.68 (C), 156.14 (C); MS (MALDI) m/z calcd for $\text{C}_{84}\text{H}_{109}\text{O}_{12}\text{S}_4\text{Ge}$ ($[\text{M}+\text{H}]^+$) 1511.6 , found: 1511.7 ; elemental analysis calcd for $\text{C}_{84}\text{H}_{108}\text{O}_{12}\text{S}_4\text{Ge}$: C, 66.79; H, 7.21, found: C, 66.60; H, 7.26.

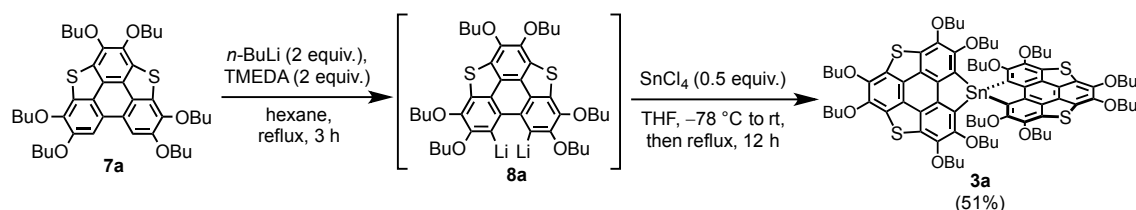
Synthesis of spirobi{2,3,6,7,10,11-hexaethoxytriphenyleno[4,5-2',3',4',5']1',1'-diphenyl-1H-germolo[8,9-b,c,d:12,1-b',c',d']dithiophene } (2b**)**



Anhydrous hexane (14 mL) and TMEDA (0.27 mL, 1.81 mmol) were added to **7b** (482 mg, 0.872 mmol) in a dried three-necked-flask under argon atmosphere. $n\text{-BuLi}$ in hexane (2.66 M, 0.66 mL, 1.76 mmol) was added dropwise to the suspension. After the suspension was refluxed for 3 hours, volatiles were removed under reduced pressure. THF (14 mL) was added to the resulting solid and the mixture was cooled to under $-70\text{ }^{\circ}\text{C}$. A toluene (10 mL) solution of tetrachlorogermane (60 μL , 0.52 mmol) was transferred to the reaction mixture via a PTFE tube over 10 min. After stirring for 30 min at $-70\text{ }^{\circ}\text{C}$, the mixture was allowed to warm to room temperature and stirred for an hour

then was refluxed for 12 hours. After cooling to room temperature, aqueous NH_4Cl was added to the mixture. The reaction mixture was extracted with chloroform and the extract was dried over anhydrous magnesium sulfate. The extract was concentrated under reduced pressure to give a brown solid. The products were purified by silica gel column chromatography (eluent, hexane : CH_2Cl_2 = 1 : 3, v/v) to give **2b** (80 mg, 69.8 μmol , 16%) as a pale yellow solid. mp. 236 $^\circ\text{C}$; ^1H NMR (500 MHz, CDCl_3) δ 0.68 (t, J = 7.0 Hz, 12H), 1.45 (t, J = 7.0 Hz, 12H), 1.59 (t, J = 7.0 Hz, 12H), 3.78 (q, J = 7.0 Hz, 8H), 4.42 (q, J = 7.0 Hz, 8H), 4.58 (q, J = 7.0 Hz, 8H); ^{13}C NMR (125 MHz, CDCl_3) δ 15.01 (CH_3), 15.91 (CH_3), 16.01 (CH_3), 66.29 (CH_2), 68.53 (CH_2), 68.87 (CH_2), 116.99 (C), 124.77 (C), 124.96 (C), 125.78 (C), 132.21 (C), 133.79 (C), 145.02 (C), 146.55 (C), 155.92 (C); MS (MALDI) m/z calcd for $\text{C}_{60}\text{H}_{60}\text{O}_{12}\text{S}_4\text{Ge}$ ($[\text{M}]^+$) 1174.2, found: 1174.6; elemental analysis calcd for $\text{C}_{60}\text{H}_{60}\text{O}_{12}\text{S}_4\text{Ge}$: C, 61.38; H, 5.15, found: C, 61.40; H, 5.00.

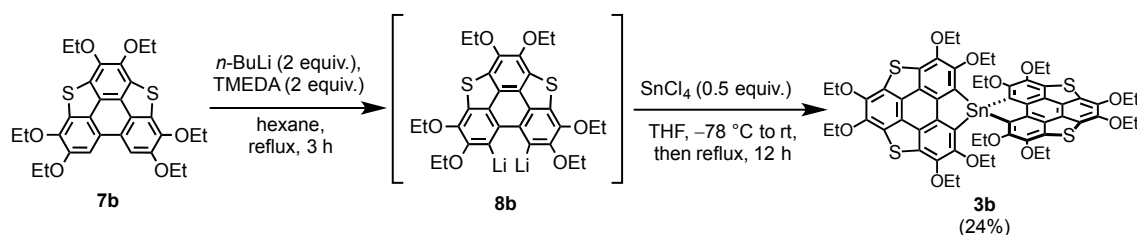
Synthesis of spirobi{2,3,6,7,10,11-hexabutoxytriphenyleno[4,5-2',3',4',5']1',1'-diphenyl-1H-stannolo[8,9-b,c,d:12,1-b',c',d']dithiophene} (3a**)**



Anhydrous hexane (4.0 mL) and TMEDA (0.37 mL, 2.48 mmol) were added to **7a** (873 mg, 1.21 mmol) in a dried three-necked-flask under argon atmosphere. $n\text{-BuLi}$ in hexane (2.66 M, 0.92 mL, 2.45 mmol) was added dropwise to the suspension. After the suspension was refluxed for 3 hours, volatiles were removed under reduced pressure. THF (18 mL) was added to the resulting solid and the mixture was cooled to under $-70\text{ }^\circ\text{C}$. A toluene (8 mL) solution of tetrachlorostannane (76 μL , 0.650 mmol) was transferred to the reaction mixture via a PTFE tube over 15 min. After stirring for 30 min at $-70\text{ }^\circ\text{C}$, the mixture was allowed to warm to room temperature and stirred for an hour. The solution was refluxed for 12 hours. After cooling to room temperature, aqueous NH_4Cl was added to the mixture. The reaction mixture was extracted with chloroform and the extract was dried over anhydrous magnesium sulfate. The extract was concentrated under reduced pressure to give an orange solid. The products were purified by silica gel column chromatography (eluent, hexane : CH_2Cl_2 = 1:1, v/v) to give **3a** (480 mg, 0.308 mmol, 51%) as a yellow solid. mp. 126 $^\circ\text{C}$ (dec.); ^1H NMR (500 MHz, CDCl_3) δ 0.04 (t, J = 7.5 Hz, 12H), 0.38 (sext, J = 7.5 Hz, 8H), 0.98 (t, J = 7.5 Hz, 12H), 1.06 (t, J = 7.5 Hz, 12H), 1.28 (quint, J = 7.5 Hz, 8H), 1.58 (sext, J = 7.5 Hz, 8H), 1.67 (sext, J = 7.5 Hz, 8H), 1.82 (quint, J = 7.5 Hz, 8H), 1.93 (quint, J = 7.5 Hz, 8H), 3.84 (t, J = 7.5 Hz, 12H), 4.34 (t, J = 6.5 Hz, 12H), 4.50 (t, J = 6.5 Hz, 12H); ^{13}C NMR (125 MHz, CDCl_3) δ 12.88 (CH_3),

14.03 (CH₃), 14.09 (CH₃), 18.19 (CH₂), 19.38 (CH₂), 19.47 (CH₂), 30.08 (CH₂), 32.45 (CH₂), 32.47 (CH₂), 70.13 (CH₂), 72.81 (CH₂), 73.01 (CH₂), 116.65 (C), 124.05 (C), 124.86 (C), 126.71 (C), 133.05 (C), 134.30 (C), 145.04 (C), 146.57 (C), 157.12 (C); ¹¹⁹Sn NMR (186 MHz, CDCl₃) δ –25.86; MS (MALDI) *m/z* calcd for C₈₄H₁₀₉O₁₂S₄Sn ([M+H]⁺) 1557.6, found: 1557.4; elemental analysis calcd for C₈₄H₁₀₈O₁₂S₄Sn: C,64.81; H, 6.99, found: C,64.82; H, 6.99.

Synthesis of spirobi{2,3,6,7,10,11-hexaethoxytriphenyleno[4,5-2',3',4',5']1',1'-diphenyl-1*H*-stannolo[8,9-*b,c,d*:12,1-*b',c',d'*]dithiophene } (3b)



Anhydrous hexane (44 mL) and TMEDA (0.76 mL, 5.10 mmol) were added to **7b** (1.36 g, 2.49 mmol) in a dried three-necked-flask under argon atmosphere. *n*-BuLi in hexane (2.66 M, 1.87 mL, 4.97 mmol) was added dropwise to the suspension. After the suspension was refluxed for 3 hours, volatiles were removed under reduced pressure. THF (44 mL) was added to the resulting solid and the mixture was cooled to under –70 °C. A toluene (15 mL) solution of tetrachlorostannane (0.15 mL, 1.28 mmol) was transferred to the reaction mixture via a PTFE tube over 10 min. After stirring for 30 min at –70 °C, the mixture was allowed to warm to room temperature and stirred for an hour. The solution was refluxed for 12 hours. After cooling to room temperature, aqueous NH₄Cl was added to the mixture. The reaction mixture was extracted with chloroform and the extract was dried over anhydrous magnesium sulfate. The extract was concentrated under reduced pressure to give a brown solid. The products were purified by silica gel column chromatography (eluent, hexane : CH₂Cl₂ = 1 : 3, v/v) to give **3b** (364 mg, 0.299 mmol, 24%) as a pale yellow solid. mp. 236 °C; ¹H NMR (500 MHz, CDCl₃) δ 0.87 (t, *J* = 7.0 Hz, 12H), 1.48 (t, *J* = 7.0 Hz, 12H), 1.59 (t, *J* = 7.0 Hz, 12H), 3.94 (q, *J* = 7.0 Hz, 8H), 4.46 (q, *J* = 7.0 Hz, 8H), 4.58 (q, *J* = 7.0 Hz, 8H); ¹³C NMR (125 MHz, CDCl₃) δ 15.21 (CH₃), 15.98 (CH₃), 16.02 (CH₃), 66.14 (CH₂), 68.59 (CH₂), 68.89 (CH₂), 117.04 (C), 124.19 (C), 124.88 (C), 126.60 (C), 132.92 (C), 134.12 (C), 145.11 (C), 146.41 (C), 156.86 (C); ¹¹⁹Sn NMR (186 MHz, CDCl₃) δ –28.72; MS (MALDI) *m/z* calcd for C₆₀H₆₀O₁₂S₄Sn ([M]⁺) 1174.2, found: 1174.6; elemental analysis calcd for C₆₀H₆₀O₁₂S₄Sn: C,61.38; H, 5.15, found: C,61.40; H, 5.00.

3. X-ray crystallographic analysis

Table S1. Crystal data for **1b**, **2b** and **3b**.

	1b	2b	3c
crystal system	triclinic	triclinic	monoclinic
space group	<i>P</i> -1	<i>P</i> -1	<i>P</i> 2 ₁ / <i>n</i>
<i>a</i> /Å	9.7198(13)	9.7680(7)	9.2728(7)
<i>b</i> /Å	13.5510(18)	11.2196(8)	21.8232(16)
<i>c</i> /Å	21.616(3)	25.3543(18)	27.505(2)
α /deg	104.724(2)	88.261(1)	—
β /deg	100.877(1)	82.947(1)	88.261(1)
γ /deg	95.266(2)	86.248(1)	—
<i>V</i> /Å ³	2674.7(6)	2751.0(3)	5523.1(7)
<i>Z</i>	2	2	4
<i>D</i> _{calc} /g cm ⁻³	1.402	1.502	1.569
<i>R</i> ₁ (<i>I</i> > 2σ(<i>I</i>))	0.0395	0.0352	0.0295
<i>R</i> ₂ (all data)	0.1104	0.0888	0.0723
goodness of fit	1.023	1.032	1.016
<i>T</i> /K	100	100	100

4. Electrochemical study

Table S2. Electrochemical properties of **9b–11b** and **1b–3b**.

	E^1_{pa} [V]	E^1_{pc} [V]	$E^1_{1/2}$ [V]	E^2_{pa} [V]	E^2_{pc} [V]	$E^2_{1/2}$ [V]
9b	0.45	0.36	0.40	NA	NA	NA
10b	0.44	0.35	0.40	0.74	—	—
11b	0.47	0.34	0.41	0.72	—	—
1b	0.44	0.37	0.40	0.57	0.50	0.54
2b	0.45	0.38	0.41	0.59	0.51	0.55
3b	0.44	0.38	0.41	0.58	0.50	0.54

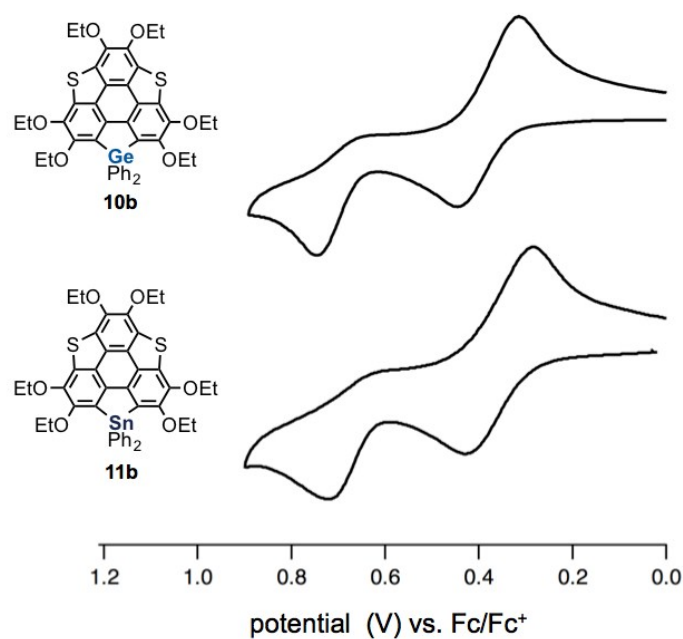


Fig. S1 Cyclic voltammograms of **10b** (0.62 mM) and **11b** (0.67 mM) in CH_2Cl_2 containing $0.1 \text{ M } n\text{-Bu}_4\text{NClO}_4$ at scan rate of 100 mV s^{-1} .

5. Chemical oxidation of spirobistannasumanene **3b**

Scheme S1 Chemical oxidation of **3b** using AgSbF₆ as an oxidant.



An anhydrous THF (1 mL) solution of silver hexafluoroantimonate(V) (8.1 mg, 23.6 μmol) was added dropwise to a THF (3 mL) solution of **9b** (26 mg, 21.3 μmol) at $-78\text{ }^{\circ}\text{C}$. After stirring for 30 min at the same temperature, the mixture was allowed to warm to room temperature and stirred for an hour. The reaction mixture was concentrated under reduced pressure to give a blue solid. The solid was extracted with dichloromethane and the extract was concentrated under reduced pressure to give a green solid (30.9 mg). An ESR spectrum of the product in CH₂Cl₂ showed a signal at g value of 2.0061 (Fig. S2).

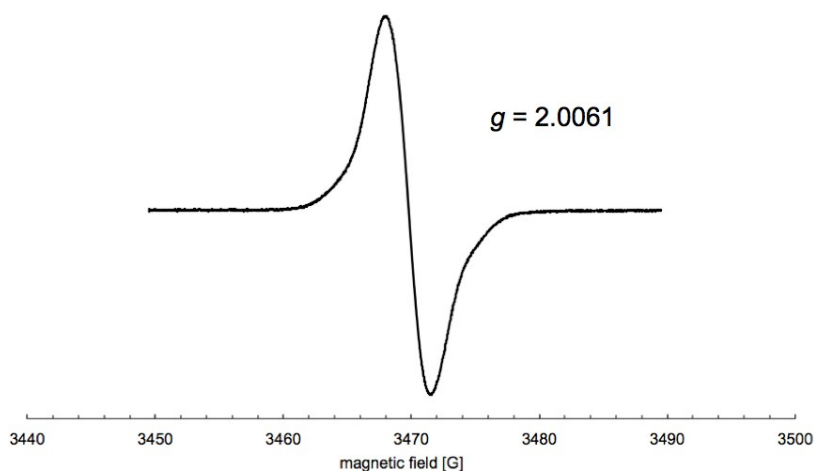


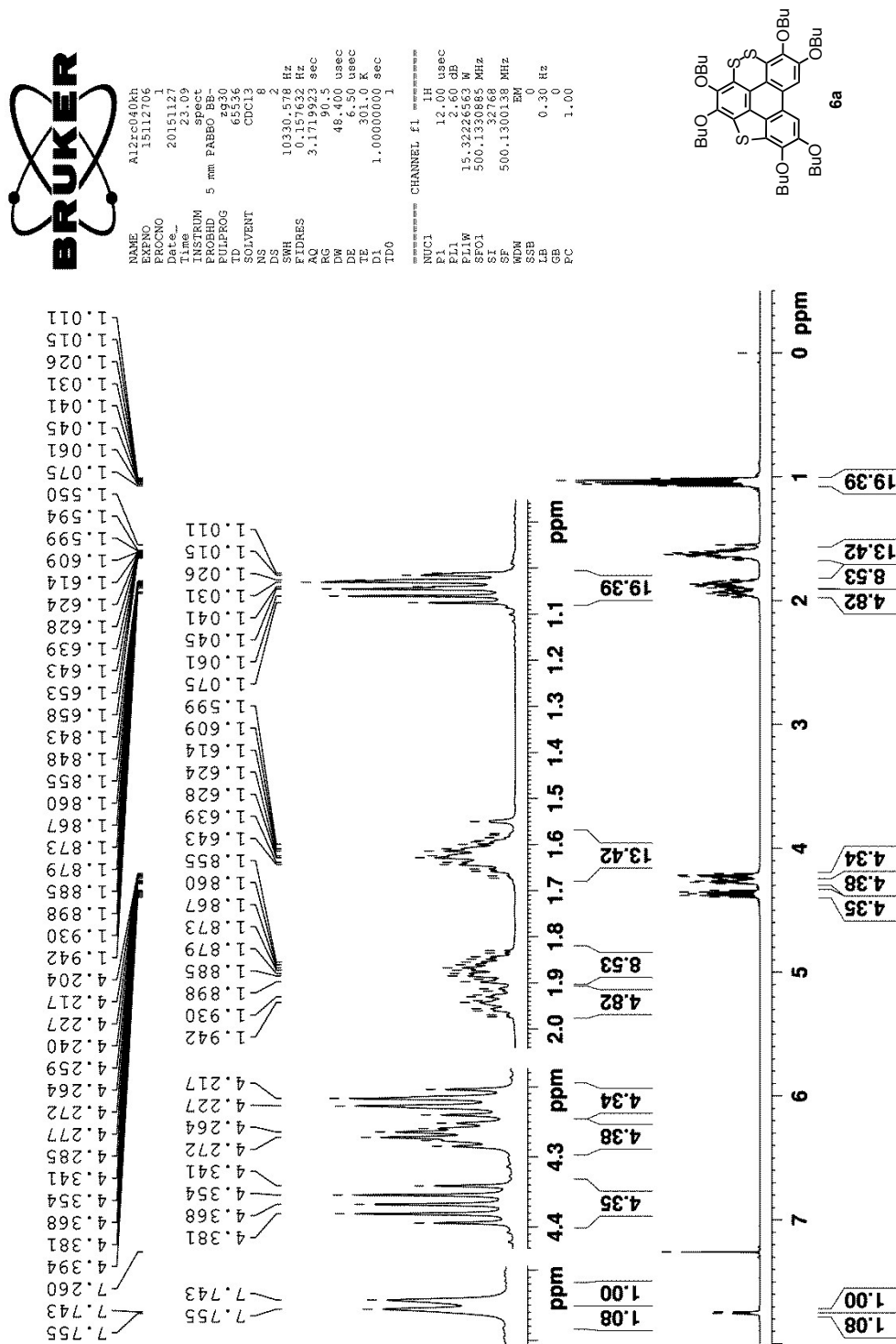
Fig. S2 ESR spectrum of the product of the oxidation reaction of **3b** using 1 equiv. of AgSbF₆, measured in CH₂Cl₂ at room temperature.

6. Reference

- 1) Wang, Y.; Zhang, C.; Wu, H.; Pu, J. *J. Mater. Chem. C.*, **2014**, 2, 1667.

7. NMR charts

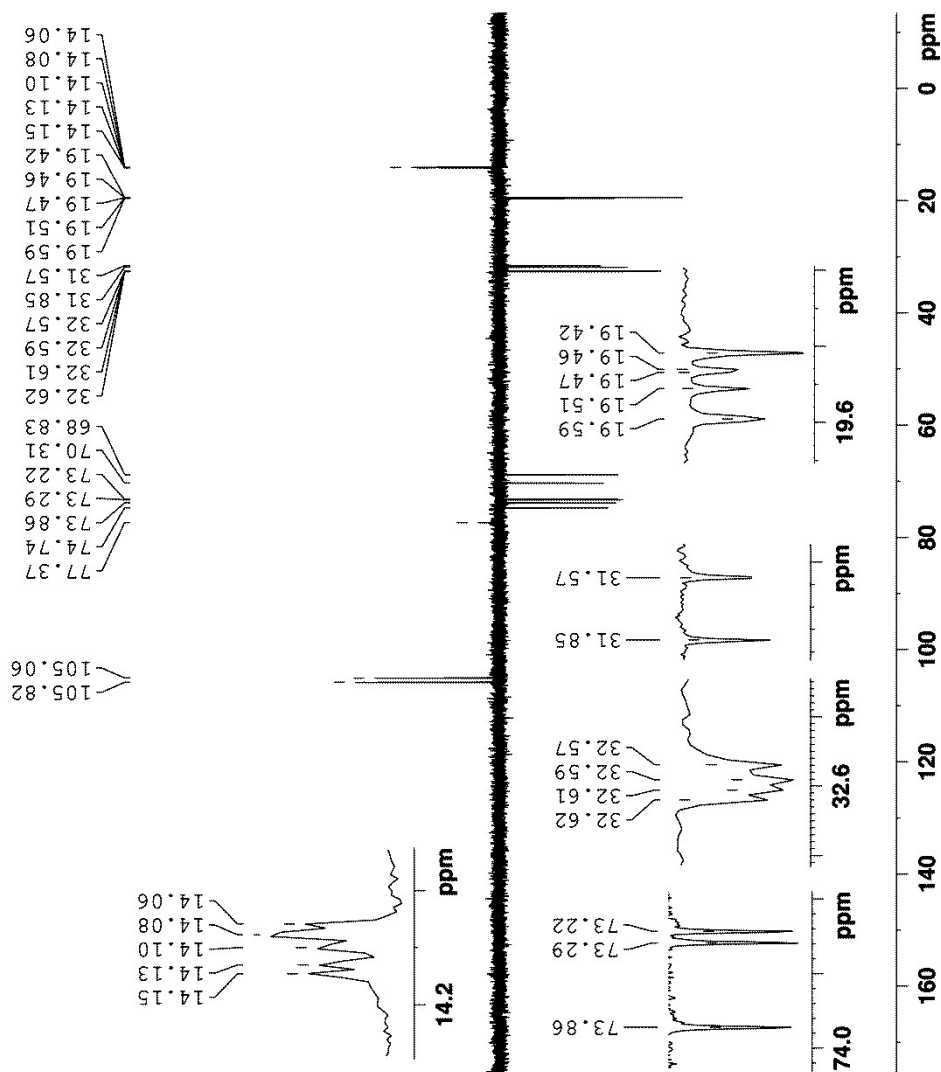
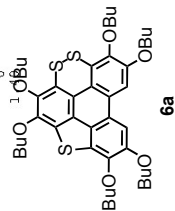
^1H NMR spectrum of 6a in CDCl_3



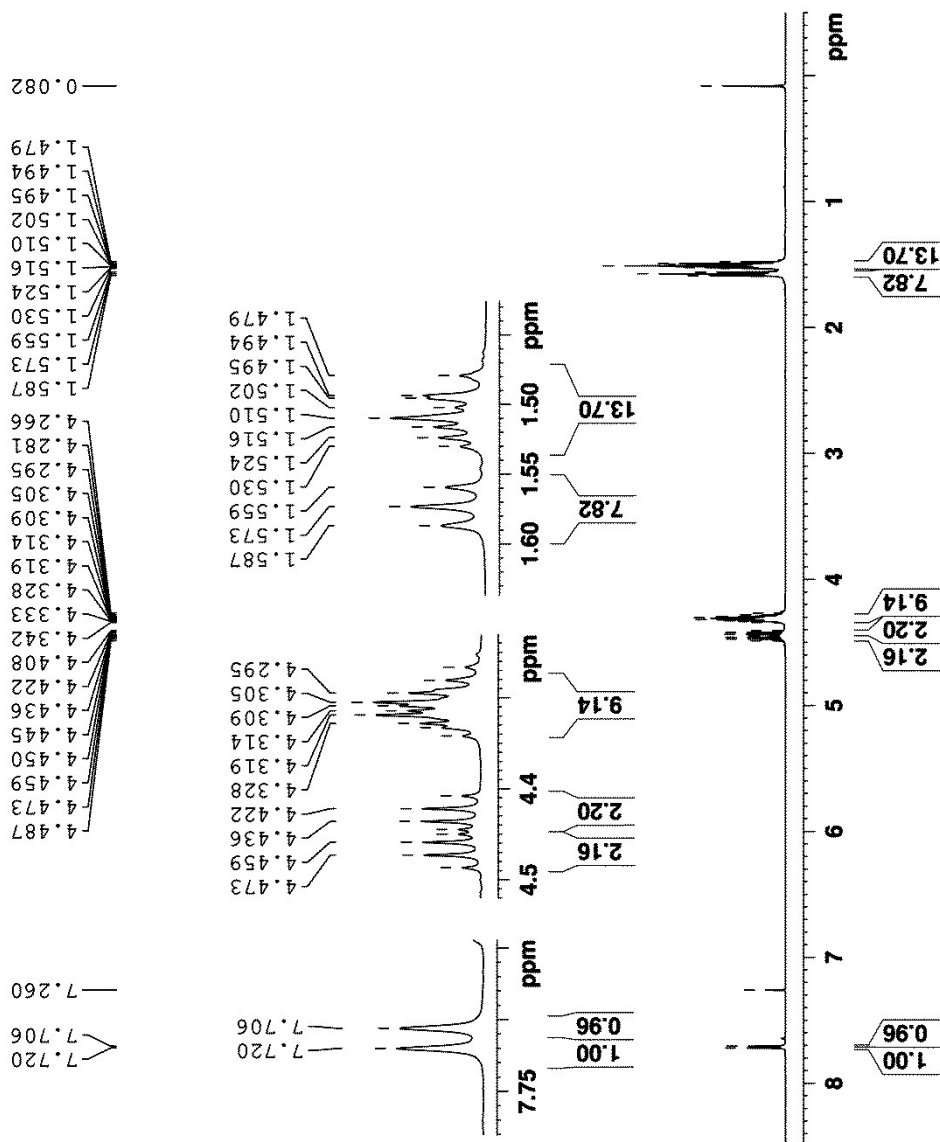
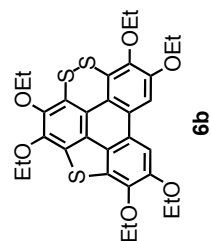
DEPT-135 of of 6a in CDCl₃

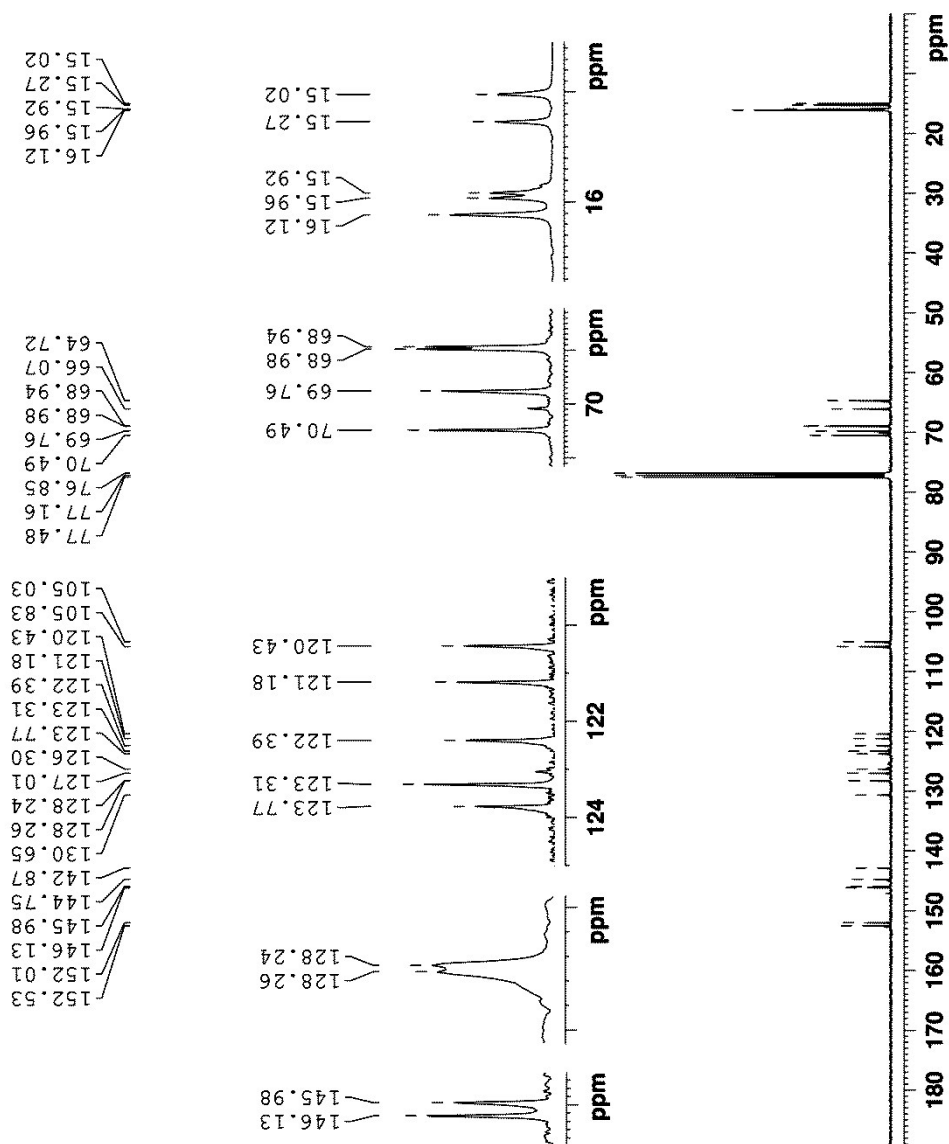
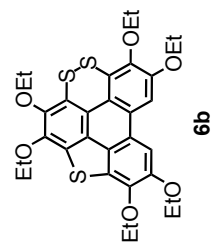


NAME AL2r040kh
 EXNO 15112708
 PROCNO 1
 Date_ 20151127
 Time 23.57
 INSTRUM spect
 PROBD 5 mm PABBO BB
 PULPROG dept135
 TD 65536
 SOLVENT CDCl₃
 NS 253
 DS 4
 SWH 29761.904 Hz
 FIDRES 0.454131 Hz
 AQ 1.1010548 sec
 RG 203
 DW 16.800 usec
 DE 6.50 usec
 TE 301.4 K
 CYS12 145.000000 sec
 D1 2.0000000 sec
 D2 0.0344828 sec
 D12 0.0002000 sec
 TD0 1
 ===== CHANNEL f1 =====
 NUC1 13C
 P1 10.00 usec
 F1 20.00 usec
 P2 1.00 dB
 PL1 93.6554230 MHz
 PLW 125.7703643 MHz
 SF01 125.7703643 MHz
 ===== CHANNEL f2 =====
 CPOPRG2 waltz16
 NUC2 1H
 P3 11.00 usec
 F3 22.00 usec
 P4 80.00 usec
 F4 1.75 dB
 PL2 18.63473365 MHz
 PLW 0.31284049 W
 SF02 500.1320005 MHz
 SI 32768
 SF 125.7577687 MHz
 WDW EM
 SSB 0
 GB 0
 PC 1.00 Hz



NAME	thendofline
EXPNO	220001
PRONO	1
Date_	20171012
Time	16:20
INSTRUM	spect
PROBHD	5 mm F4BBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SMNH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1719923 sec
RG	71.8
DE	48.400 usec
TE	6.50 usec
TD0	302.1 K
TD1	1.00000000 sec
TD0	1
NAME	CHANNEL f1
MUCL	1H
PR1	12.00 usec
PR2	2.60 dB
PR1P1W	15.322265563 W
SF01	500.130895 MHz
SI	32768
SF	500.1300132 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

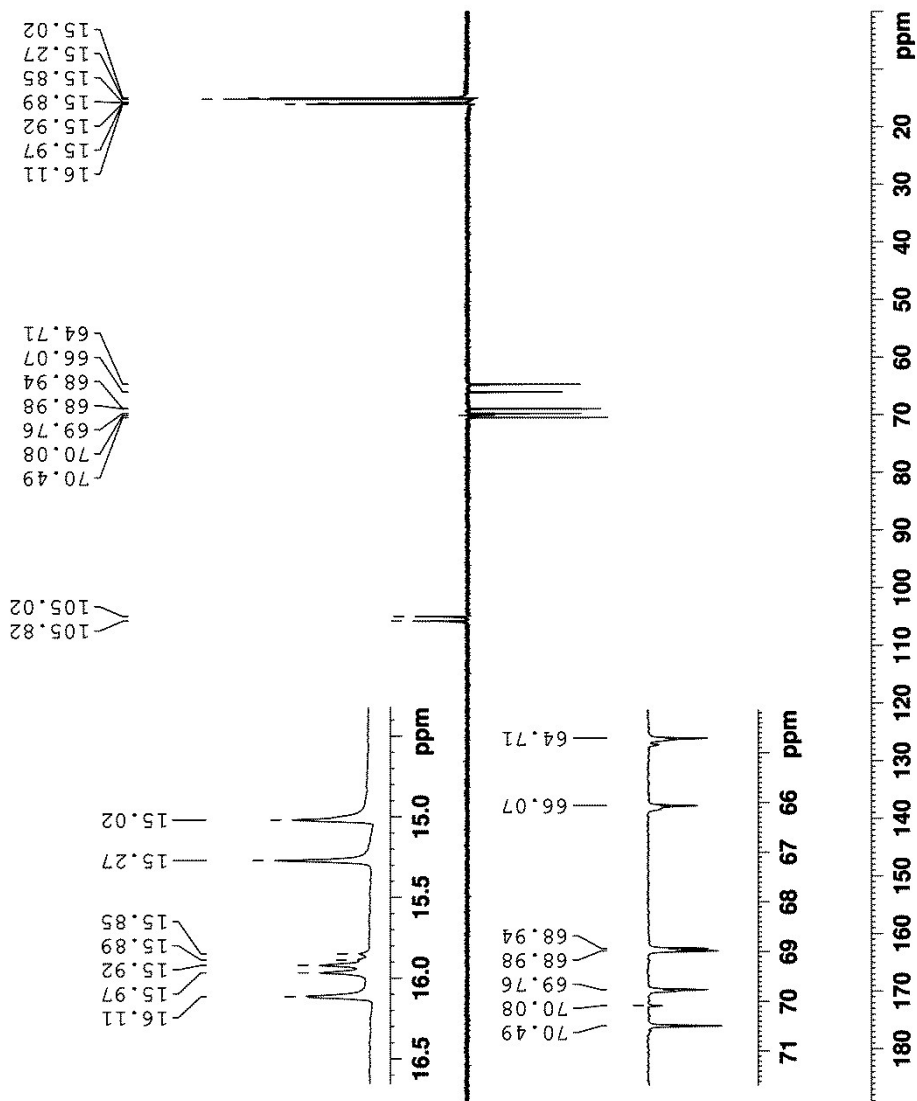
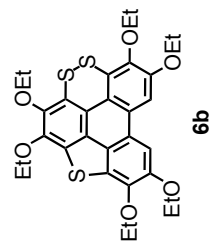




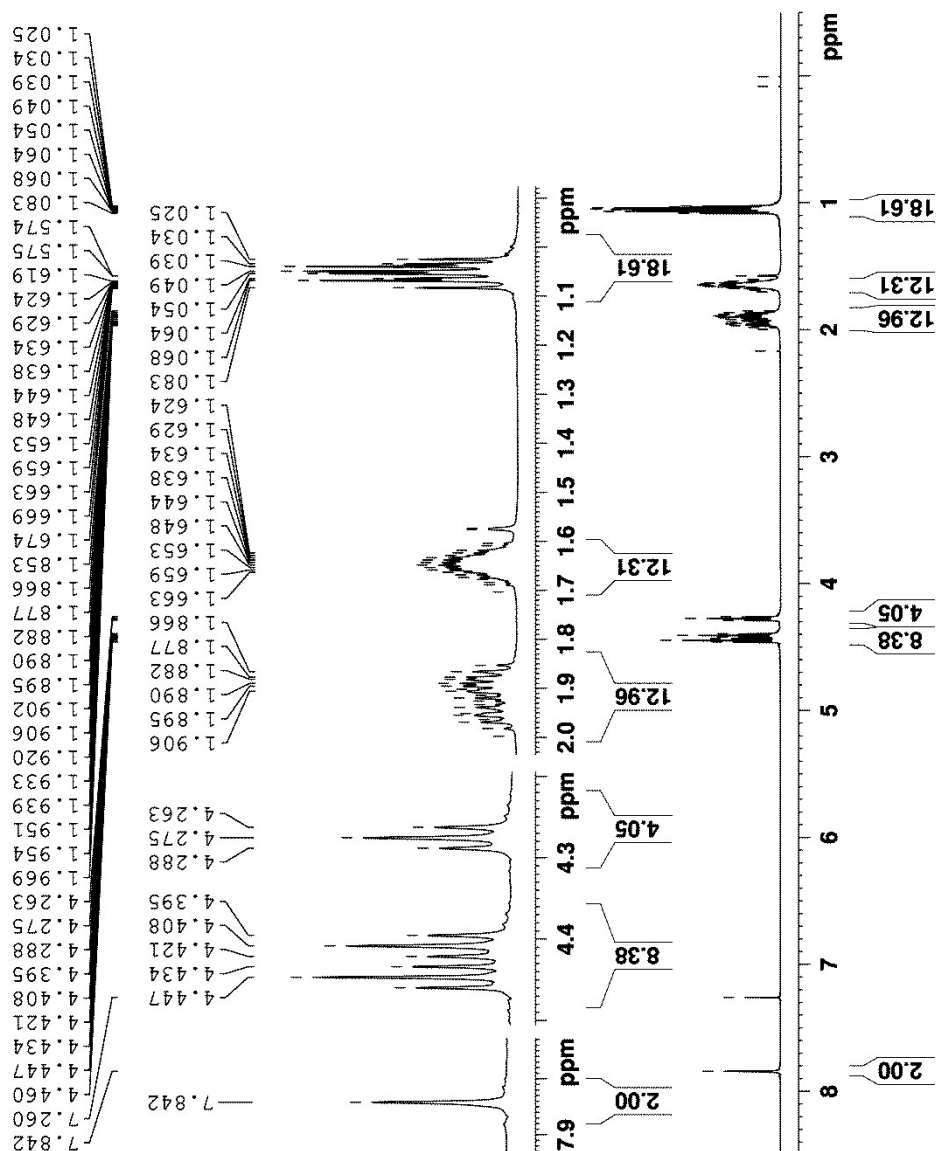
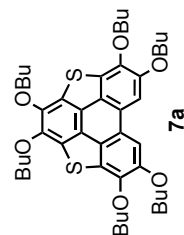
DEPT-135 of of 6b in CDCl₃



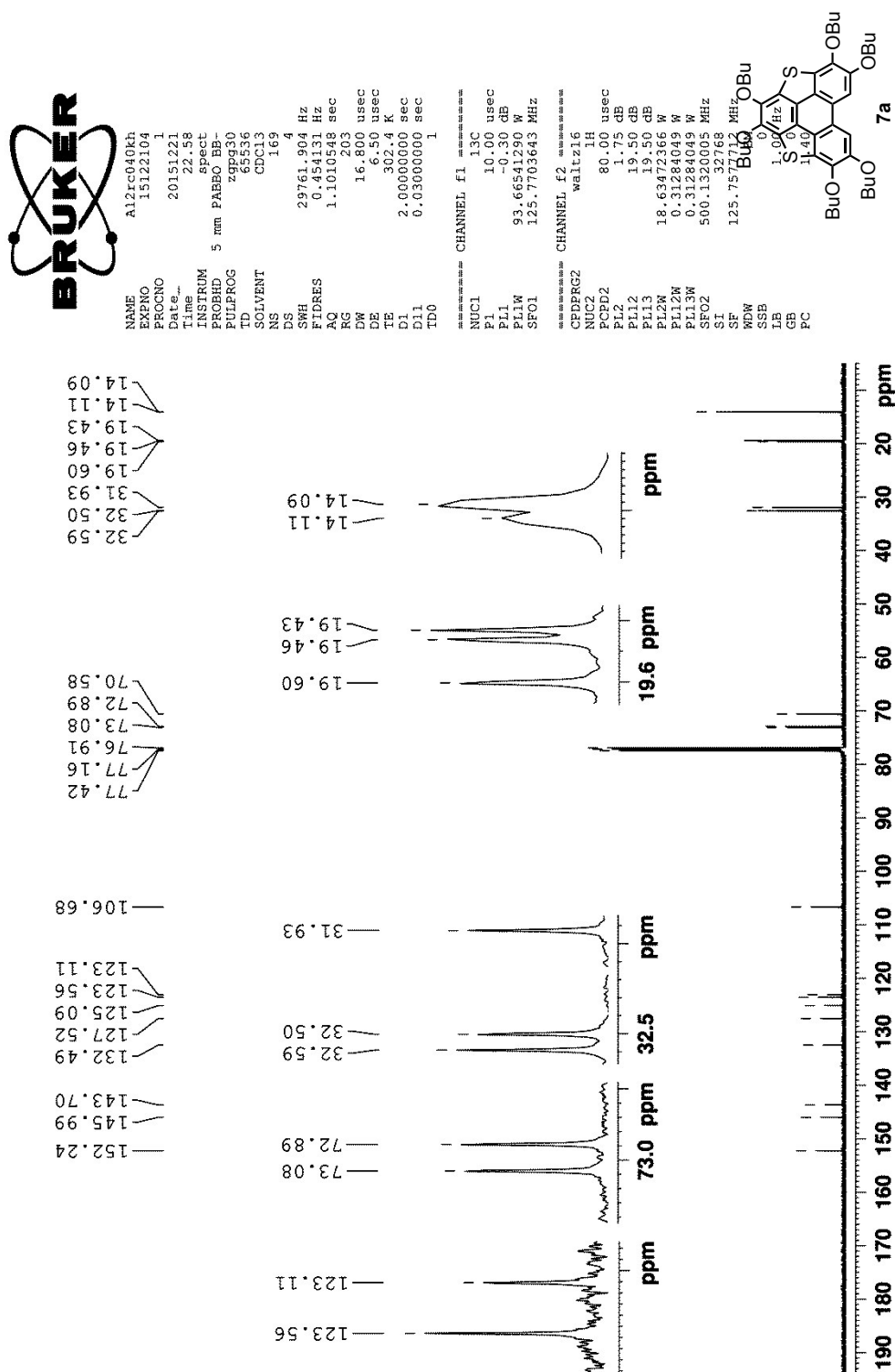
NAME thienodithine
EXPNO 71
PROCNO 1
Date_ 20171013
Time 12.12
INSTRUM spect
PROBHD 5 mm CPQNP 1H/
PULPROG dept135
TD 65536
SOLVENT CDCl3
NS 28
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 110.67
DW 16.800 usec
DE 18.00 usec
TE 300.0 K
CNS12 145.0000000 sec
D1 2.0000000 sec
D2 0.0034828 sec
D12 0.00002000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 12.00 usec
P2 24.00 usec
SI 32768
SF 100.6127570 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



NAME	VALUE	UNIT	CHANNEL
AL2rc040th			
191221i			
PRONO	20151221		
Date	20151221		
Time	22:46		
INSTRUM	spec		
PROBHD	5 mm F4BBO BB-		
PULPROG	zg30		
ID	65536		
SOLVENT	CDCl3		
NS	2		
DS	2		
SWH	10330.578	Hz	
FIDRES	0.157632	Hz	
RG	3.1719923	sec	
RG	71.8		
DC	48.400	usec	
DE	6.50	usec	
TE	301.4	K	
DI	1.000000000	sec	
ID0	1		
NUC1	1H		
P1	12.00	usec	
P13	2.60	dB	
P1P1W	15.32226563	W	
SFO1	500.1330885	MHz	
S1	327.68		
SF0	500.1300138	MHz	
SW	END		
GB	0.30	Hz	
GB	0		
PC	1.00		



¹³C NMR spectrum of 7a in CDCl₃



DEPT-135 of of 7a in CDCl₃

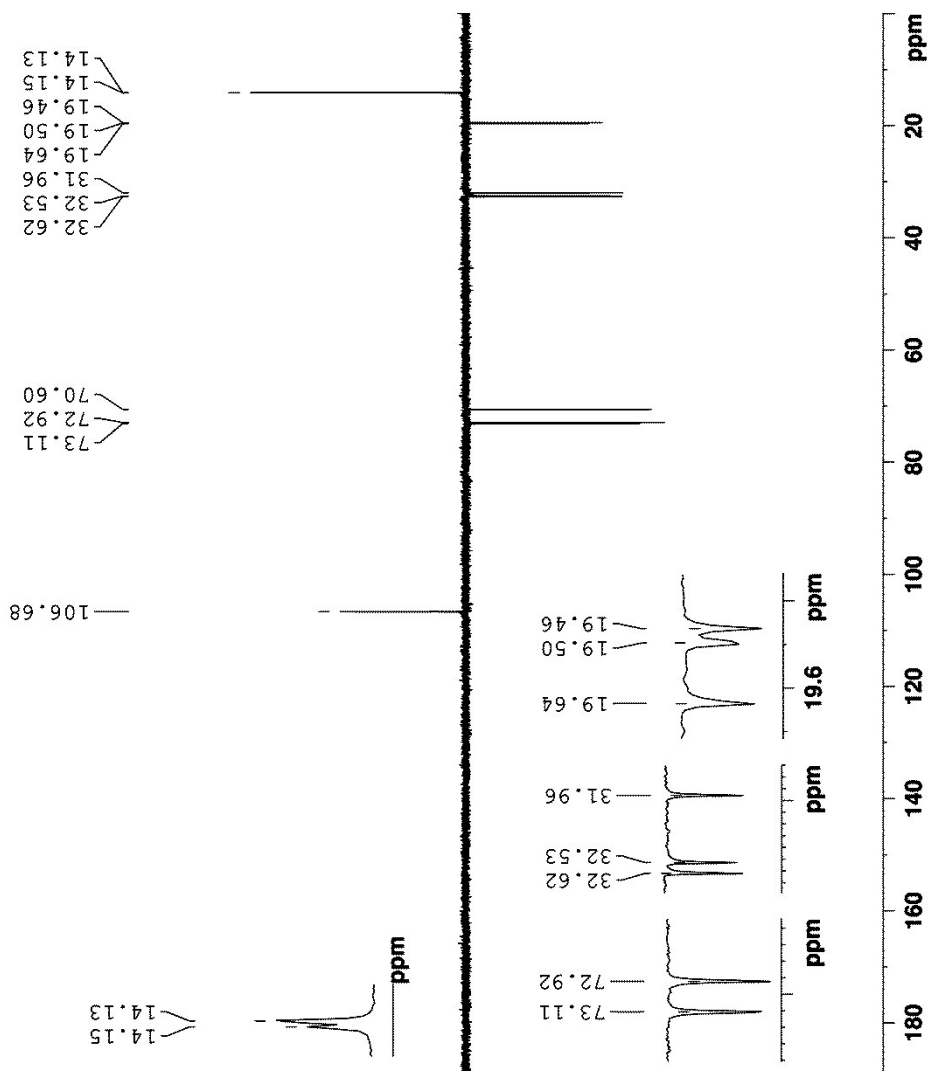
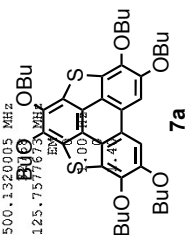


NAME Al2rc040kh
 EXPNO 15122105
 PROCNO 1
 Date_ 20151221
 Time 23:07
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG dept135
 ID 6535
 SOLVENT CDCl₃
 NS 88
 DS 4
 SWH 29761.904 Hz
 FIDRES 0.454131 Hz
 AQ 1.1010548 sec
 RG 203
 DW 16.800 usec
 DE 6.50 usec
 TE 301.6 K
 CNST2 145.0000000
 D1 2.00000000 sec
 D2 0.0034828 sec
 D12 0.0000200 sec
 TD0 1

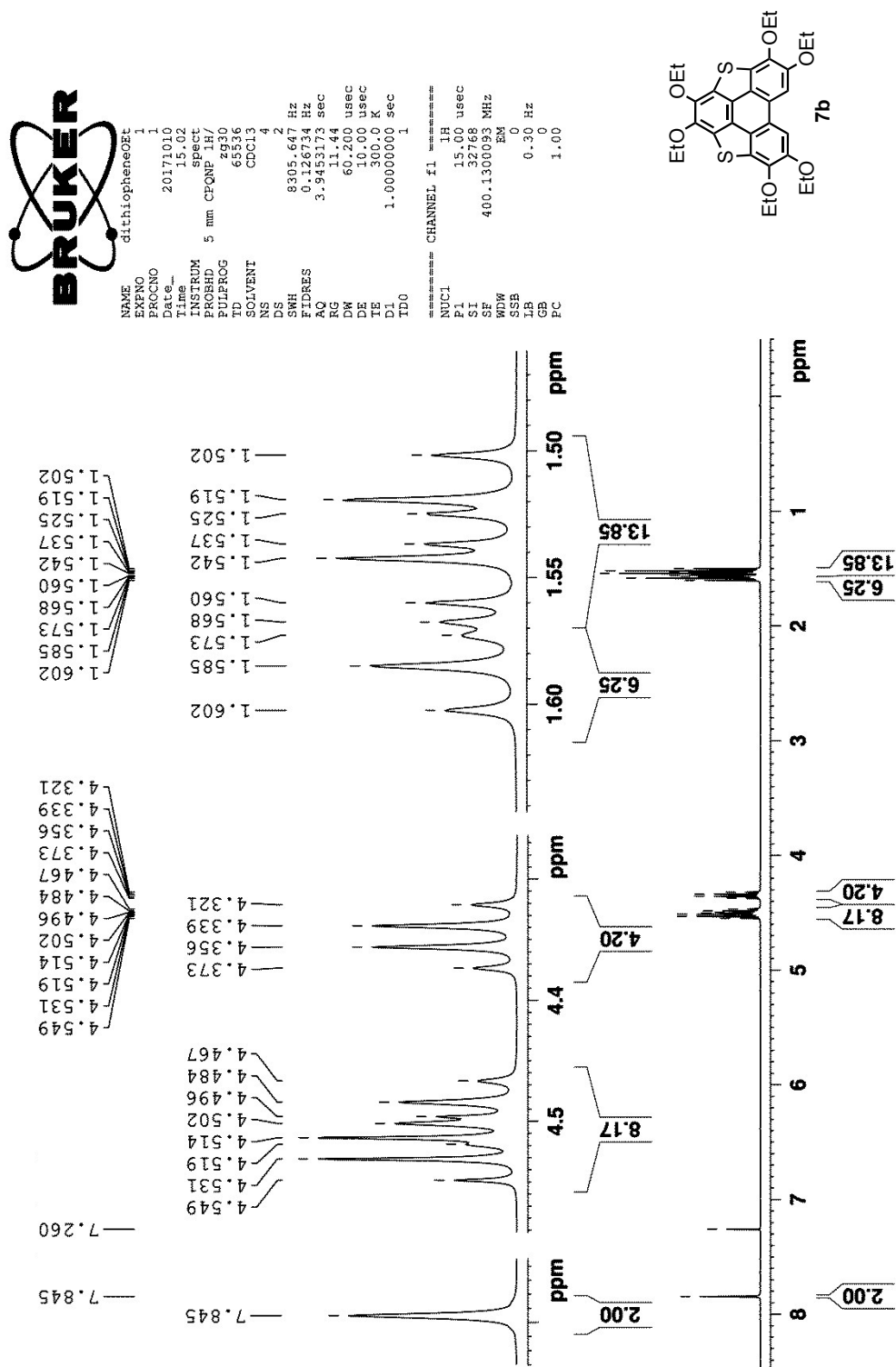
CHANNEL f1 13C
 NUC1 13C
 P1 10.00 usec
 P2 20.00 usec
 PL1 -0.30 dB
 PL1W 93.66541290 W
 SF01 125.7703643 MHz

CHANNEL f2 waitz16
 CPDPRG2 waitz16
 NUC2 1H
 F3 11.00 usec
 F4 22.00 usec
 FCPD2 80.00 usec
 PL2 1.75 dB
 PL12 19.50 dB
 PL1W 18.6341256 W
 PL12W 18.374256 W
 SF02 500.132005 MHz
 SI 125.757783 MHz

WDM
 SSB
 LB
 GB
 PC



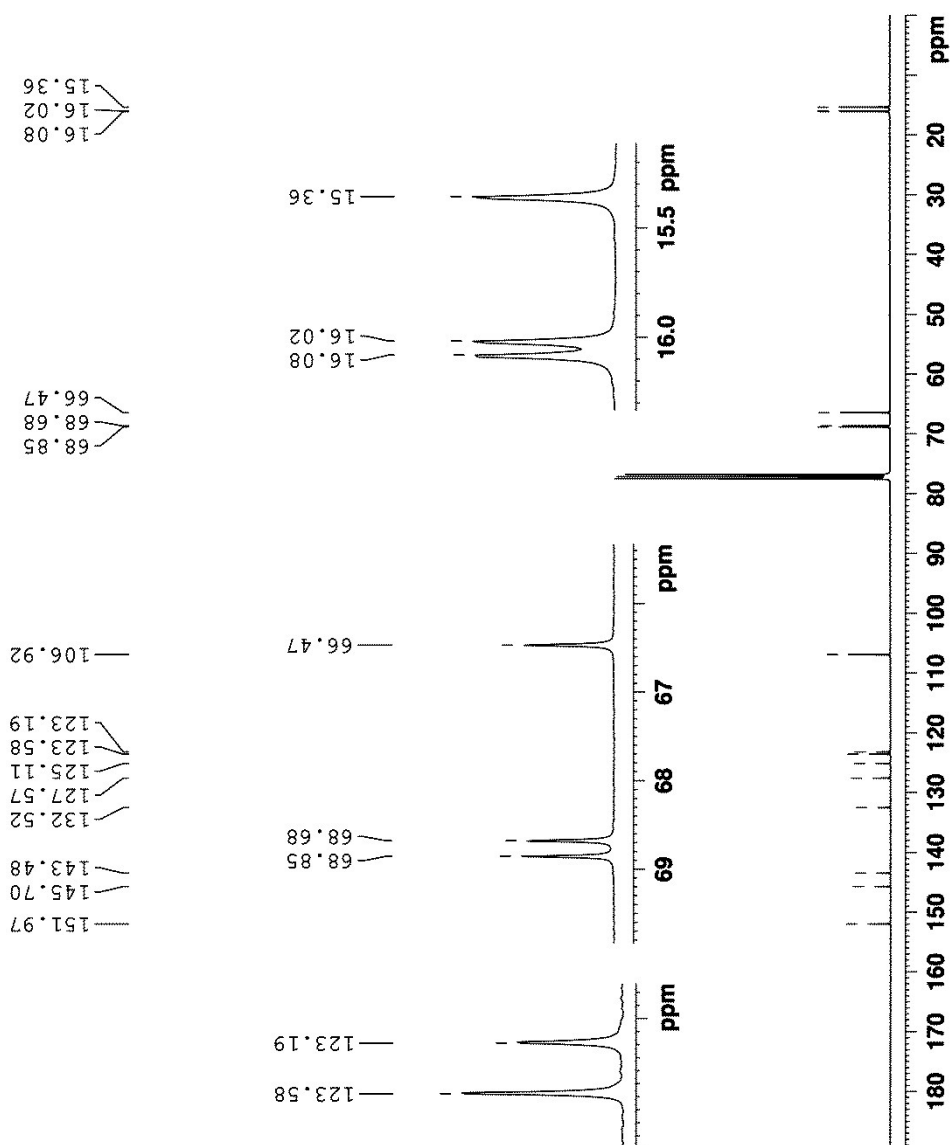
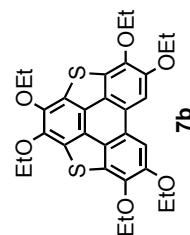
¹H NMR spectrum of 7b in CDCl₃



¹³C NMR spectrum of 7b in CDCl₃



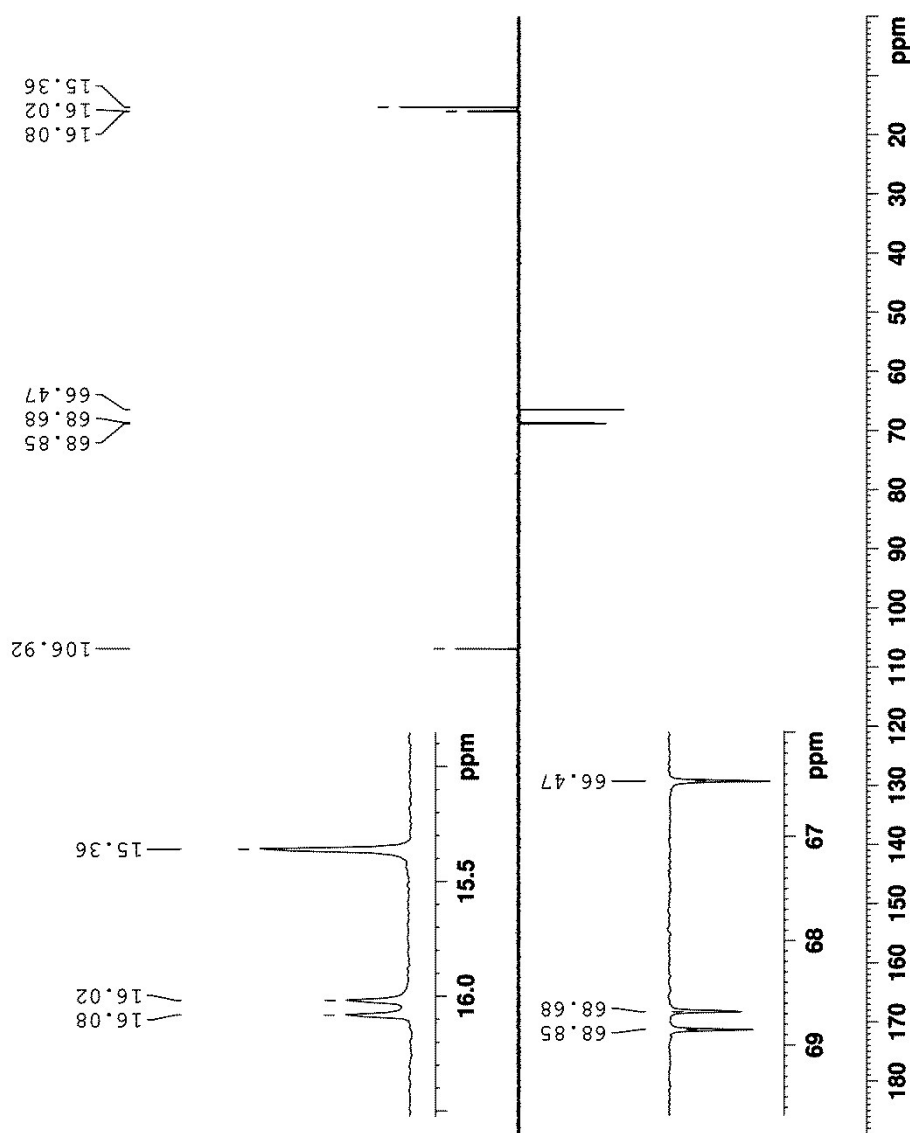
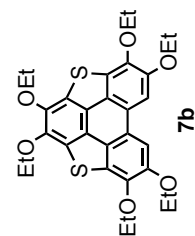
NAME 16mc125kh
 EXPNO 13
 PROCNO 1
 Date_ 20171010
 Time 15.08
 INSTRUM spect
 PROBHD 5 mm CPQNP 1H/
 PULPROG zgpg30
 ID 65536
 SOLVENT CDCl₃
 NS 1024
 DS 2
 SWH 29761.904 Hz
 FIDRES 0.454131 Hz
 AQ 1.1010948 sec
 RG 126.99
 DW 16.800 usec
 DE 18.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 D10 1
 ===== CHANNEL f1 =====
 NUC1 13C
 P1 12.00 usec
 PL 32768
 SF 100.6127546 MHz
 ZGPG 64
 SSB 0
 AB 2.00 Hz
 GB 1.40
 FC



DEPT-135 of of 7b in CDCl₃



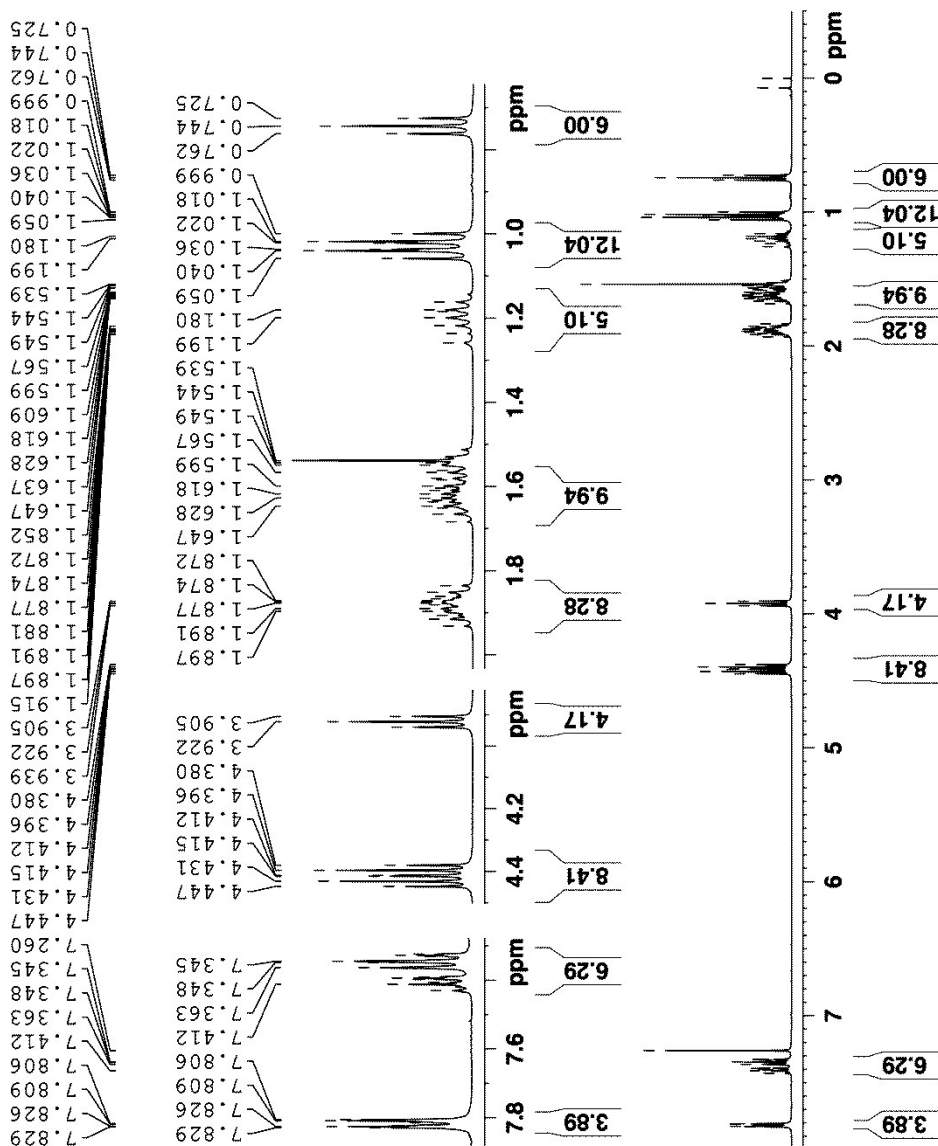
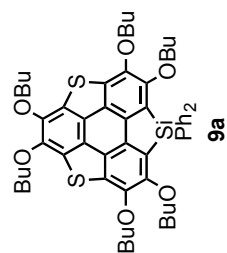
NAME 16mcl25kh
EXPNO 135
PROCNO 1
Date_ 20171010
Time 15.04
Spect
INSTRUM 5 mm CPQNP 1H/
PROBHD dept-135
PULPROG zgpg30
TD 65536
F2 65536
SOLVENT CDCl₃
NS 64
DS 4
SMH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 110.67
DW 16.800 usec
DE 18.00 usec
TE 300.0 K
CNS12 145.0000000
D1 2.00000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 12.00 usec
P2 24.00 usec
SI 32768
SF 100.6127547 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



¹H NMR spectrum of 9a in CDCl₃



Current Data Parameters
 NAME A16mc125kh
 EXPNO 171100404
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20171004
 Time 20.19
 INSTRUM spect
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 2
 SWH 8305.647 Hz
 FIDRES 0.126734 Hz
 AQ 3.9453173 sec
 RG 60.721 usec
 DE 10.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1
 ===== CHANNEL f1 =====
 NUC1 1H
 P1 15.00 usec
 PL1 5.1993981 W
 SF01 400.1324708 MHz
 F2 - Processing parameters
 SI 32768
 SF 400.1300093 MHz
 WDW EM
 SSB 0
 LB 0
 GB 0
 PC 1.00



¹³C NMR spectrum of 9a in CDCl₃



Current Data Parameters
NAME Al6mc125kh
EXPNO 17100405
PROCNO 1

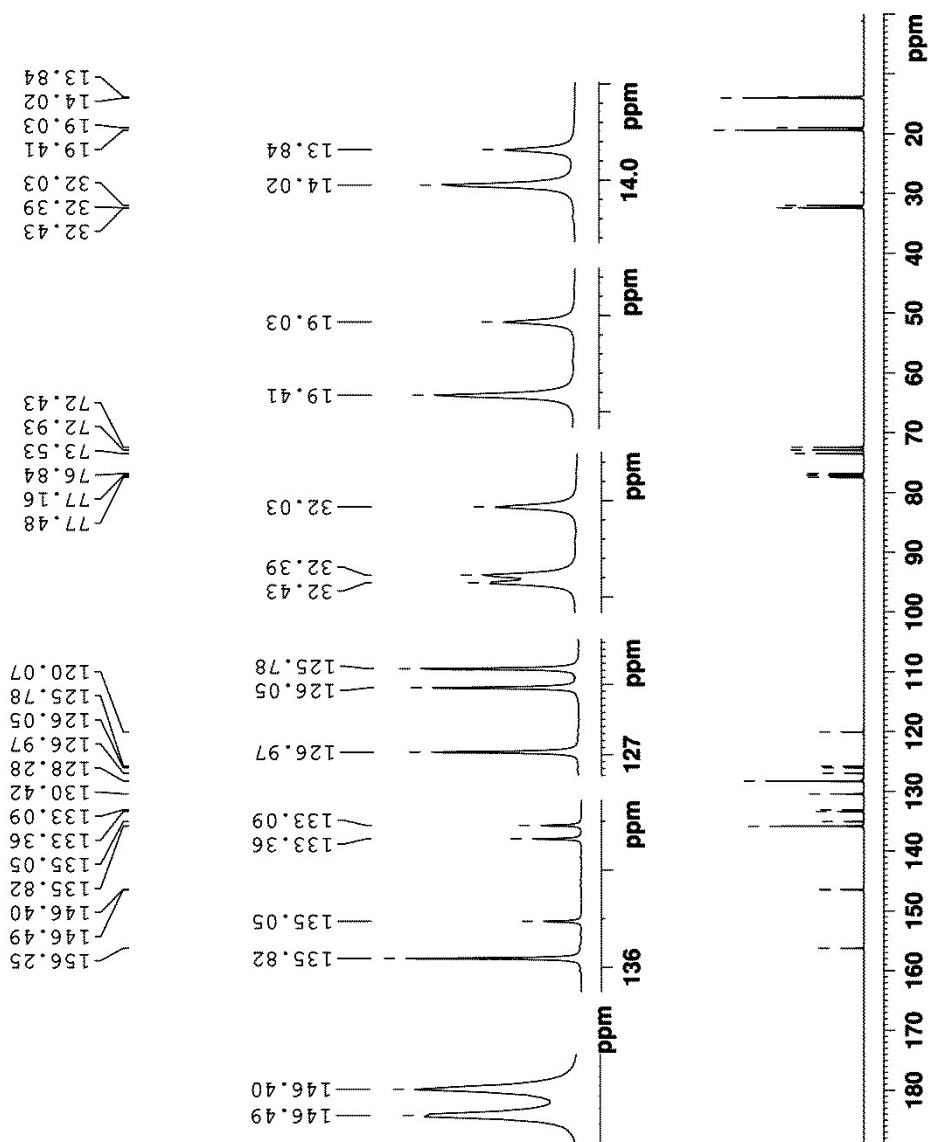
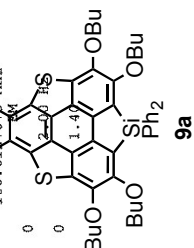
F2 - Acquisition Parameters
Date_ 20171004
Time 21:00

INSTRUM spect
PROBHD 5 mm CPONE-HY
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 431
DS 2
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 126.99
DM 16.800 usec
DE 18.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

CHANNEL f1 13C
NUC1 13C
P1 12.00 usec
PLW1 15.50000000 W
SFO1 100.6248425 MHz

CHANNEL f2 waitz16
CPDPRG2 1H
NUC2 1H
PCPD2 90.00 usec
PLW2 5.19999981 W
PLM2 0.1444000 W
PLM3 0.11700000 W
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 125.761 MHz
WDW EM
SSB 0
LB 0
GB 0
PC



DEPT-135 of of 9a in CDCl₃



Current Data Parameters
 Name: 17100601
 EXPNO: 1
 PROCNO: 1

F2 - Acquisition Parameters
 Date_: 20171006
 Time: 16.13

INSTRUM: spect
 PROBHD: 5 mm CPQNP 1H/
 PULPROG: zgpg30
 TD: 65536
 SOLVENT: CDCl₃

NS: 130
 DS: 4

SWH: 29761.904 Hz
 FIDRES: 0.454131 Hz
 AQ: 1.1010548 sec

RG: 110.67
 DW: 16.800 usec
 DE: 15.000 usec
 TE: 300.2 K

CNST2: 145.0000000 sec
 D1: 2.00000000 sec
 D2: 0.00344828 sec

D12: 0.00002000 sec
 TD0: 1

CHANNEL f1
 NUC1: 13C
 P1: 12.00 usec
 F1: 24.00 usec

PL1: 15.50000000 W
 SFO1: 100.6248425 MHz

CHANNEL f2
 NUC2: 1H
 P2: 15.00 usec
 F2: 30.00 usec

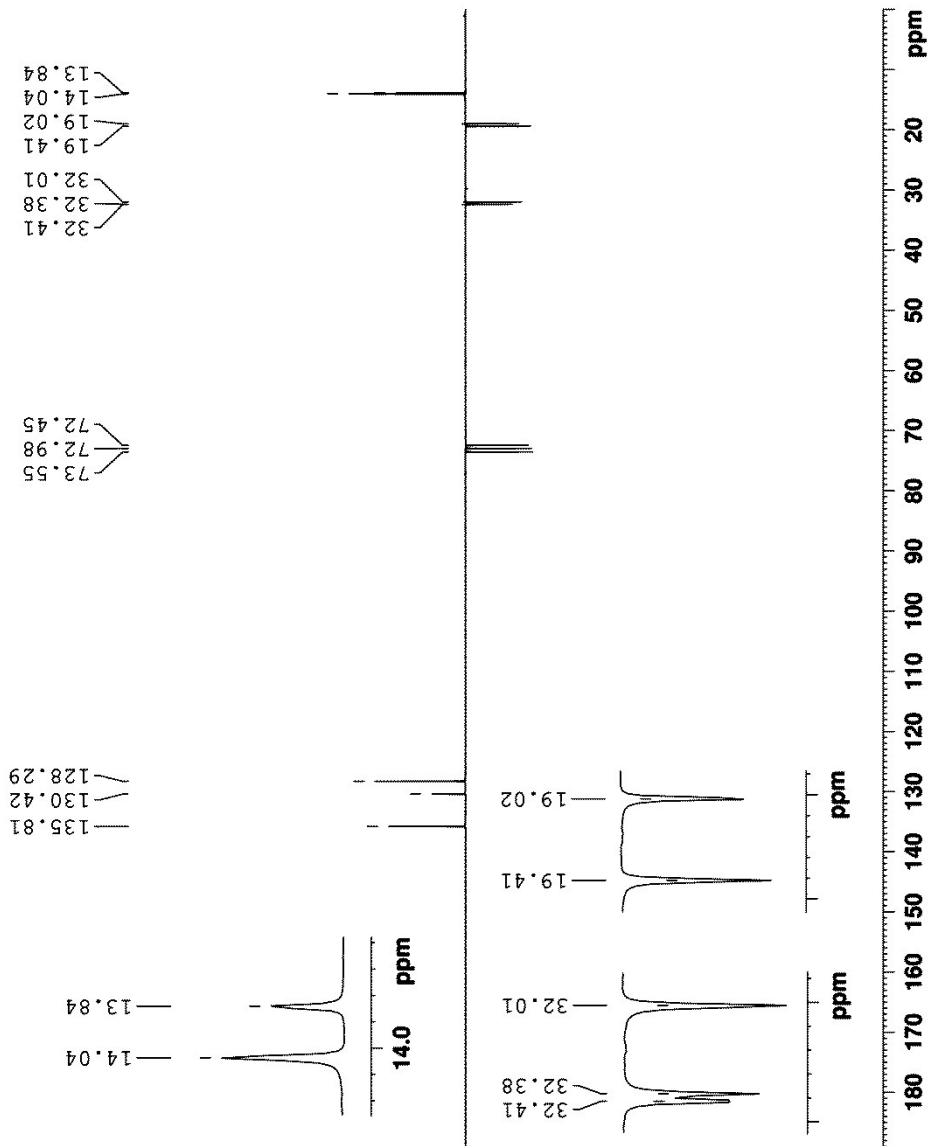
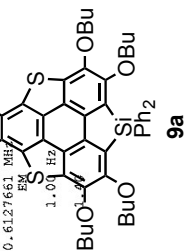
PL2: 5.19999981 W
 SFO2: 400.1316005 MHz

CEPDEG2: wallz16
 NUC2: 1H
 P3: 15.00 usec
 F3: 30.00 usec

PL3: 5.19999981 W
 SFO3: 400.1316005 MHz

PLM12: 0.14444000 W
 SFO2: 400.1316005 MHz

F2 - Processing parameters
 SI: 32768
 SF: 100.6127661 MHz
 WDM: 0
 SSF: 0
 GB: 0
 PC: 0

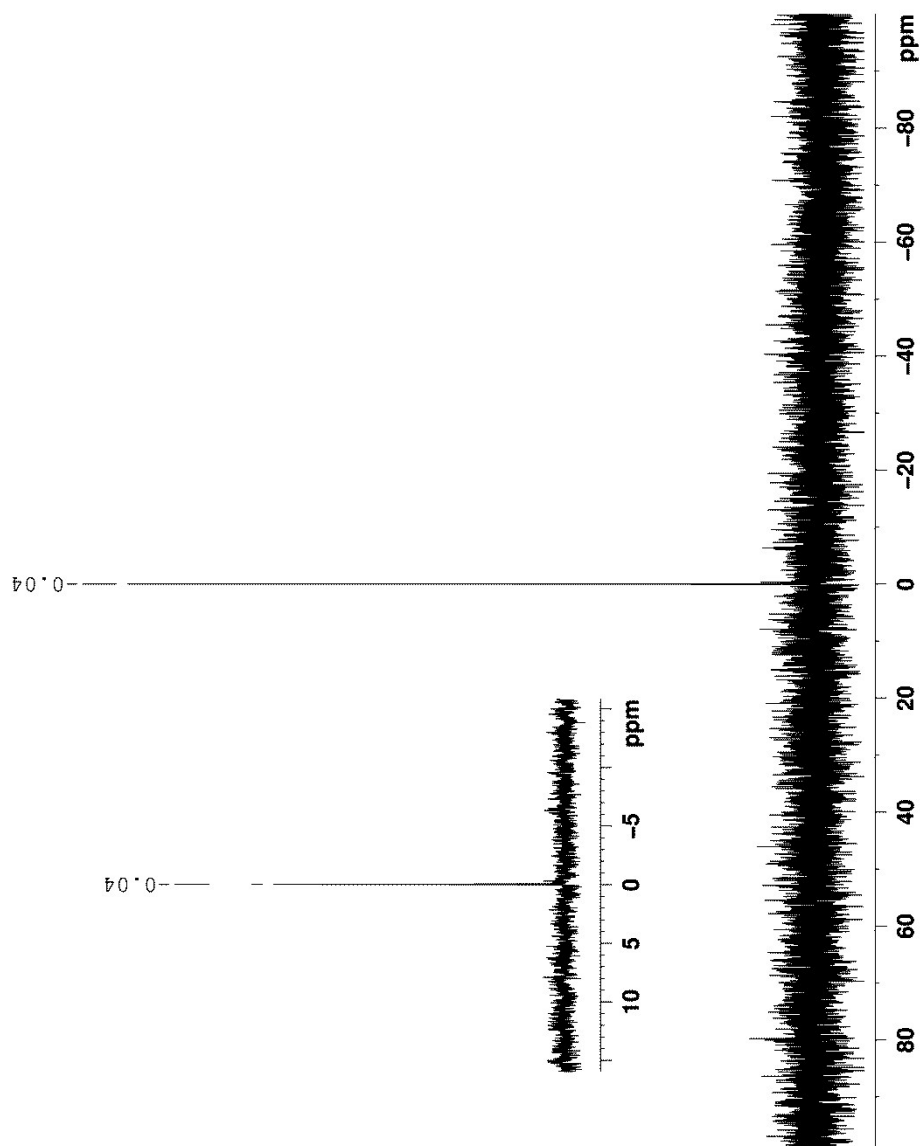
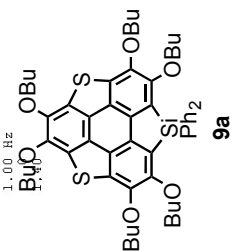


²⁹Si NMR spectrum of 9a in CDCl₃



```

NAME      AL7mcl26cm
EXPNO     2017100429
PROCNO    1
Date_     20171004
Time      21.03
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgig
TD         65536
SOLVENT   CDCl3
NS         20
DS         4
SWH        39682.539 Hz
FIDRES     0.60507 Hz
AQ         0.8258036 sec
RG         203
DW         12.600 usec
DE         6.50 usec
TE         300.3 K
D1         5.00000000 sec
D11        0.03000000 sec
TD0        1
===== CHANNEL f1 =====
NUC1       29Si
P1         15.00 usec
PL1        3.00 dB
PL1W       37.76776123 W
SF01       99.3319610 MHz
===== CHANNEL f2 =====
CFDPRG2    waltz16
NUC2        1H
PCPD2       80.00 usec
PL2         2.40 dB
PL2W       15.17711735 W
PL12W      0.33051354 W
SF02       500.0320001 MHz
SI         32768
SF         99.3418950 MHz
WDW         EM
SSB         0
LB          0
GB          0
PC          0
  
```

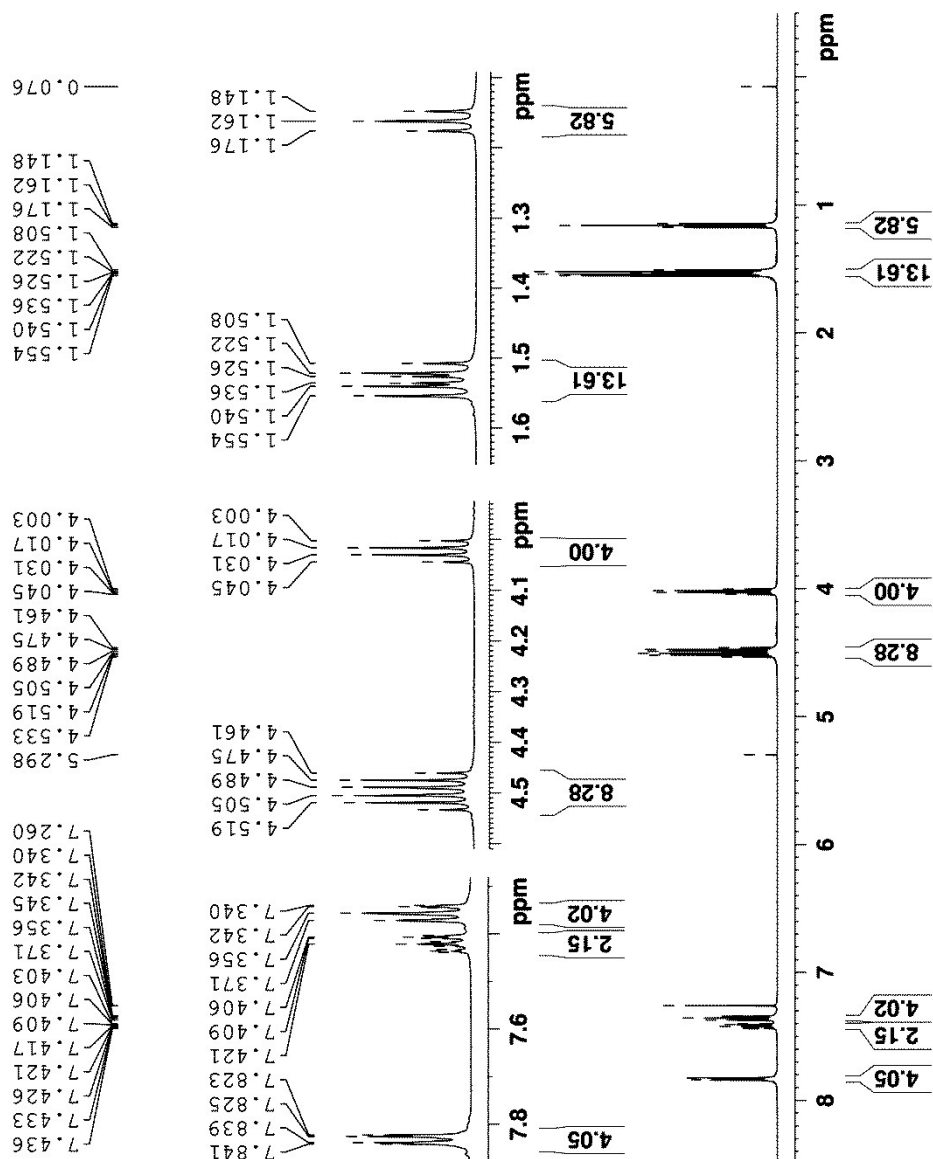
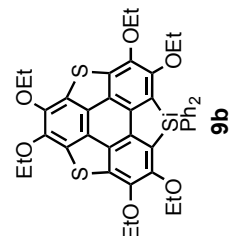


¹H NMR spectrum of 9b in CDCl₃

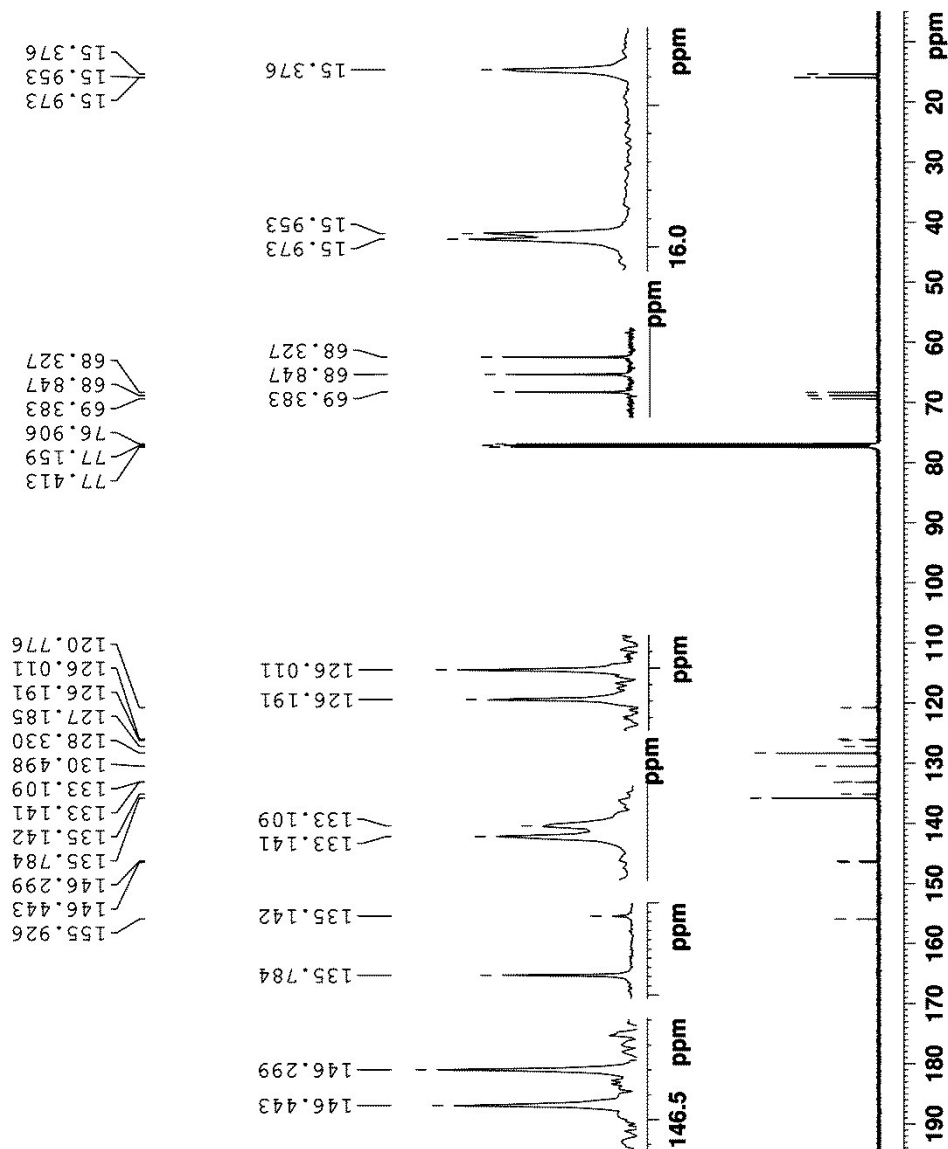
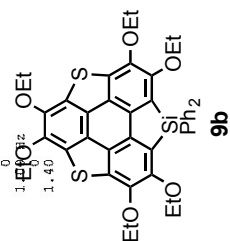


NAME Sisumanene
EXPNO 111
PROCNO 1
Date_ 20171005
Time 23.05
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
FIDRES 0.157632 Hz
RG 3.1719923 sec
AQ 144
RG 144
DW 48.400 usec
DE 6.50 usec
TE 301.5 K
D1 1.00000000 sec
D11 1
TD0 1

CHANNEL f1
NUC1 1H
P1 12.00 usec
PL1 0 dB
PL12 15.32265653 dB
SFO1 500.1300885 MHz
SI 32768
SF 500.1300136 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



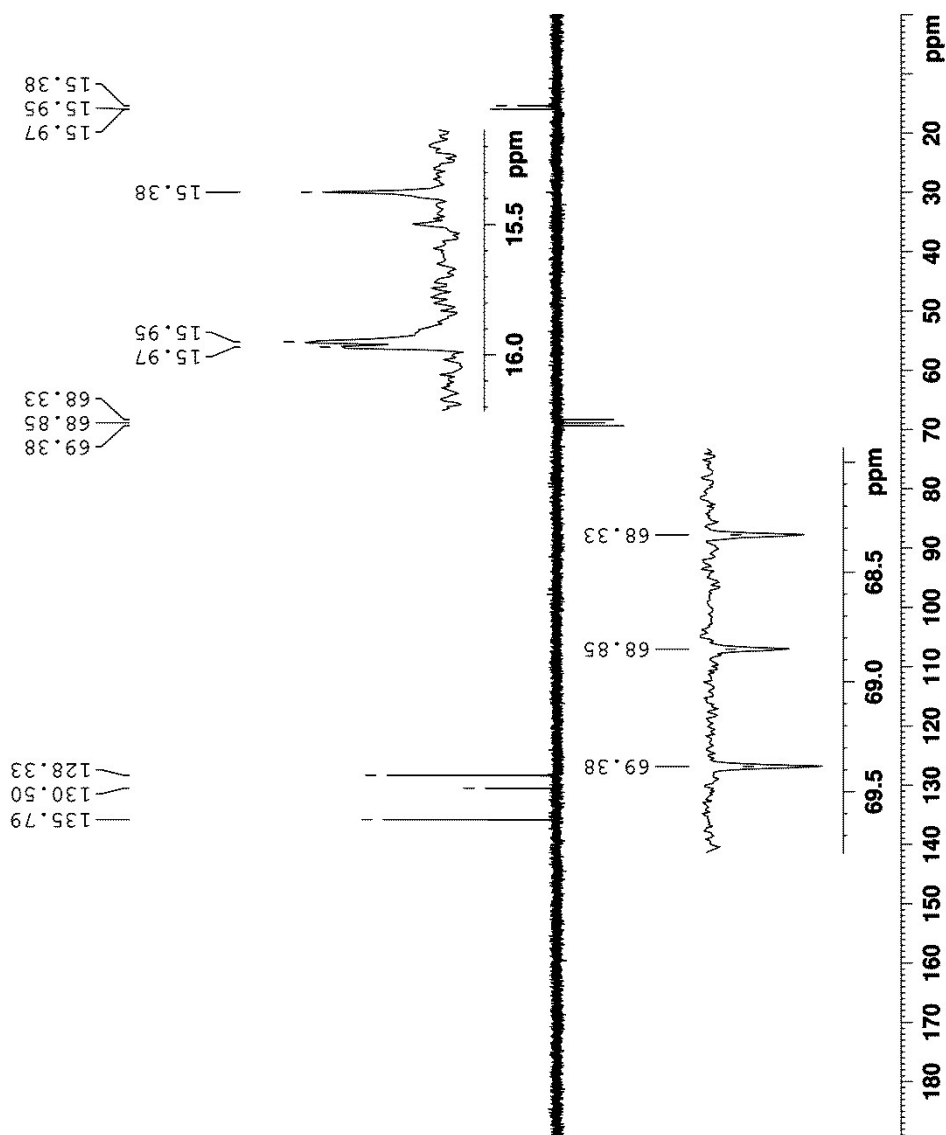
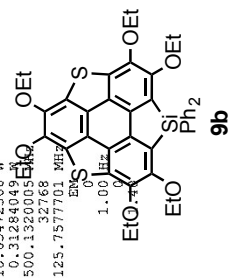
NAME	UNIT	VALUE	UNIT	NAME	UNIT	VALUE	UNIT
CHANNEL #1	13C			CHANNEL #2	16		
NUC1				CPDPK2			
PI1	10.00 useC			NUC2	1H	80.00 useC	
PP1	-0.30 dB			PCPD2		1.75 dB	
PIW1	93.66541290 W			PI12	19.50 dB		
SF01	125.7757703 MHz			PI13	19.50 dB		
				PI14	18.6341200 W		
				PI15	0.3724009 W		
				PI16	0.31284039 W		
				PI17	0.31284039 W		
				PI18	500.1320005 MHz		
				SI	32768		
				SI	125.7577703 MHz		
				WDW	EM		
				SSB	0		
				LB	100 Hz		
				PC	1.40		



DEPT-135 of of 9b in CDCl₃



NAME 16mc125kh
EXPNO 135
PROCNO 1
Date_ 20171009
Time 20.38
INSTRUM spect
PROBHD 5 mm F4BBO BB-
PULPROG dept135
TD 65536
F2 32768
SOLVENT CDCl₃
RG 41
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 304.6 K
CNS2 145.0000000
D1 2.00000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
TD0 1
CHANNEL f1 13C
NUC1 13C
P1 10.00 usec
P2 20.00 usec
PL1 -0.30 dB
PL1W 93.66541290 W
SFO1 125.7703643 MHz
CHANNEL f2 1H
waltz16
CFPRG2 1H
NUC2 1H
P3 11.00 usec
P4 22.00 usec
PCPD2 80.00 usec
P12 1.75 dB
P12W 15.50 dB
F12W 18.6342366 W
F12W 50.3182000 W
SFO2 500.1320045 MHz
SF 32768
SF 125.7577701 MHz
EMW 1.00 Hz
SSB 0
GB 0
PC

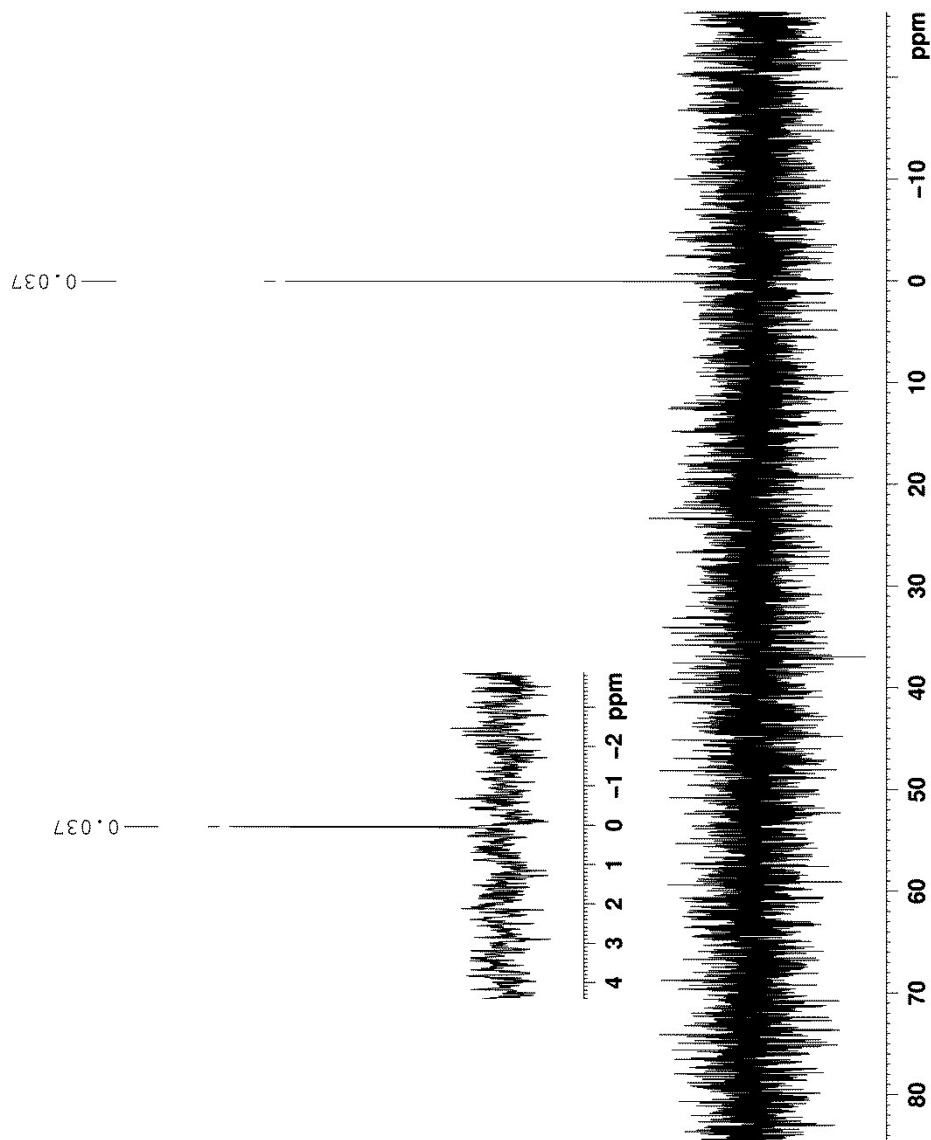
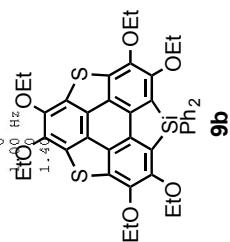


²⁹Si NMR spectrum of 9b in CDCl₃



```

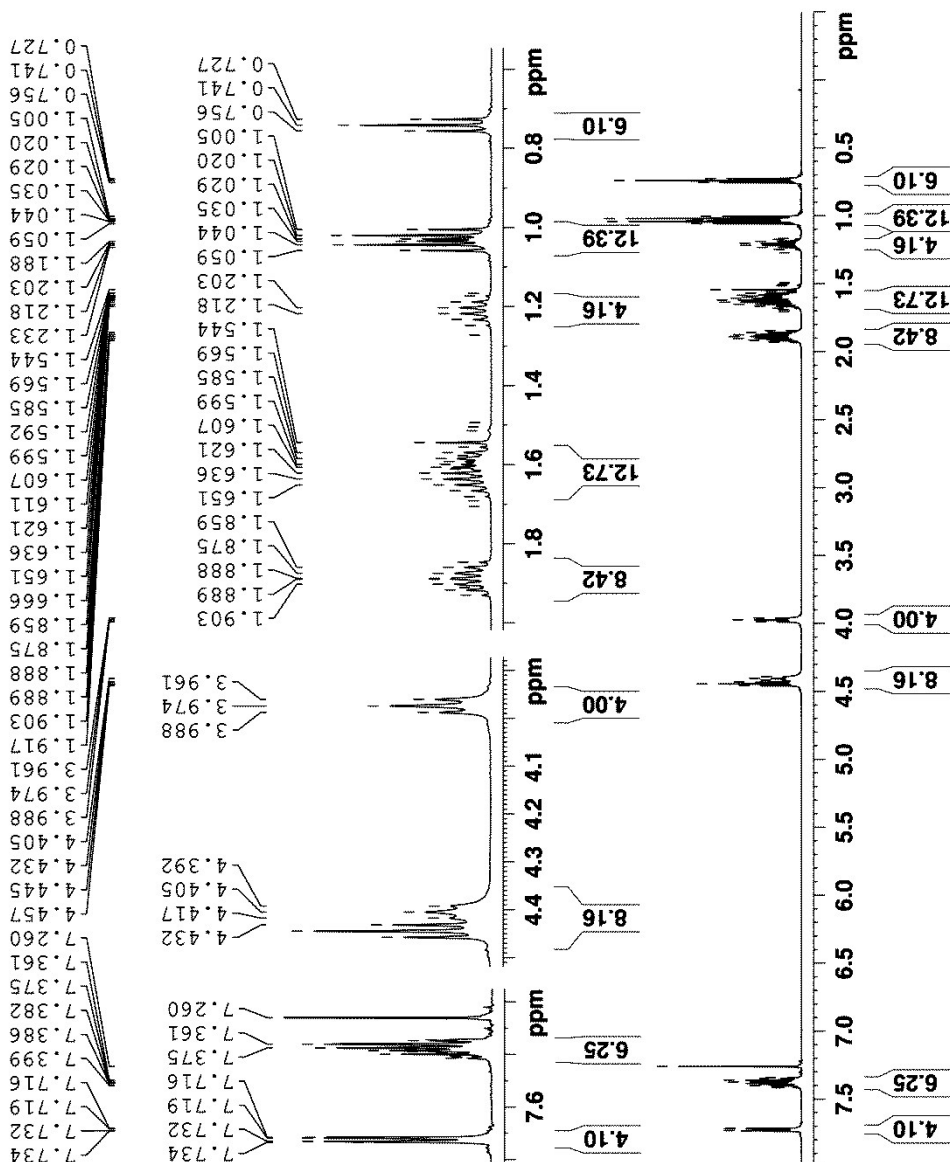
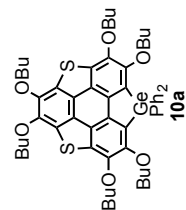
NAME          Sisurancene
EXPNO         29
PROCNO        1
Date_         20171005
Time_         22.39
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            1024
DS            0
SWH           39682.539 Hz
FIDRES        0.605507 Hz
AQ            0.8258036 sec
RG            203
DW            12.600 usec
DE            6.50 usec
TE            301.6 K
D1            10.0000000 sec
D11           0.0500000 sec
TD0           1
===== CHANNEL f1 =====
NUC1          29Si
P1            10.00 usec
PL1           3.00 dB
SFO1          99.3518262 MHz
===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           1.75 dB
PL12          19.50 dB
PL12W         18.63472366 W
PL12W         0.31284949 W
SFO2          500.1320005 MHz
SI            32768
SF            99.3617620 MHz
EM            0
WDW            EM
SSB            0
GB            0
PC            0
  
```



¹H NMR spectrum of 10a in CDCl₃



NAME A12rc040kh
EXPNO 16010705
PROCNO 1
Date_ 20160107
Time 20.12
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl₃
NS 8
DS 4
SWH 10330.576 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 128
DW 48.400 usec
DE 6.50 usec
TE 301.3 K
D1 1.00000000 sec
D10 1
CHANNEL f1
NUC1 1H
P1 12.00 usec
PL1 2.60 dB
PL1W 15.3222563 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300138 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



¹³C NMR spectrum of 10a in CDCl₃

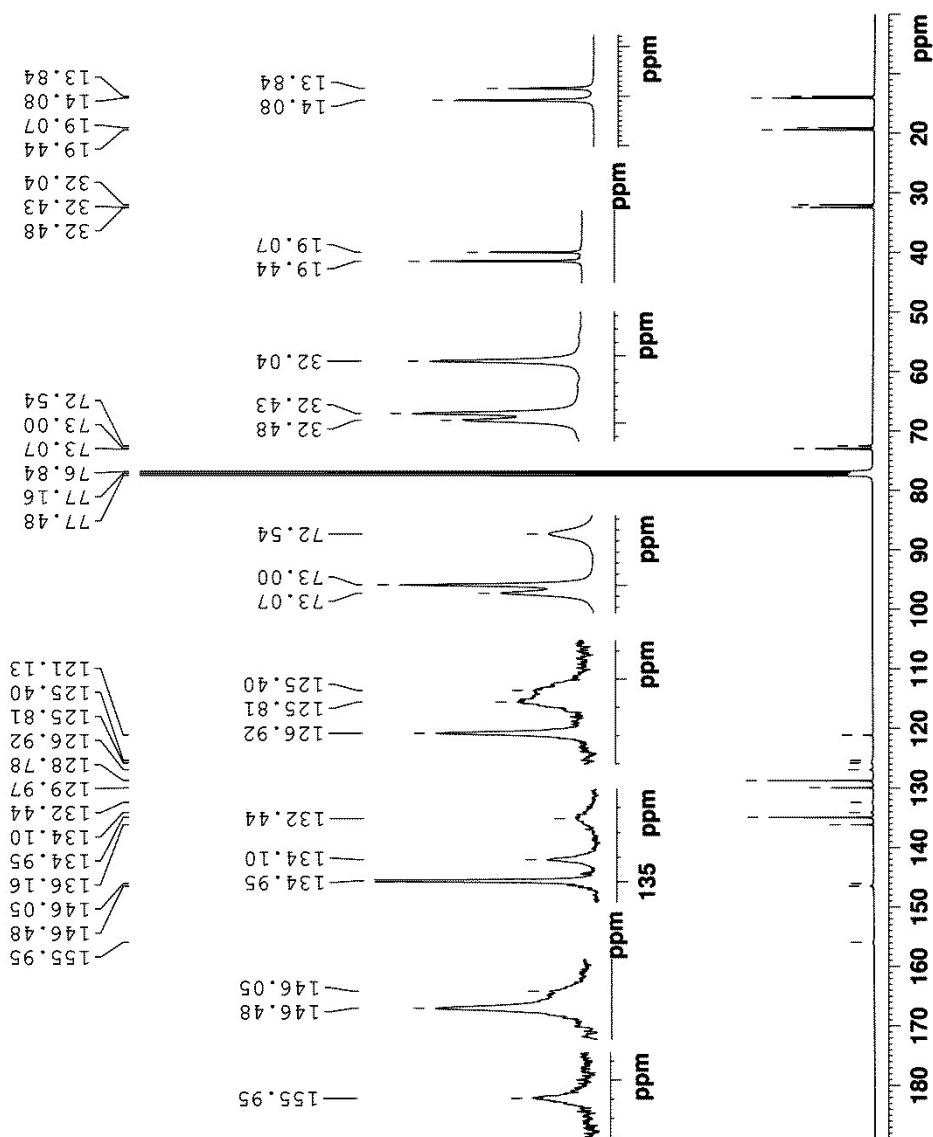
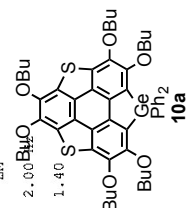


Current Data Parameters
NAME Al3mc15kh
EXPNO 999013002
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160112
Time_ 20.13
INSTRUM spect
PROBHD 5 mm CPQNP 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 14399
DS 2
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 126.99
DW 16.800 usec
DE 18.00 usec
TE 300.0 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PLM1 15.50000000 W
SF01 100.6248425 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLM2 5.1999981 W
PLM12 0.1444000 W
PLM13 0.1170000 W
SF02 400.1316005 MHz

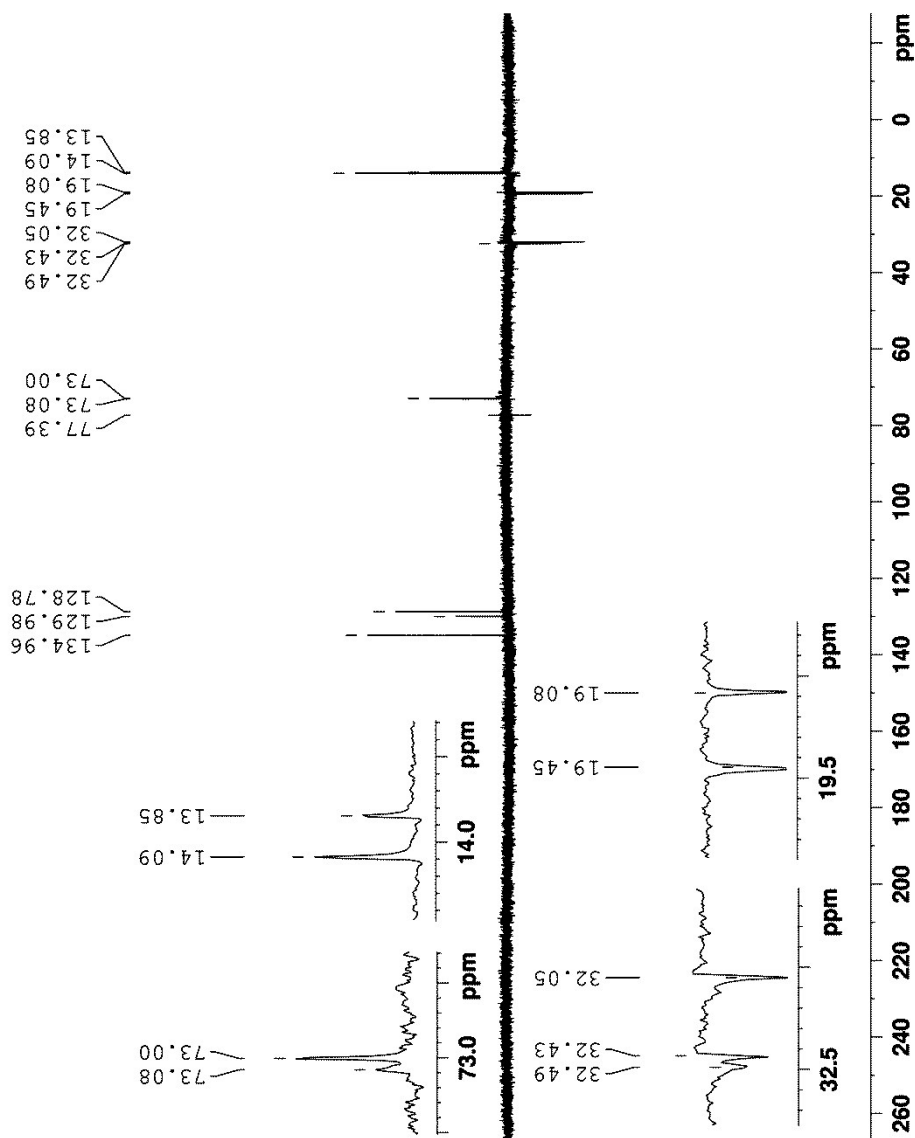
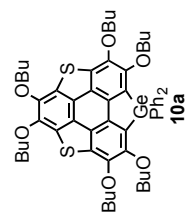
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 0
GB 0
PC



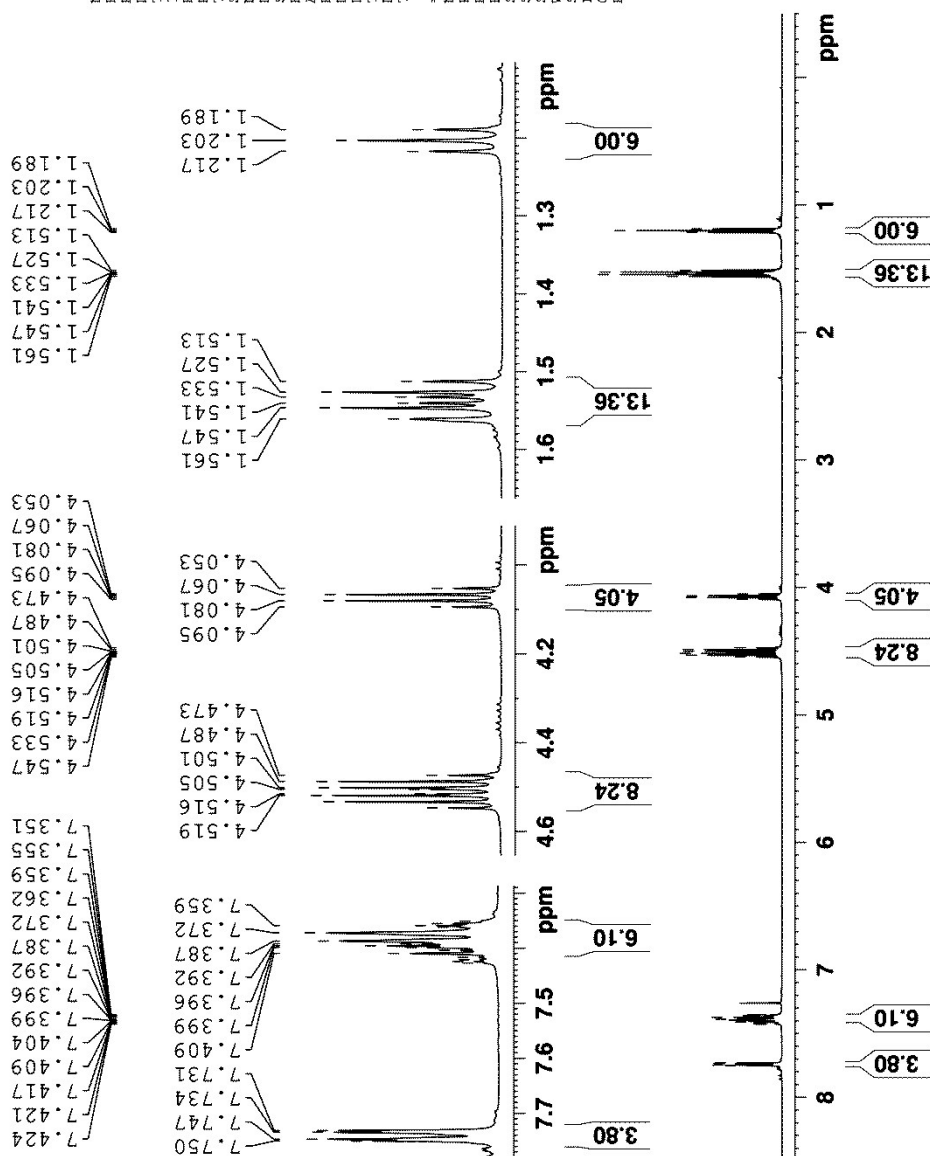
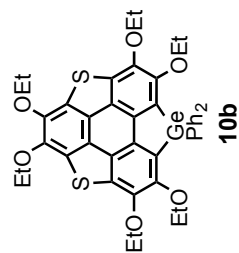
DEPT-135 of of 10a in CDCl₃



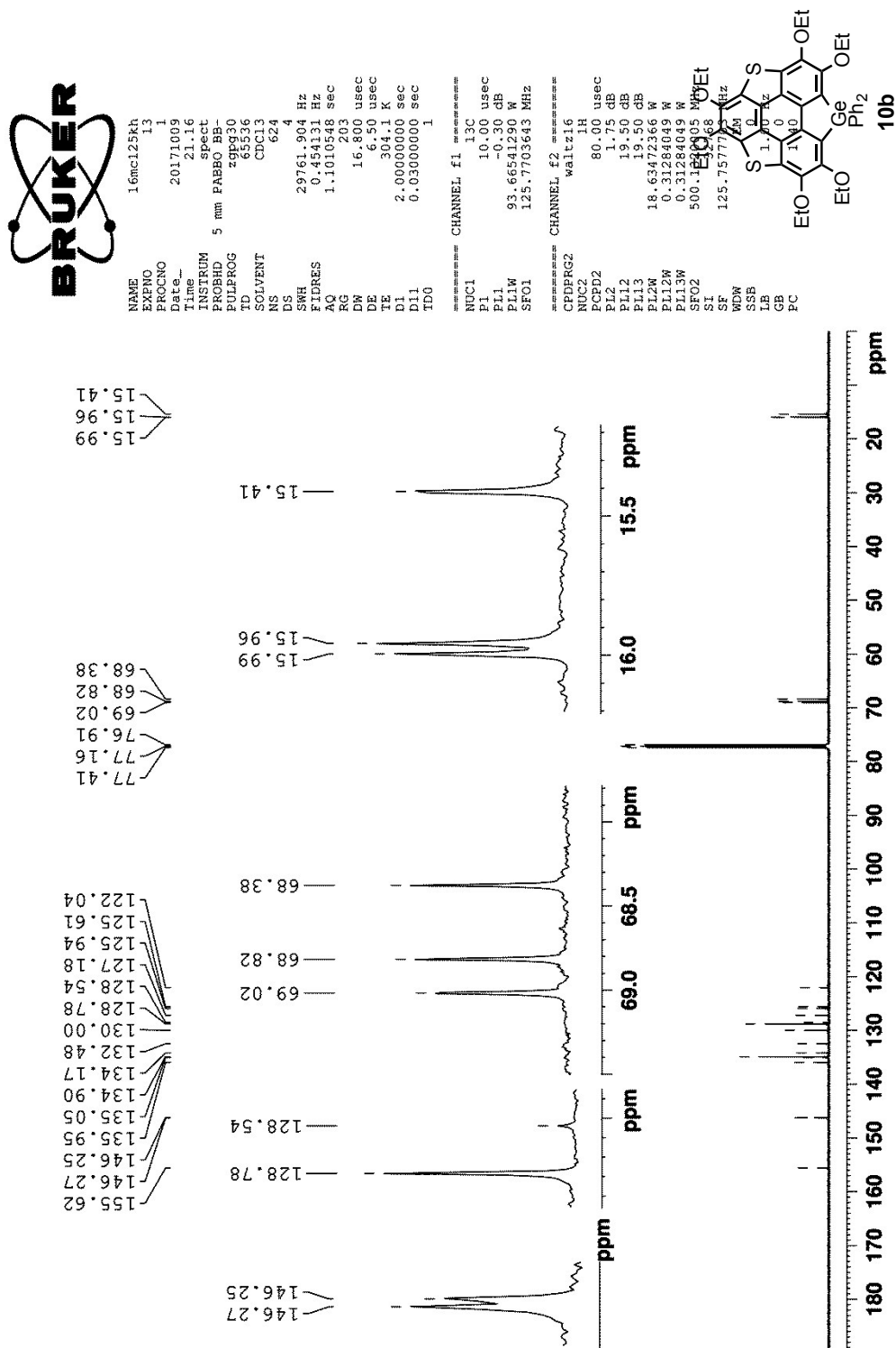
NAME 16mc125kh
 EXPNO 999135001
 PROCNO 1
 Date_ 20160106
 Time 20.34
 INSTRUM spect
 PULPROG zgpg30
 FIDRES 5 mm CPQNE 1H/13C
 TD 65536
 SOLVENT CDCl₃
 NS 100
 DS 4
 SWH 29761.904 Hz
 FIDRES 0.454131 Hz
 AQ 1.1010548 sec
 RG 110.67
 DW 16.800 usec
 DE 18.00 usec
 TE 300.0 K
 CN3T2 145.0000000
 D1 2.00000000 sec
 D2 0.0034828 sec
 D12 0.0000200 sec
 ID0 1
 CHANNEL f1 13C
 NUC1 13C
 P1 12.00 usec
 P2 24.00 usec
 SI 32768
 SF 100.6127528 MHz
 EM 0
 WDW 0
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



NAME	Geसानुने
EXPNO	1
PROCNO	1
Time	20171009
Date_	15.29
INSTRUM	spect
PROBHD	5 mm F4BBO BB-
PULPROG	zg30
TD	6536
SOLVENT	CDCl3
NS	8
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1719923 sec
RG	114
DW	48.400 usec
DE	6.50 usec
TE	301.9 K
D1	1.00000000 sec
TD0	1
CHANNEL f1	=====
NUC1	1H
P1	12.00 usec
PL1	2.60 dB
PL1W	15.32226563 W
SFO1	500.1330885 MHz
SI	32768
SF	500.1330131 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
FC	1.00



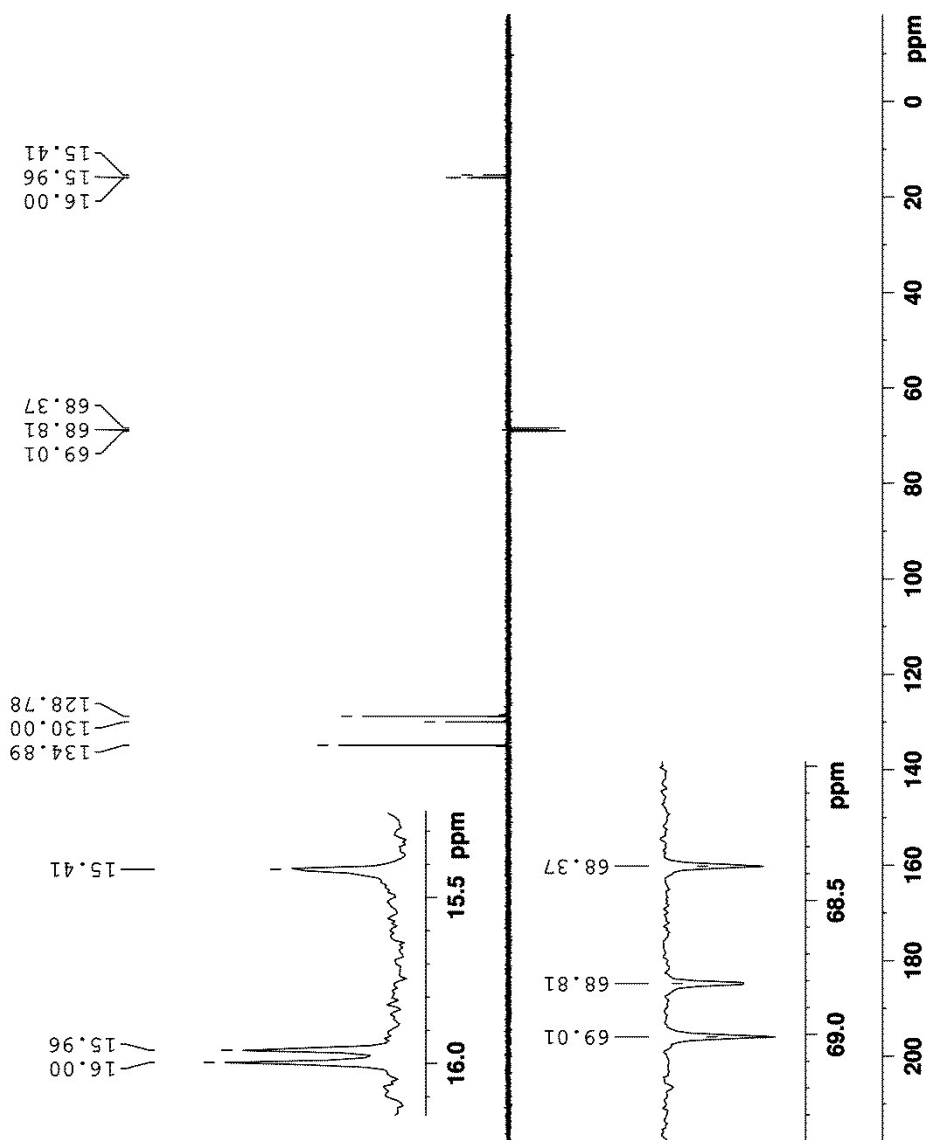
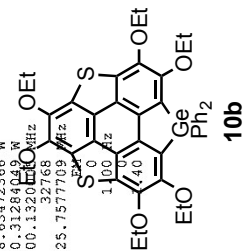
¹³C NMR spectrum of 10b in CDCl₃



DEPT-135 of of 10b in CDCl₃



NAME 16mci25kh
EXPNO 135
PROCNO 1
Date_ 20171009
Time 21:21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG dept135
TD 65536
SOLVENT CDCl₃
NS 92
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DM 16.500 usec
DE 303.3 K
TE 303.3 K
CNST2 145.000000 sec
D1 2.00000000 sec
D2 0.00344828 sec
D12 0.00002050 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 10.00 usec
P2 20.00 usec
PL1 -0.30 dB
PL1W 93.66541290 W
SF01 125.7703643 MHz
CHANNEL f2
waitz16
NUC2 1H
P3 11.00 usec
P4 22.00 usec
PCPD2 50.00 usec
PL2 19.75 dB
PL2W 19.50 dB
PL3W 18.63472366 W
PL4W 0.31284049 W
SF02 500.1320190 MHz
SI 32768
SF 125.7577709 MHz
WDW SSB
LB 1B
GB 1B
PC 1B

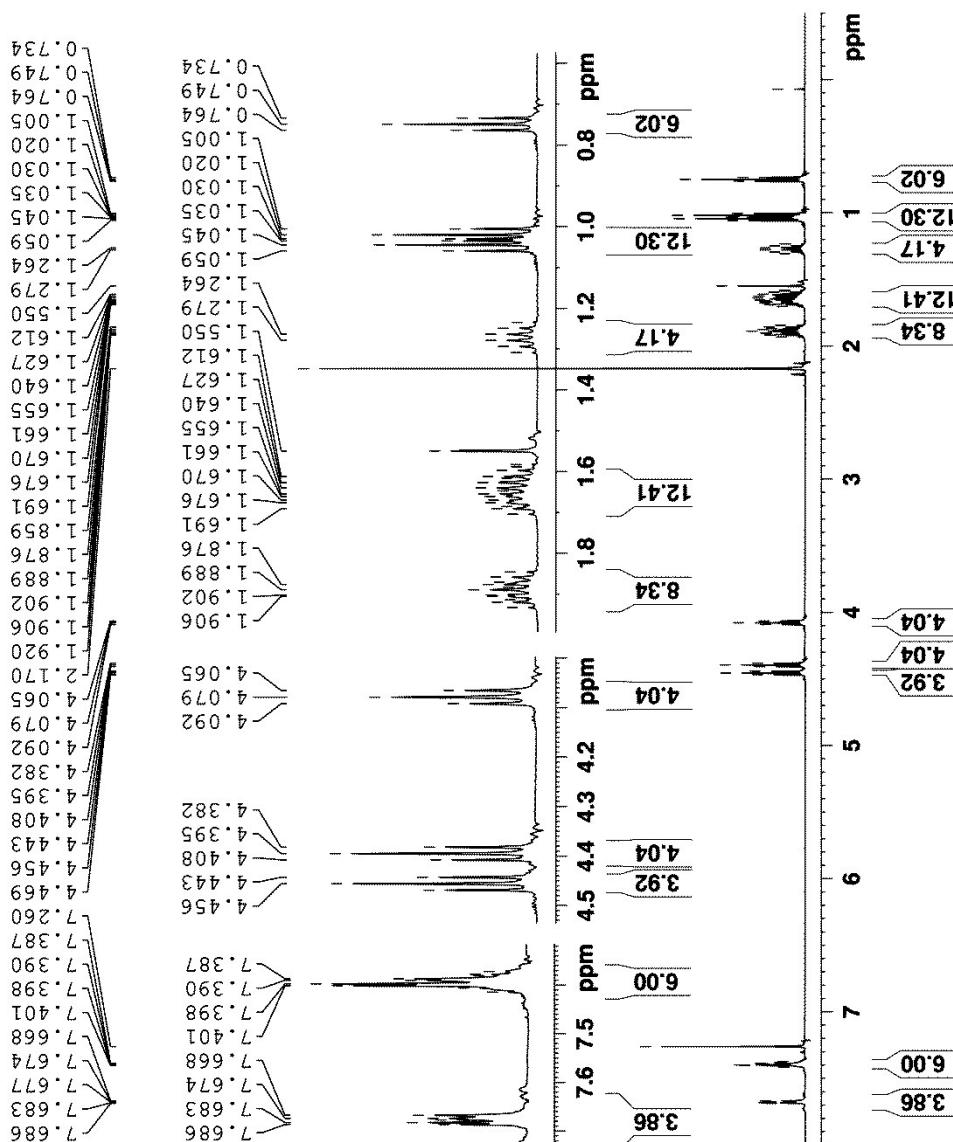
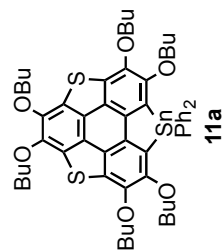



```
Current Data Parameters
=====
NAME      A12C040H
EXPNO     16010509
PROCNO    1

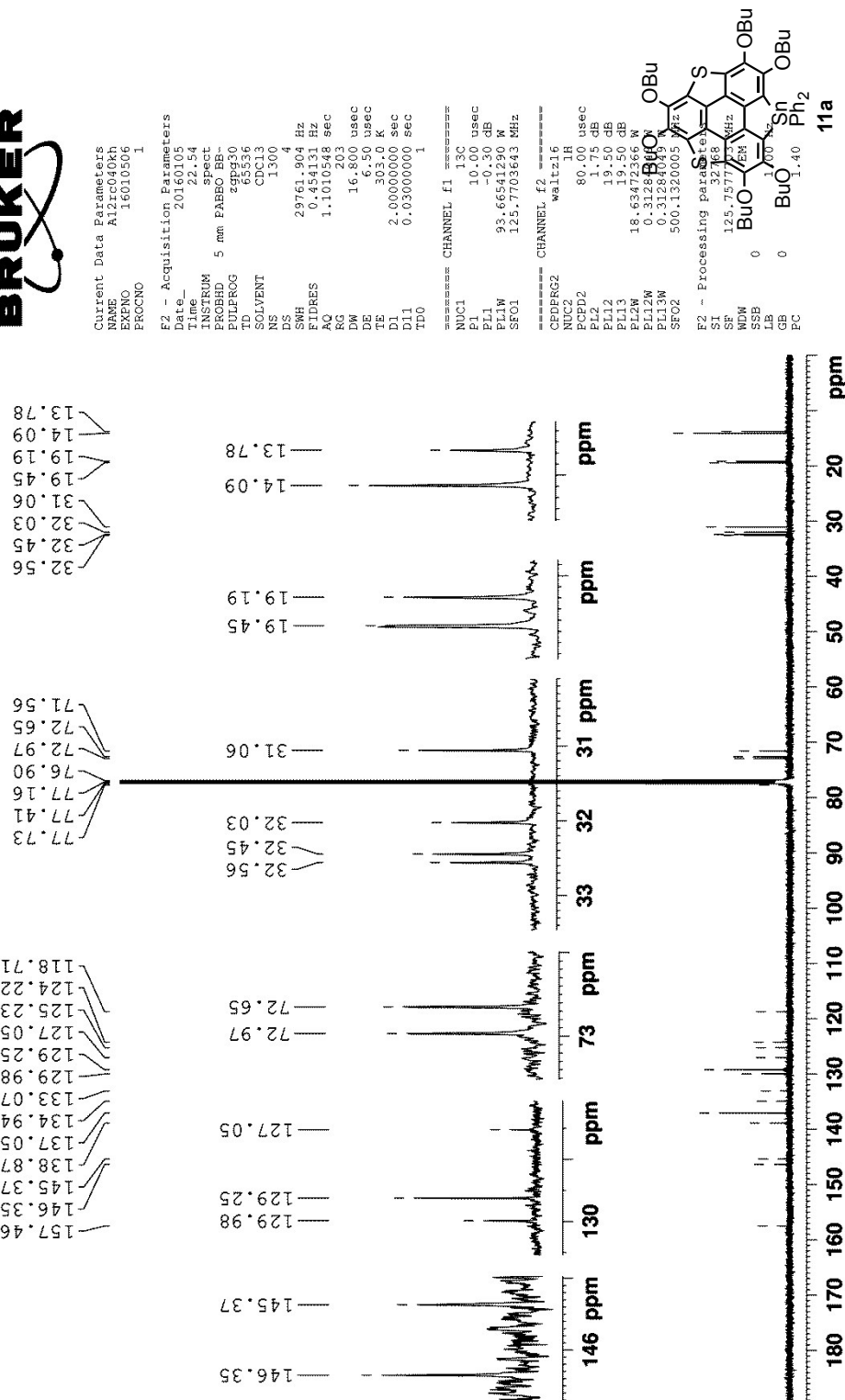
P2 - Acquisition Parameters
=====
Date_     20160105
Time      21.41
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPRG    zg30
F2 - F1    zg30
NUC1       65Cu
NUC2       65Cu
SOLVENT    CDCl3
NS         8
DS         8
SWH         10330.576 Hz
AQ          0.157632 Hz
RG          3.1710923 Hz
WDW         144
SSB         0
GB          0
PC          1.00000000 sec
TE         301.7 K
TD0        1

=====
CHANNEL f1 1H
=====
NUC1       1H
P1         12.00 usec
PR1        2.60 dB
IR1        15.32228563 W
FIDPRG1    W
SFO1       500.1330885 MHz

P2 - Processing parameters
=====
SI         32768
SF         500.1330138 MHz
WDW         EM
SSB         0
GB          0
PC          1.00
```



¹³C NMR spectrum of 11a in CDCl₃



DEPT-135 of of 11a in CDCl₃



¹¹⁹Sn NMR spectrum of 11a in CDCl₃

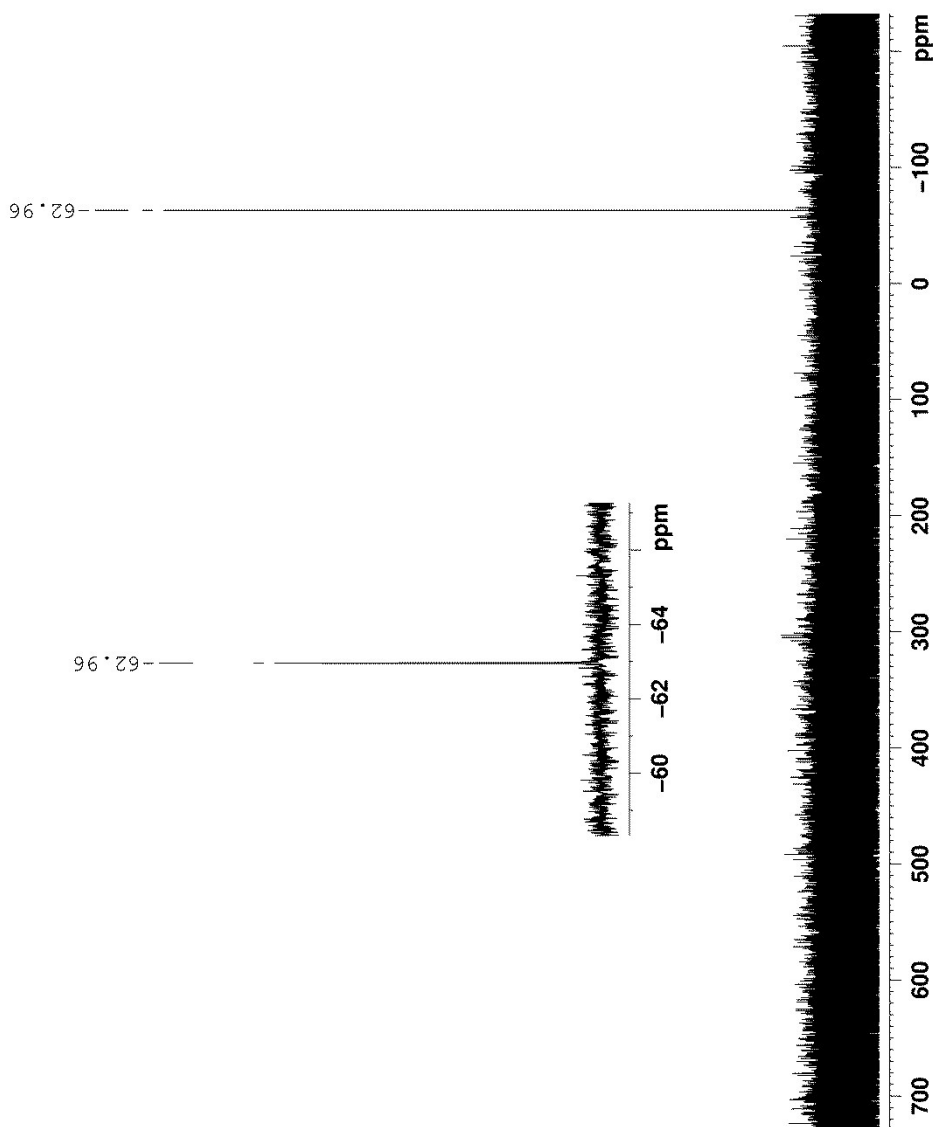
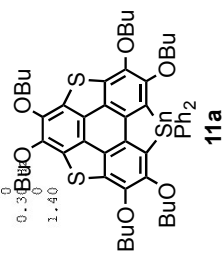


```

NAME      Al2rc040kh
EXPNO     16010601
PROCNO    1
Date_     20160106
Time      20.22
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD        320000
SOLVENT   CDCl3
NS        680
DS        4
SWH       187500.000 Hz
FIDRES    0.585938 Hz
AQ         0.8533834 sec
RG         203
DW         2.667 usec
DE         6.50 usec
TE         301.7 K
D1         4.0000000 sec
D11        0.0300000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       119Sn
P1         10.50 usec
PL1        1.50 dB
SFO1       186.5482634 MHz

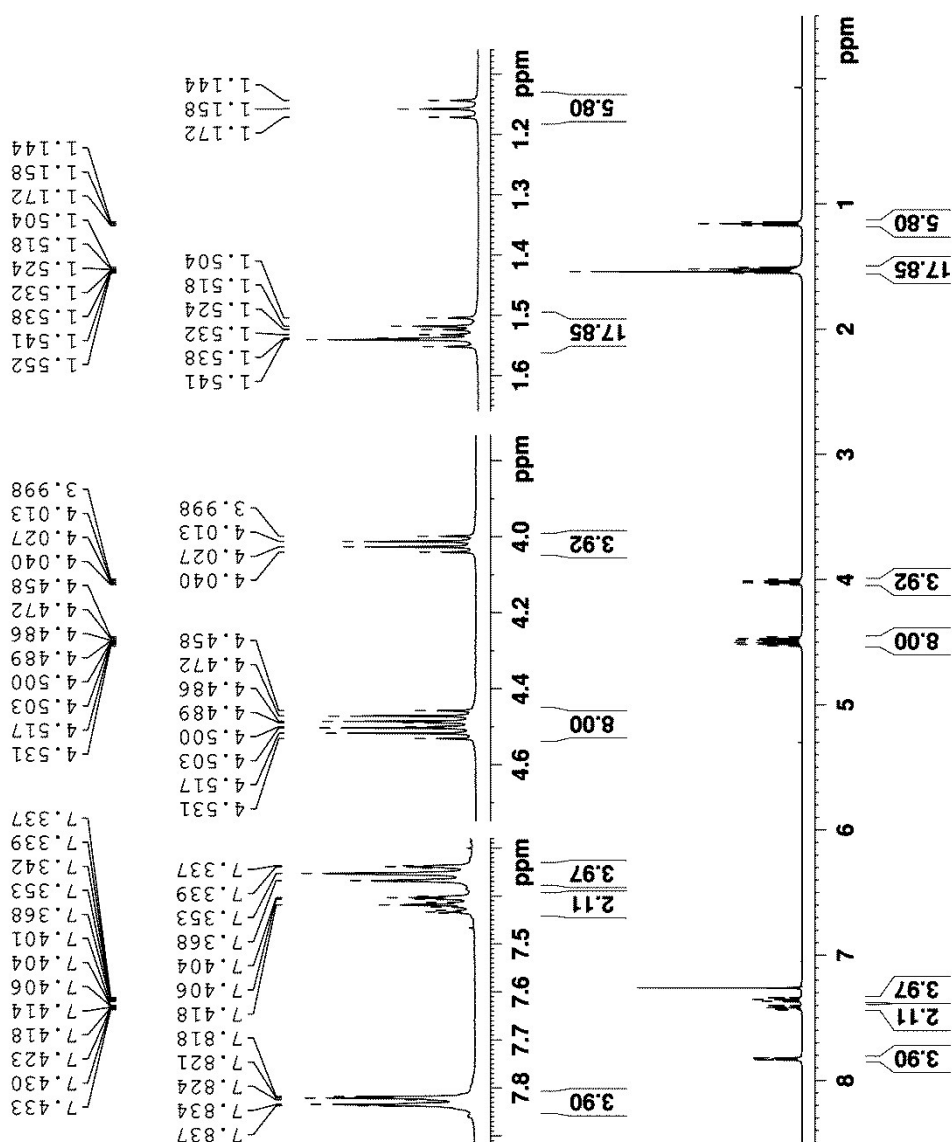
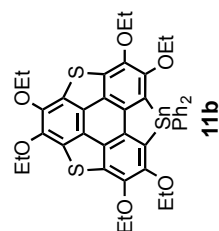
===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2        1H
PCPD2      80.00 usec
PL2         1.75 dB
PL12       19.50 dB
PL12W      18.63472366 W
PL12W      0.31284049 W
SFO2       500.1320005 MHz
SI         131072
SF         186.5017555 MHz
MDW        EM
LB         0
GB         0
PC         0
  
```

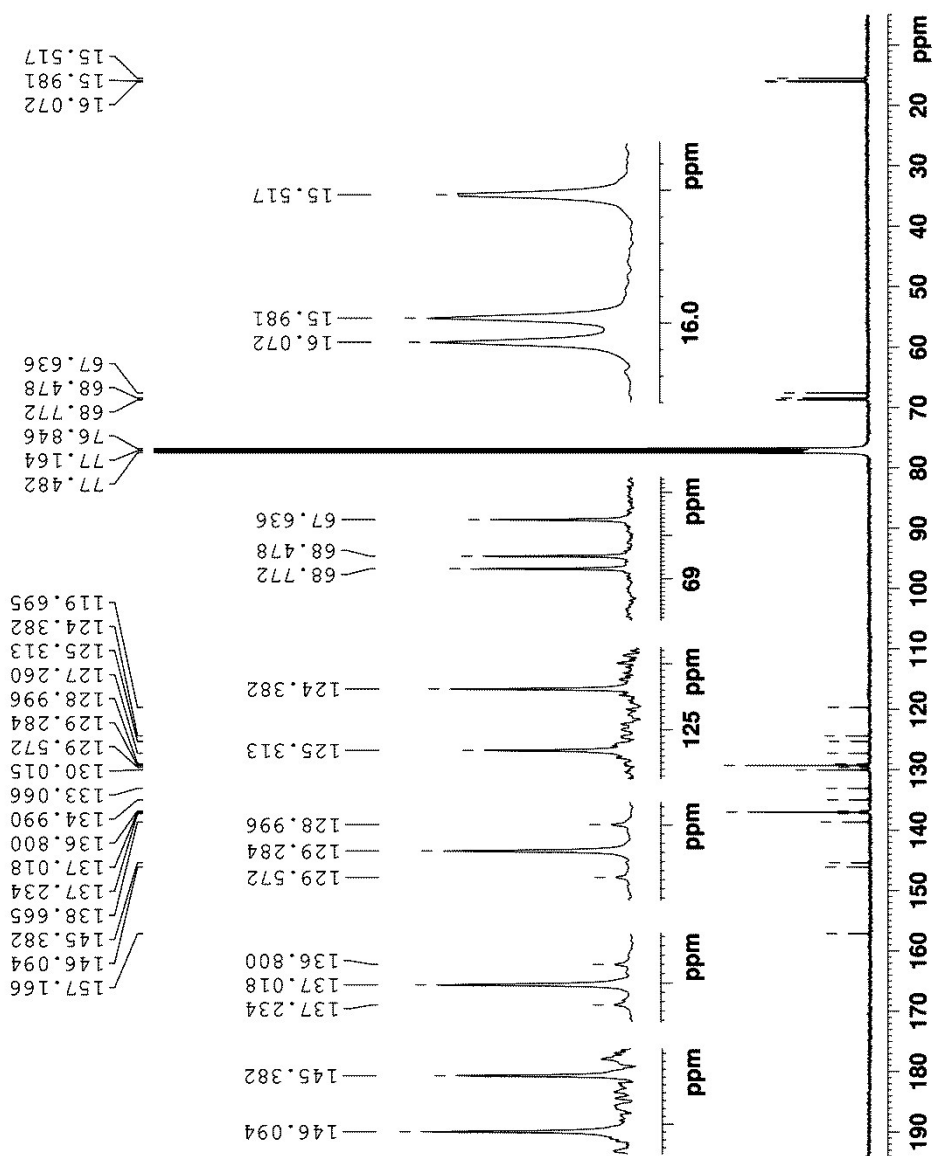
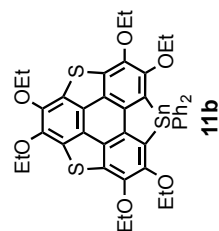


¹H NMR spectrum of 11b in CDCl₃



NAME Snsmanene
EXPNO 1
PROCNO 1
Date_ 20171005
Time 12.11
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl₃
NS 8
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.171923 sec
RG 655
DW 48.400 usec
DE 6.50 usec
TE 301.7 K
D1 1.00000000 sec
D11
TD0 1
===== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 2.60 dB
PL1W 15.32226563 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300136 MHz
WDW EM
SSB 0
GB 0.30 Hz
PC 1.00

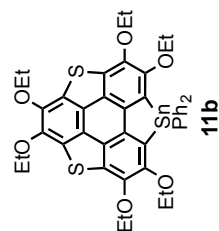
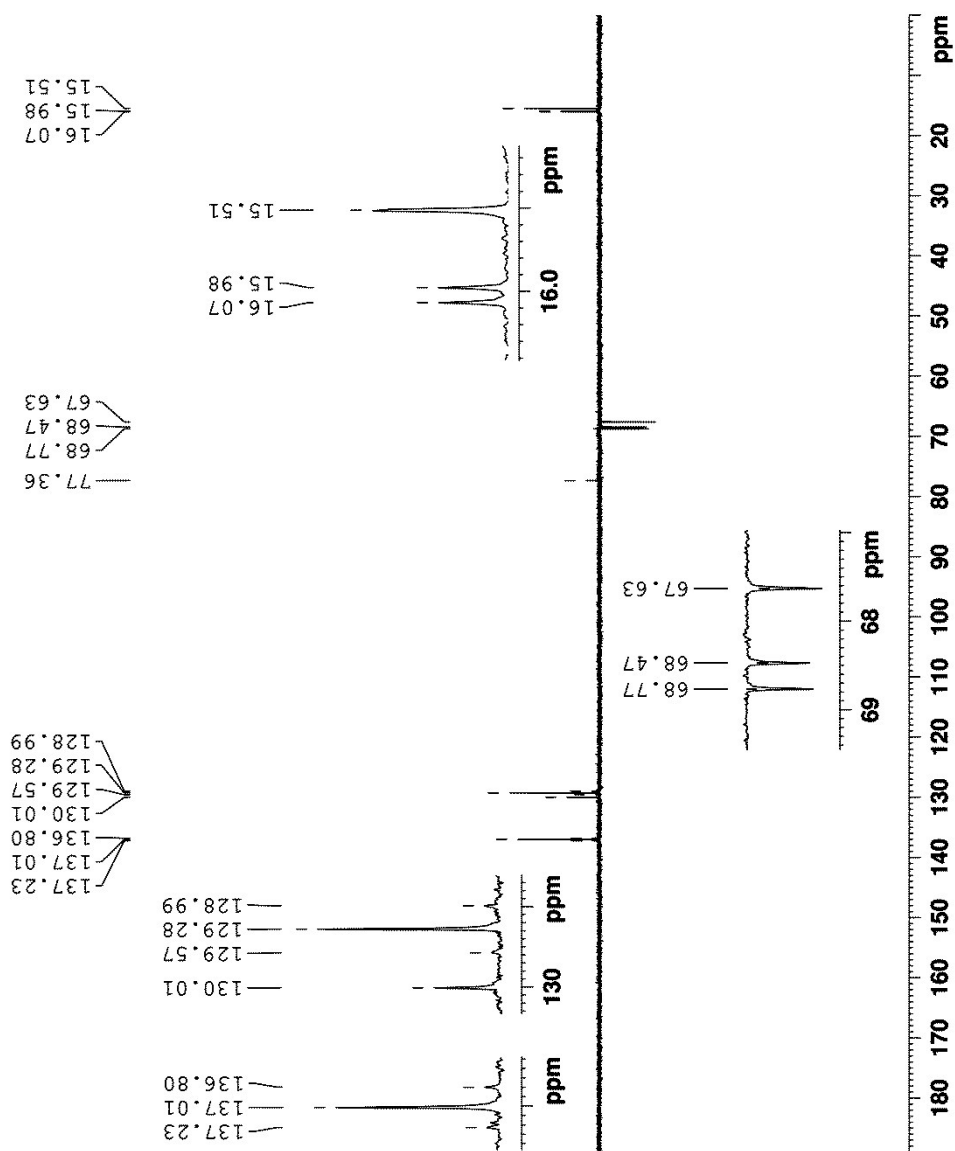


[illegible]

DEPT-135 of of 11b in CDCl₃



NAME 16m125kh
EXPNO 1
PROCNO 155
Date_ 20171004
Time 16:31
INSTRUM spect
PROBHD 5 mm CPNP 1H/
PULPROG dept135
TD 65536
SOLVENT CDCl3
NS 480
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 163.82
DE 16.00 usec
TE 300.0 K
CNS2 145.0000000
D1 2.00000000 sec
D2 0.00344828 sec
D3 0.00002000 sec
TD0 1
===== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL 0.00 dB
SI 32768
SF 100.6127532 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



¹¹⁹Sn NMR spectrum of 11b in CDCl₃

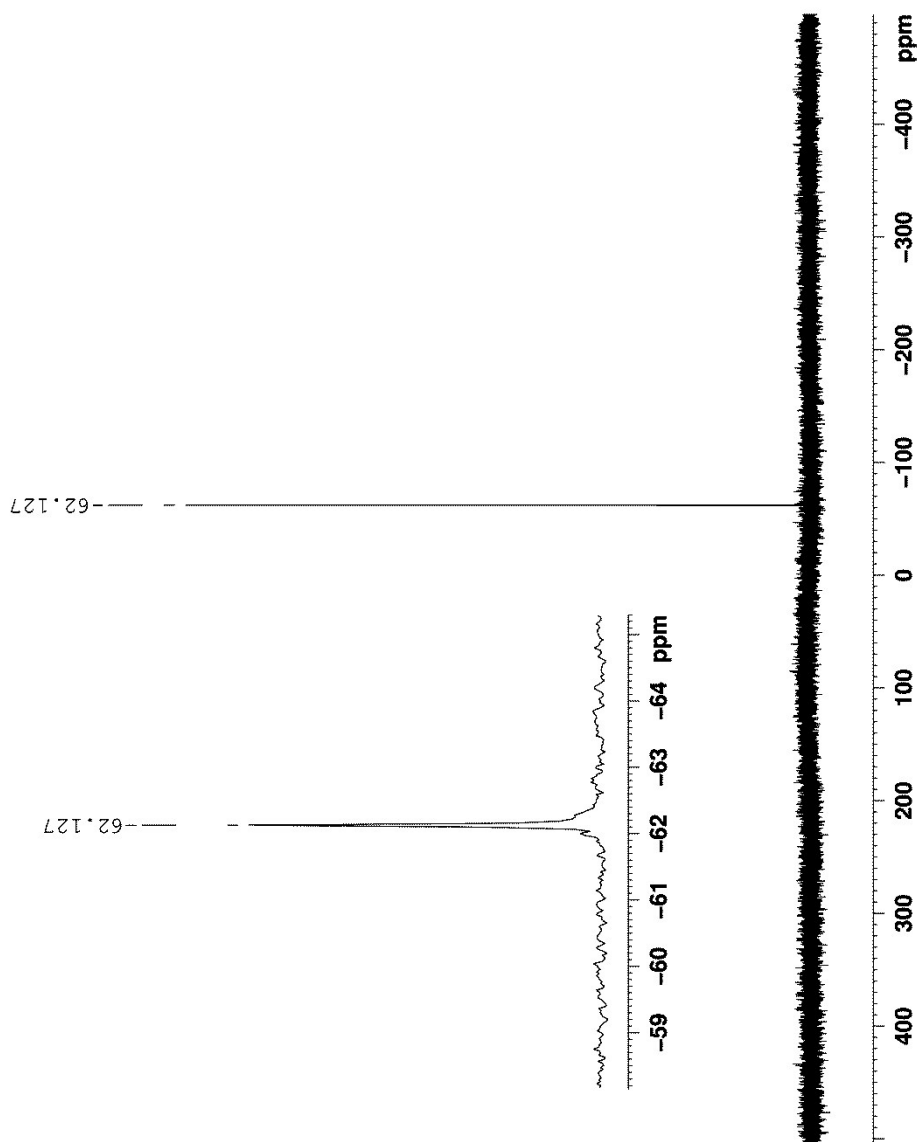
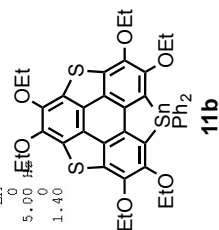


```

NAME                               Snsunanene
EXPNO                               111
PROCNO                              1
Date_                                20170608
Time_                                17.11
INSTRUM                             spect
PROBHD 5 mm PABBO BB-
PULPROG                             zgpg30
TD                                  240000
SOLVENT                             CDCl3
NS                                  2224
DS                                  4
SWH 187500.000 Hz
FIDRES 0.781250 Hz
AQ 0.6400500 sec
RG 203
DW 2.667 usec
DE 6.50 usec
TE 300.1 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 119Sn
P1 15.00 usec
PL1 3.50 dB
PL1W 33.66055298 W
SF01 186.4652793 MHz

===== CHANNEL f2 =====
CPDPRG2 waitz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.40 dB
PL12 19.02 dB
PL12W 15.17711735 W
PL12W 0.33051354 W
SF02 500.0320001 MHz
SI 32768
SF 186.4652793 MHz
WDW EM
SSB 0
LB 0
GB 0
PC 0
  
```



¹H NMR spectrum of 1a in CDCl₃

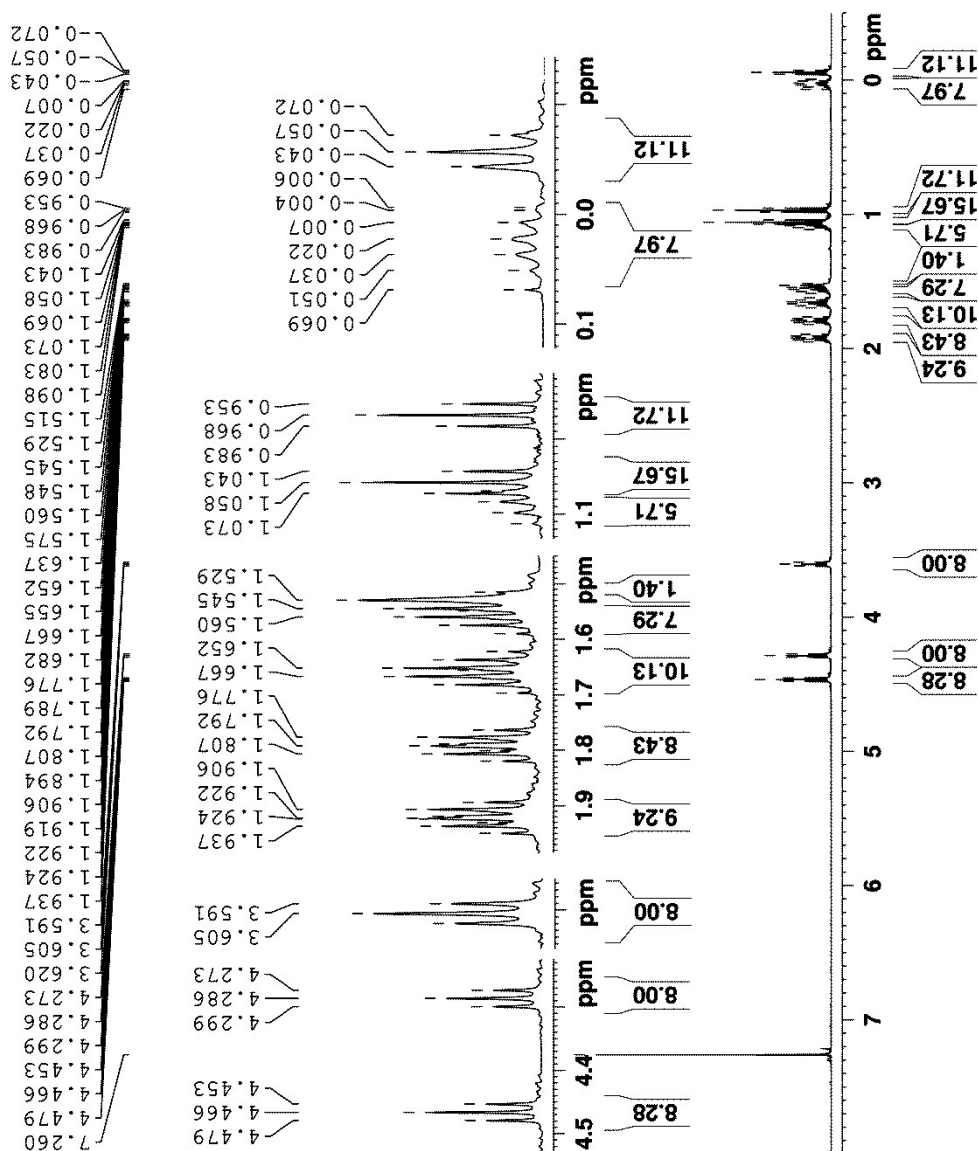
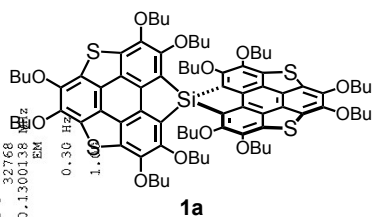


Current Data Parameters
NAME A12rc040Kh
EXPNO 16012702
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160127
Time 23:16
INSTRUM spect
PROBHD 5 mm PABBO-5B-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 161
DE 48.400 usec
TE 299.6 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 2.60 dB
PL1W 15.32246563 W
SFO1 500.1350885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300138 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1



DEPT-135 of of 1a in CDCl₃



Current Data Parameters
NAME AL2rc040Kh
EXPNO 16012705
PROCNO 1

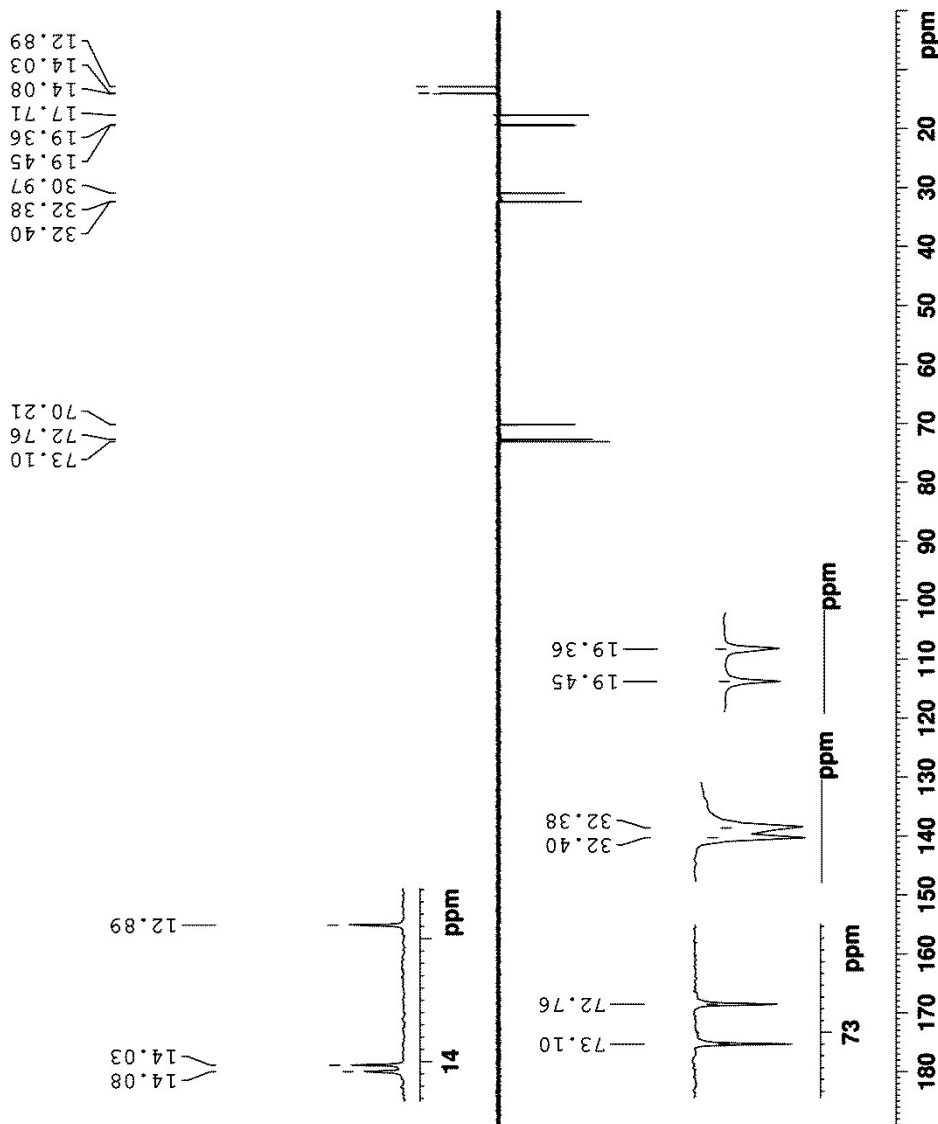
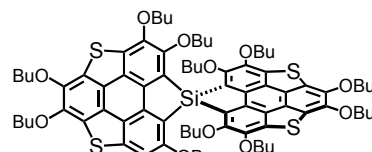
F2 - Acquisition Parameters
Date_ 20160127
Time 23:48
INSTRUM spect
PROBHD 5 mm PABD-BB-
PULPROG dept135
TD 65536
SOLVENT CDCl₃
NS 80
DS 4

SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DE 16.500 usec
TE 299.9 K
CNST2 145.0000000
D1 2.00000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
TD0 1

CHANNEL f1
NUC1 ¹³C
P1 10.00 usec
P2 20.00 usec
PL1 -0.30 dB
PL1W 93.66541290 W
SFO1 125.7703643 MHz

CHANNEL f2
CPDPRG2 waltz16
NUC2 ¹H
P3 11.00 usec
P4 22.00 usec
PCPD2 90.00 usec
PL2 19.75 dB
PL12 19.50 dB
PL2W 18.63472366 W
PL12W 0.31284049 W
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577724 MHz
WDW EM
SSB 0
GB 0
PC 1.40



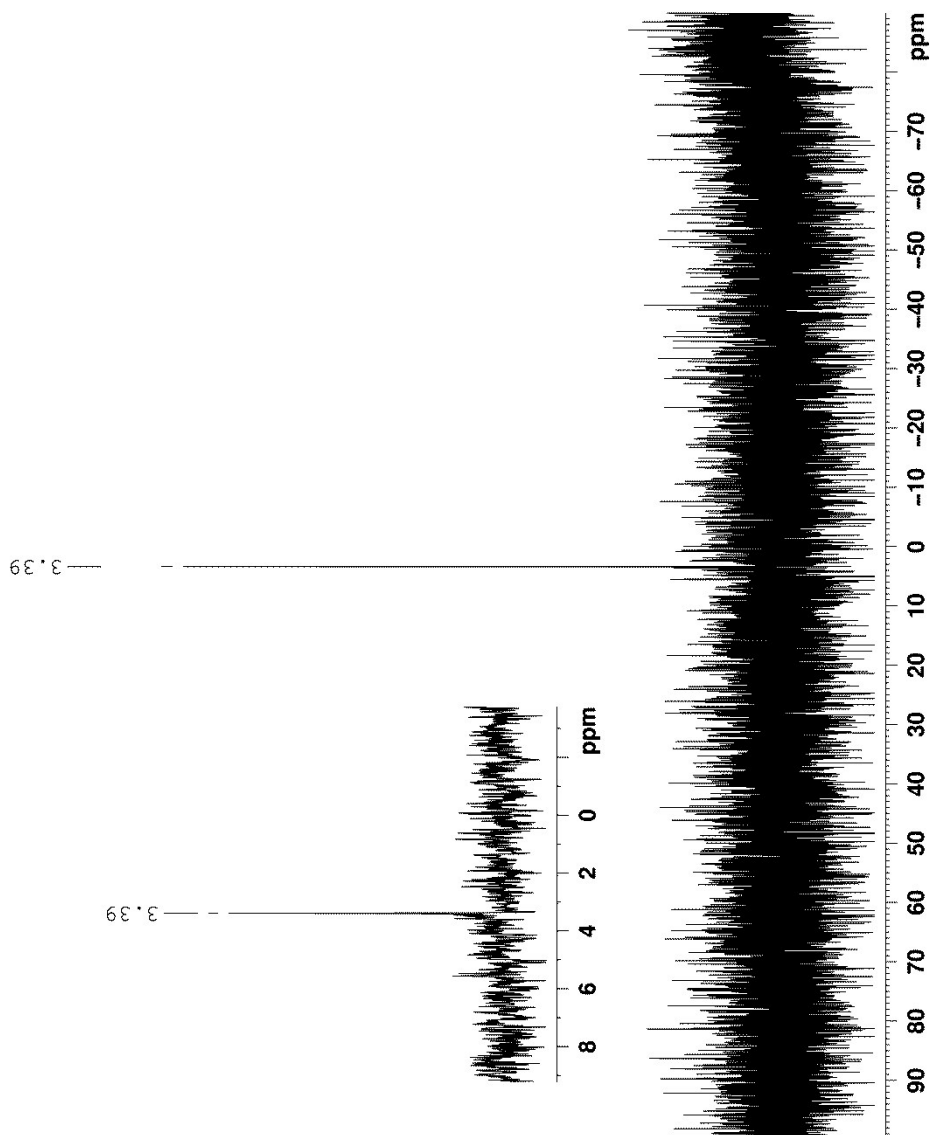
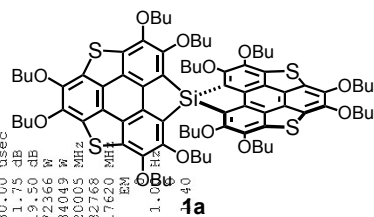
²⁹Si NMR spectrum of 1a in CDCl₃



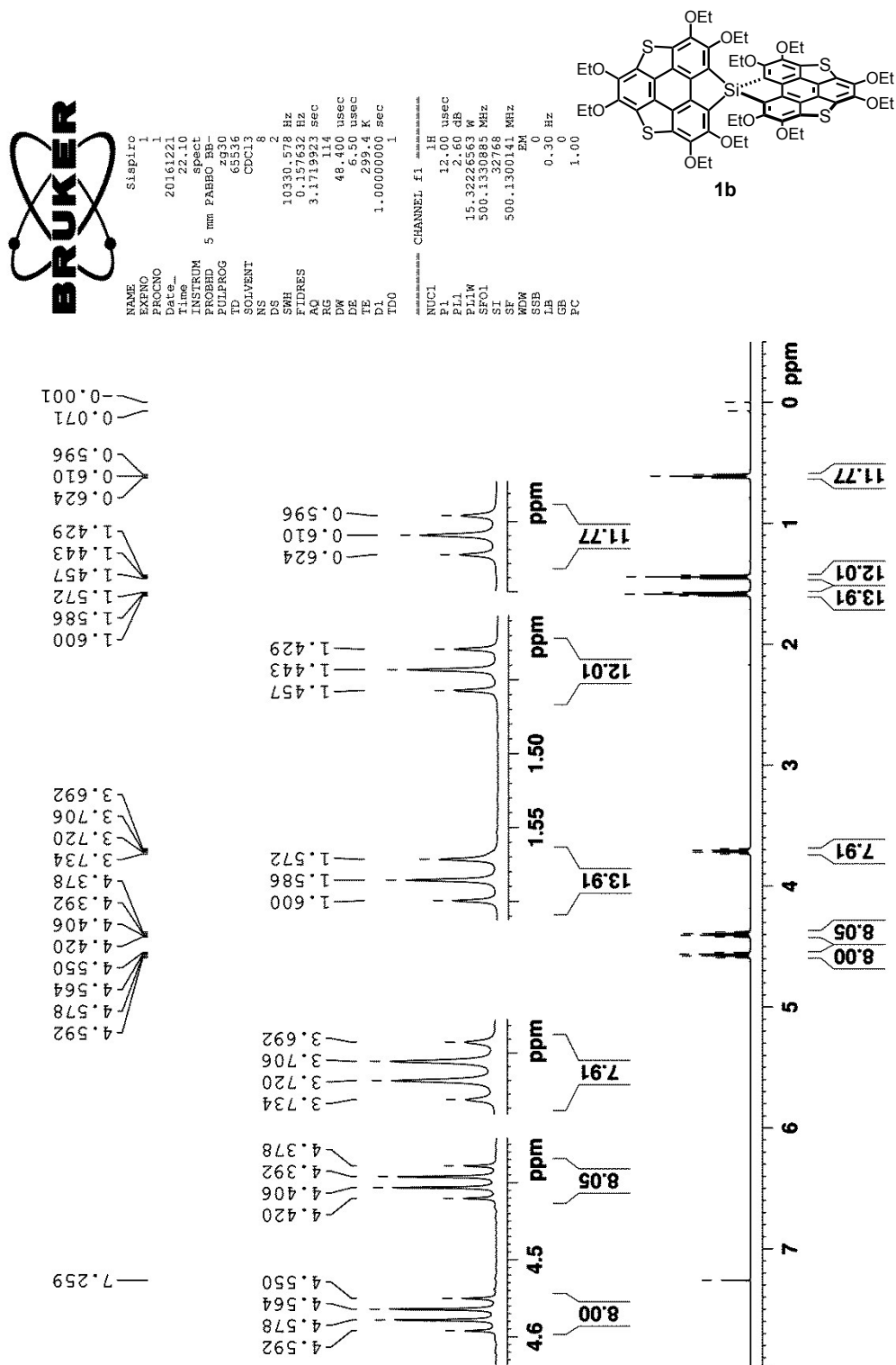
NAME A12rc040kh
 EXPNO 16012706
 PROCNO 1
 Date_ 20160127
 Time 23.58
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 ID 65536
 SOLVENT CDCl₃
 NS 85
 DS 0
 SWH 39682.537 Hz
 FIDRES 0.405907 Hz
 AQ 0.8258036 sec
 RG 203
 DW 12.600 usec
 DE 6.50 usec
 TE 299.7 K
 D1 10.0000000 sec
 D11 0.0300000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 29Si
 P1 10.00 usec
 PL1 3.00 dB
 SF01 99.3518262 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 1.75 dB
 PL12 19.50 dB
 PL2W 18.63472366 W
 PL2W 0.31284049 W
 SFO2 500.1320005 MHz
 ST 3268
 SI 99.3617620 MHz
 EM 1.00
 NMW 40
 SSB 40
 LB 40
 GB 40
 PC 40



¹H NMR spectrum of 1b in CDCl₃

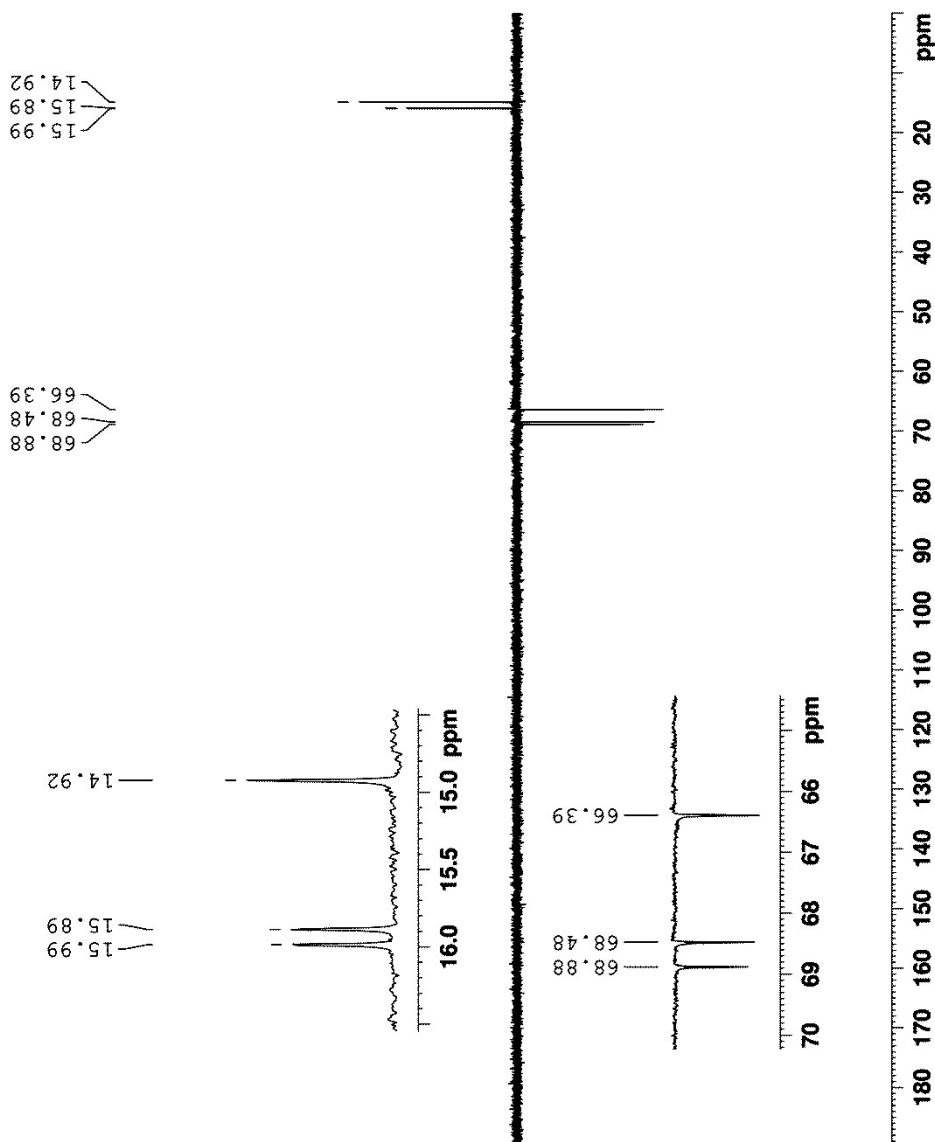
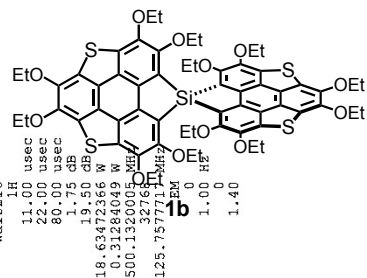


DEPT-135 of of 1b in CDCl₃



```

NAME      16mcl25kh
EXPNO     1
PROCNO    135
Date_     20161117
Time      22.31
INSTRUM   spect
PROBHD    5 mm PABEO BB-
PULPROG   dept135
TD         65536
SOLVENT   CDCl3
NS         116
DS         4
SWH        29761.904 Hz
FIDRES     0.454131 Hz
AQ         1.1010548 sec
RG         203
DW         16.800 usec
DE         6.50 usec
TE         299.6 K
CNS12     145.0000000
D1         2.00000000 sec
D2         0.00344828 sec
D12        0.00002000 sec
TD0        1
===== CHANNEL f1 =====
NUC1       13C
P2         10.00 usec
P3         20.00 usec
PL1        -0.30 dB
PL1W       93.66541230 W
SF01       125.7703643 MHz
===== CHANNEL f2 =====
CPOPRG2    waltz16
NUC2       1H
P3         11.00 usec
P4         22.00 usec
PCPD2      80.00 usec
PL2        1.5 dB
PL2W       1.5 dB
PL12W      18.63472356 W
PL12W      0.31284049 W
SF02       500.1320005 MHz
SI         32768
SF         125.7577711 MHz
WDW         EM
SSB         0
LB         1.00 Hz
GB         0
EC         1.40
  
```



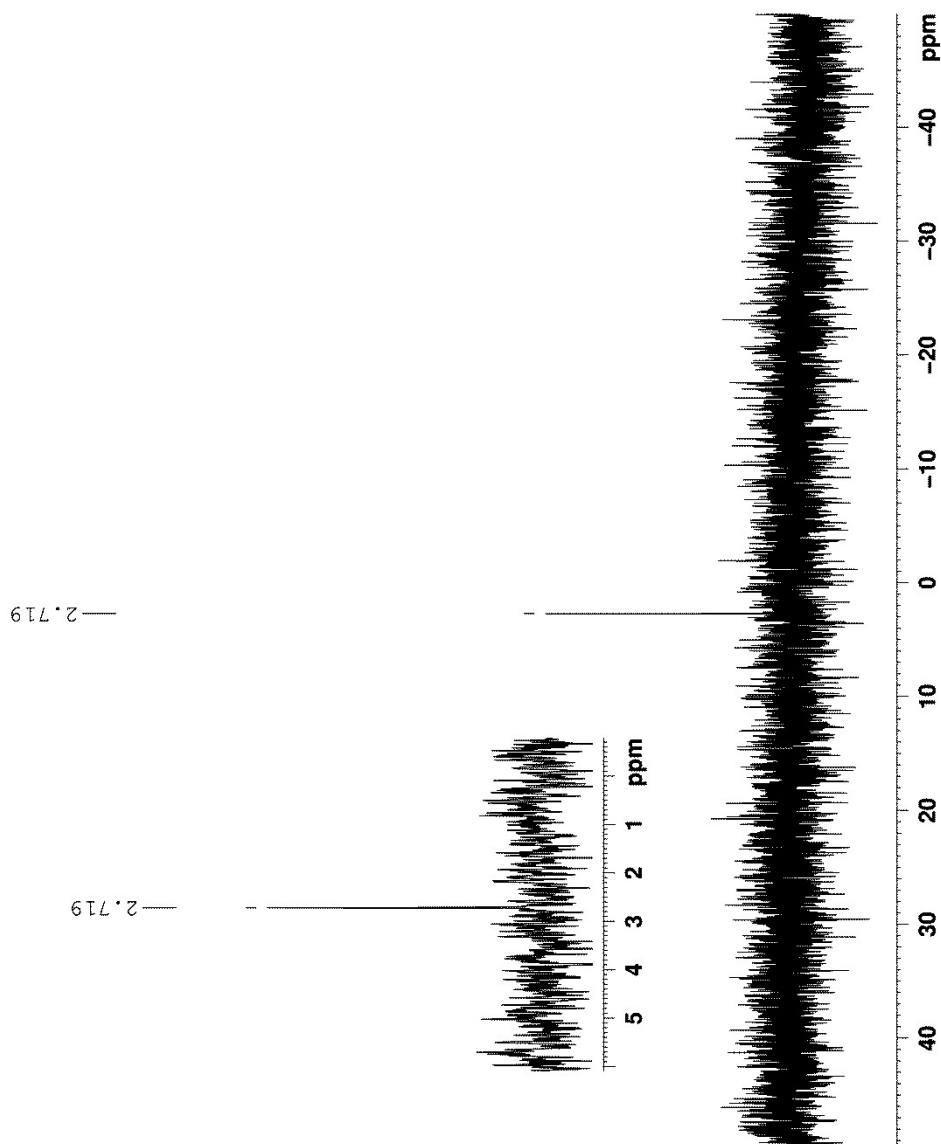
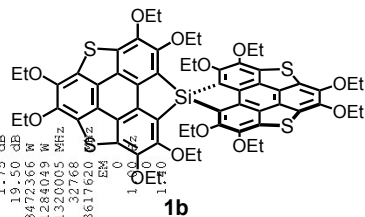
²⁹Si NMR spectrum of 1b in CDCl₃



NAME Sispiro
 EXENO 29
 PROCNO 1
 Date_ 20161110
 Time 21.59
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 ID 65536
 CDCl₃
 SOLVENT
 NS 480
 DS 0
 SWH 9820.635 Hz
 FIDRES 0.151377 Hz
 AQ 3.3030643 sec
 RG 203
 DW 50.400 usec
 DE 6.50 usec
 TE 299.7 K
 D1 10.0000000 sec
 D11 0.0300000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 29Si
 P1 10.00 usec
 PL1 3.00 dB
 SFO1 99.3617620 MHz

===== CHANNEL f2 =====
 CDEPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 1.75 dB
 PL12 18.6347250 dB
 PL13 0.3128409 W
 SFO2 500.132005 MHz
 SI 32768
 SF 99.3617620 MHz
 WDW EM
 SSB 0
 LB 1.40
 GB 0
 PC



¹H NMR spectrum of 2a in CDCl₃



Current Data Parameters
NAME AL2604065
EXPNO 16012301
PROCNO 1

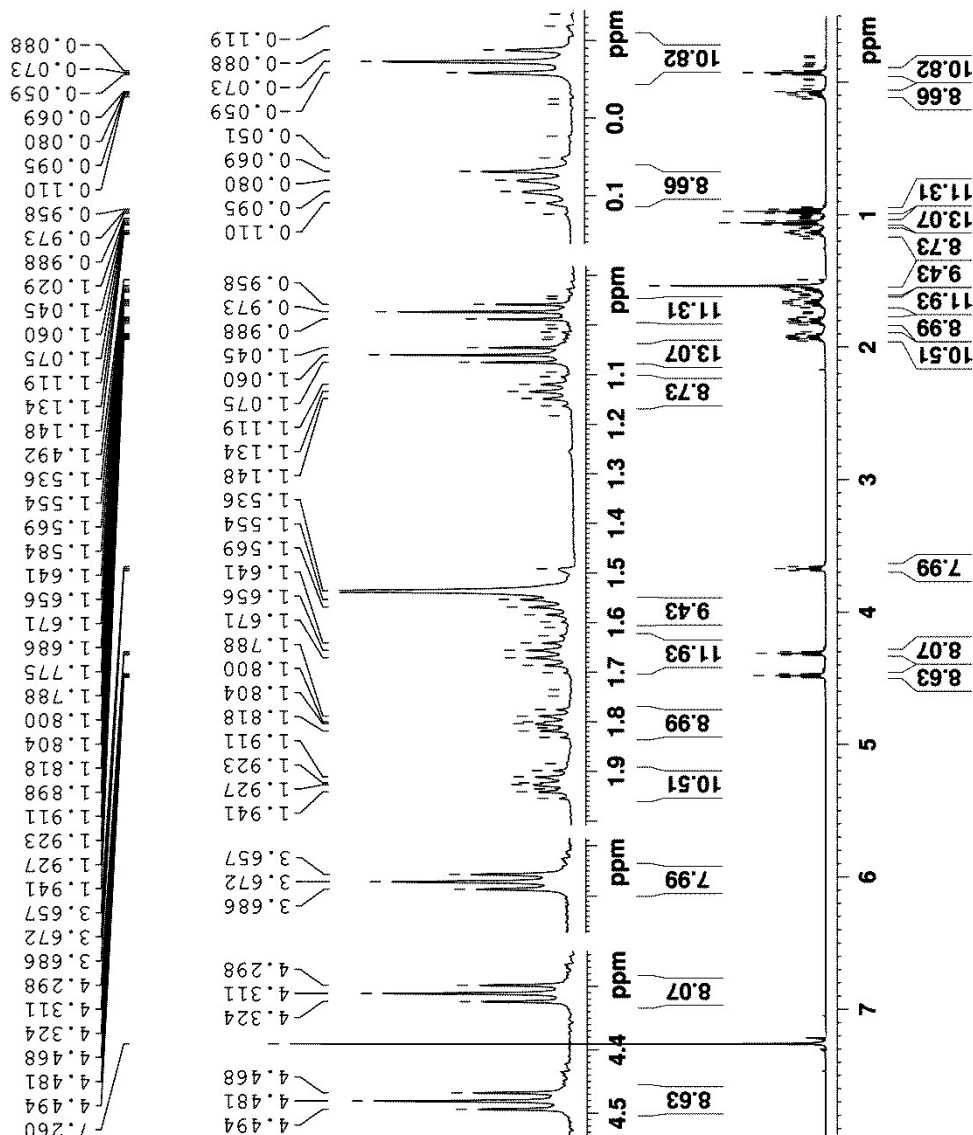
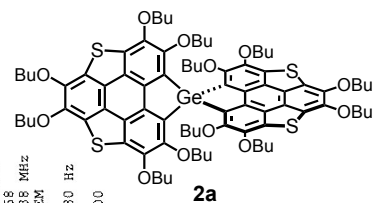
F2 - Acquisition Parameters

Date_ 20160122
Time_ 19.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 10330.578 Hz
FIDRES 0.151682 Hz
AQ 3.171923 sec
RG 203
DM 48.400 usec
DE 6.50 usec
TE 300.4 K
D1 1.00000000 sec
TD0 1

CHANNEL f1
NUC1 1H
P1 12.00 usec
PL1 2.60 dB
PL1W 15.3226663 W
SFO1 500.1330885 MHz

F2 - Processing parameters

SF 500.1330885 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



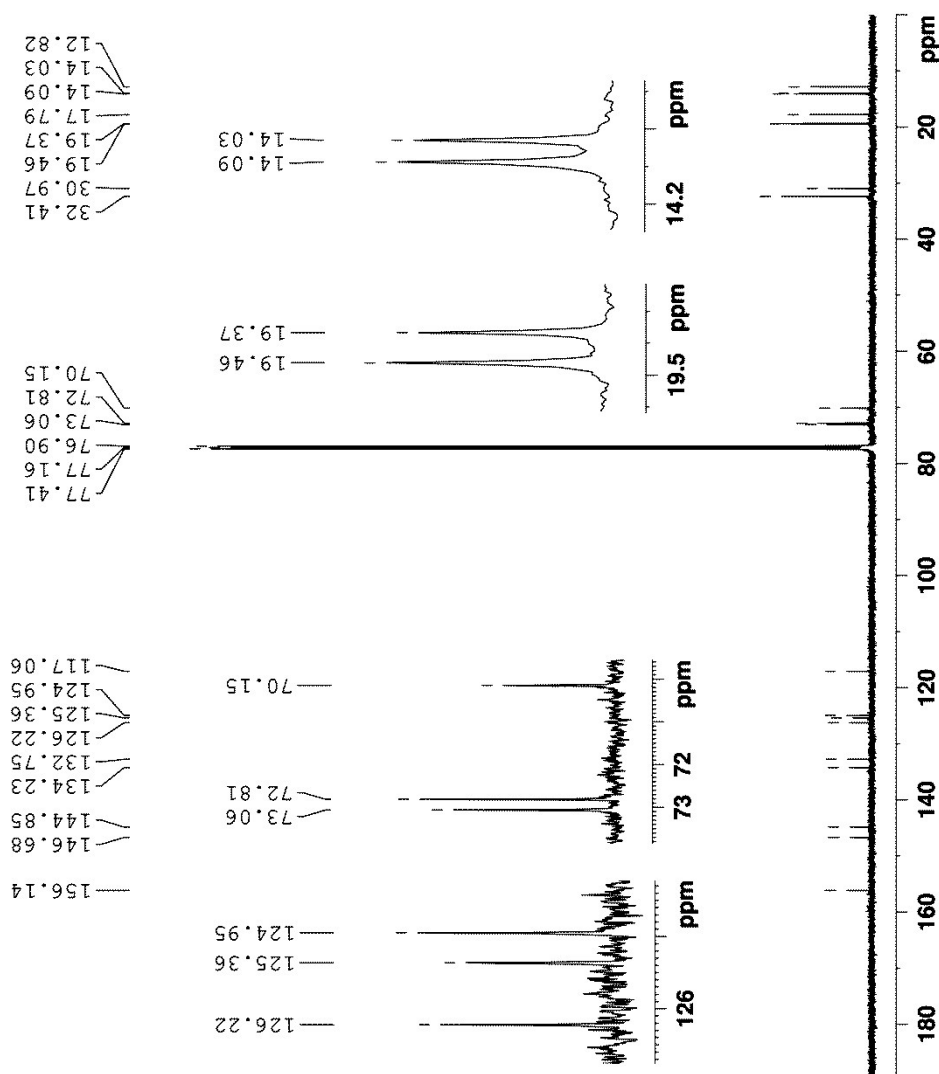
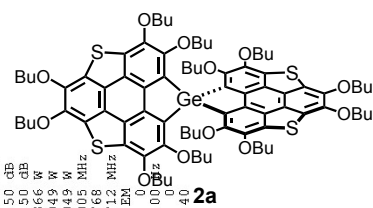
¹³C NMR spectrum of 2a in CDCl₃



NAME A12rc040kh
EXPNO 16020106
PROCNO 1
Date_ 20160202
Time 0.05
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
F4 564
SOLVENT CDCl₃
NS 2
DS 29761.904 Hz
SWH 0.454131 Hz
FIDRES 1.1010548 sec
AQ 203
RG 16.800 usec
DW 6.50 usec
DE 301.1 K
TE 2.00000000 sec
D1 0.03000000 sec
D11 1
TD0

CHANNEL f1 13C
NUC1 13C
P1 10.00 usec
PL1 -0.30 dB
PL1W 93.66541290 W
SF01 125.7703643 MHz

CHANNEL f2
waltz16
NUC2 1H
PCPD2 80.00 usec
P12 1.75 dB
PL2 19.50 dB
PL13 19.50 dB
PL12W 18.63472366 W
P112W 0.31284049 W
P113W 0.31284049 W
SF02 500.1320005 MHz
SI 32768
SF 125.7577712 MHz
EM 0
SSB 0
LB 0
GB 0
PC



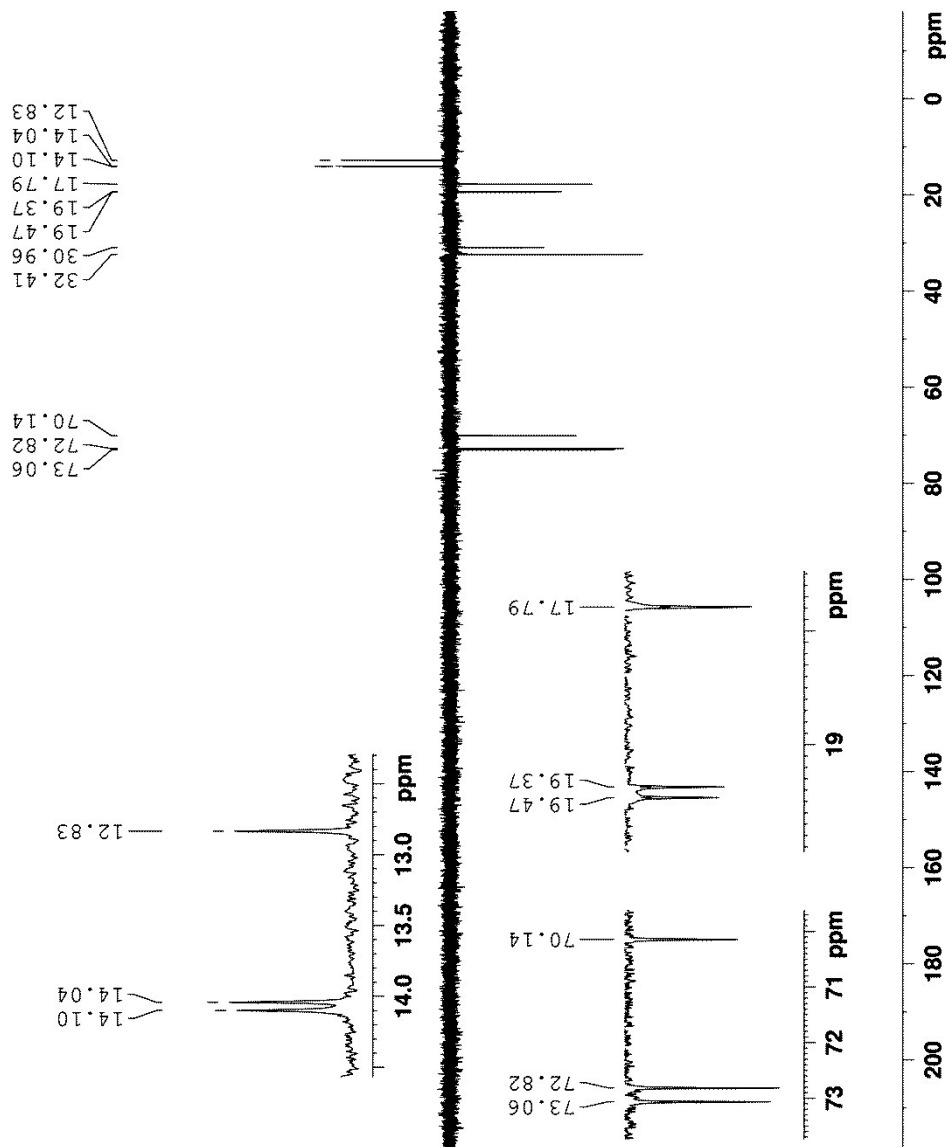
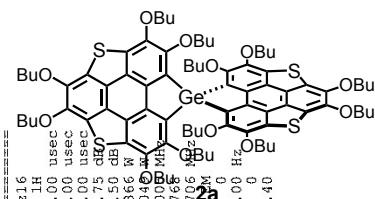
DEPT-135 of of 2a in CDCl₃



NAME A12rc040kh
EXPNO 16020105
PROCNO 1
Date_ 20160201
Time 23.33
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG dept135
TD 65536
SOLVENT CDCl₃
NS 168
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 300.5 K
CNS12 145.000000
D1 2.00000000 sec
D2 0.00344828 sec
D3 0.00002000 sec
TD0 1

CHANNEL f1 13C
NUC1 13C
P1 10.00 usec
P2 20.00 usec
PL1 -0.30 dB
PL1W 93.66541290 W
SFO1 125.7703643 MHz

CHANNEL f2 waltz16
CPDPRG2
NUC2 1H
P3 11.00 usec
P4 22.00 usec
PCPD2 80.00 usec
PL2 1.75 dB
PL12 19.50 dB
PL2W 18.63472366 W
PL12W 0.31284040 W
SFO2 500.132000000 MHz
SI 32768
SF 125.757706 MHz
WDW EM
SSB 0
LB 0
GB 0
PC 1.40



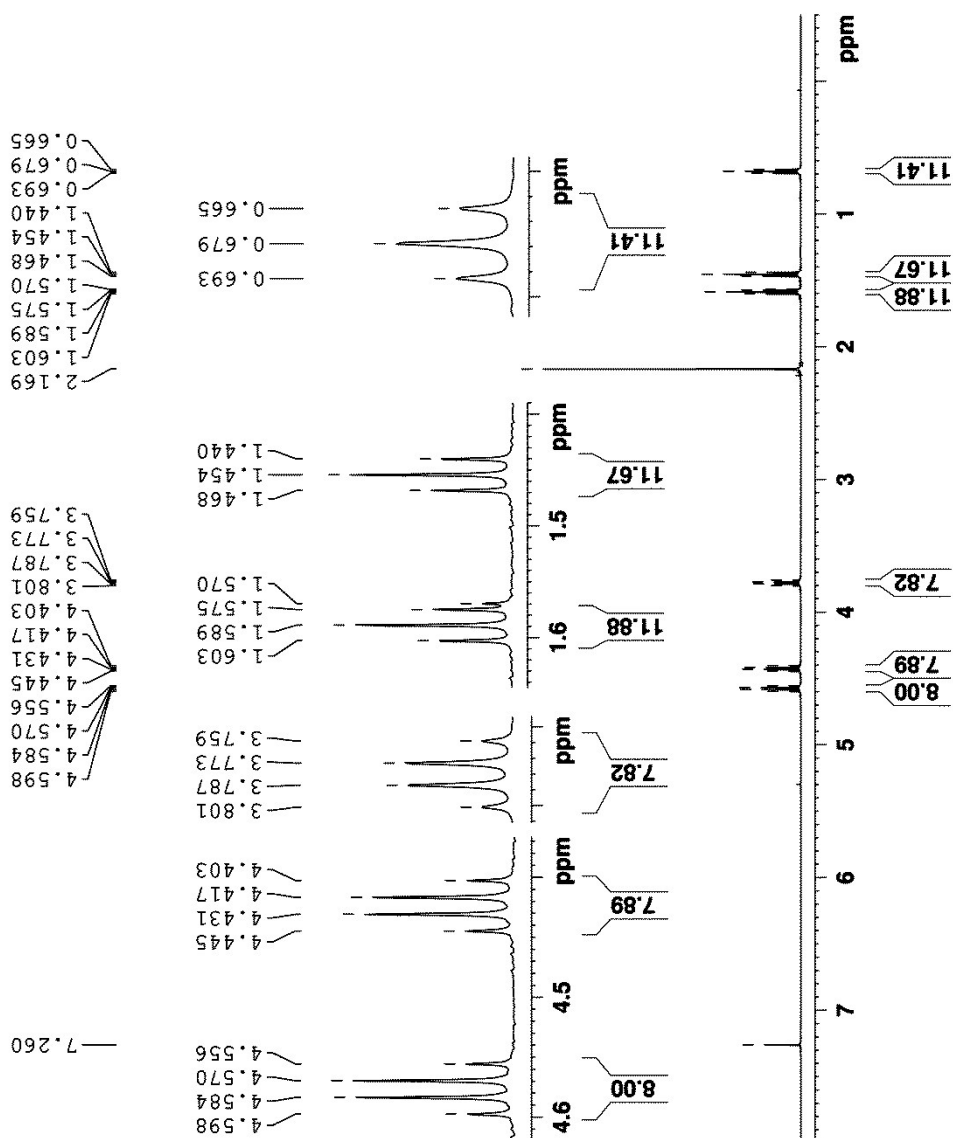
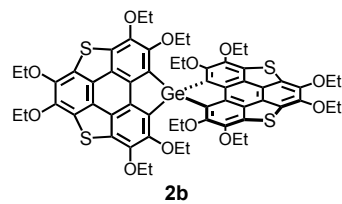
¹H NMR spectrum of 2b in CDCl₃



```

NAME      Gespiro
EXPNO     1
PROCNO    1
Date_     20161214
Time      21.00
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         8
DS         2
F2H1      10330.577 Hz
FIDRES    0.175632 Hz
AQ         3.171923 sec
RG         128
DC         48.400 usec
DE         6.50 usec
TE         299.3 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         12.00 usec
PL         2.00 dB
FLL1       15.3226563 MHz
SFO1       500.132988 MHz
SF         500.132988 MHz
SE         0.30 Hz
WDW         EM
SSB         0
LB         0
GB         0
PC         1.00
  
```





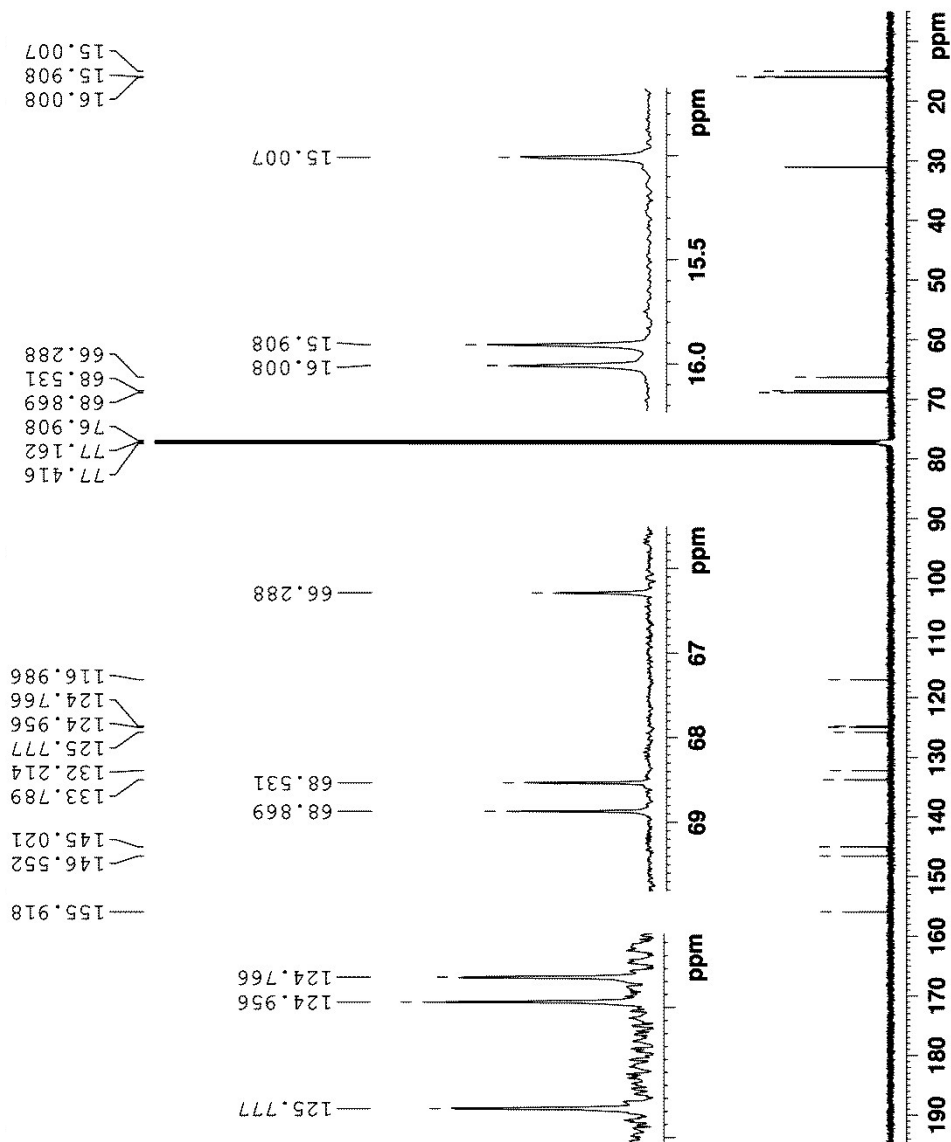
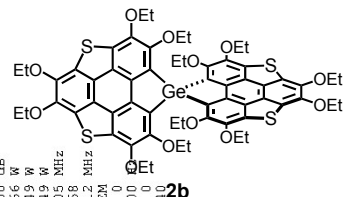
NAME	EXPNO	PROCNO	Date_	Time	INSTRUM	PROBHD	PULPROG	TD0	SOLVENT	NS	DS	SWH	FIDRES	RG	DM	DE	TE	DELTA	DELTA1	DELTA2	TD1	TD0	TD1	TD0
Caspino	13	1	20161214	21.57	5 mm	PABBO B8-	zgpg30	65536	CDCl3	1020	4	29761.904 Hz	0.454131 Hz	1.1010548 sec	203	16,800 usec	36.50 usec	2.00000000 sec	0.03000000 sec	0.03000000 sec	1	1	1	1

```
===== CHANNEL #1 =====
NUC1      13C
P1         10.00 usec
P11        -0.30 dB
PL1W       93.6541290 W
SF01       125.7703643 MHz
```

```
===== CHANNEL f2 =====
CPDPRG2      waitz16
NPGC2        1H
PCPD2        80.00 usec
PL2          1.75 dB
PL12         19.50 dB
PL13         19.50 dB
PL2W         18.63472366 W
PL12W        0.31840409 W
PL13W        0.31840409 W
SFG2         500.1320005 MHz
```

SI	32768	
SE	125.7577712	MHz
SF		
WDW	EM	
SSB	0	
LB	1.00	
GB	0	
PC	1.40	

2b



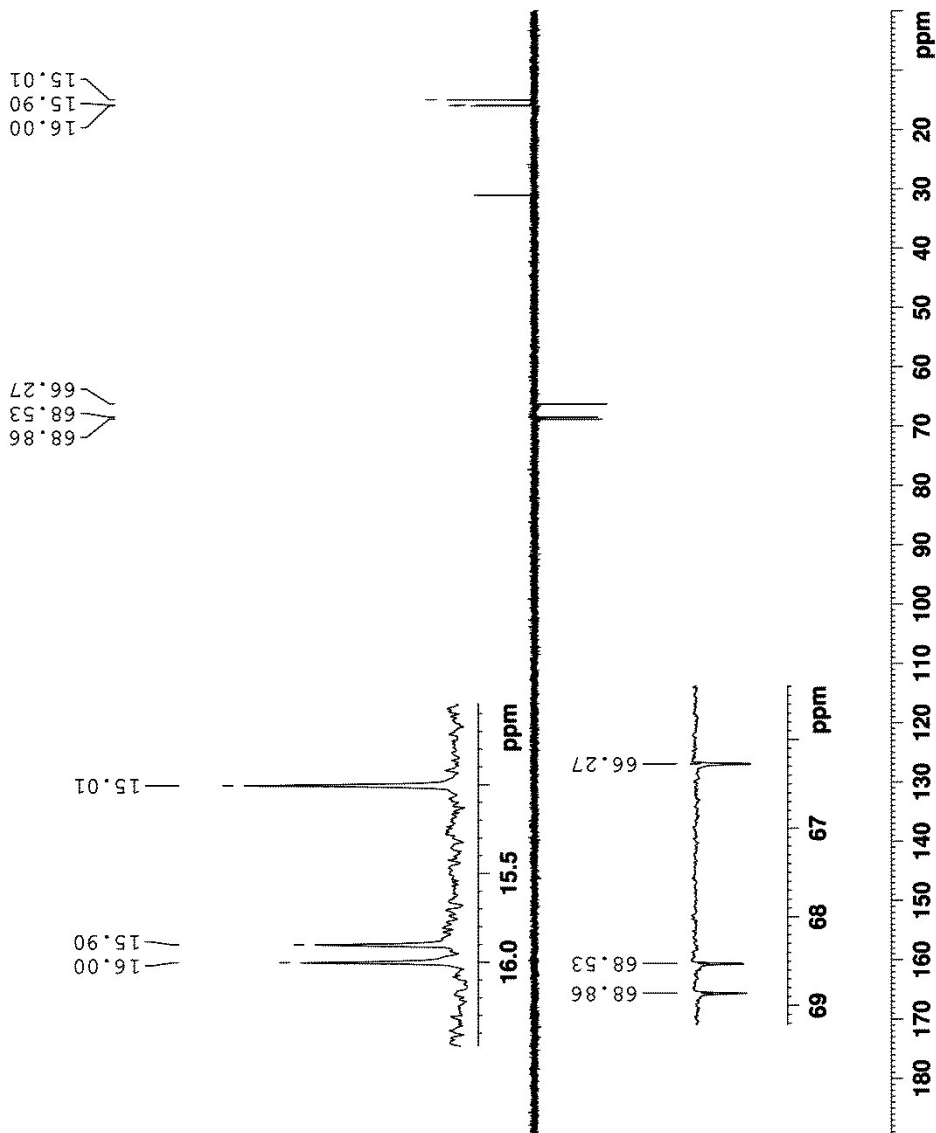
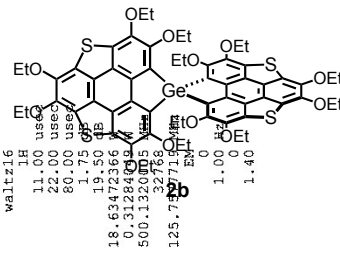
DEPT-135 of of 2b in CDCl₃



NAME 15mcl25kh
EXPNO 135
PROCNO 1
Date_ 20161214
Time 22.05
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG dept135
TD 65536
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DE 16.800 usec
TE 295.8 K
CNST2 145.0000000
D1 2.00000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
TD0 1

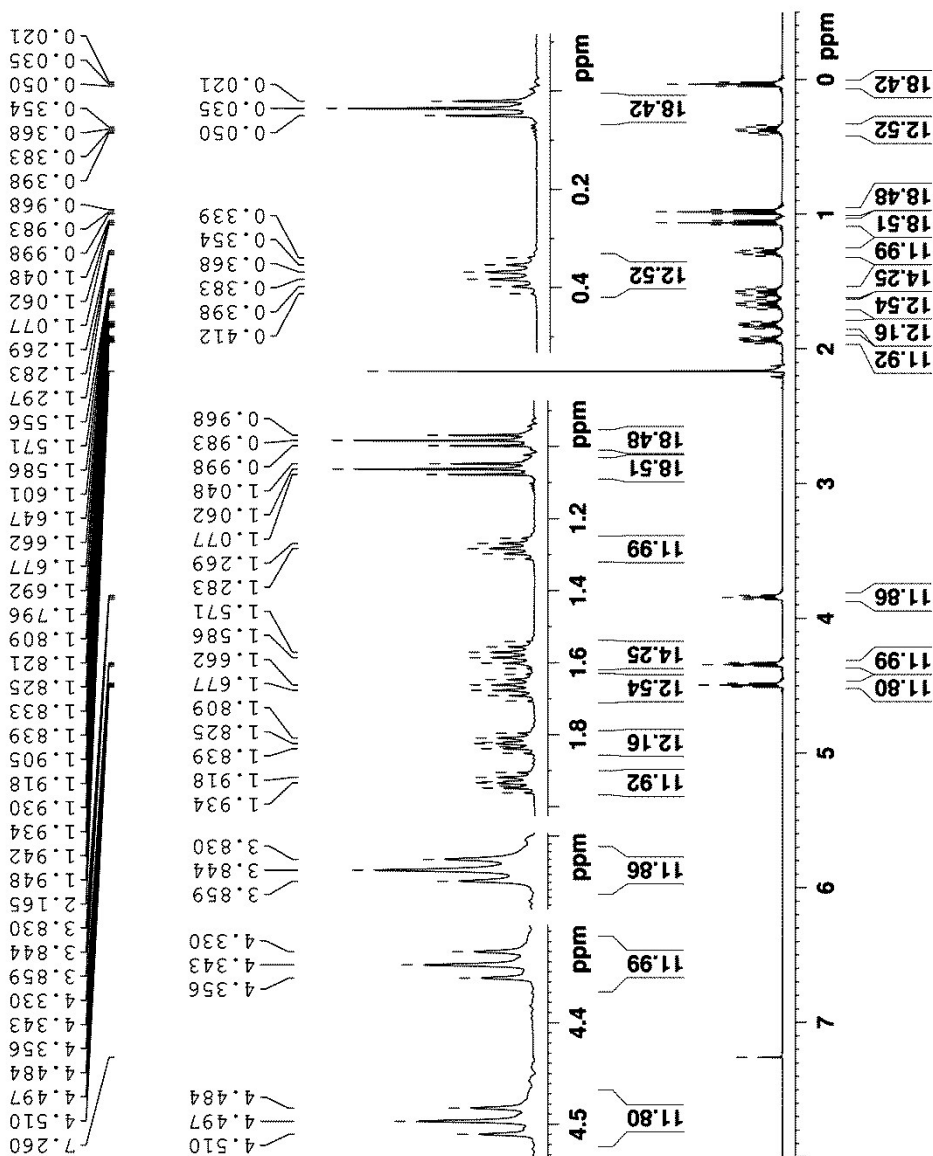
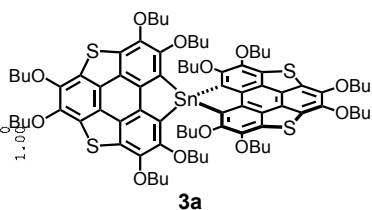
CHANNEL f1
NUC1 13C
P1 10.00 usec
P2 20.00 usec
PL1 -0.30 dB
PL1W 93.66541290 W
SFO1 125.7703643 MHz

CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
P3 11.00 usec
P4 22.00 usec
PCPD2 80.00 usec
PL2 1.75 dB
PL12 19.50 dB
PL2W 18.63472366 W
PL12W 0.31284014 W
SFO2 500.132005 MHz
SI 32768
SF 125.7703643 MHz
WDW ENT
SSB 0
LB 0
GB 0
PC 1.40



NAME	A12csc04Rht
EXPNO	16010901
PROCNO	1
Date_	20160108
Time	21.43
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	10330.575 Hz
FIDRES	0.157623 Hz
AQ	3.171923 sec
RMG	64
RG	48.400 usec
DE	6.50 usec
TE	301.3 K
D1	1.00000000 sec
ID0	1

CHANNEL	f1	f2	f3	f4	f5	f6	f7	f8	f9	f10	f11	f12	f13	f14	f15	f16	f17	f18	f19	f20	f21	f22	f23	f24	f25	f26	f27	f28	f29	f30	f31	f32	f33	f34	f35	f36	f37	f38	f39	f40	f41	f42	f43	f44	f45	f46	f47	f48	f49	f50	f51	f52	f53	f54	f55	f56	f57	f58	f59	f60	f61	f62	f63	f64	f65	f66	f67	f68	f69	f70	f71	f72	f73	f74	f75	f76	f77	f78	f79	f80	f81	f82	f83	f84	f85	f86	f87	f88	f89	f90	f91	f92	f93	f94	f95	f96	f97	f98	f99	f100	f101	f102	f103	f104	f105	f106	f107	f108	f109	f110	f111	f112	f113	f114	f115	f116	f117	f118	f119	f120	f121	f122	f123	f124	f125	f126	f127	f128	f129	f130	f131	f132	f133	f134	f135	f136	f137	f138	f139	f140	f141	f142	f143	f144	f145	f146	f147	f148	f149	f150	f151	f152	f153	f154	f155	f156	f157	f158	f159	f160	f161	f162	f163	f164	f165	f166	f167	f168	f169	f170	f171	f172	f173	f174	f175	f176	f177	f178	f179	f180	f181	f182	f183	f184	f185	f186	f187	f188	f189	f190	f191	f192	f193	f194	f195	f196	f197	f198	f199	f200	f201	f202	f203	f204	f205	f206	f207	f208	f209	f210	f211	f212	f213	f214	f215	f216	f217	f218	f219	f220	f221	f222	f223	f224	f225	f226	f227	f228	f229	f230	f231	f232	f233	f234	f235	f236	f237	f238	f239	f240	f241	f242	f243	f244	f245	f246	f247	f248	f249	f250	f251	f252	f253	f254	f255	f256	f257	f258	f259	f260	f261	f262	f263	f264	f265	f266	f267	f268	f269	f270	f271	f272	f273	f274	f275	f276	f277	f278	f279	f280	f281	f282	f283	f284	f285	f286	f287	f288	f289	f290	f291	f292	f293	f294	f295	f296	f297	f298	f299	f300	f301	f302	f303	f304	f305	f306	f307	f308	f309	f310	f311	f312	f313	f314	f315	f316	f317	f318	f319	f320	f321	f322	f323	f324	f325	f326	f327	f328	f329	f330	f331	f332	f333	f334	f335	f336	f337	f338	f339	f340	f341	f342	f343	f344	f345	f346	f347	f348	f349	f350	f351	f352	f353	f354	f355	f356	f357	f358	f359	f360	f361	f362	f363	f364	f365	f366	f367	f368	f369	f370	f371	f372	f373	f374	f375	f376	f377	f378	f379	f380	f381	f382	f383	f384	f385	f386	f387	f388	f389	f390	f391	f392	f393	f394	f395	f396	f397	f398	f399	f400	f401	f402	f403	f404	f405	f406	f407	f408	f409	f410	f411	f412	f413	f414	f415	f416	f417	f418	f419	f420	f421	f422	f423	f424	f425	f426	f427	f428	f429	f430	f431	f432	f433	f434	f435	f436	f437	f438	f439	f440	f441	f442	f443	f444	f445	f446	f447	f448	f449	f450	f451	f452	f453	f454	f455	f456	f457	f458	f459	f460	f461	f462	f463	f464	f465
---------	----	----	----	----	----	----	----	----	----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------	------



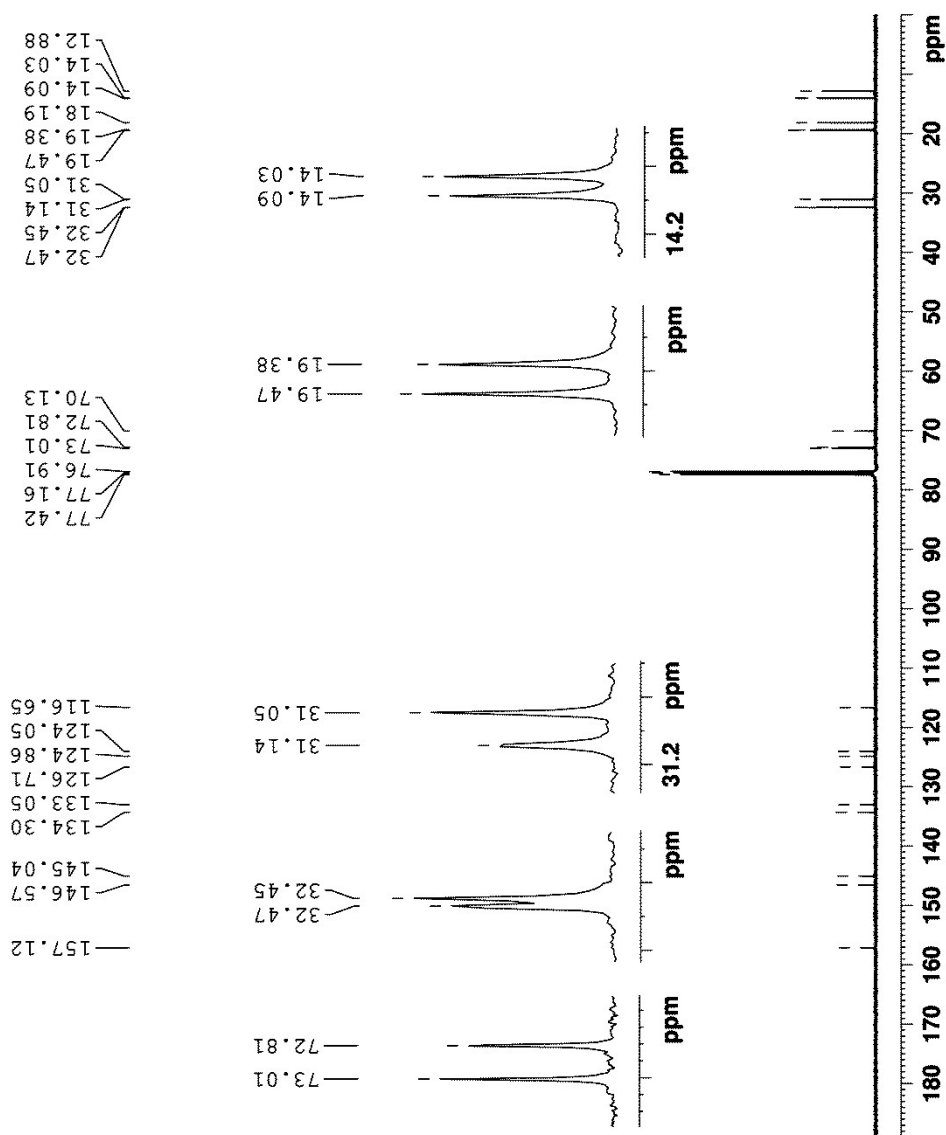
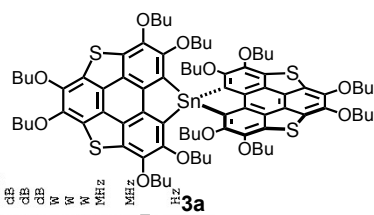
¹³C NMR spectrum of 3a in CDCl₃



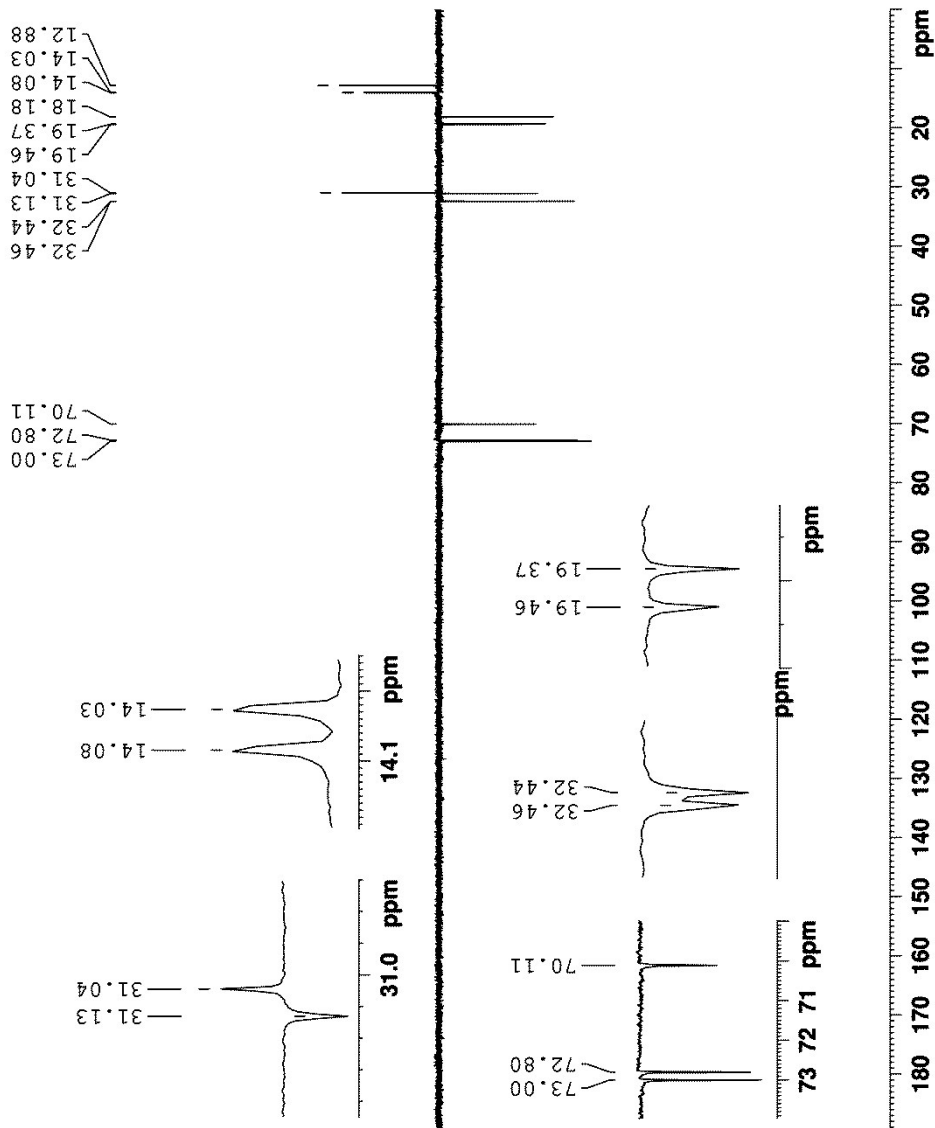
NAME AL2rc040kb
EXPNO 16010902
PROCNO 1
Date_ 20160108
Time_ 21:56
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 240
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010348 sec
RG 203
DW 16.800 usec
DE 36.53 usec
TE 300.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 10.00 usec
PL1 -0.30 dB
PL1W 93.66541290 W
SF01 125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.75 dB
PL12 19.50 dB
PL13 18.63412320 dB
PL14 0.31284049 W
PL12W 0.31284049 W
PL13W 500.1320005 MHz
SF02 327.68 MHz
SI 125.7577712 MHz
SF 0
WDW EM
SSB 0
LB 0
GB 0
PC 1.40



DEPT-135 of of 3a in CDCl₃



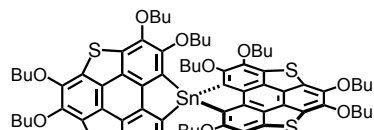
NAME AL2rc040kh
 EXPNO 16010903
 PROCNO 1
 Date_ 20160108
 Time 22.02
 INSTRUM spect
 FROBHD 5 mm PABBO BB
 PULPROG dept135
 TD 65536
 SOLVENT CDCl3
 NS 97
 DS 4
 SWH 29761.904 Hz
 FIDRES 0.454131 Hz
 AQ 1.1010548 sec
 RG 203
 DW 16.800 usec
 DE 6.50 usec
 IE 301.7 K
 CNST2 145.0000000 sec
 D1 2.00000000 sec
 D2 0.0034828 sec
 D12 0.00062000 sec
 TD0 1

CHANNEL f1
 NUC1 13C
 P1 10.00 usec
 PL1 0.00 dB
 F1 93.66541230 MHz
 PL1W 0.30 dB
 SFO1 125.7703643 MHz

CHANNEL f2
 waitz16
 NUC2 1H
 P2 11.00 usec
 PL2 0.00 dB
 F2 400.1461000 MHz
 SFO2 500.1320055 MHz

CPDPRG2 waitz16
 NUC2 1H
 P3 11.00 usec
 PL3 0.00 dB
 F3 400.1461000 MHz
 SFO3 500.1320055 MHz

18.63472366 Hz
 0.31284199 MHz
 500.1320055 MHz
 32768 Hz
 125.7577723 MHz
 0 Hz
 1.00 Hz
 0
 1.40
 PC



¹¹⁹Sn NMR spectrum of 3a in CDCl₃



```

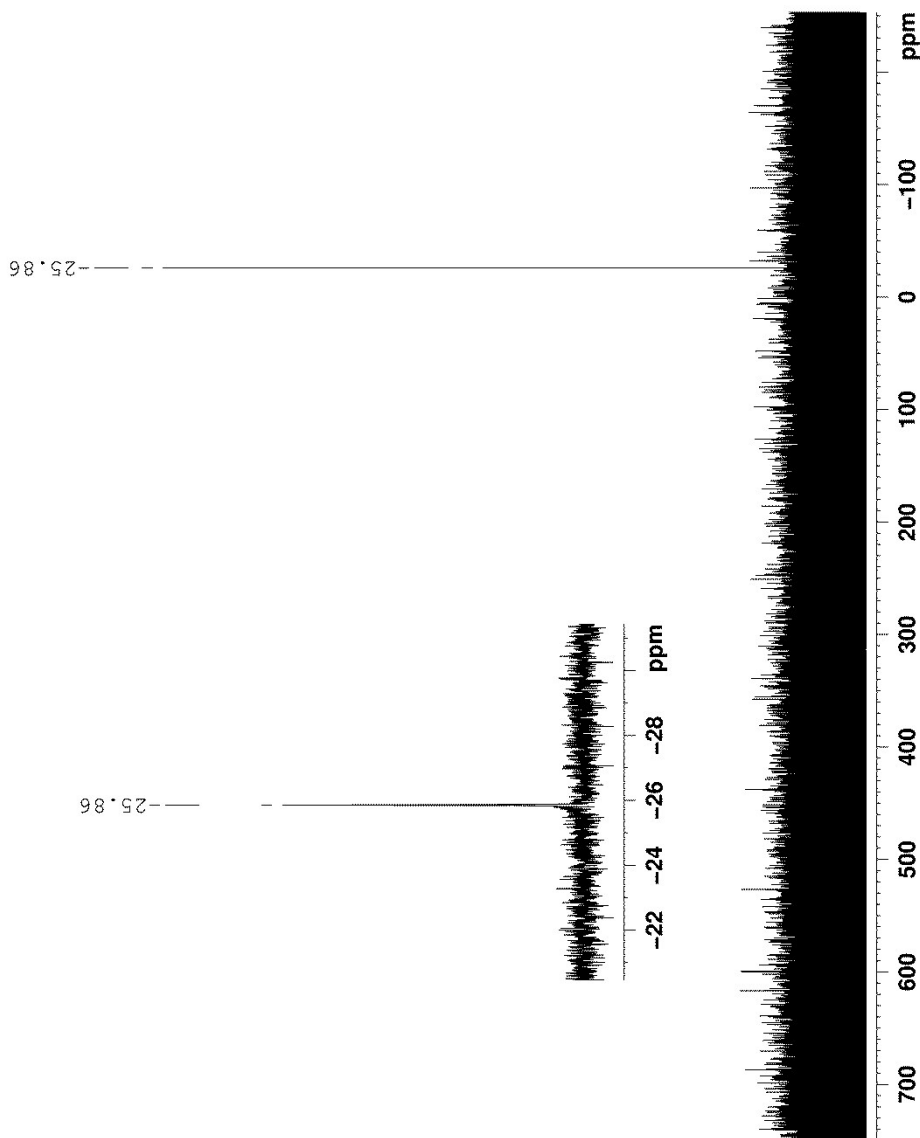
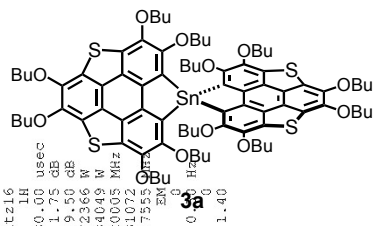
NAME      Al2rc040kh
EXPNO     16010904
PROCNO    1
Date_     20160108
Time      22.11
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD        320000
SOLVENT   CDCl3
NS        80
DS        4
SWH        187500.000 Hz
FIDRES     0.536938 Hz
AQ          0.8533834 sec
RG          303
DW          2.667 usec
DE          6.50 usec
TE         301.5 K
C1         4.00000000 sec
D11        0.03000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1       119Sn
P1         10.50 usec
PL1        1.50 dB
SFO1       186.5462634 MHz
  
```

```

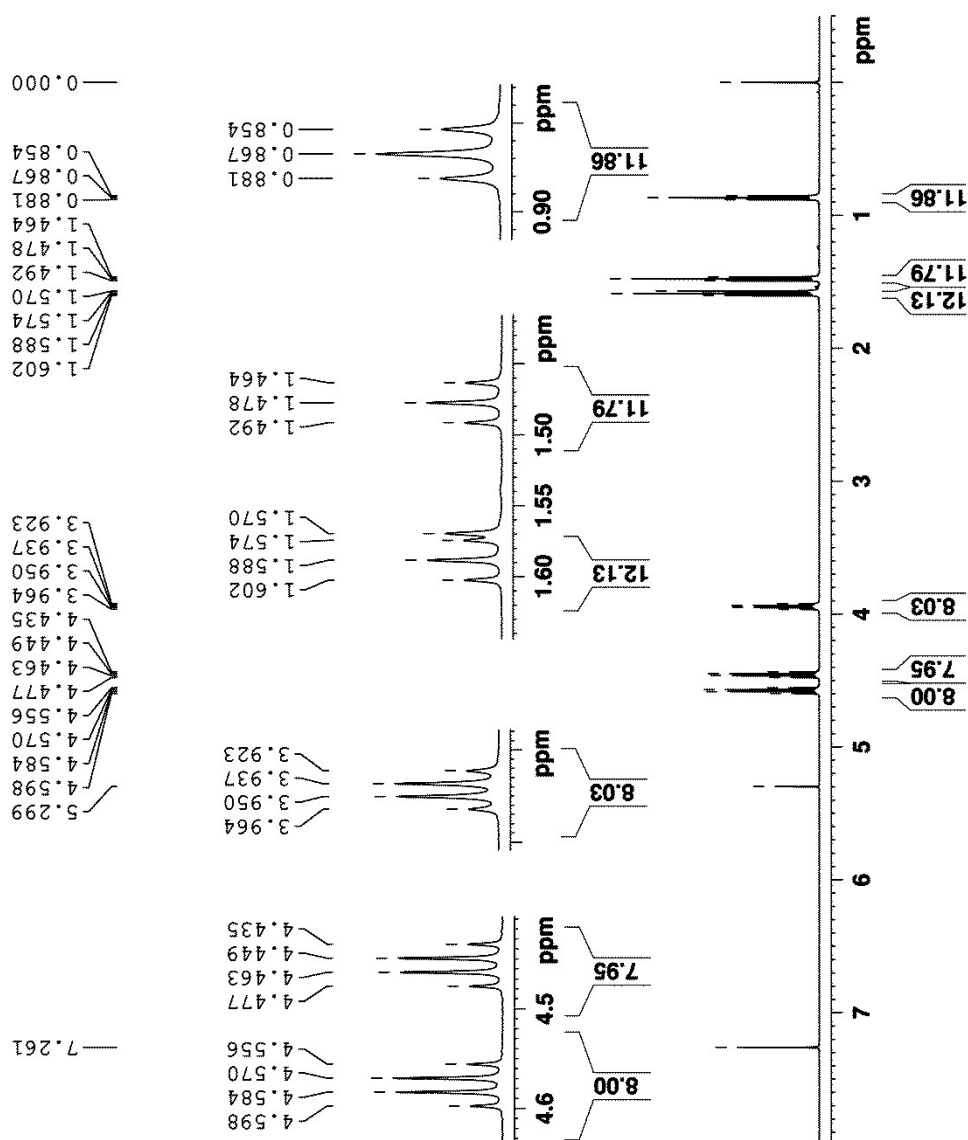
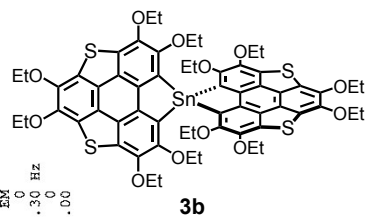
===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2        1H
PCPD2       80.00 usec
PL2         1.70 dB
PL12        19.50 dB
PL12W       18.63472366 W
SFO2        0.31284049 MHz
SI          131072
SF          186.5017555 MHz
WDW          EM
SSB          0
GB          0
PC          1.40
  
```



¹H NMR spectrum of 3b in CDCl₃



NAME Snapiro
EXPNO 1
PROCNO 1
Date_ 20161026
Time 14.55
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 8
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.171923 sec
RG 128
DW 48.400 usec
DE 6.50 usec
TE 299.6 K
D1 1.0000000 sec
TD0 1
===== CHANNEL f1 =====
NUC1 ¹H
P1 12.00 usec
PL1 0.00 dB
FLLW 15.3226883 MHz
SFO1 500.132688 MHz
SF 500.132688 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



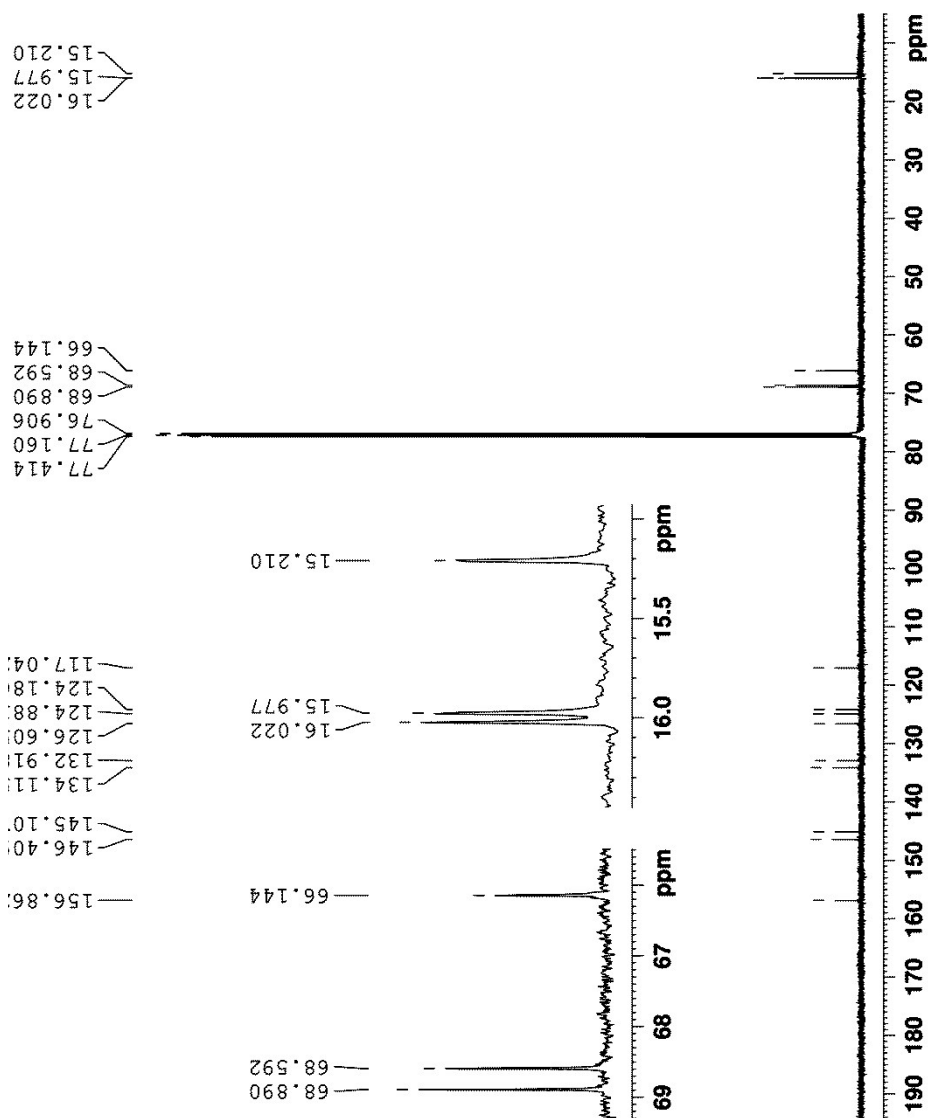
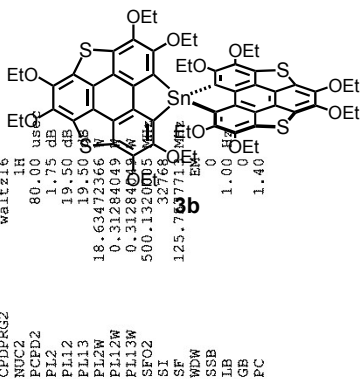
¹³C NMR spectrum of 3b in CDCl₃



NAME Snspiro
EXPNO 13
PROCNO 1
Date_ 20161028
Time 15:24
INSTRUM spect
PROBHD 5 mm PABBO BBO
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 512
DS 4
SWH 28761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 300.6 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

CHANNEL f1
NUC1 ¹³C
P1 10.00 usec
PL1 -0.30 dB
PL1W 93.66541290 W
SFO1 125.7703643 MHz

CHANNEL f2
waitz16

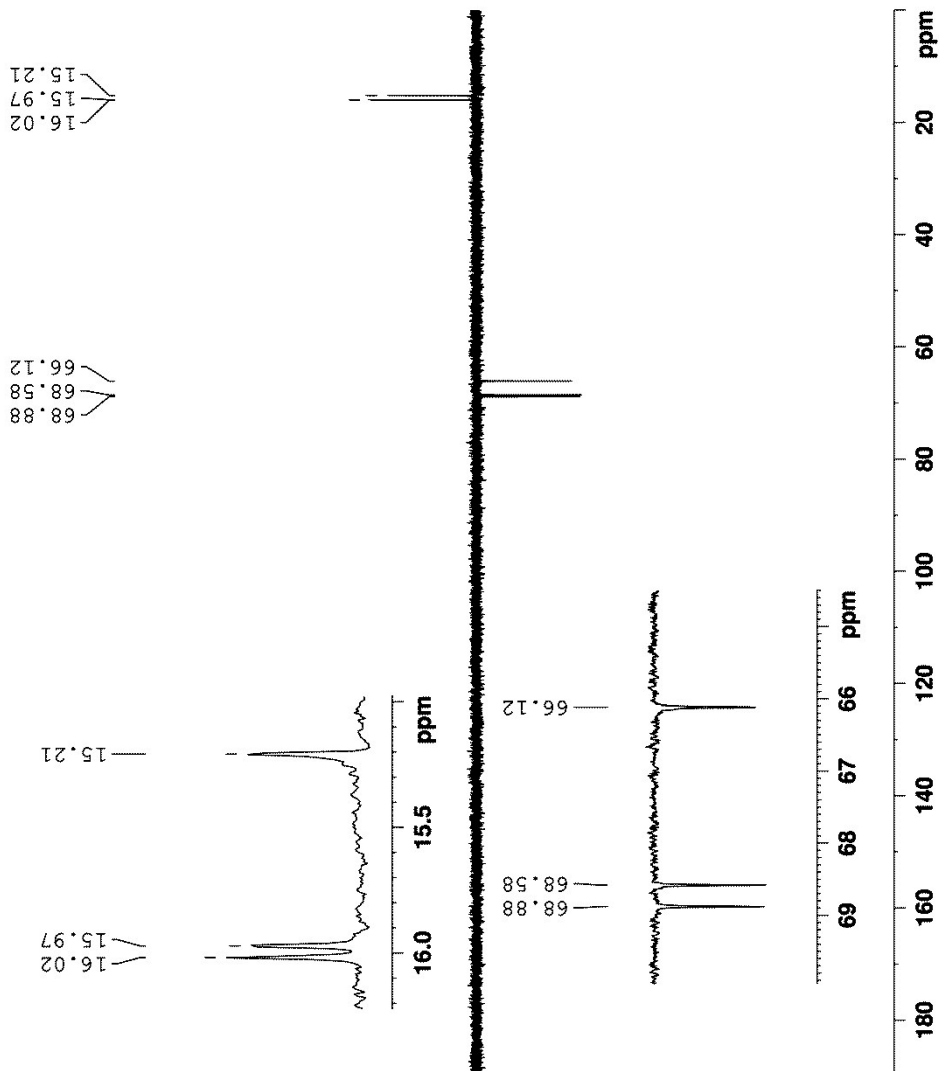
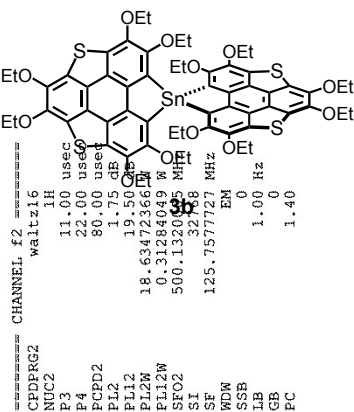


DEPT-135 of of 3b in CDCl₃



NAME 16mcl25kh
EXPNO 135
PROCNO 1
Date_ 20161118
Time 12.27
INSTRUM spect
PROBHD 5 mm F4BBO BB-
PULPROG dept135
ID 65536
SOLVENT CDCl3
NS 60
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DM 16.800 usec
DE 6.50 usec
TE 299.6 K
CNST2 145.0000000
D1 2.80000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
TD0 1

CHANNEL f1
NUC1 13C
P1 10.00 usec
F2 20.00 usec
PL1 -0.30 dB
PL1W 93.66541290 W
SFO1 125.7703643 MHz



¹¹⁹Sn NMR spectrum of 3b in CDCl₃



NAME Snapiro
 EXNO 119
 PROCNO 1
 Date_ 20161107
 Time 21.54
 INSTRUM spect
 PROBD 5 mm PABBO BB-
 PULPROG zgpg
 TD 320000
 SOLVENT CDCl₃
 NS 1244
 DS 4
 SWH 187500.000 Hz
 FIDRES 0.585938 Hz
 AQ 0.8533834 sec
 RG 203
 DW 2.667 usec
 DE 6.50 usec
 TE 299.8 K
 IE 4.00000000 sec
 D1 0.03000000 sec
 D11 4
 TDO

===== CHANNEL f1 =====
 NUC1 119Sn
 P1 10.50 usec
 PL1 1.50 dB
 SFO1 186.4964160 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 1.75 dB
 PL12 19.50 dB
 PL12W 18.63472366 W
 PL12W 0.31284049 W
 SFO2 500.1320005 MHz
 SI 131072
 SF 186.5017555 MHz
 WDW EM
 SSB 0
 LB 0.30
 GB 1.40
 PC

