Supplementary Information for Ferroelectric Columnar Assemblies Arising from Bowl-to-Bowl Invertible Aromatic Cores

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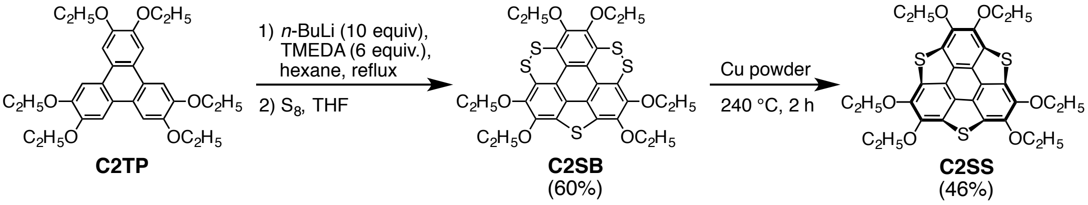
1. General consideration for the synthesis and characterization

NMR spectra were recorded on a Bruker AVANCE500 spectrometer (^{1}H NMR, 500 MHz; ^{13}C NMR, 126 MHz) and a Bruker AVANCE400 spectrometer (^{1}H NMR, 400 MHz; ^{13}C NMR, 100 MHz). Chemical shifts for ^{1}H 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 77.00). The data are presented as follows: chemical shift, multiplicity (t = triplet, q = quartet, quint = quintet, m = multiplet and/or multiple resonances), coupling constant in hertz (Hz), and integration. Mass spectra were obtained with a Bruker AutoflexIII (MALDI-TOF) mass spectrometer. Melting points were measured by Yanaco MP-S3 instrument. Column chromatography was performed using Kanto silica gel 60N. Thin layer chromatography was performed using Merck Silica Gel 60 F254, TLC Plates.

2. Synthetic procedures for all compounds

Synthesis of C2SS



N,*N*,*N′*,*N′*-Tetramethylethylenediamine (14.5 mL, 97.3 mmol) was added to a suspension of 2,3,6,7,10,11-hexaethoxytriphenylene (**C2TP**) (8.02 g, 16.3 mmol) in hexane (380 mL). *n*-BuLi in hexane (2.66 M, 62 mL, 165 mmol) was added dropwise to the suspension for 5 min at room temperature, then the mixture was refluxed for 3.5 hours. After cooling to room temperature, volatiles were removed under reduced pressure. THF (300 mL) was added to the resulting solid and then cooled below −60 °C. To the mixture, sulfur powder (8.36 g, 261 mmol) was added in one portion and the mixture was stirred for 30 min at the same temperature. Then the mixture was allowed to warm to room temperature and stirred for 7 hours. After stirring, aqueous NH_4Cl was added to the mixture, and the reaction mixture was extracted with dichloromethane. The extract was dried over anhydrous magnesium sulfate and concentrated under reduced pressure to give a crude product. The products were purified by silica gel column chromatography (hexane/chloroform) to give thienotriphenylenobis(dithiin) **C2SB** (6.31 g, 9.75 mmol, 60%) as a yellow solid.

Copper powder (11.9 g, 187 mmol) was added to **C2SB** (4.04 g, 6.24 mmol), then the mixture was stirred at 240 °C for 2 hours. After cooling to room temperature, dichloromethane was added to the reaction mixture. Insoluble solids were removed by filtration, then volatiles were removed under reduced pressure to obtain crude products. The products were purified by silica gel column chromatography (hexane/dichloromethane) to give **C2SS** (1.69 g, 2.90 mmol, 46%) as a pale yellow solid. Unreacted starting material **2** (1.57 g, 2.43 mmol, 39%) was also recovered.

Compounds data

C2SB: yellow solid, mp. 148–150 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.48–1.53 (m, 18 H), 4.32 (q, *J* = 6.8 Hz, 8H), 4.48 (q, *J* = 6.8 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 15.90 (CH_3), 15.92 (CH_3), 16.10 (CH_3), 69.19 (CH_2), 70.32 (CH_2), 70.62 (CH_2), 122.10 (C), 122.48 (C), 124.32 (C), 126.30 (C), 128.49 (C), 129.05 (C), 146.59 (C), 147.07 (C), 148.66 (C); MS (MALDI) *m*/*z* calcd for $C_{30}H_{30}O_6S_5$ (M^+) 646.1, found: 646.0; elemental analysis calcd for $C_{30}H_{30}O_6S_5$: C, 55.70; H, 4.67, found: C, 55.79; H, 4.49.

C2SS: pale yellow solid, 204-205 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.55 (t, *J* = 6.8 Hz, 18 H), 4.48

(q, J = 6.8 Hz, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 15.99 (CH_3), 69.16 (CH_2), 129.97 (C), 130.81 (C), 146.40 (C); MS (MALDI) *m*/*z* calcd for $C_{30}H_{30}O_6S_3$ (M^+) 582.1, found: 582.3; elemental analysis calcd for $C_{30}H_{30}O_6S_3$: C, 61.83; H, 5.19, found: C, 61.72; H, 5.03.

Synthesis of trithiasumanenes bearing long alkyl chains C*n*SS

C*n*SS derivatives (n = 6, 8, 10, and 16) were synthesized by the following general procedure described in the main text (see Methods section).

Synthesis of C6SS

For the dealkylation, **C2SS** (150 mg, 0.26 mmol) in dichloromethane (5 mL) was treated with boron tribromide in dichloromethane (1.0 mol L^{-1}, 2.6 mL, 2.6 mmol). 1-Bromohexane (0.3 mL, 1.54 mmol), potassium carbonate (305 mg, 2.21 mmol) and DMF (3 mL) were used for the realkylation step. **C6SS** (166 mg, 0.18 mmol, 70%) was obtained as a pale yellow solid. mp. 121 ºC, ^{1}H NMR (400 MHz; $CDCl_3$) δ 0.94 (t, J = 7.2 Hz, 18H), 1.41 (br, 24H), 1.58 (quint, J = 7.2 Hz, 12H), 1.90 (quint, J = 6.4 Hz, 12H), 4.40 (t, J = 6.4 Hz, 12H); ^{13}C NMR (101 MHz; $CDCl_3$) δ 14.21 (CH_3), 22.81 (CH_2), 25.89 (CH_2), 30.38 (CH_2), 31.81 (CH_2), 73.71 (CH_2), 130.26 (C), 130.97 (C), 146.76 (C); MS (MALDI) *m*/*z* calcd for $C_{54}H_{78}O_6S_3$ (M^+) 918.5, found: 919.0; elemental analysis calcd for $C_{66}H_{102}O_6S_3$: C, 70.54; H, 8.55, found: C, 70.14; H, 8.55.

Synthesis of C8SS

For the dealkylation, **C2SS** (150 mg, 0.26 mmol) in dichloromethane (5 mL) was treated with boron tribromide in dichloromethane (1.0 mol L^{-1}, 2.6 mL, 2.6 mmol). 1-Bromooctane (0.35 mL, 2.02 mmol), potassium carbonate (350 mg, 2.54 mmol) and DMF (3 mL) were used for the realkylation step. **C8SS** (117 mg, 0.11 mmol, 42%) was obtained as a pale yellow solid. mp. 92.5 ºC, ^{1}H NMR (400 MHz; $CDCl_3$) δ 0.93 (t, 18H), 1.43 (br, 48H), 1.62 (quint, J = 7.2 Hz, 12H), 1.93 (quint, J = 7.2 Hz, 12H), 4.41 (t, J = 6.8 Hz, 12H);^{13}C NMR (101 MHz; $CDCl_3$) δ 14.26 (CH_3), 22.86 (CH_2), 29.27 (CH_2), 29.51 (CH_2), 29.64 (CH_2), 30.47 (CH_2), 32.05 (CH_2), 73.67 (CH_2), 129.96 (C), 130.70 (C), 146.64 (C); MS (MALDI) *m*/*z* calcd for $C_{66}H_{102}O_6S_3$ (M^+) 1086.7, found: 1087.2; elemental analysis calcd for $C_{66}H_{102}O_6S_3$: C, 72.88; H, 9.45, found: C, 72.60; H, 9.70.

Synthesis of C10SS

For the dealkylation, **C2SS** (200 mg, 0.34 mmol) in dichloromethane (8 mL) was treated with boron tribromide in dichloromethane (1.0 mol L^{-1}, 3.5 mL, 3.5 mmol). 1-Bromodecane (0.38 mL, 1.84 mmol), potassium carbonate (260 mg, 1.88 mmol) and DMF (5 mL) were used for the realkylation step. **C10SS** (165 mg, 0.13 mmol, 39%) was obtained as an off-white solid. mp. 83.0 ºC; ^{1}H NMR

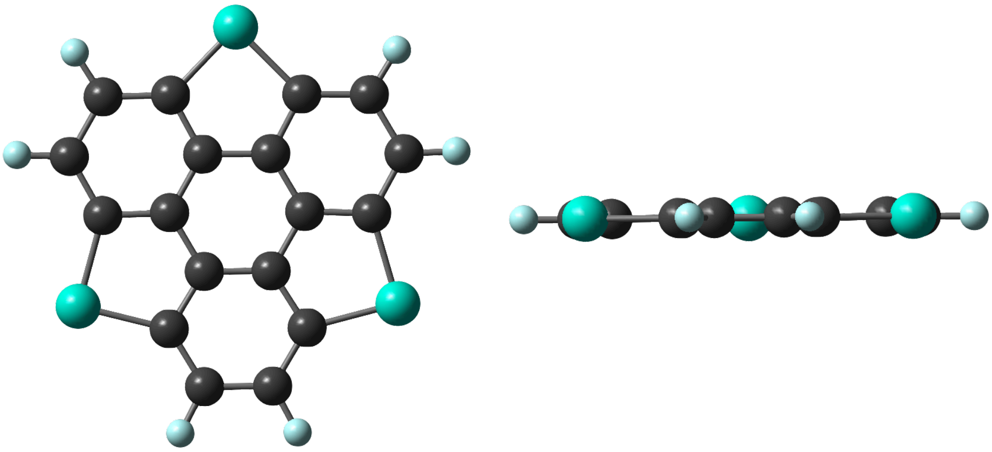
(500MHz; $CDCl_3$) δ 0.89 (t, *J* = 7.2 Hz, 18H), 1.29–1.45 (m, 72H), 1.59 (quint, *J* = 7.2 Hz, 12H), 1.91 (quint, *J* = 7.2 Hz, 12H), 4.39 (t, *J* = 6.4 Hz, 12H);^{13}C NMR (101 MHz; $CDCl_3$) δ 14.26 (CH_3), 22.86 (CH_2), 26.28 (CH_2), 29.55 (CH_2), 29.69 (CH_2), 29.82 (CH_2), 29.87 (CH_2), 30.48 (CH_2), 32.11 (CH_2), 73.64 (CH_2), 129.99 (CH_3), 130.72 (CH_3), 146.66 (CH_3); MS (MALDI) *m*/*z* calcd for $C_{78}H_{126}O_6S_3$ (M^+) 1254.9, found: 1255.5; elemental analysis calcd for $C_{78}H_{126}O_6S_3$: C, 74.59; H, 10.11, found: C, 74.46; H, 10.40.

Synthesis of C16SS

For the dealkylation, **C2SS** (194 mg, 0.33 mmol) in dichloromethane (8 mL) was treated with boron tribromide in dichloromethane (1.0 mol L^{-1}, 3.5 mL, 3.5 mmol). 1-Bromohexadecane (0.47 mL, 1.54 mmol), potassium carbonate (186 mg, 1.35 mmol) and DMF (5 mL) were used for the realkylation step. **C16SS** (73 mg, 0.04 mmol, 12%) was obtained as a white solid. mp. 70.0 ºC; ^{1}H NMR (500MHz; $CDCl_3$) δ 0.88 (t, *J* = 7.1 Hz, 18H), 1.26–1.45 (m, 144 H), 1.57 (quint, *J* = 7.6 Hz, 12H), 1.90 (quint, *J* =7.6 Hz, 12H), 4.39 (t, *J* = 6.4 Hz, 12H); ^{13}C NMR (101 MHz; $CDCl_3$) δ 14.26 (CH_3), 22.68 (CH_2), 22.86 (CH_2), 23.02 (CH_2), 26.27 (CH_2), 26.42 (CH_2), 29.54 (CH_2), 29.68 (CH_2), 29.87 (CH_2), 29.91 (CH_2), 30.46 (CH_2), 31.93 (CH_2), 32.10 (CH_2), 32.27 (CH_2), 73.67 (CH_2), 130.14 (C), 130.85(C), 146.72 (C); MS (MALDI) *m*/*z* calcd for $C_{114}H_{198}O_6S_3$ (M^+) 1759.4, found: 1760.4; elemental analysis calcd for $C_{114}H_{198}O_6S_3$: C, 77.75; H, 11.33, found: C, 77.24; H, 11.40.

3. The optimized structures of triselenasumanene

The geometry optimization of triselenasumanene was performed using Gaussian 09 program[1] at the B3LYP/cc-pVDZ level of theory.

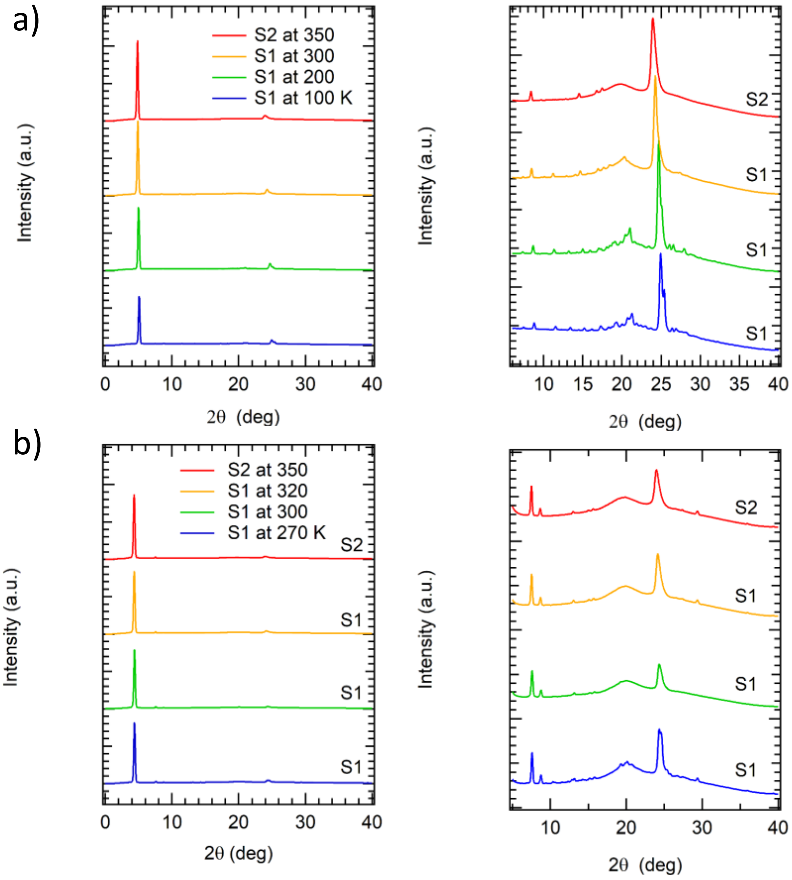


Supplementary Figure 1. The optimized structure of triselenasumanene: top view (left) and side view (right).

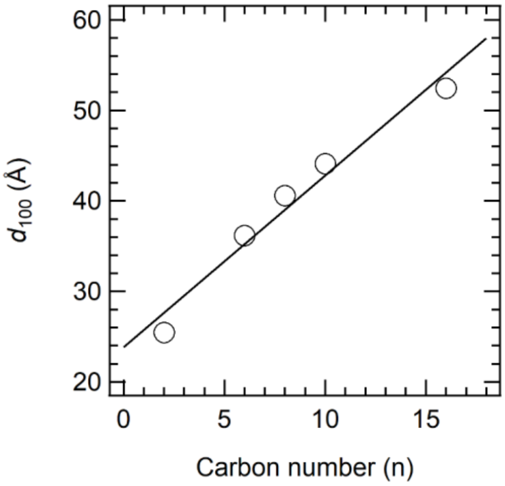
Table S1. Theoretically optimized coordinates of triselenasumanene (Cartesian coordinates in Å)

atom	*x*	*y*	*z*
C	0.710683	3.612464	0
C	-0.710683	3.612464	0
C	-1.435544	2.405058	0
C	-0.697602	1.221221	0
C	0.697602	1.221221	0
C	1.435544	2.405058	0
Se	3.329432	1.922249	0
C	-3.483828	-1.190763	0
C	-2.773144	-2.421702	0
C	-1.365069	-2.445746	0
C	-0.708808	-1.214751	0
C	-1.406409	-0.00647	0
C	-2.800613	0.040689	0
Se	-3.329432	1.922249	0
C	2.773144	-2.421702	0
C	3.483828	-1.190763	0
C	2.800613	0.040689	0
C	1.406409	-0.00647	0
C	0.708808	-1.214751	0
C	1.365069	-2.445746	0
Se	0	-3.844497	0
H	3.353812	-3.34616	0
H	4.574766	-1.231406	0
H	1.220954	4.577566	0
H	-1.220954	4.577566	0
H	-4.574766	-1.231406	0
H	-3.353812	-3.34616	0

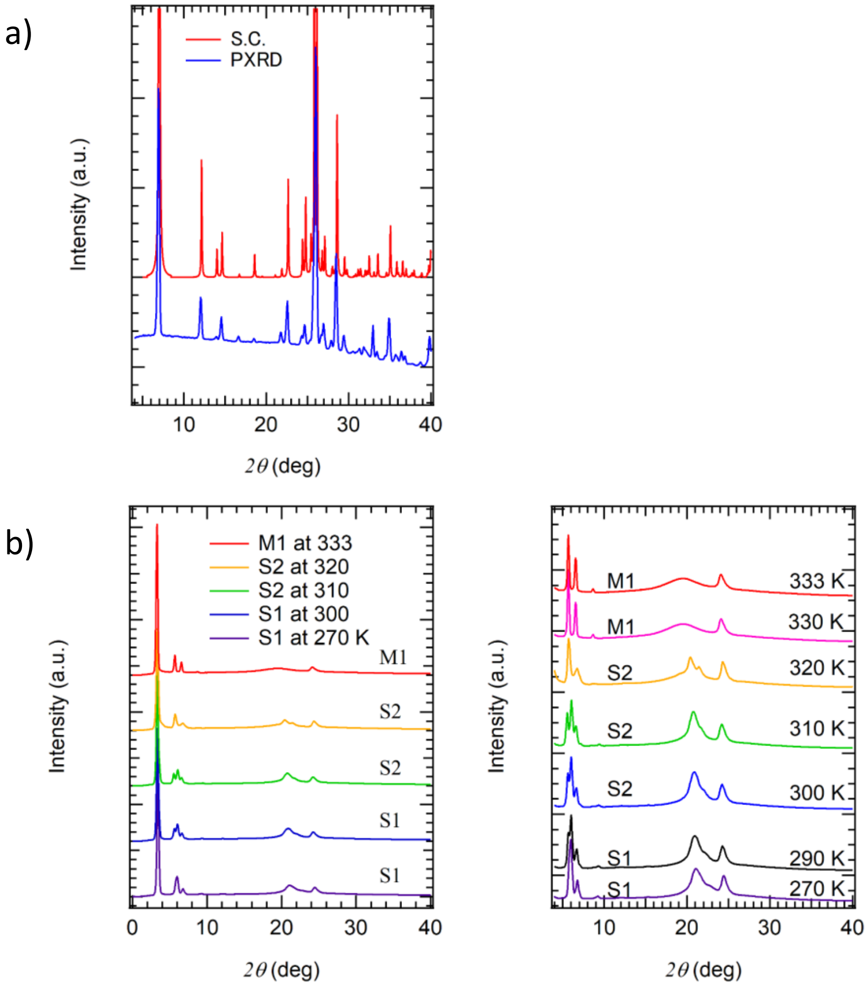
3. PXRD analysis of C*n*SS derivatives



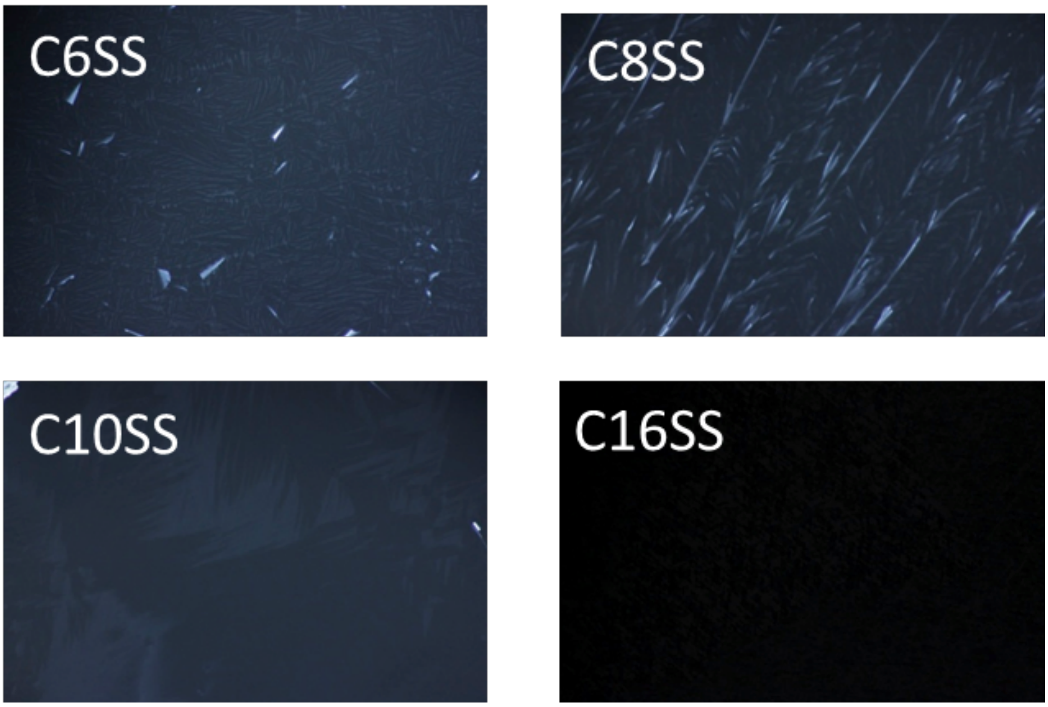
Supplementary Figure 2. Temperature-dependent PXRD patterns of a) **C6SS** and b) **C8SS** at S1 and S2 phases with the index assignments of 100, 110, 200, and 001 reflections. Right figure is expanded PXRD pattern at 2θ range from 5 to 40°.



Supplementary Figure 3. A linear relationship of and the number of carbon atoms of alkyl chains (*n* = 2, 6, 8, 10 and 16) and d_{100} spacing.



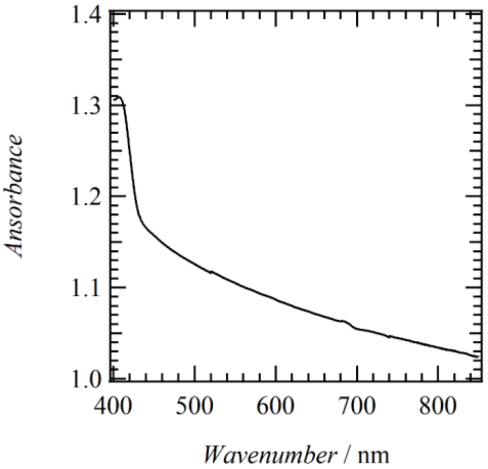
Supplementary Figure 4. Crystal structures of **C2SS** and **C16SS**. a) PXRD pattern (blue) and a simulated one (red) based on the single crystal X-ray crystal structural analysis of **C2SS**. b) Temperature dependent changes in the PXRD patterns at S1, S2 and M1 phases. Right figure shows the magnified diffraction patterns in the range of 2*θ* between 5° and 40°.



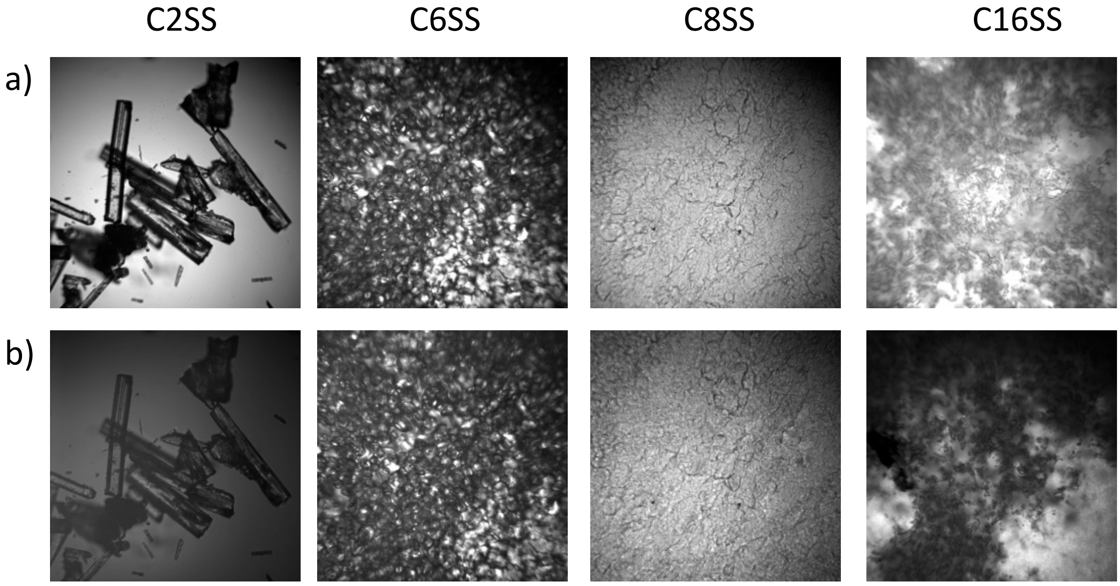
Supplementary Figure 5. POM images of S2 phase of **C6SS**, **C8SS**, **C10SS**, and **C16SS**. Dark images under the cross-Nicol optical arrangement were consistent with the homeotropic orientation of each column.

SHG measurements of C*n*SS.

The intensity of the SHG activity was measured using the Kurtz powder method. Powder samples were sandwiched between glass plates, with a 58 μm gap, and the temperature-dependent SHG signal was measured five times by comparison with sucrose. Temperature–dependent powder SHG measurement was conducted on glass substrate equipped with temperature controller and the SHG intensity was relative magnitude with sucrose. Although the SHG intensity was not strong due to insufficient polar domain orientation in the absence of the application of electric filed and/or poling procedure, the SHG signal could be observed in the ferroelectric phase without the poling process through the phase transition from non-polar paraelectric to polar ferroelectric phase. A femtosecond pulsed beam (wavelength 1400 nm) from an optical parametric amplifier (Spectra-Physics, OPA-800C) pumped by a beam from a Ti:sapphire regenerative amplifier (Spectra-Physics, Spitfire) was used as the light source. The pulse duration was typically 150–200 fs, and the repetition rate was 1 kHz. The average incident power was 0.2 mW. The incident beam was focused by a plano-convex lens (f = 120 mm). The output SHG signals at wavelength of 700 nm were detected by a photomultiplier tube (Hamamatsu, Model SR250) and processed using a boxcar average (Stanford Research, Model SR250).

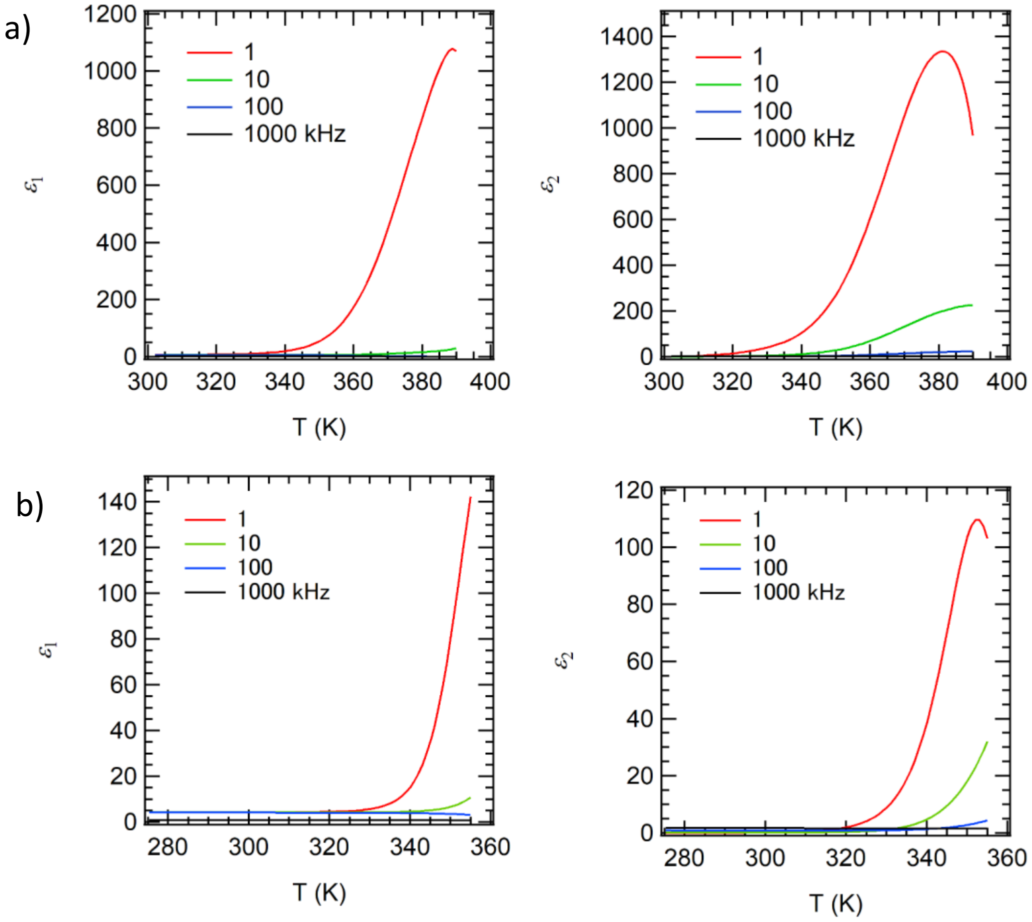


Supplementary Figure 6. Solid state diffuse scattering reflection spectra of C10SS at 298 K to examine the resonance energy region for SHG measurement.

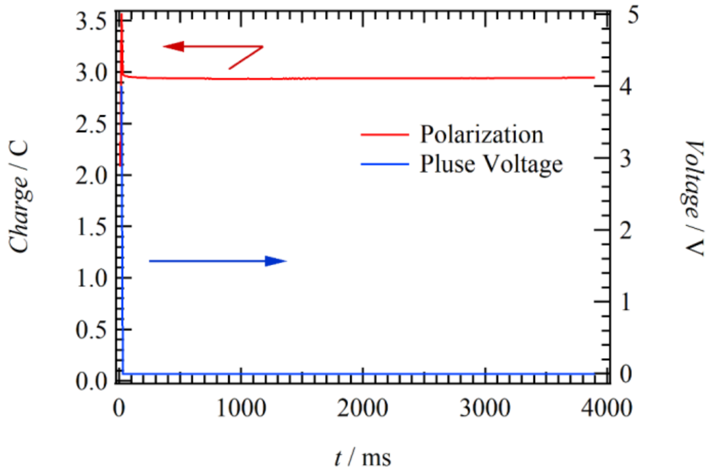


Supplementary Figure 7. POM images of **C*n*SS** crystals a) with polarizer and b) without polarizer. The SHG activity was clearly observed at **C2SS** with an intensity of sucrose.

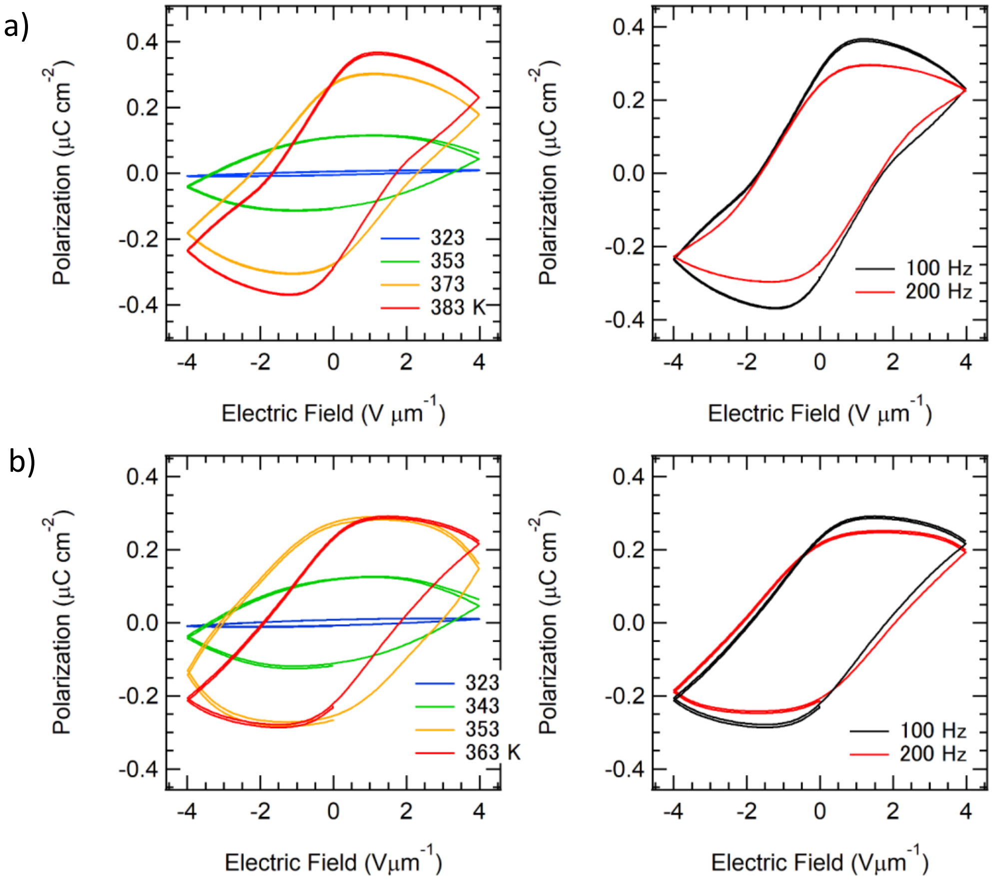
4. Dielectric properties of C*n*SS derivatives



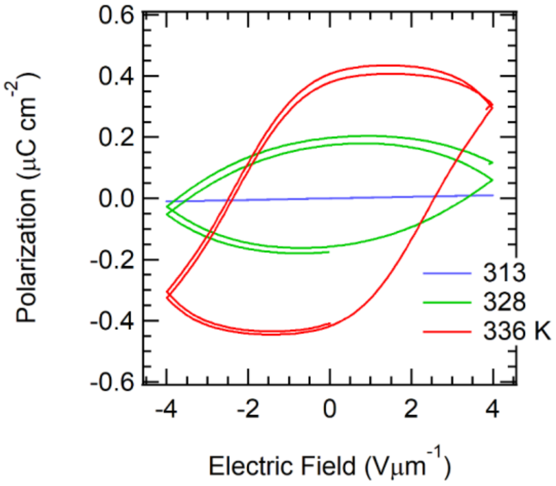
Supplementary Figure 8. Dielectric properties of a) **C6SS** and b) **C8SS**. *T*- and *f*-dependent real part ε_1 (left) and imaginary part ε_2 (right) dielectric constants in the heating process.



Supplementary Figure 9. Time-dependent polarization behavior of **C10SS** at 343K. The pulse voltage was applied at 4 V during 10 msec, then polarization change was monitored.

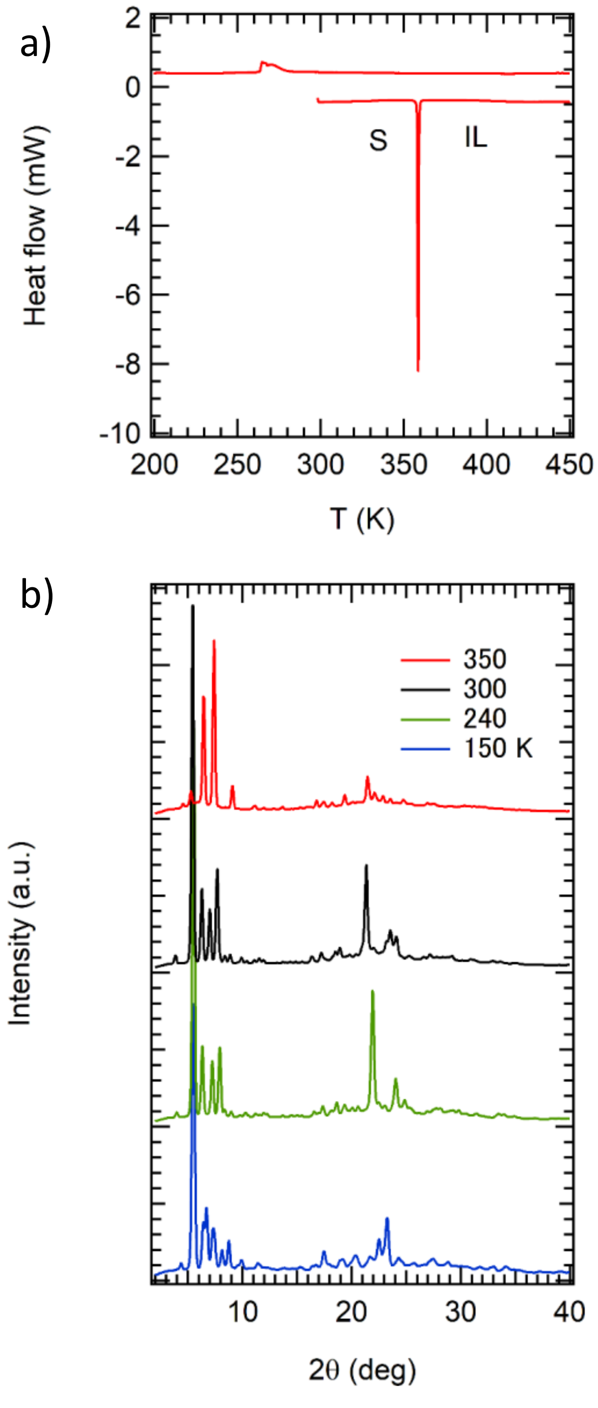


Supplementary Figure 10. *T*-dependent (left) and *f*-dependent (right) *P*-*E* hysteresis curves of a) **C6SS** and b) **C8SS**. The *T*-dependent curves were obtained at *f* = 100 Hz. The *f*-dependent curves were obtained at 383 K for **C6SS** and 363 K for **C8SS**.

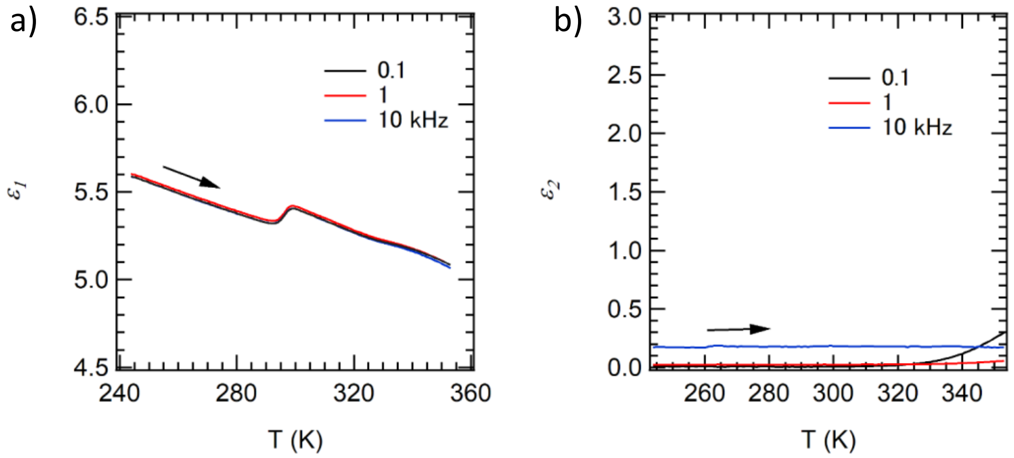


Supplementary Figure 11. *T*-dependent *P*-*E* hysteresis curves at *f* = 100 Hz of **C16SS**.

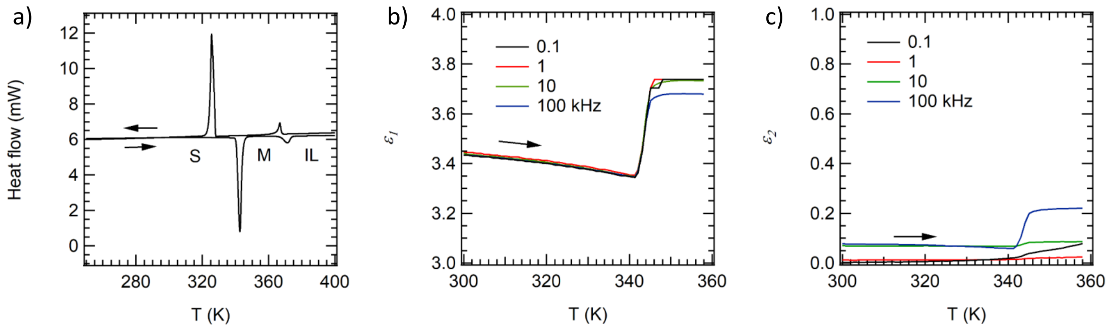
5. Properties of the reference compounds, C4SeS and C6TP



Supplementary Figure 12. Phase transition behavior and crystallinity of **C4SeS**. a) DSC chart in the first cycle and b) *T*-dependent PXRD pattern at $T = 150$, 240, 300, and 350 K.



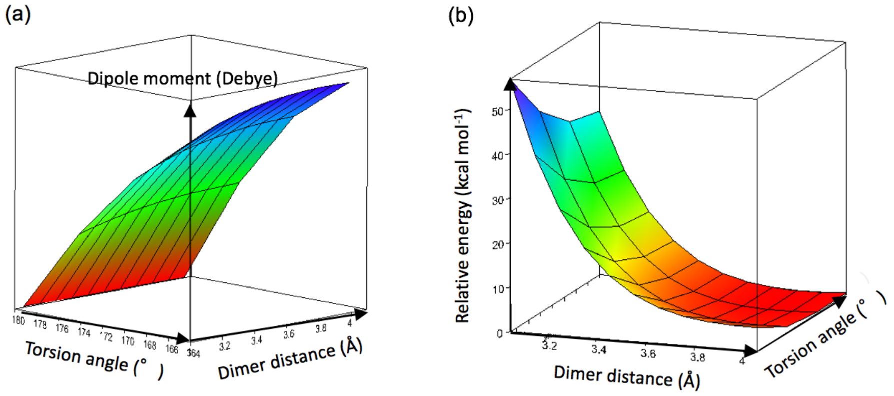
Supplementary Figure 13. Temperature and frequency dependent a) real part dielectric constant ε_1 and b) imaginary part dielectric constant ε_2 of **C4SeS** at $f = 0.1$, 1, and 10 kHz.



Supplementary Figure 14. Phase transition and dielectric behavior of **C6TP**. a) The S-M and M-IL phase transition in DSC chart. *T*- and *f*-dependent b) real part dielectric constant ε_1 and c) imaginary part dielectric constant ε_2 at $f = 0.1$, 1, 10, and 100 kHz.

Theoretical calculations of molecular dynamics.

Theoretical DFT calculation based on a basis function of B3LYP/6-31G(d) was utilized for each **C*n*SS** model calculations. Molecular structures was optimized by fixed angle at C-C-C-S (torsion angle), where the atomic coordinates of central benzene-ring were fixed at the result form the single crystal X-ray structural analysis and the torsion angle of six –OCH_3 groups to the benzene-ring was fixed at zero. The skew angel was obtained at **C1SS** dimer model with a fixed inter-dimer distance along the translational operation of the atomic coordinates, where the upper molecule was rotated and obtained the total energy. The energy minimum was defined as a zero for the relative energy scale at each figure.



Supplementary Figure 15. 3D diagram of stable molecular arrangement. (a) Torsion angle (x) – Dimer distance (y) - dipole moment (z) plot and (b) dimer distance (x) – torsion angle (y) – relative energy (z) plot.

Supplementary References

1. Gaussian 09, Revision B.01, M. J. Frisch, G. W. Trucks, H. B. Schlegel, G. E. Scuseria, M. A. Robb, J. R. Cheeseman, G. Scalmani, V. Barone, B. Mennucci, G. A. Petersson, H. Nakatsuji, M. Caricato, X. Li, H. P. Hratchian, A. F. Izmaylov, J. Bloino, G. Zheng, J. L. Sonnenberg, M. Hada, M. Ehara, K. Toyota, R. Fukuda, J. Hasegawa, M. Ishida, T. Nakajima, Y. Honda, O. Kitao, H. Nakai, T. Vreven, J. A. Montgomery, Jr., J. E. Peralta, F. Ogliaro, M. Bearpark, J. J. Heyd, E. Brothers, K. N. Kudin, V. N. Staroverov, T. Keith, R. Kobayashi, J. Normand, K. Raghavachari, A. Rendell, J. C. Burant, S. S. Iyengar, J. Tomasi, M. Cossi, N. Rega, J. M. Millam, M. Klene, J. E. Knox, J. B. Cross, V. Bakken, C. Adamo, J. Jaramillo, R. Gomperts, R. E. Stratmann, O. Yazyev, A. J. Austin, R. Cammi, C. Pomelli, J. W. Ochterski, R. L. Martin, K. Morokuma, V. G.

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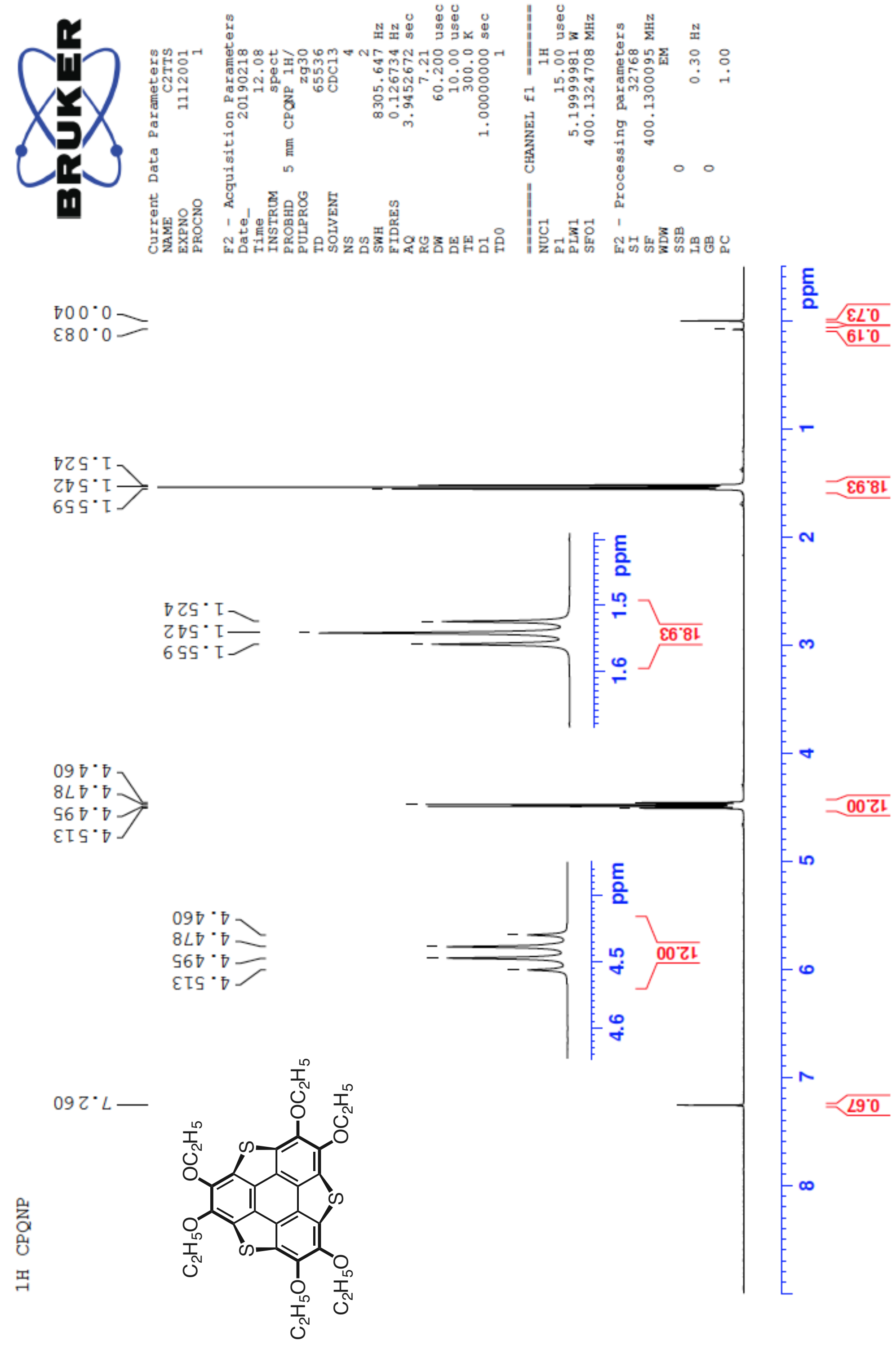
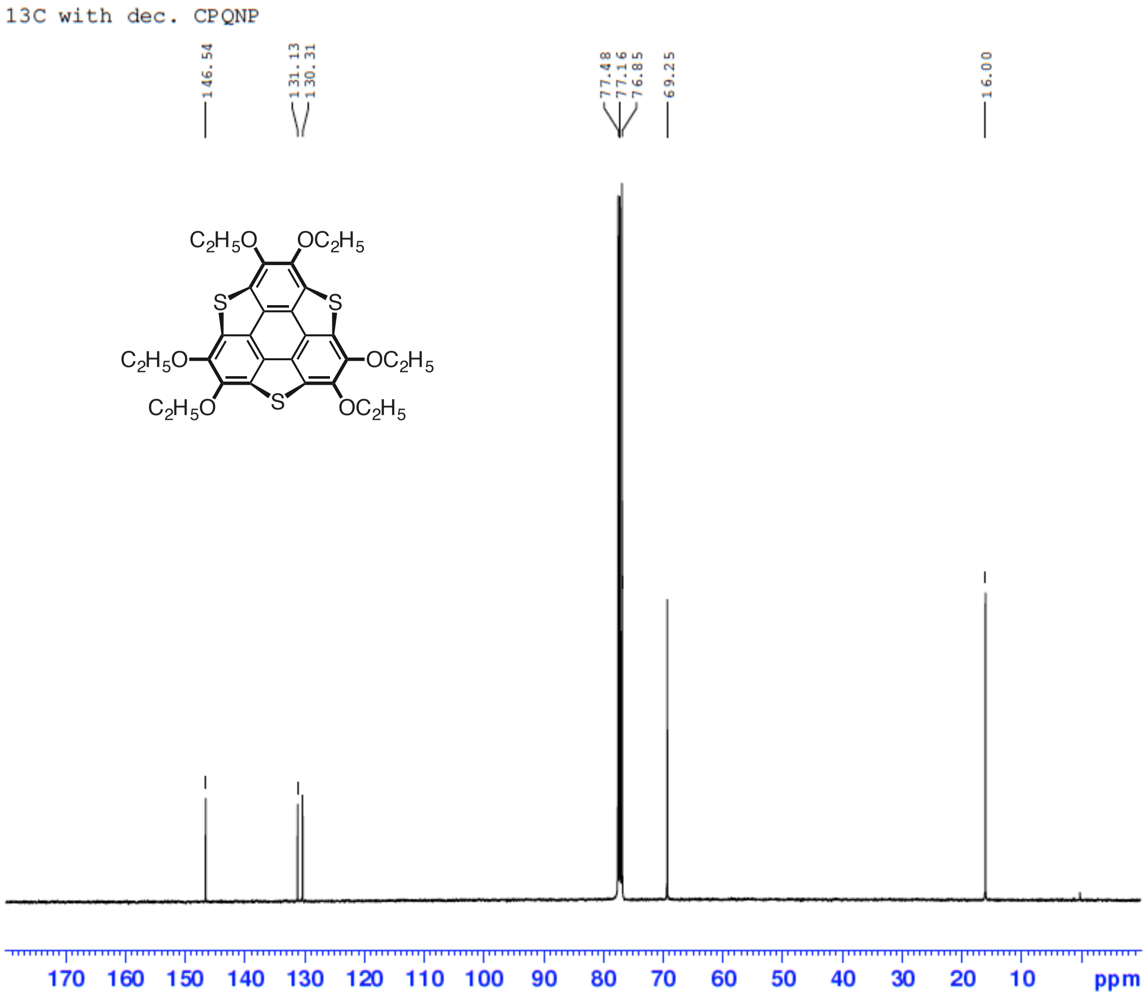
7. NMR charts

^{1}H NMR spectrum of C2SS in $CDCl_3$



^{13}C NMR spectrum of C2SS in $CDCl_3$



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DE                  18.00 usec
TE                  300.0 K
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D11            0.03000000 sec
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======== CHANNEL f2 ========
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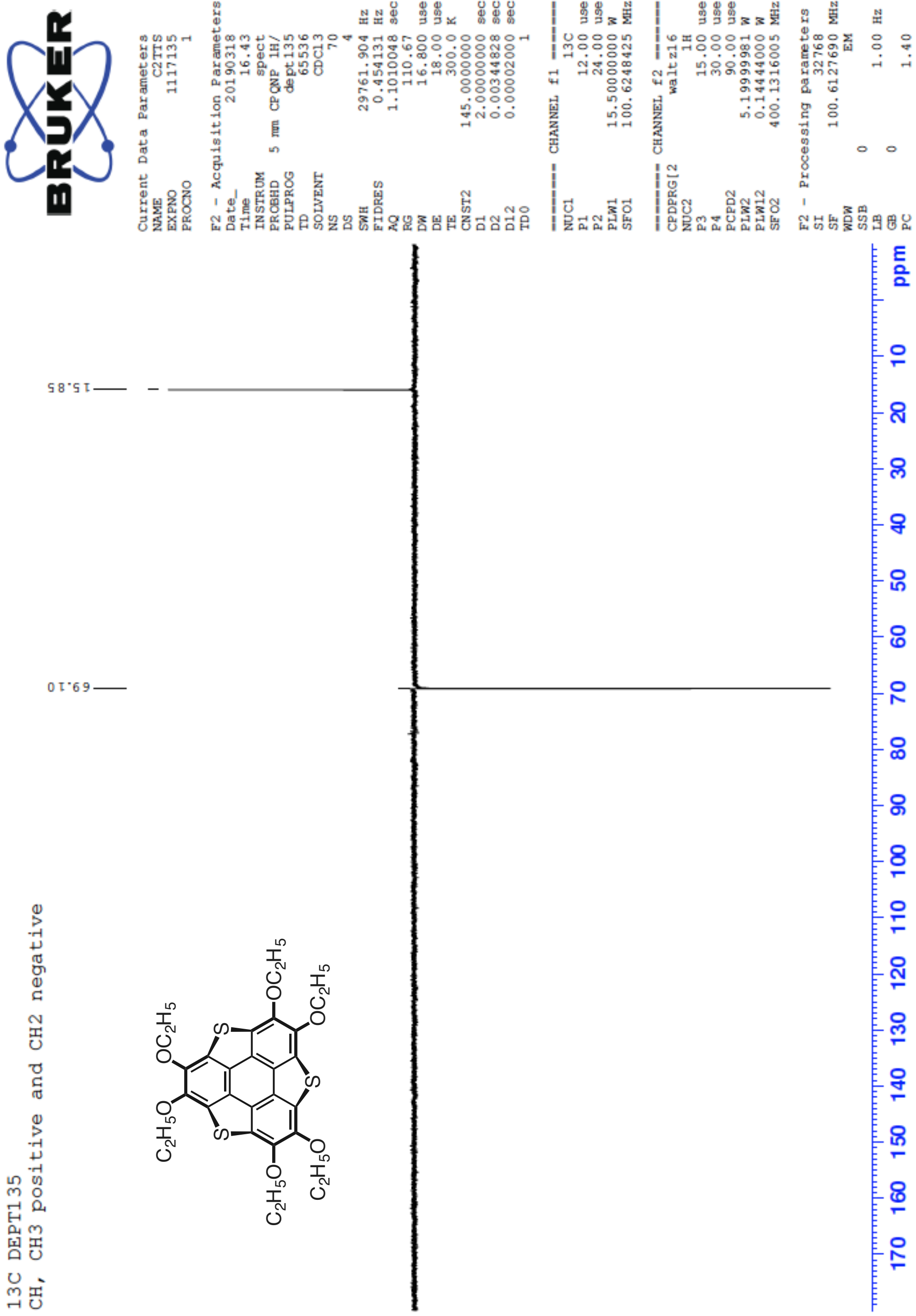

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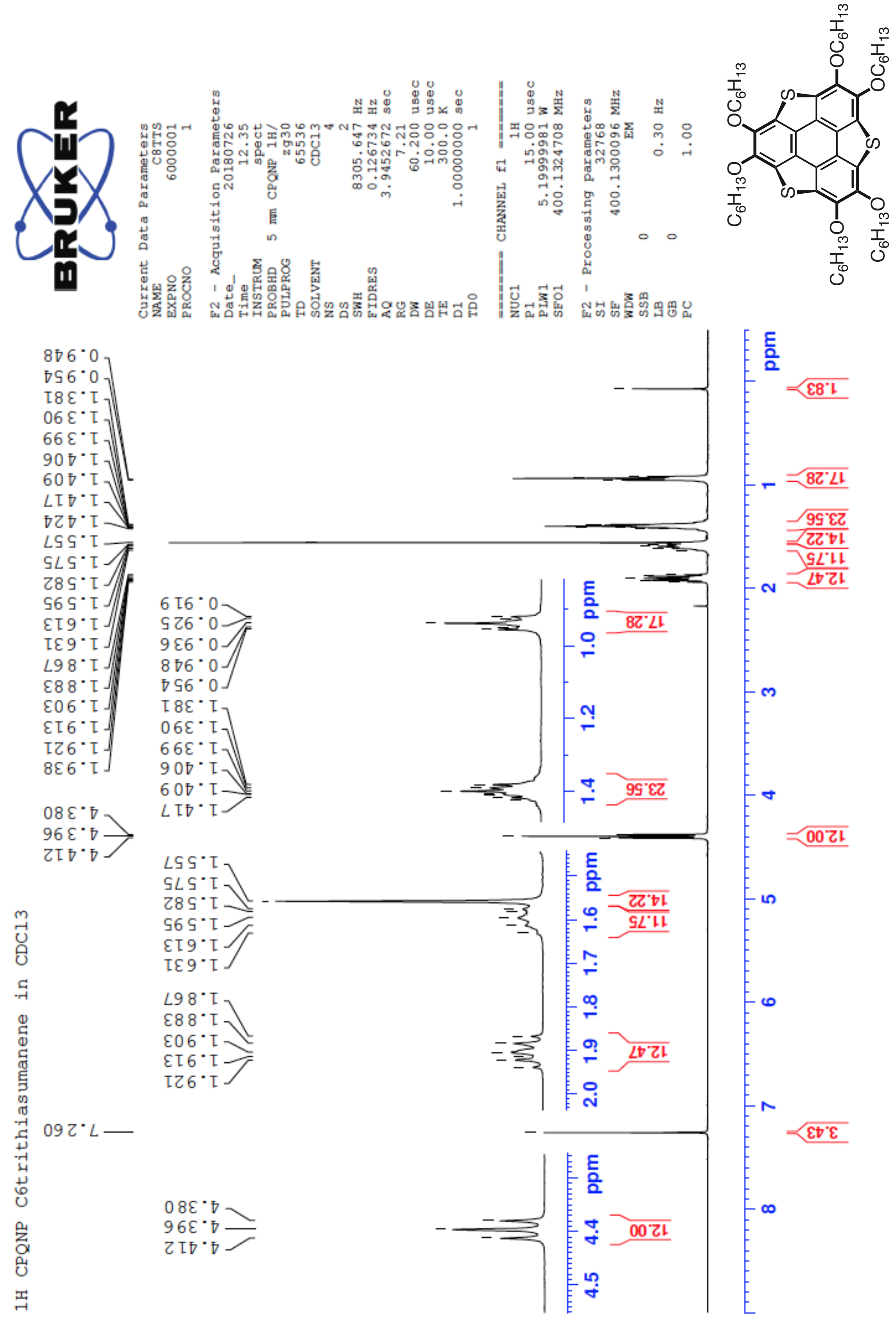
Time              16.43
INSTRUM           spect
PROBHD   5 mm CPQNP 1H/
PULPROG         dept135
TD                65536
SOLVENT           CDCl3
NS                   70
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010048 sec
RG               110.67
DW               16.800 usec
DE                18.00 usec
TE                300.0 K
CNST2       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
PLW1        15.50000000 W
SFO1        100.6248425 MHz

======== CHANNEL f2 ========
CPDPRG[2        waltz16
NUC2                 1H
P3                15.00 usec
P4                30.00 usec
PCPD2             90.00 usec
PLW2         5.19999981 W
PLW12        0.14444000 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127690 MHz
WDW                  EM
SSB      0
LB       0         1.00 Hz
GB       0
PC                 1.40
```

^{1}H NMR spectrum of C6SS in $CDCl_3$



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Current Data Parameters
NAME          C8TTS
EXPNO       6000001
PROCNO            1

F2 - Acquisition Parameters
Date_      20180726
Time          12.35
INSTRUM       spect
PROBHD   5 mm CPQNP 1H/
PULPROG        zg30
TD            65536
SOLVENT       CDCl3
NS                4
DS                2
SWH        8305.647 Hz
FIDRES     0.126734 Hz
AQ        3.9452672 sec
RG             7.21
DW           60.200 usec
DE            10.00 usec
TE            300.0 K
D1       1.00000000 sec
TD0               1

======== CHANNEL f1 ========
NUC1             1H
P1            15.00 usec
PLW1     5.19999981 W
SFO1   400.1324708 MHz

F2 - Processing parameters
SI            32768
SF      400.1300096 MHz
WDW              EM
SSB               0
LB             0.30 Hz
GB                0
PC             1.00
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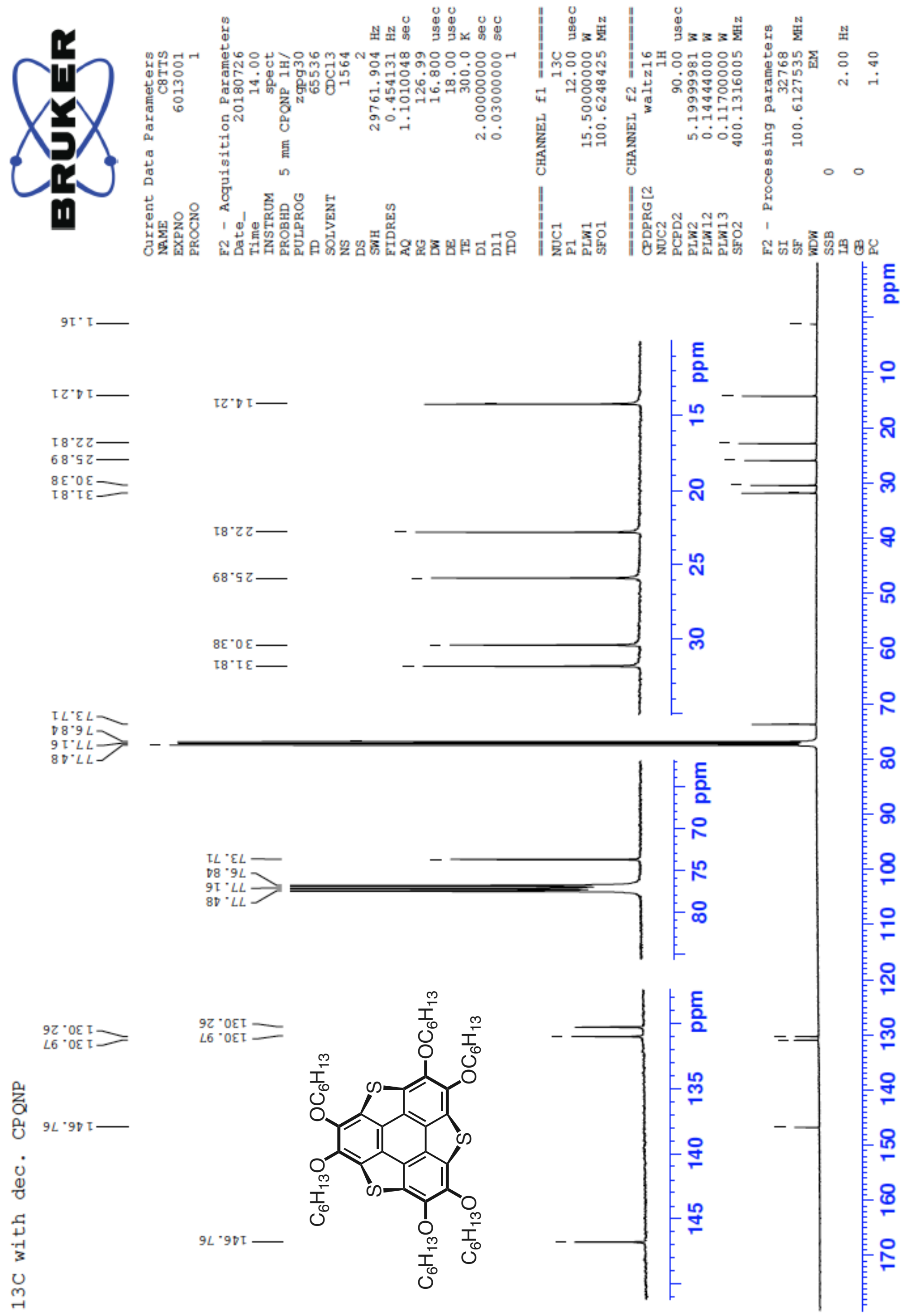
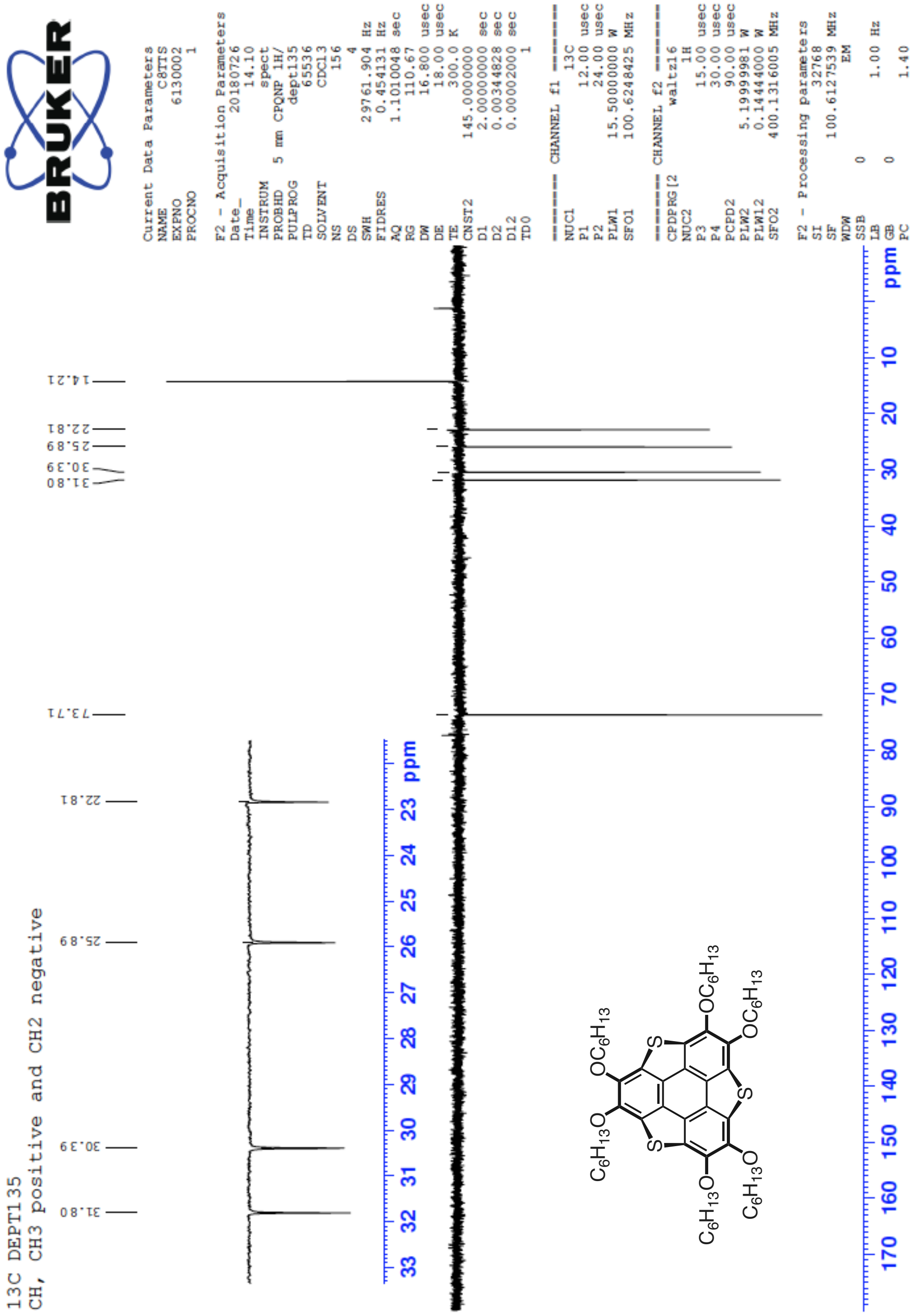
^{13}C NMR spectrum of C6SS in $CDCl_3$



DEPT-135 of of C6SS in CDCl$_3$



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Current Data Parameters
NAME          C8TTS
EXPNO       6130002
PROCNO            1

F2 - Acquisition Parameters
Date_      20180726
Time          14.10
INSTRUM       spect
PROBHD   5 mm CPQNP 1H/
PULPROG     dept135
TD            65536
SOLVENT       CDCl3
NS              156
DS                4
SWH       29761.904 Hz
FIDRES     0.454131 Hz
AQ        1.1010048 sec
RG           110.67
DW           16.800 usec
DE            18.00 usec
TE            300.0 K
CNST2   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
PLW1    15.50000000 W
SFO1    100.6248425 MHz

======== CHANNEL f2 ========
CPDPRG[2    waltz16
NUC2             1H
P3            15.00 usec
P4            30.00 usec
PCPD2         90.00 usec
PLW2     5.19999981 W
PLW12    0.14444000 W
SFO2    400.1316005 MHz

F2 - Processing parameters
SI            32768
SF      100.6127539 MHz
WDW              EM
SSB      0
LB             1.00 Hz
GB       0
PC             1.40
```
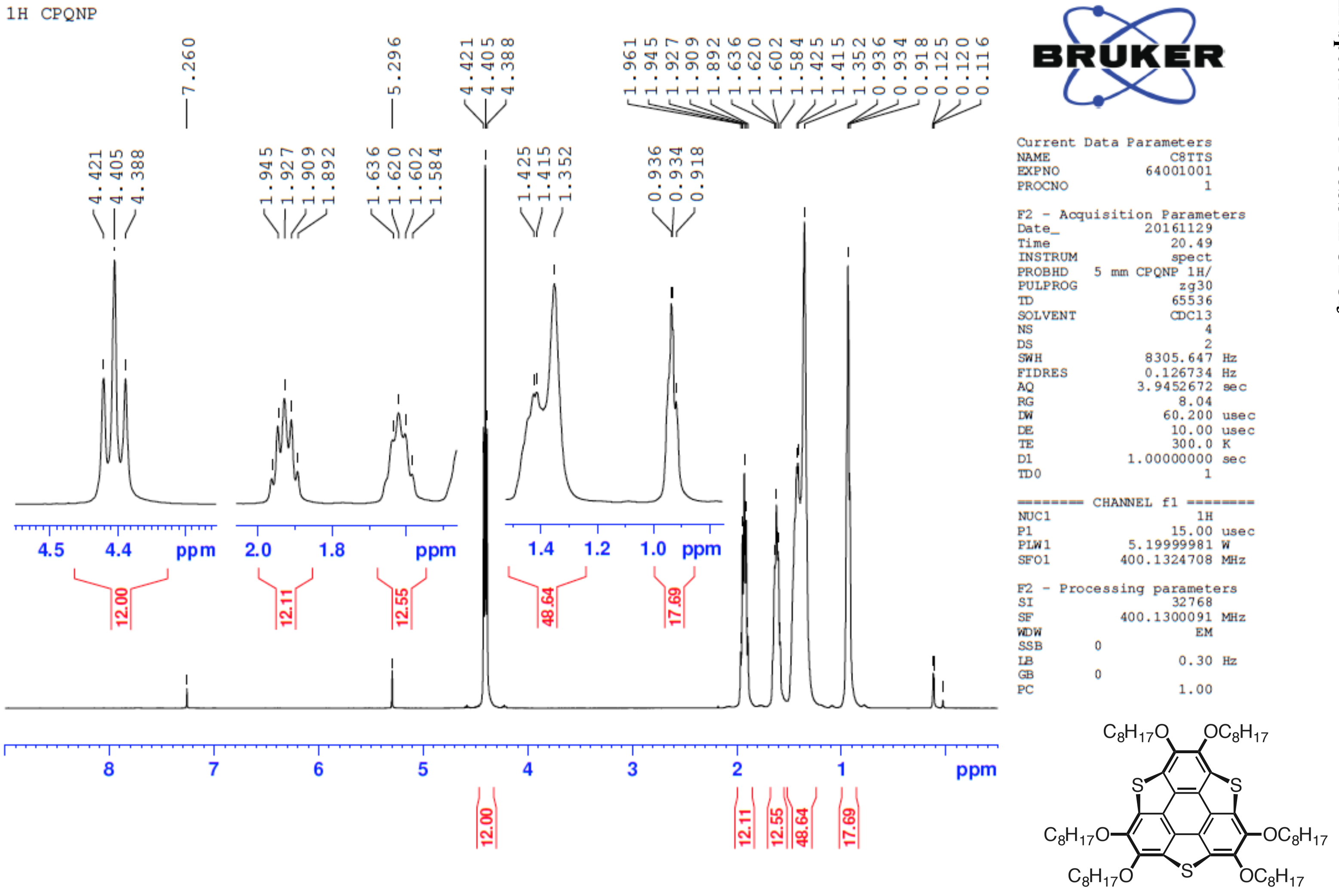

^{1}H NMR spectrum of C8SS in $CDCl_3$



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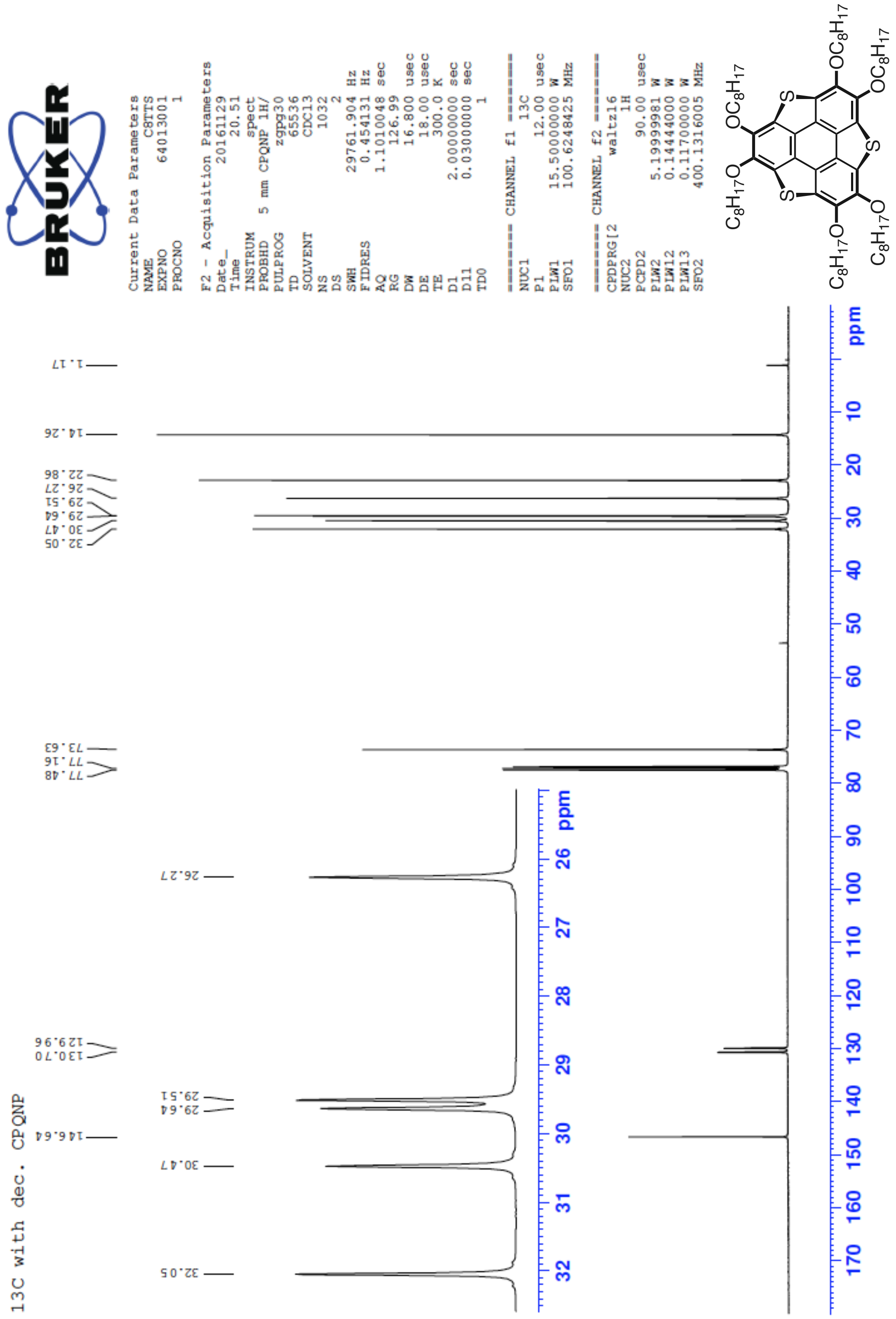
Current Data Parameters
NAME                C8TTS
EXPNO            64001001
PROCNO                  1

F2 - Acquisition Parameters
Date_            20161129
Time                20.49
INSTRUM             spect
PROBHD    5 mm CPQNP 1H/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      4
DS                      2
SWH              8305.647 Hz
FIDRES           0.126734 Hz
AQ              3.9452672 sec
RG                   8.04
DW                 60.200 usec
DE                  10.00 usec
TE                  300.0 K
D1             1.00000000 sec
TD0                     1

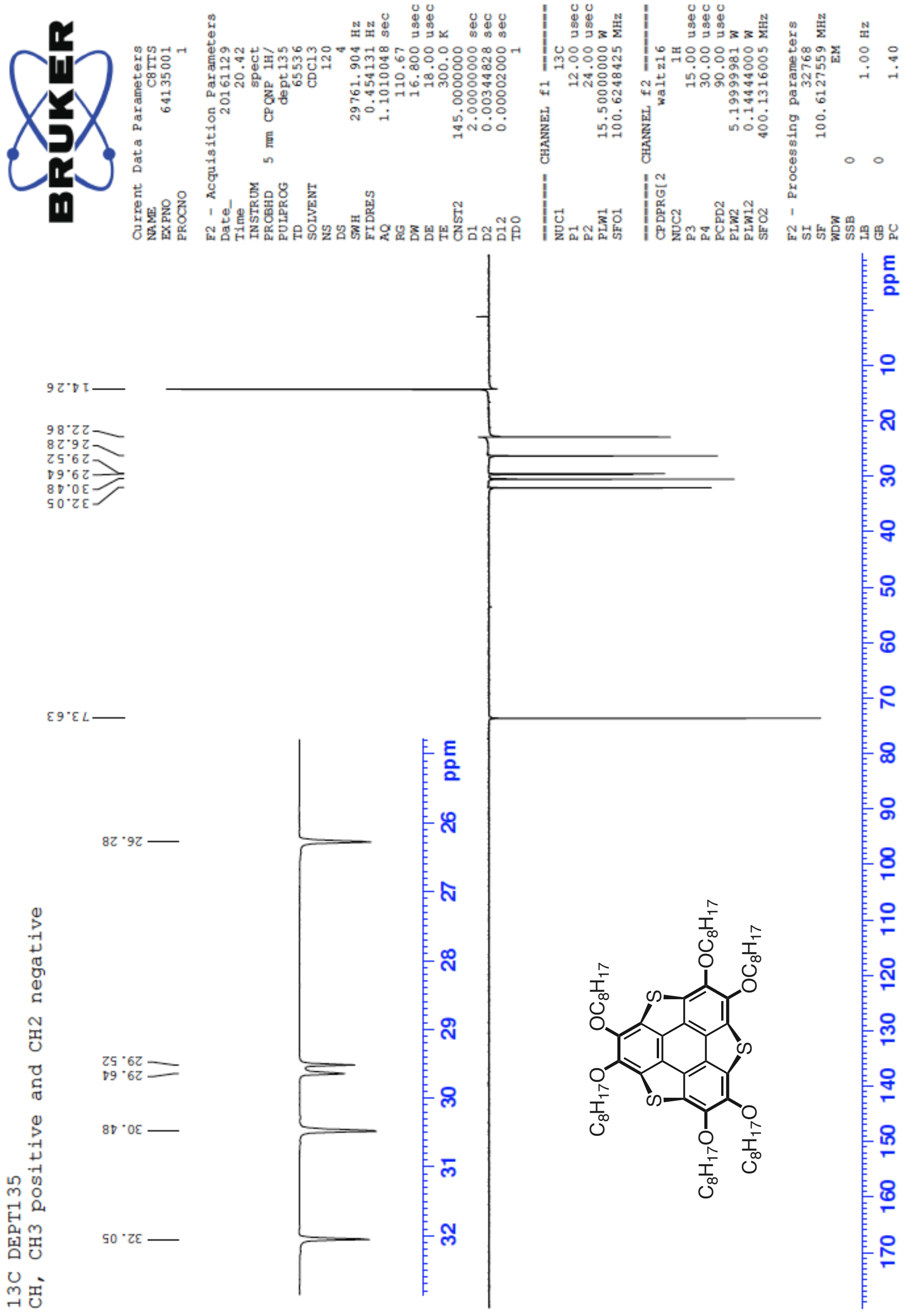
======== CHANNEL f1 ========
NUC1                   1H
P1                  15.00 usec
PLW1           5.19999981 W
SFO1          400.1324708 MHz

F2 - Processing parameters
SI                  32768
SF            400.1300091 MHz
WDW                    EM
SSB       0
LB                   0.30 Hz
GB        0
PC                   1.00
```

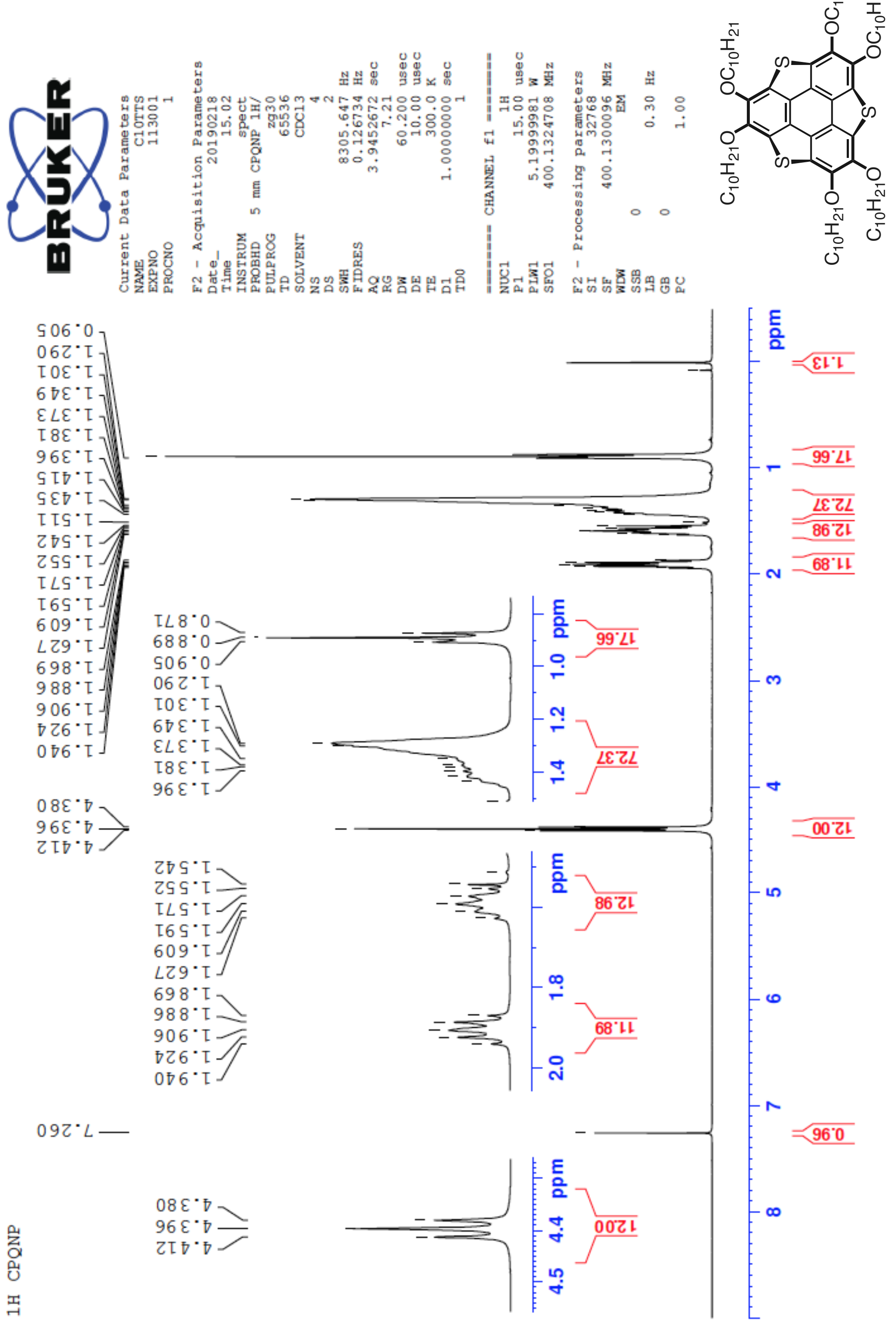
^{13}C NMR spectrum of C8SS in $CDCl_3$



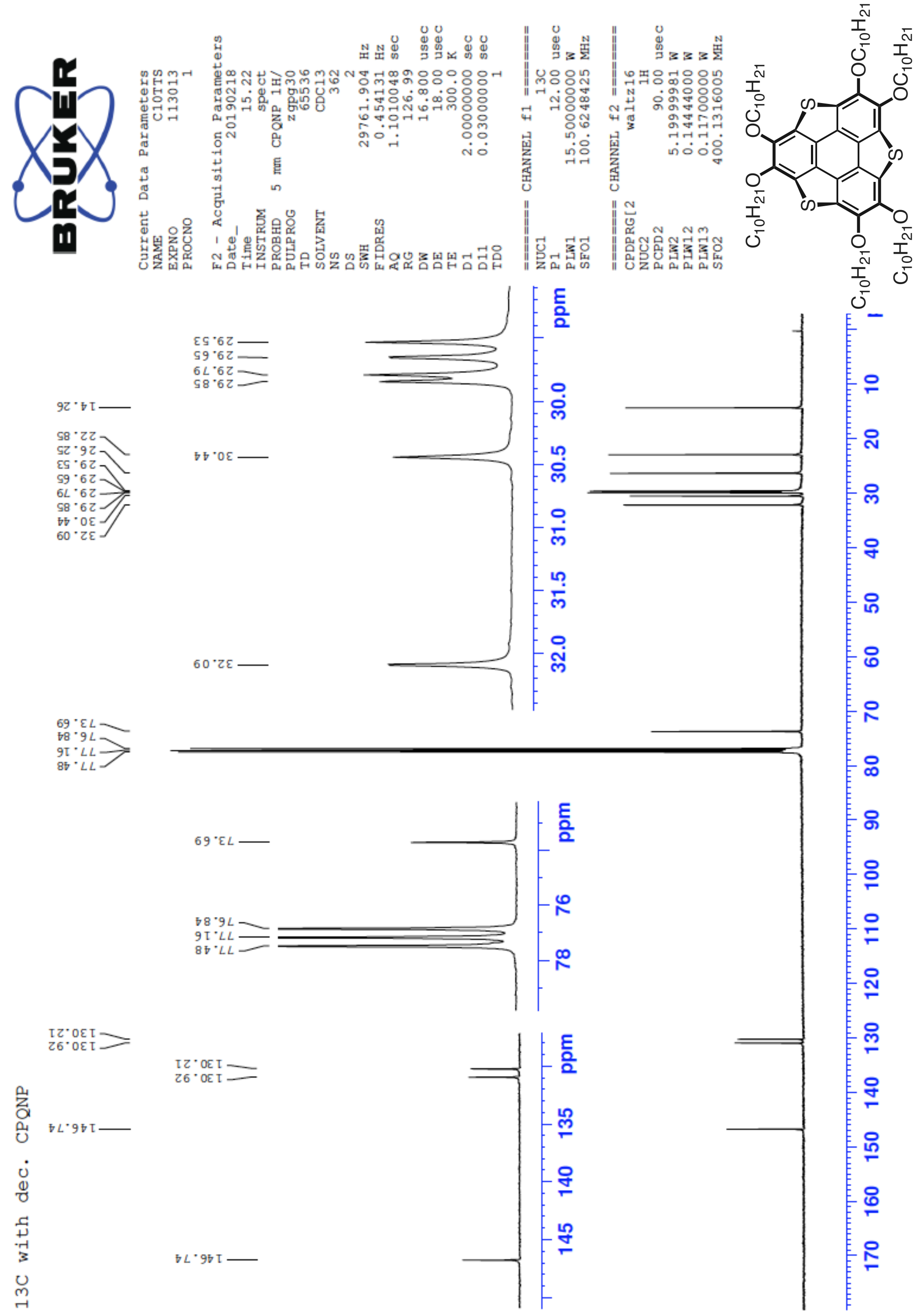
DEPT-135 of C8SS in $CDCl_3$



^{1}H NMR spectrum of C10SS in $CDCl_3$



^{13}C NMR spectrum of C10SS in $CDCl_3$



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Current Data Parameters
NAME            C10TTS
EXPNO           113013
PROCNO               1

F2 - Acquisition Parameters
Date_         20190218
Time             15.22
INSTRUM          spect
PROBHD   5 mm CPQNP 1H/
PULPROG        zgpg30
TD               65536
SOLVENT          CDCl3
NS                 362
DS                   2
SWH          29761.904 Hz
FIDRES        0.454131 Hz
AQ           1.1010048 sec
RG              126.99
DW              16.800 usec
DE               18.00 usec
TE               300.0 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                  1

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

======== CHANNEL f2 ========
CPDPRG[2       waltz16
NUC2                1H
PCPD2            90.00 usec
PLW2        5.19999981 W
PLW12       0.14444000 W
PLW13       0.11700000 W
SFO2       400.1316005 MHz
```
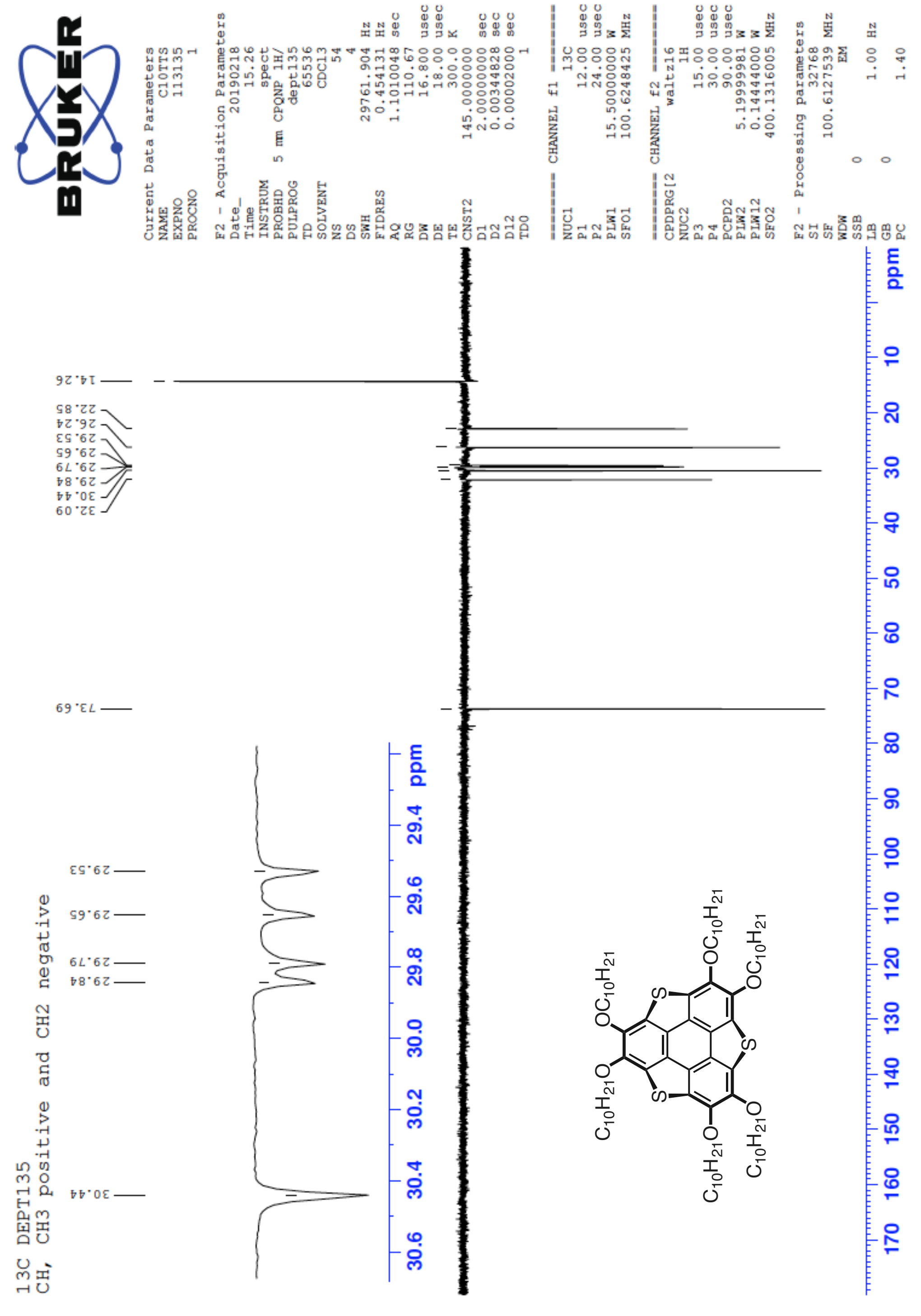
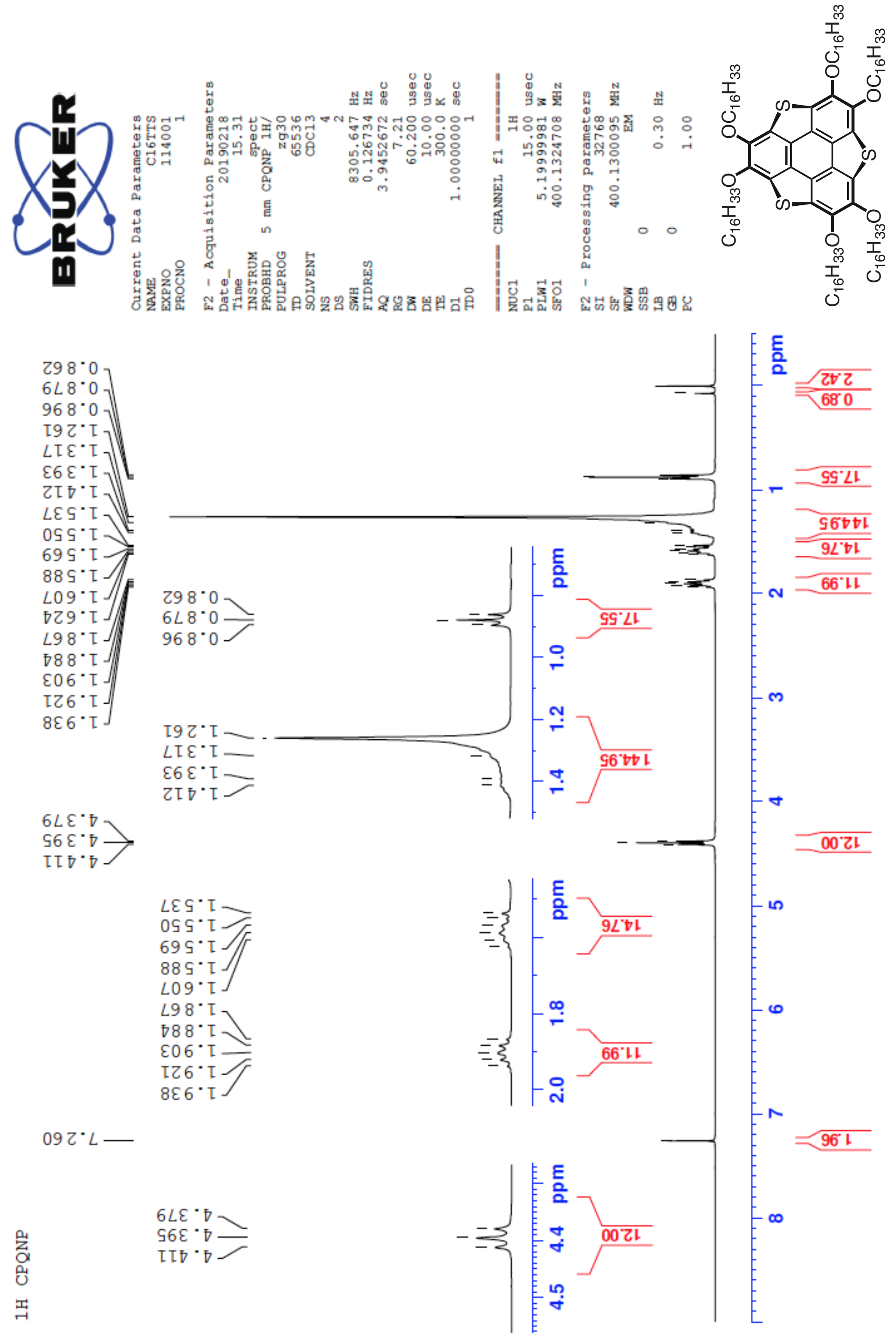
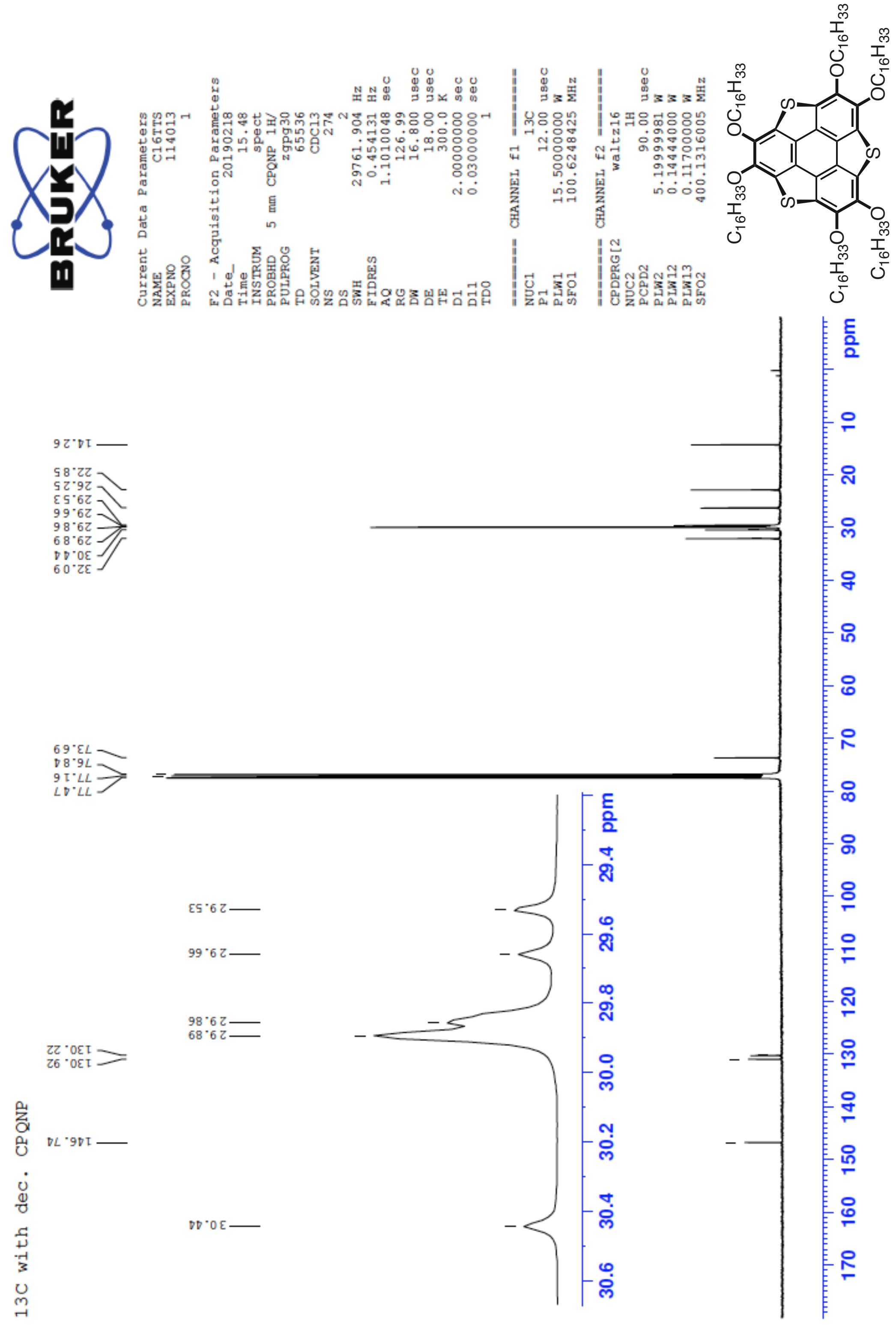

DEPT-135 of C10SS in $CDCl_3$



^{1}H NMR spectrum of C16SS in $CDCl_3$



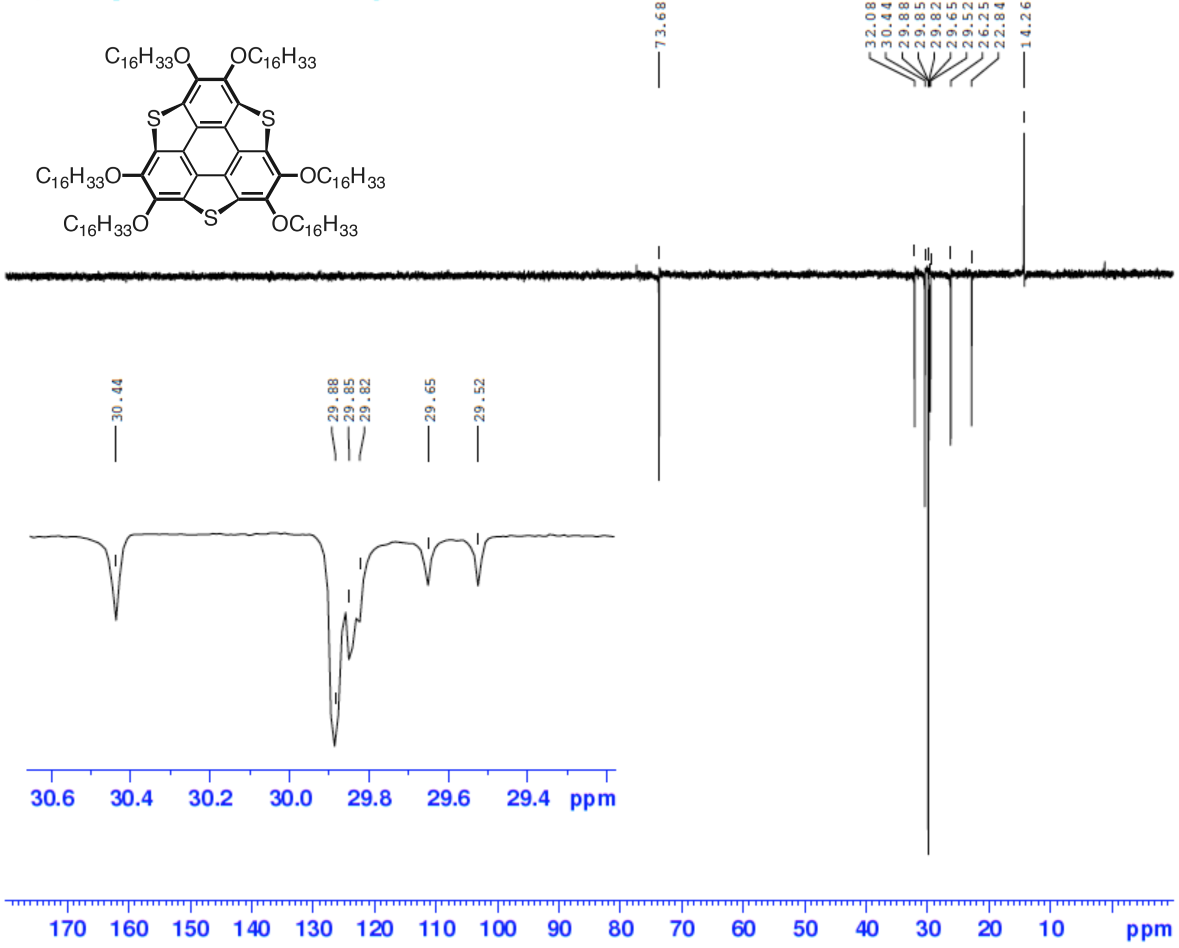
^{13}C NMR spectrum of C16SS in $CDCl_3$



```
Current Data Parameters
NAME              C16TTS
EXPNO             114013
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190218
Time               15.48
INSTRUM            spect
PROBHD    5 mm CPQNP 1H/
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   274
DS                     2
SWH            29761.904 Hz
FIDRES          0.454131 Hz
AQ             1.1010048 sec
RG                126.99
DW                16.800 usec
DE                 18.00 usec
TE                 300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

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

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                  1H
PCPD2              90.00 usec
PLW2          5.19999981 W
PLW12         0.14444000 W
PLW13         0.11700000 W
SFO2         400.1316005 MHz
```

DEPT-135 of C16SS in CDCl$_3$

13C DEPT135
CH, CH3 positive and CH2 negative



```
Current Data Parameters
NAME              C16TTS
EXPNO             114135
PROCNO                 1

F2 - Acquisition Parameters
Date_           20190218
Time               15.56
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG         dept135
TD                 65536
SOLVENT            CDCl3
NS                   128
DS                     4
SWH            29761.904 Hz
FIDRES          0.454131 Hz
AQ             1.1010048 sec
RG                110.67
DW                16.800 usec
DE                 18.00 usec
TE                 300.0 K
CNST2        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
PLW1         15.50000000 W
SFO1         100.6248425 MHz

======== CHANNEL f2 ========
CPDPRG[2         waltz16
NUC2                  1H
P3                 15.00 usec
P4                 30.00 usec
PCPD2              90.00 usec
PLW2          5.19999981 W
PLW12         0.14444000 W
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127544 MHz
WDW                   EM
SSB      0
LB                  1.00 Hz
GB       0
PC                  1.40
```