Supporting Information

A Novel Synthesis of Calix[4]thiophenes and Calix[4]furans

Gon-Ann Lee,* Wen-Chieh Wang, Minghuey Shieh and Ting-Shen Kuo

Department of Chemistry, Fu Jen Catholic University, Hsinchuang, Taipei 24205, Taiwan, Republic of China

E-mail: 016850@mail.fju.edu.tw

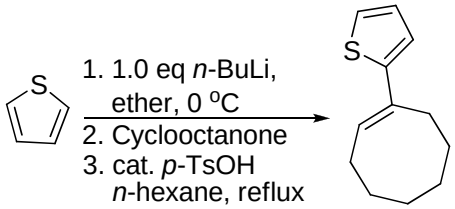
Table of Contents

General

Melting points were measured with a Barloworld SMP3 melting point apparatus and were uncorrected. NMR spectra were measured in $CDCl_3$ solution with a Bruker AC-300 MHz NMR spectrometer, with $CHCl_3$ as the internal standard. Chemical shifts (δ) are expressed in ppm downfield from tetramethylsilane. Coupling constants (J) are expressed in hertz (Hz). Mass spectra were measured with a Finnigan/Thermo Quest MAT 95XL mass spectrometer. The elemental analyses were recored on a Elementar vario EL III elemental analyzer. Infrared spectra were recorded on KBr pellets. Flash column chromatography was performed on silica gel (230 mesh). Solvents are of reagent grade.

Experimental procedures and characterization data for all compounds.

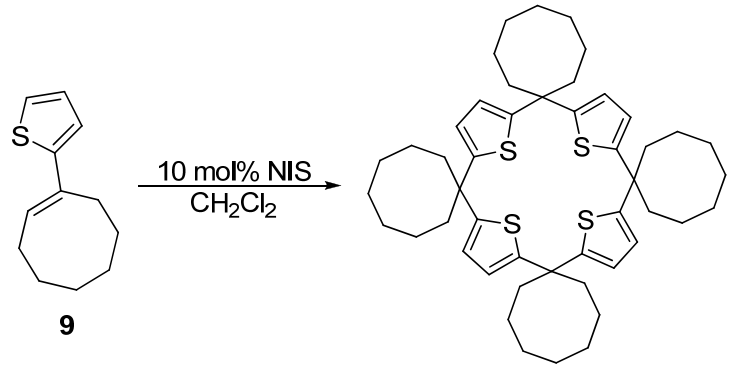
Synthesis of 1-(2-thienyl)cyclooctanene (9)



9, 81%

To a stirred solution of thiophene (10.0 g, 0.12 mol) in 100 mL of dry ether at 0 °C was added *n*-butyllithium (1.6 M in hexane, 75 mL, 0.12 mol) dropwise from a syringe. The mixture was stirred at 0 °C for 1 h, and cyclooctanone (15.14 g, 0.12 mol) was added. The mixture was allowed to warm to rt and stirred overnight. The mixture was poured into a 250-mL beaker with 100 g of crushed ice. The organic layer was separated and washed with water and brine, and then dried over anhydrous magnesium sulfate. After filtration, the solvent was removed on a rotary evaporator. To a stirred solution of crude alcohol in 100 mL of *n*-hexane was added catalytic amount of *p*-TsOH and the mixture was refluxed for 12 h. The *n*-hexane solution was dried over anhydrous K_2CO_3. After filtration, the solvent was removed and distilled to give **9** (81-82 °C / 0.1 torr, 18.7 g, 81%). δ_H(300 MHz; $CDCl_3$) 7.08 (1 H, dd, J 1.7 and 4.6, thiophene α-H), 6.98-6.94 (2 H, m, thiophene β-H), 6.17 (1 H, t, J 8.3, olefinic), 2.66-2.62 (2 H, m, cyclooctyl), 2.30-2.23 (2 H, m, cyclooctyl), 1.72-1.64 (2 H, m, cyclooctyl), 1.62-1.50 (6 H, m, cyclooctyl); δ_C(75 MHz; $CDCl_3$) 147.1 (C), 134.3 (C), 127.2 (CH), 126.8 (CH), 122.8 (CH), 121.7 (CH), 30.2 (CH_2), 28.9 (CH_2), 28.4 (CH_2), 27.3 (CH_2), 26.9 (CH_2), 26.0 (CH_2).

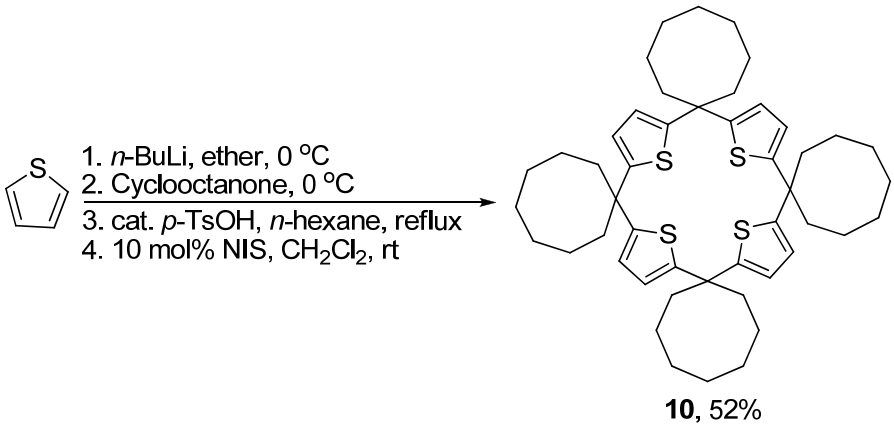
NIS-catalyzed tetramerization of 1-(2-thienyl)cyclooctene.



9 **10**, 61%

To a stirred solution of compound **9** (1g, 5.2 mmol) in 10 mL of CH_2Cl_2 was added 10 mol% of NIS (0.117 g, 0.52 mmol) and the mixture was stirred at rt for 72 h. The solvent was removed under reduced pressure and the residue was purified by flash column chromatography (5% EtOAc in toluene) to afford white solid **10** (0.61 g, 61%). mp 361-362 ^{o}C; (Found: C, 74.58; H, 8.37. $C_{48}H_{64}S_4$ requires C, 74.94; H, 8.39%); ν_{max}(film)/cm^{-1} 3095, 3079, 3063, 2922, 2850, 1638, 1618, 1477, 1446, 785, 759; δ_H(300 MHz; $CDCl_3$) 6.61 (8 H, s, thiophene β-H), 2.31-2.13 (16 H, br m, cyclooctyl), 1.66-1.42 (40 H, br m, cyclooctyl); δ_C(75 MHz; $CDCl_3$) 155.3 (C), 120.2 (CH), 47.6 (C), 35.8 (CH_2), 28.5 (CH_2), 24.9 (CH_2), 22.9 (CH_2); MS (FAB) *m/z* 768 (M^+, 4), 136 (100); HRMS (FAB) Calcd for $C_{48}H_{64}S_4$ *m/z* 768.3891 (M^+), found 768.3887. CCDC 768516.

General procedure for the synthesis of calix[4]arenes (10, 14-20) from thiophene and furan. Exemplified for compound 10.

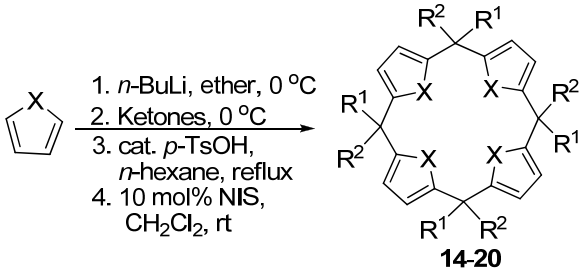


10, 52%

To a stirred solution of thiophene (1.0 g, 11.9 mmol) in 100 mL of dry ether at 0 °C was added *n*-butyllithium (1.6 M in hexane, 7.4 mL, 11.9 mmol) dropwise from a syringe. The mixture was stirred at 0 °C for 1 h, and cyclooctanone (1.5 g, 11.9 mmol) was added. The mixture was allowed to warm to rt and stirred overnight. The mixture was poured into a 250-mL beaker with 100 g of crushed ice. The organic layer was separated and washed with water and brine, and then dried over anhydrous magnesium sulfate. After filtration, the

solvent was removed on a rotary evaporator. To a stirred solution of crude alcohol in 100 mL of *n*-hexane was added catalytic amount of *p*-TsOH and the mixture was refluxed for 12 h. The *n*-hexane solution was dried over anhydrous K_2CO_3. After filtration, the solvent was removed under reduced pressure. The alkene was used without further purification. To a CH_2Cl_2 (20 mL) solution of crude 1-(2-thienyl)cyclooctene (1.97 g) was added 10 mol% of NIS (0.25 g) and the mixture was stirred at rt for 72 h. The residue was purified by flash column chromatography to afford white solid **10** (1.19 g, 52%).

Table 1 The synthesis of calix[4]arenes **14-20** from thiophene and furan.



14-20

Entry	X	R^1	R^2	Product	Yield
1	S	-$(CH_2)_6$-		**14**	56
2	S	Et	Et	**15**	62
3	S	*n*-Pr	*n*-Pr	**16**	58
4	S	*n*-Bu	*n*-Bu	**17**	66
5	O	-$(CH_2)_6$-		**18**	42
6	O	-$(CH_2)_7$-		**19**	51
7	O	*n*-Bu	*n*-Bu	**20**	47

Tetraspirocycloheptyl calix[4]thiophene (14)

mp 338-339 °C; (Found: C, 74.31; H, 7.76. $C_{44}H_{56}S_4$ requires C, 74.10; H, 7.91%); ν_{max} (film)/cm^{-1} 3092, 3072, 3060, 2932, 2920, 2850, 1638, 1618, 1462, 1443, 795, 786, 755; δ_H(300 MHz; $CDCl_3$) 6.59 (8 H, s, thiophene β-H), 2.33-2.19 (16 H, br m, cycloheptyl), 1.68-1.50 (32 H, br m, cycloheptyl); δ_C(75 MHz; $CDCl_3$) 156.0 (C), 119.5 (CH), 47.5 (C), 41.9 (CH_2), 29.8 (CH_2), 23.4 (CH_2); MS (FAB) *m/z* 712 (M^+, 10), 136 (100); HRMS (FAB) Calcd for $C_{44}H_{56}S_4$ *m/z* 712.3265 (M^+), found 712.3270. CCDC 768514.

Octaethyl calix[4]thiophene (15)

mp 317.5-318 °C; (Found: C, 70.98; H, 7.65. $C_{36}H_{48}S_4$ requires C, 71.00; H, 7.94%); ν_{max} (film)/cm^{-1} 3100, 3079, 3066, 2965, 2933, 2875, 1638, 1617, 1477, 1457, 1378, 789, 780; δ_H(300 MHz; $CDCl_3$) 6.58 (8 H, s, thiophene β-H), 2.0 (16 H, q, *J* 7.3, CH_2CH_3), 0.71 (24 H, t, *J* 7.3, CH_2CH_3); δ_C(75 MHz; $CDCl_3$) 154.5 (C), 120.5 (CH), 47.2 (C), 29.8 (CH_2), 8.1 (CH_3); MS (EI) *m/z* 608 (M^+, 9), 579 (100); HRMS (EI) Calcd for $C_{36}H_{48}S_4$ *m/z* 608.2639 (M^+), found 608.2635. CCDC 768517.

Octapropyl calix[4]thiophene (16)

mp 341.5-342.5 °C; (Found: C, 73.08; H, 8.79. $C_{44}H_{64}S_4$ requires C, 73.27; H, 8.94%); ν_{max} (film)/cm^{-1} 3096, 3076, 2956, 2932, 2870, 1638, 1618, 1475, 1464, 1457, 1379, 796, 773; δ_H(300 MHz; $CDCl_3$) 6.55 (8 H, s, thiophene β-H), 1.96-1.90 (16 H, m, $CH_2CH_2CH_3$), 1.18-1.05 (16 H, m, $CH_2CH_2CH_3$), 0.86 (24 H, t, *J* 7.2, $CH_2CH_2CH_3$); δ_C(75 MHz; $CDCl_3$) 154.9 (C), 120.0 (CH), 46.8 (C), 40.3 (CH_2), 17.1 (CH_2), 14.5 (CH_3); MS (EI) *m/z* 720 (M^+, 6), 677 (100); HRMS (EI) Calcd for $C_{44}H_{64}S_4$ *m/z* 720.3891 (M^+), found 720.3886. CCDC 768512.

Octabutyl calix[4]thiophene (17)

mp 286.5-287.5 °C; (Found: C, 75.01; H, 9.67. $C_{52}H_{80}S_4$ requires C, 74.94; H, 9.67%); ν_{max} (film)/cm^{-1} 3097, 3077, 2954, 2932, 2862, 1638, 1618, 1465, 1378, 789; δ_H(300 MHz; $CDCl_3$) 6.55 (8 H, s, thiophene β-H), 1.96-1.91 (16 H, m, $CH_2CH_2CH_2CH_3$), 1.25 (16 H, sextet, *J* 7.2, $CH_2CH_2CH_2CH_3$), 1.15-0.98 (16 H, br m, $CH_2CH_2CH_2CH_3$), 0.81 (24 H, t, *J* 7.2, $CH_2CH_2CH_2CH_3$); δ_C(75 MHz; $CDCl_3$) 155.1 (C), 120.0 (CH), 46.7 (C), 37.5 (CH_2), 25.9 (CH_2), 22.9 (CH_2), 13.9 (CH_3); MS (FAB) *m/z* 833 ($[M + 1]^+$, 1.4), 154 (100); HRMS (FAB) Calcd for $C_{52}H_{81}S_4$ *m/z* 833.5221 $[M + H]^+$, found 833.5234. CCDC 768518.

Tetraspirocycloheptyl calix[4]furan (18)

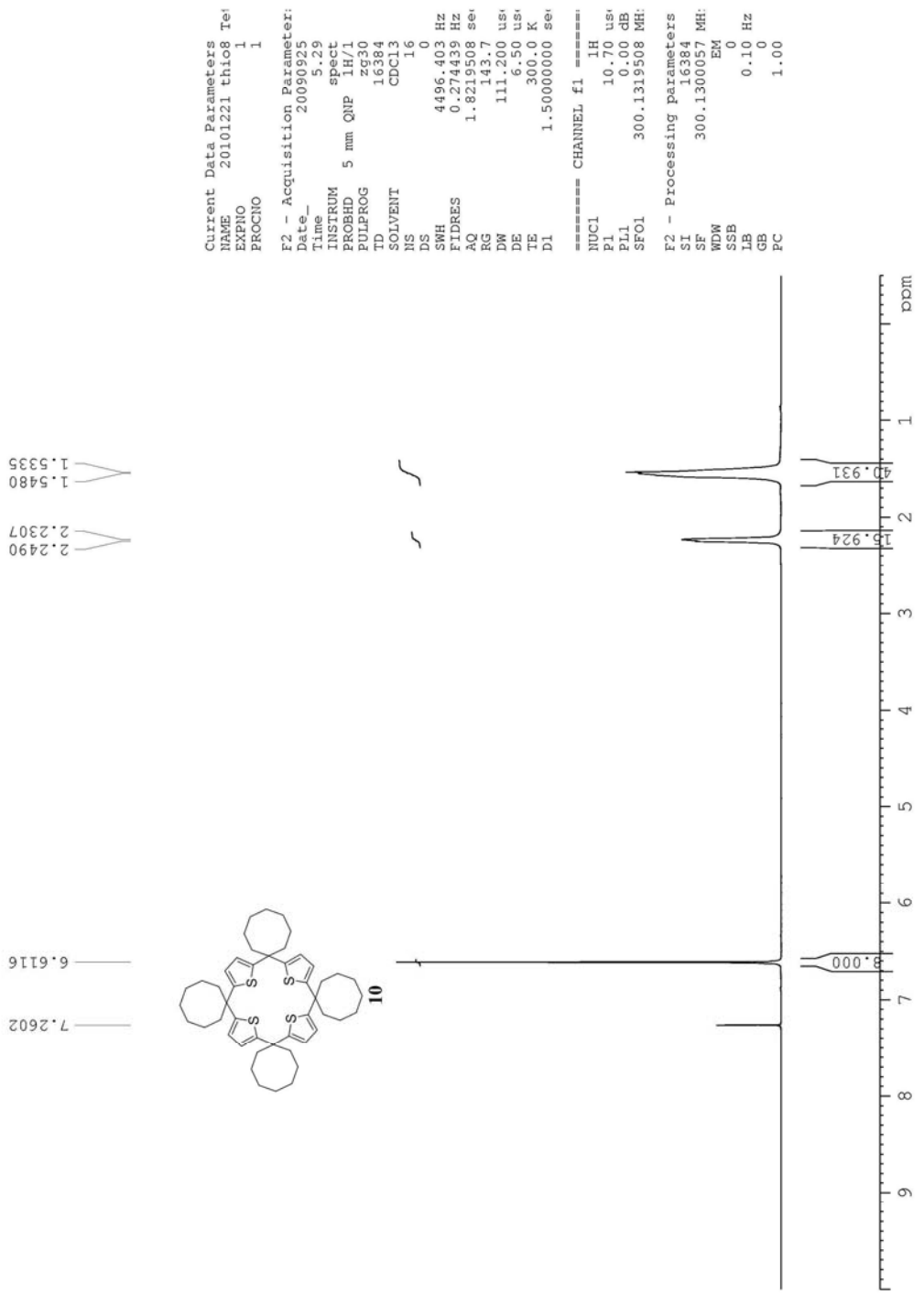
mp 233-234 ^{o}C; (Found: C, 81.57; H, 9.08. $C_{44}H_{56}O_4$ requires C, 81.44; H, 8.70%); ν_{max} (film)/cm^{-1} 2942, 2920, 2856, 1638, 1617, 1548, 1464, 1446, 1201, 1020, 776, 763; δ_H(300 MHz; $CDCl_3$) 5.87 (8 H, s, furan β-H), 2.10-1.99 (16 H, br m, cycloheptyl), 1.56-1.37 (32 H, br m, cycloheptyl); δ_C(75 MHz; $CDCl_3$) 158.3 (C), 103.7 (CH), 44.3 (C), 35.6 (CH_2), 29.7 (CH_2), 22.6 (CH_2); MS (EI) *m/z* 648 (M^+, 100); HRMS (EI) Calcd for $C_{44}H_{56}O_4$ *m/z* 648.4179 (M^+), found 648.4185. CCDC 768513.

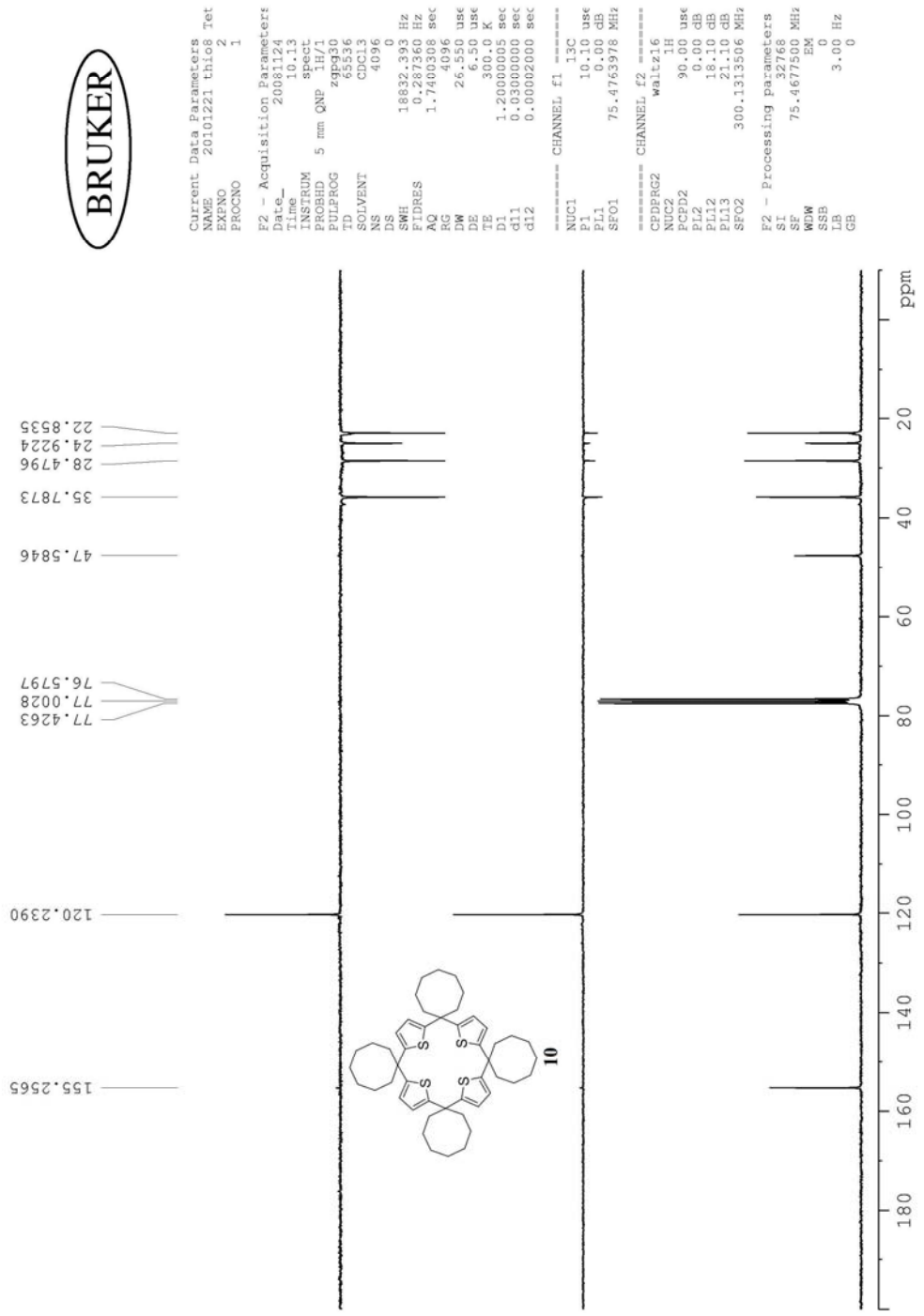
Tetraspirocyclooctyl calix[4]furan (19)

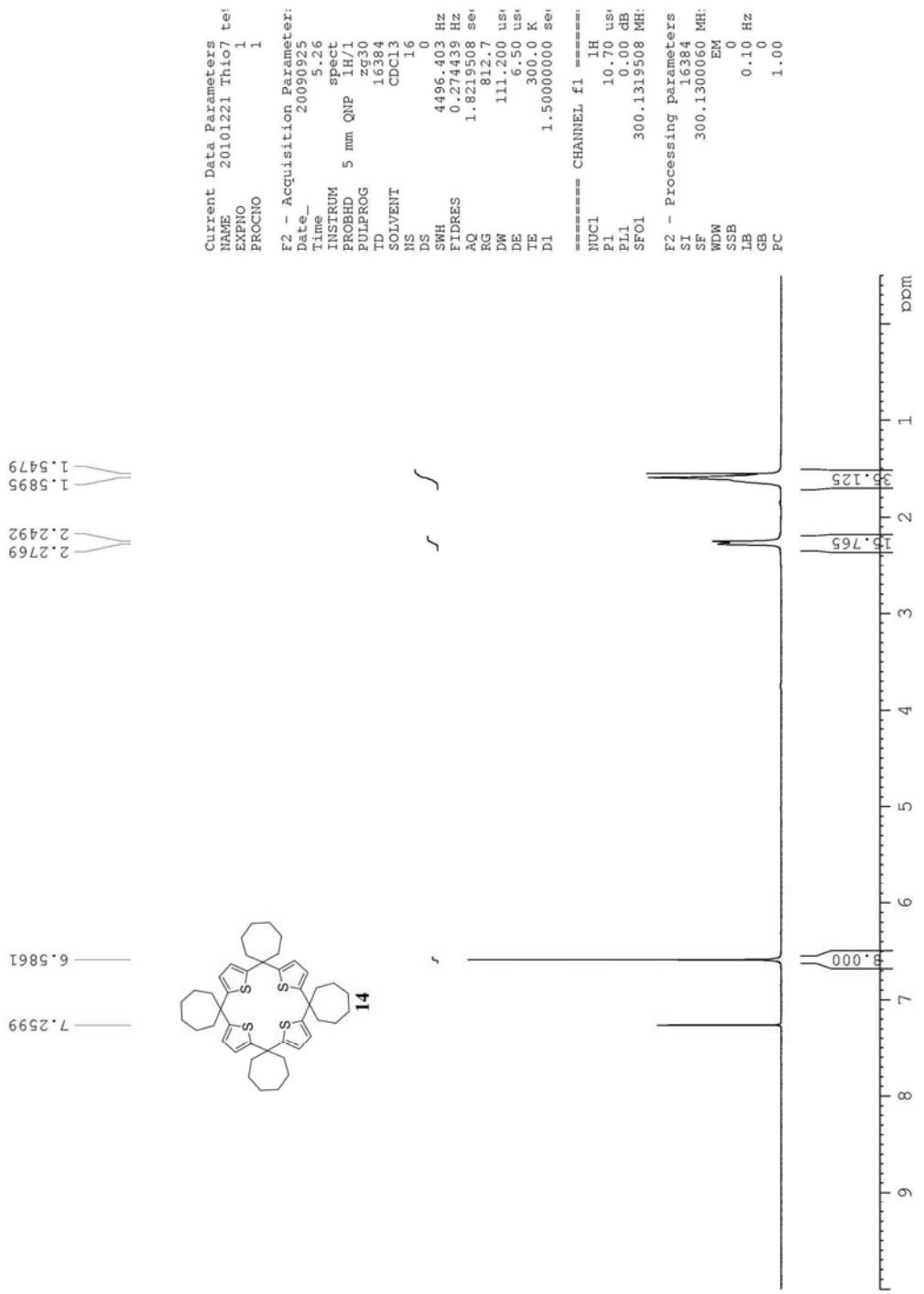
mp 306-307 ^{o}C; (Found: C, 82.01; H, 9.21. $C_{48}H_{64}O_4$ requires C, 81.77; H, 9.15%); ν_{max} (film)/cm^{-1} 2947, 2916, 2846, 1638, 1618, 1551, 1479, 1446, 1188, 1059, 1027, 774; δ_H(300 MHz; $CDCl_3$) 5.91 (8 H, s, furan β-H), 2.09-1.97 (16 H, br m, cyclooctyl), 1.58-1.41 (24 H, br m, cyclooctyl), 1.38-1.24 (16 H, br m, cyclooctyl); δ_C(75 MHz; $CDCl_3$) 157.2 (C), 104.6 (CH), 44.4 (C), 29.9 (CH_2), 28.5 (CH_2), 25.0 (CH_2), 22.1 (CH_2); MS (EI) *m/z* 704 (M^+, 100); HRMS (EI) Calcd for $C_{48}H_{64}O_4$ *m/z* 704.4805 (M^+), found 704.4807. CCDC 768515.

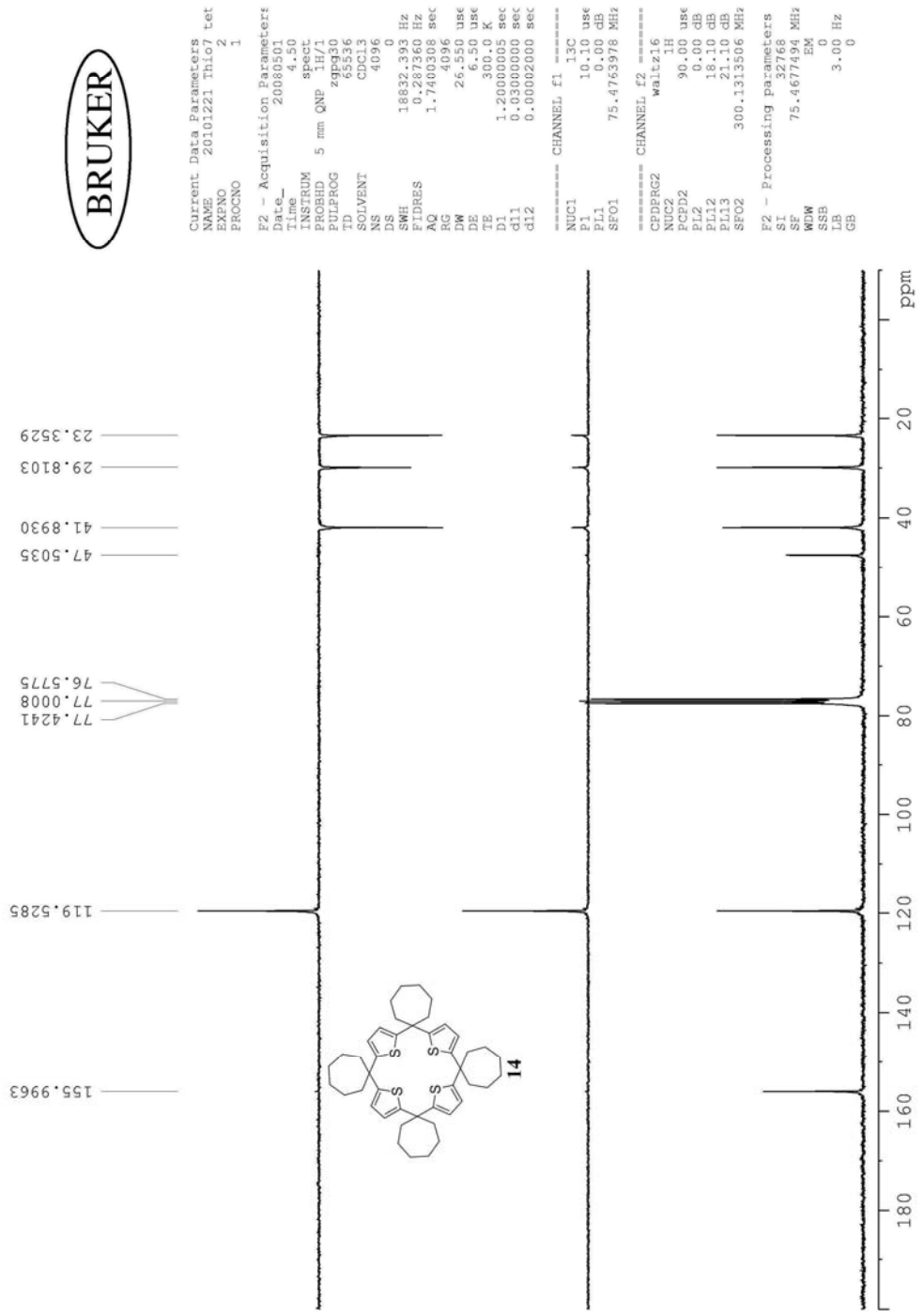
Octabutyl calix[4]furan (20)

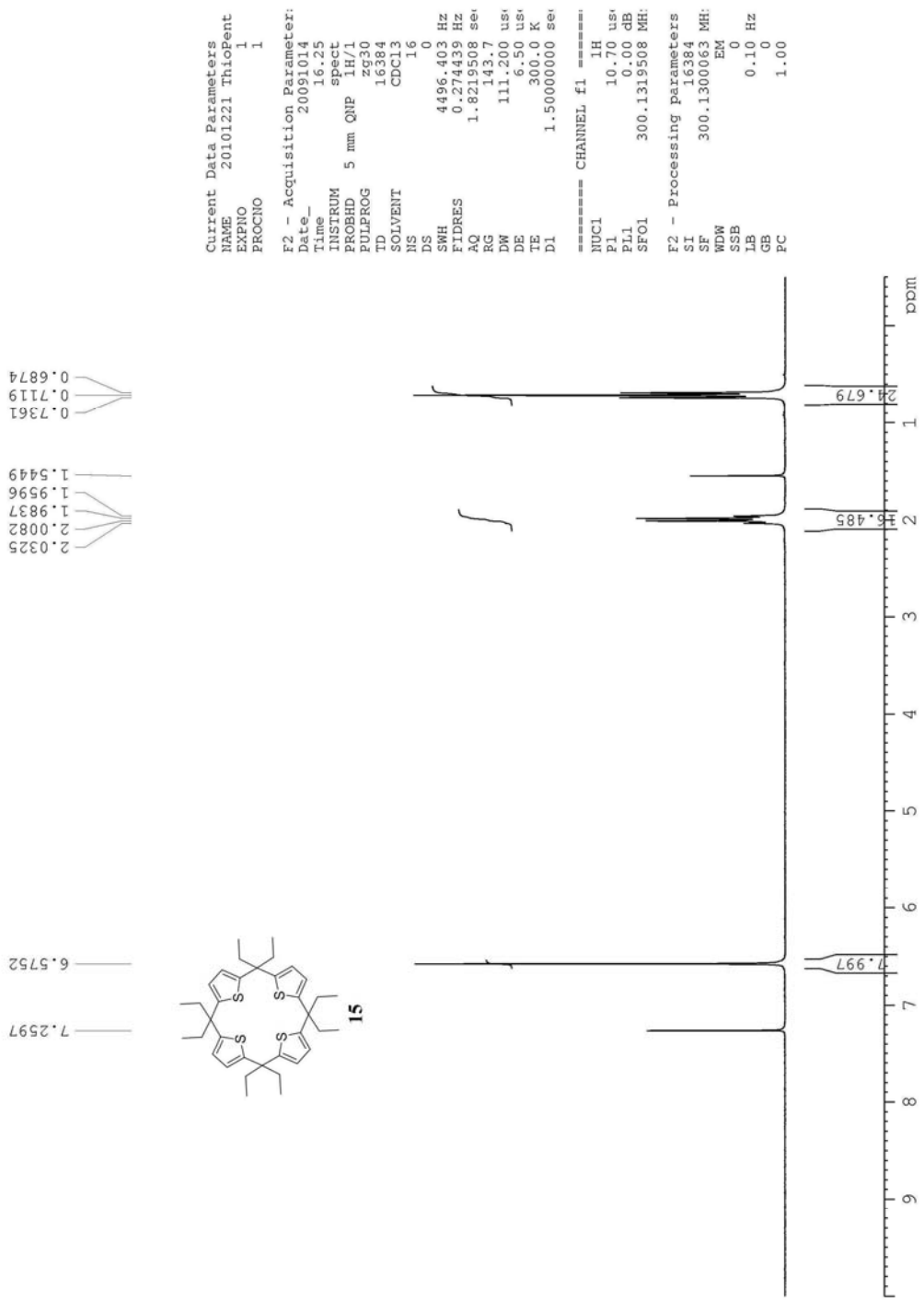
mp 271.5-272.5 ^{o}C; (Found: C, 81.27; H, 10.58. $C_{52}H_{80}O_4$ requires C, 81.20; H, 10.48%); ν_{max} (film)/cm^{-1} 2956, 2918, 2872, 2859, 1639, 1617, 1601, 1551, 1467, 1379, 1198, 1023, 777, 754; δ_H(300 MHz; $CDCl_3$) 5.87 (8 H, s, furan β-H), 1.87-1.73 (16 H, br m, C*H*$_2$CH$_2$CH$_2$CH$_3$), 1.22 (16 H, sextet, *J* 7.3, CH$_2$CH$_2$C*H*$_2$CH$_3$), 0.99-0.80 (16 H, m, CH$_2$C*H*$_2$CH$_2$CH$_3$), 0.82 (24 H, t, *J* 7.3, CH$_2$CH$_2$CH$_2$C*H*$_3$); δ_C(75 MHz; $CDCl_3$) 156.8 (C), 104.6 (CH), 43.5 (C), 32.0 (CH_2), 25.5 (CH_2), 23.2 (CH_2), 14.1 (CH_3); MS (FAB) *m/z* 768 (M^+, 3), 154 (100); HRMS (FAB) Calcd for $C_{52}H_{80}O_4$ *m/z* 768.6057 (M^+), found 768.6058. CCDC 768519.

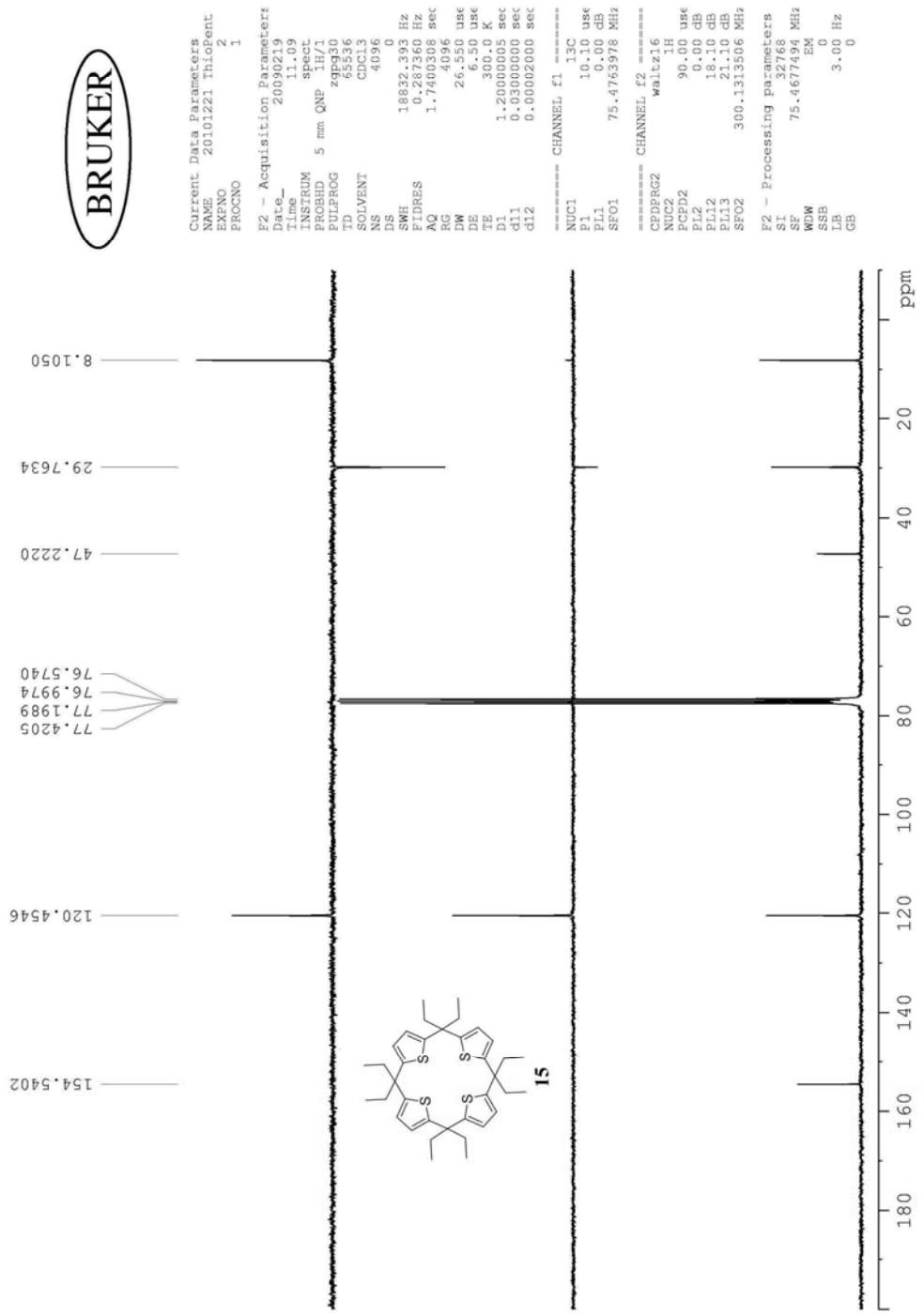

Current Data Parameters
NAME 20101221 thio8 Te
EXPNO 1
PROCNO 1
F2 - Acquisition Parameter
Date_ 20090925
Time 5.29
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 se
RG 143.7
DW 111.200 us
DE 6.50 us
TE 300.0 K
D1 1.50000000 se
======== CHANNEL f1 ======
NUC1 1H
P1 10.70 us
PL1 0.00 dB
SFO1 300.1319508 MH
F2 - Processing parameters
SI 16384
SF 300.1300057 MH
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1.5335
1.5480
2.2307
2.2490
6.6116
7.2602
40.931
15.924
8.000
10
ppm

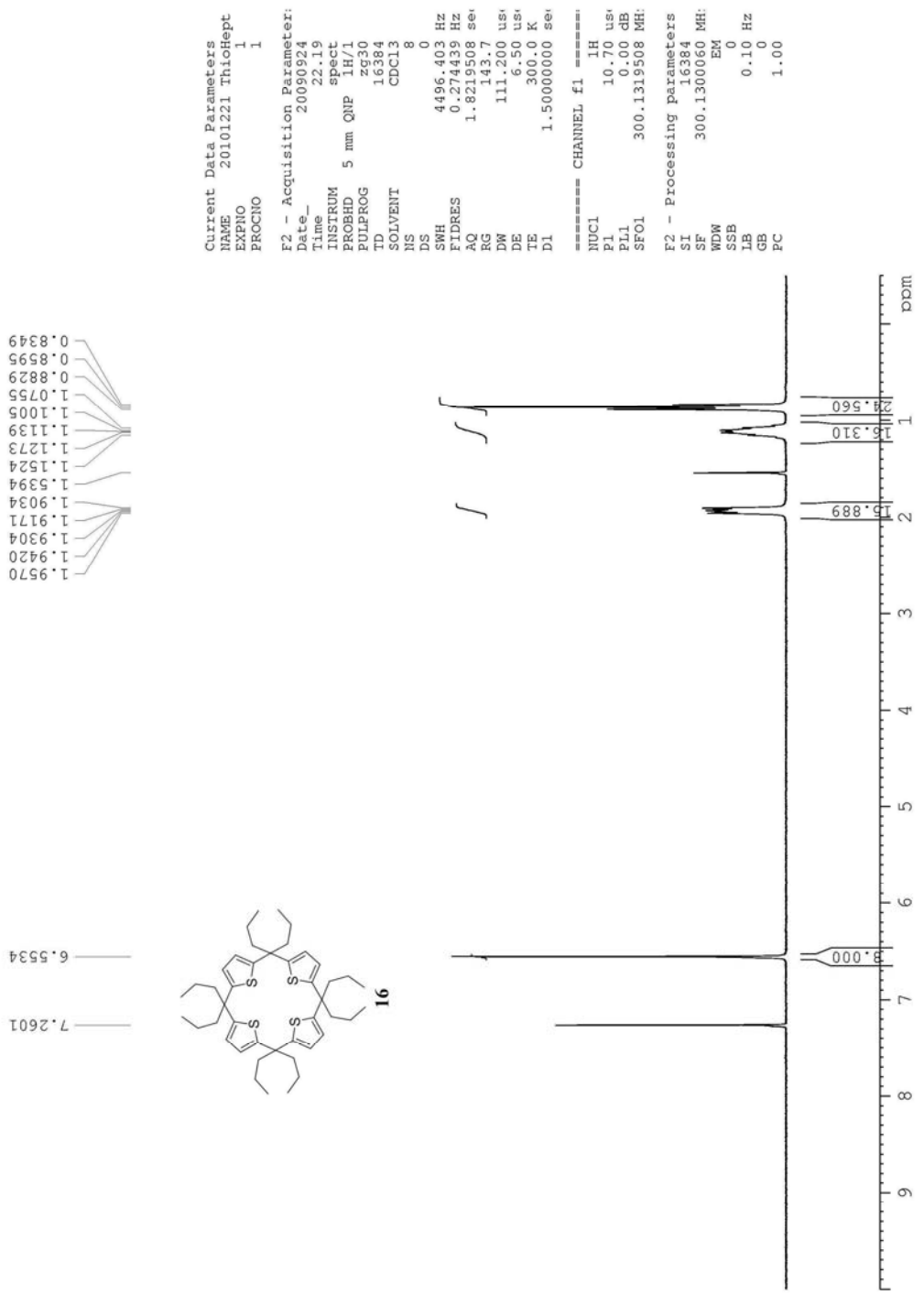

BRUKER
Current Data Parameters
NAME 20101221 thio8 Tet
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081124
Time 10.13
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4096
DS 0
SWH 18832.393 Hz
FIDRES 0.287360 Hz
AQ 1.7400308 sec
RG 4096
DW 26.550 use
DE 6.50 use
TE 300.0 K
D1 1.20000005 sec
d11 0.03000000 sec
d12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 10.10 use
PL1 0.00 dB
SFO1 75.4763978 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 use
PL2 0.00 dB
PL12 18.10 dB
PL13 21.10 dB
SFO2 300.1313506 MHz
F2 - Processing parameters
SI 32768
SF 75.4677500 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
155.2565
120.2390
77.4263
77.0028
76.5797
47.5846
35.7873
28.4796
24.9224
22.8535
10
ppm

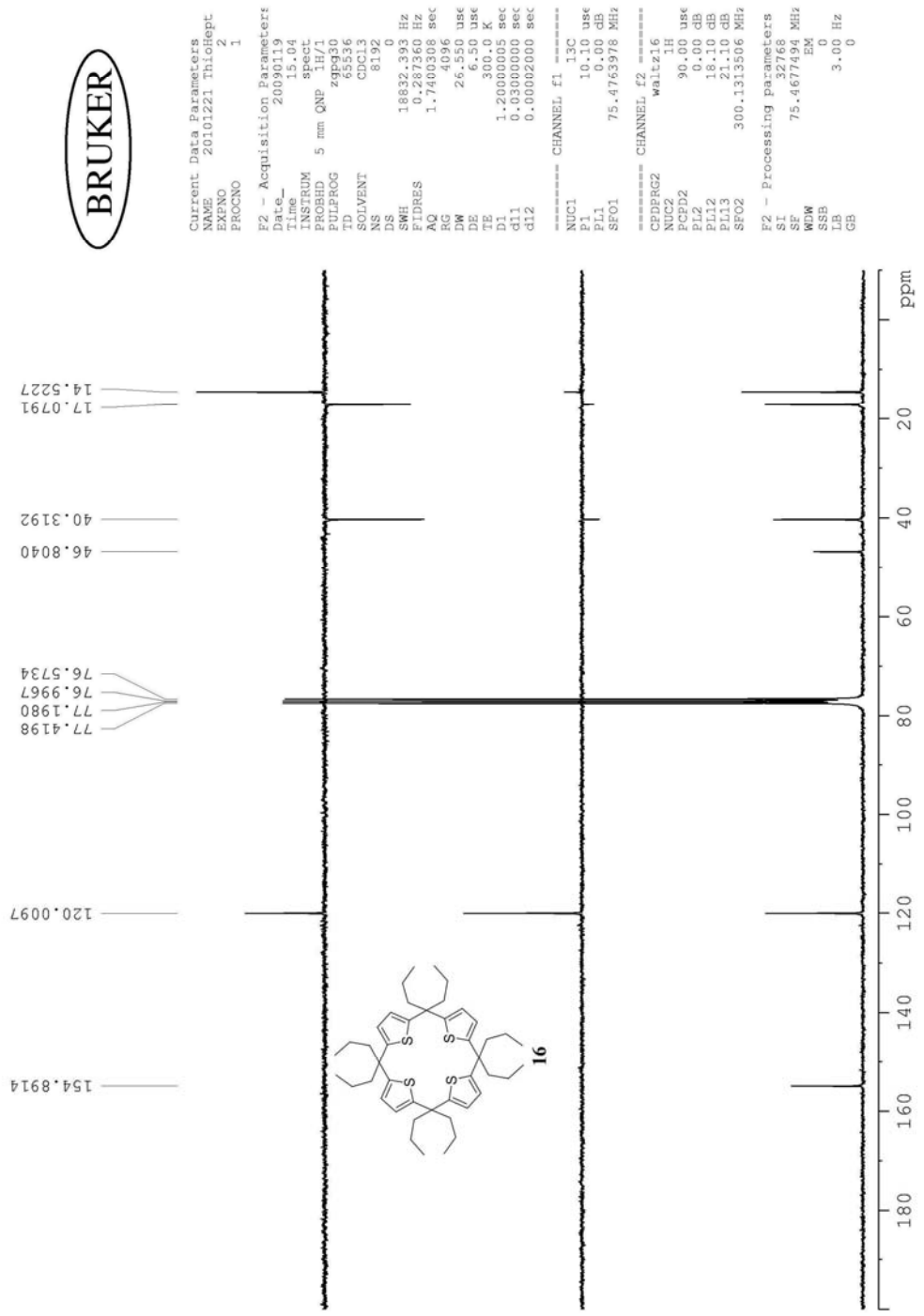

Current Data Parameters
NAME 20101221 Thio7 te
EXPNO 1
PROCNO 1
F2 - Acquisition Parameter
Date_ 20090925
Time 5.26
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 16384
SOLVENT CDC13
NS 16
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 se
RG 812.7
DW 111.200 us
DE 6.50 us
TE 300.0 K
D1 1.50000000 se
======== CHANNEL f1 ======
NUC1 1H
P1 10.70 us
PL1 0.00 dB
SFO1 300.1319508 MH
F2 - Processing parameters
SI 16384
SF 300.1300060 MH
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1.5479
1.5895
2.2769
2.2492
6.5861
7.2599
14
8.000
15.765
15.125
ppm

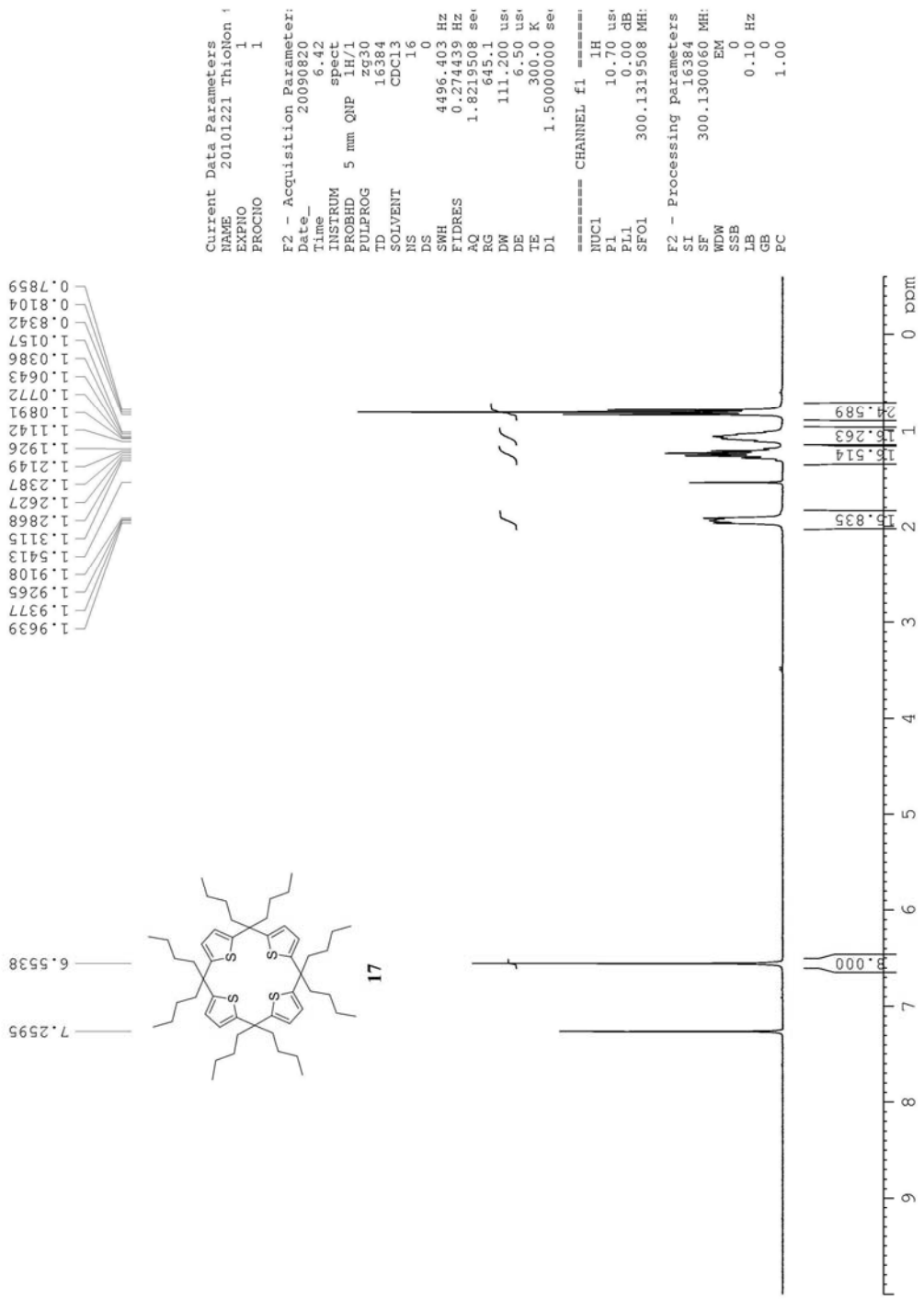

BRUKER
Current Data Parameters
NAME 20101221 Thio7 tet
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080501
Time 4.50
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4096
DS 0
SWH 18832.393 Hz
FIDRES 0.287360 Hz
AQ 1.7400308 sec
RG 4096
DW 26.550 use
DE 6.50 use
TE 300.0 K
D1 1.20000005 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.10 use
PL1 0.00 dB
SFO1 75.4763978 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 use
PL2 0.00 dB
PL12 18.10 dB
PL13 21.10 dB
SFO2 300.1313506 MHz
F2 - Processing parameters
SI 32768
SF 75.4677494 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
23.3529
29.8103
41.8930
47.5035
76.5775
77.0008
77.4241
119.5285
155.9963
14
ppm
20
40
60
80
100
120
140
160
180

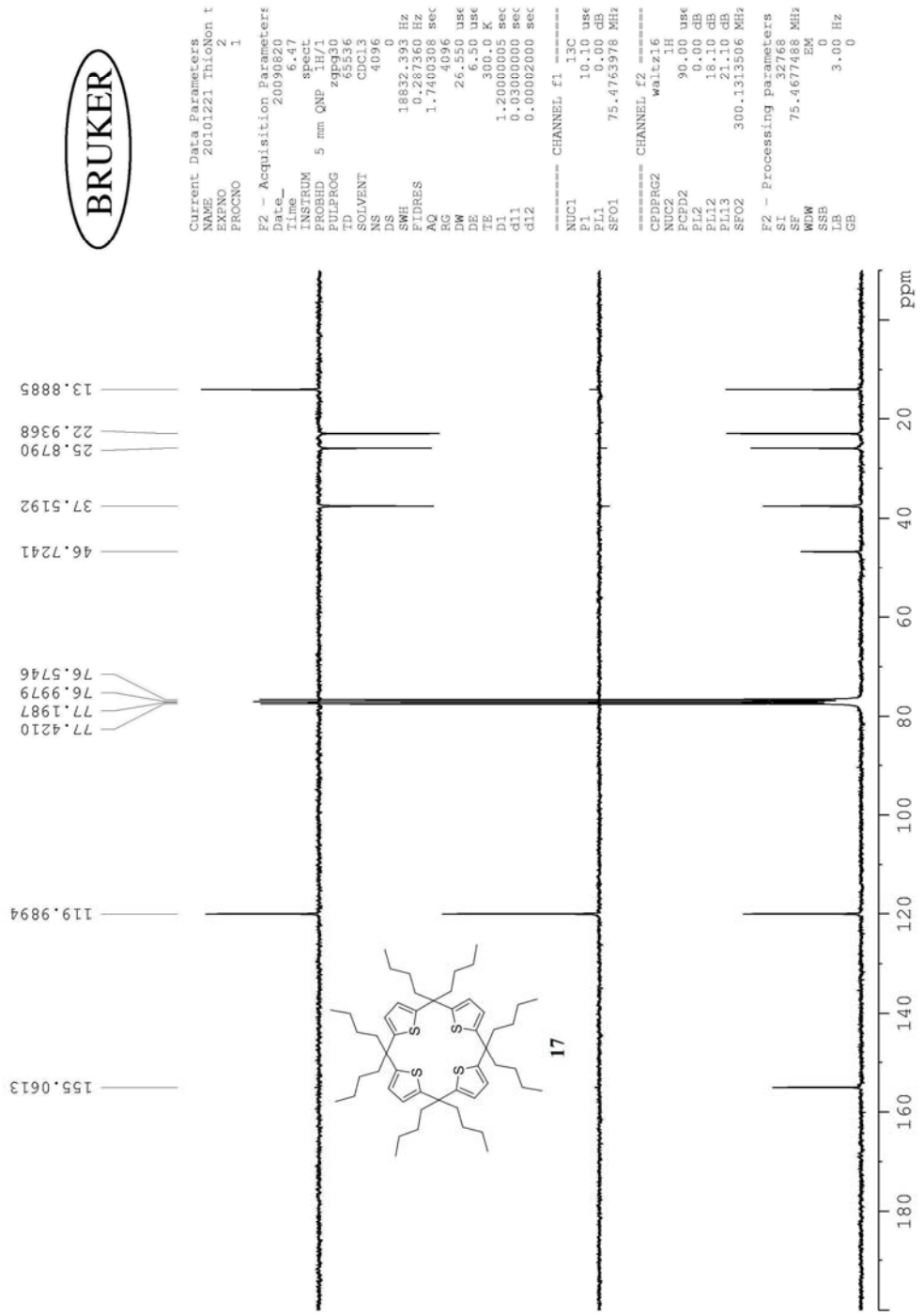

Current Data Parameters
NAME 20101221 ThioPent
EXPNO 1
PROCNO 1
F2 - Acquisition Parameter
Date_ 20091014
Time 16.25
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 se
RG 143.7
DW 111.200 us
DE 6.50 us
TE 300.0 K
D1 1.50000000 se
======== CHANNEL f1 ======
NUC1 1H
P1 10.70 us
PL1 0.00 dB
SFO1 300.1319508 MH
F2 - Processing parameters
SI 16384
SF 300.1300063 MH
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
0.7361
0.7119
0.6874
1.5449
1.9596
1.9837
2.0082
2.0325
7.2597
6.5752
24.679
16.485
7.997
ppm
15

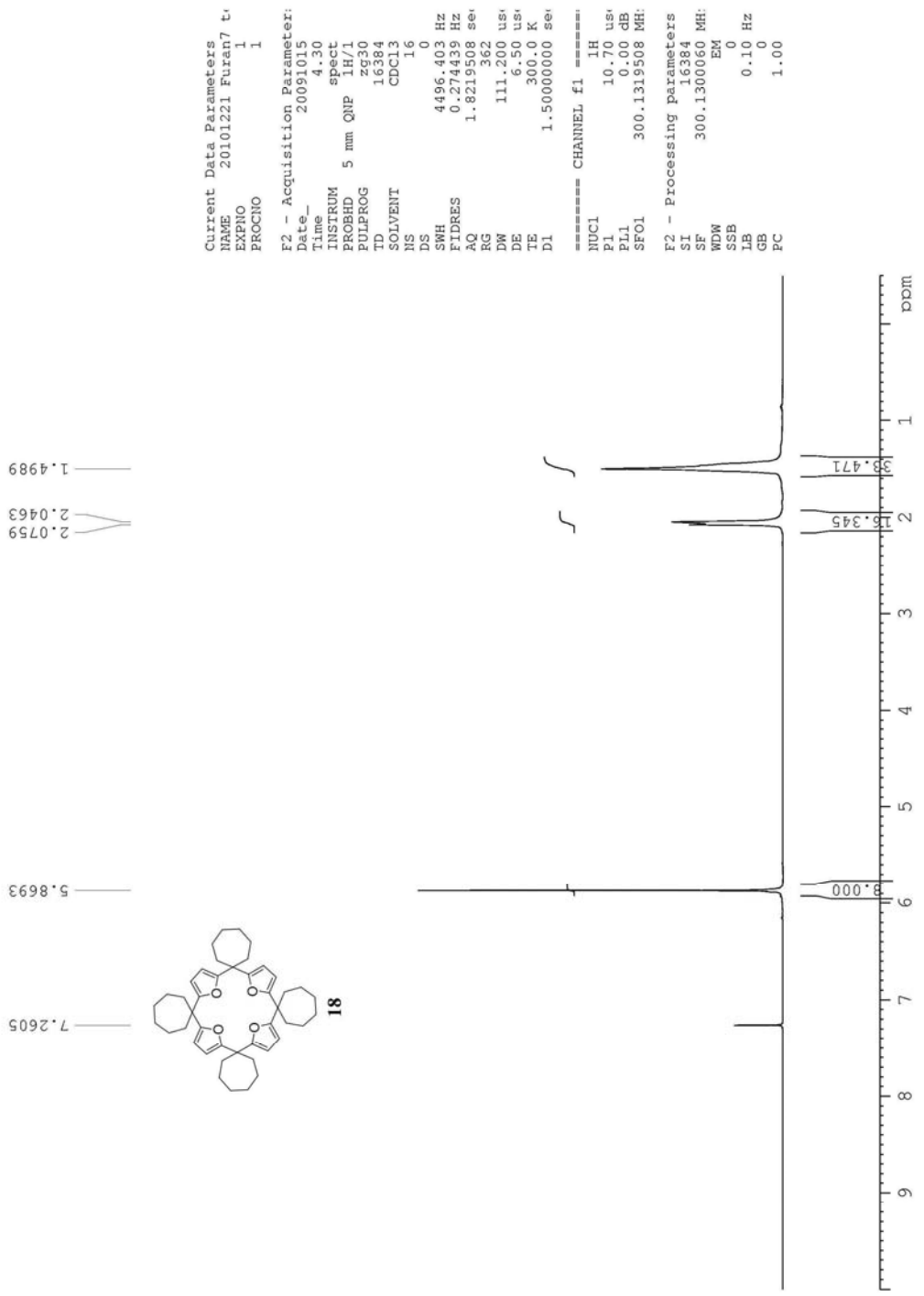

BRUKER
Current Data Parameters
NAME 20101221 ThioPent
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090219
Time 11.09
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4096
DS 0
SWH 18832.393 Hz
FIDRES 0.287360 Hz
AQ 1.7400308 sec
RG 4096
DW 26.550 use
DE 6.50 use
TE 300.0 K
D1 1.20000005 sec
d11 0.03000000 sec
d12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 10.10 use
PL1 0.00 dB
SFO1 75.4763978 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 use
PL2 0.00 dB
PL12 18.10 dB
PL13 21.10 dB
SFO2 300.1313506 MHz
F2 - Processing parameters
SI 32768
SF 75.4677494 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
8.1050
29.7634
47.2220
76.5740
76.9974
77.1989
77.4205
120.4546
154.5402
ppm
20
40
60
80
100
120
140
160
180
15

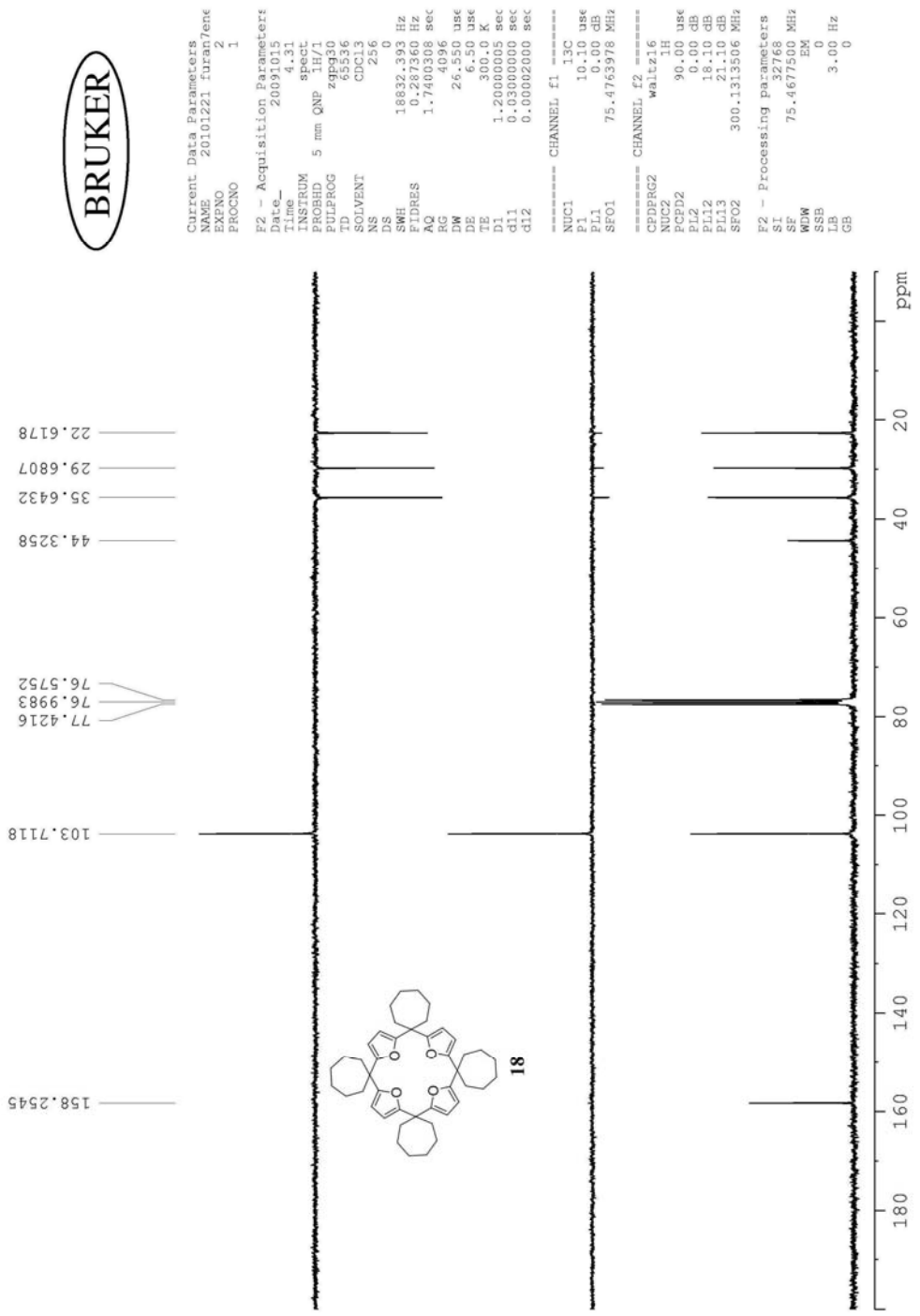

Current Data Parameters
NAME 20101221 ThioHept
EXPNO 1
PROCNO 1
F2 - Acquisition Parameter
Date_ 20090924
Time 22.19
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 8
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 se
RG 143.7
DW 111.200 us
DE 6.50 us
TE 300.0 K
D1 1.50000000 se
======== CHANNEL f1 ======
NUC1 1H
P1 10.70 us
PL1 0.00 dB
SFO1 300.1319508 MH
F2 - Processing parameters
SI 16384
SF 300.1300060 MH
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
24.560
16.310
15.889
8.000
7.2601
6.5534
1.9570
1.9420
1.9304
1.9171
1.9034
1.5394
1.1524
1.1273
1.1139
1.1005
1.0755
0.8829
0.8595
0.8349
16
ppm

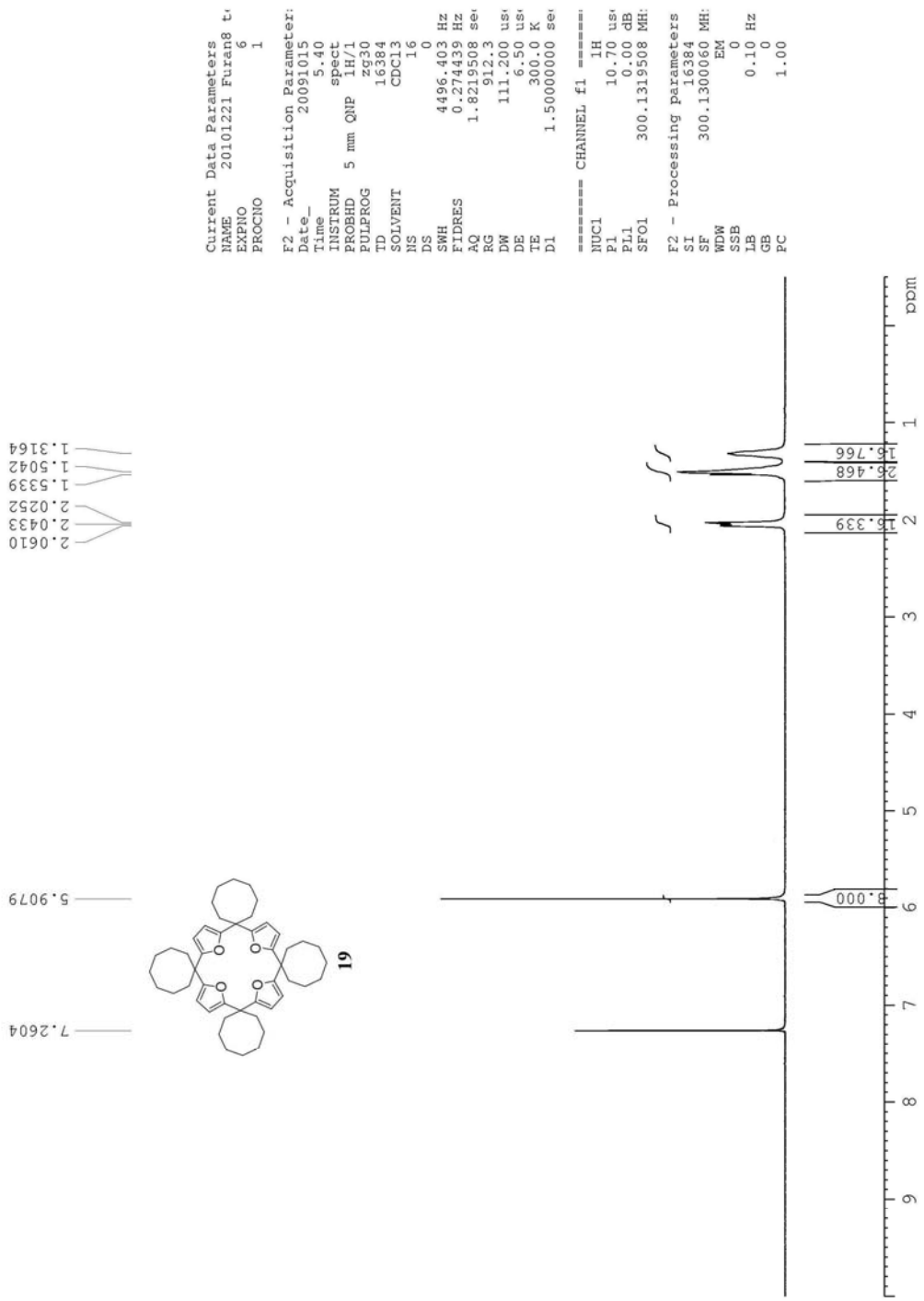

BRUKER
Current Data Parameters
NAME 20101221 ThioHept
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090119
Time 15.04
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 8192
DS 0
SWH 18832.393 Hz
FIDRES 0.287360 Hz
AQ 1.7400308 sec
RG 4096
DW 26.550 use
DE 6.50 use
TE 300.0 K
D1 1.20000005 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.10 use
PL1 0.00 dB
SFO1 75.4763978 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 use
PL2 0.00 dB
PL12 18.10 dB
PL13 21.10 dB
SFO2 300.1313506 MHz
F2 - Processing parameters
SI 32768
SF 75.4677494 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
14.5227
17.0791
40.3192
46.8040
76.5734
76.9967
77.1980
77.4198
120.0097
154.8914
ppm
20
40
60
80
100
120
140
160
180
16

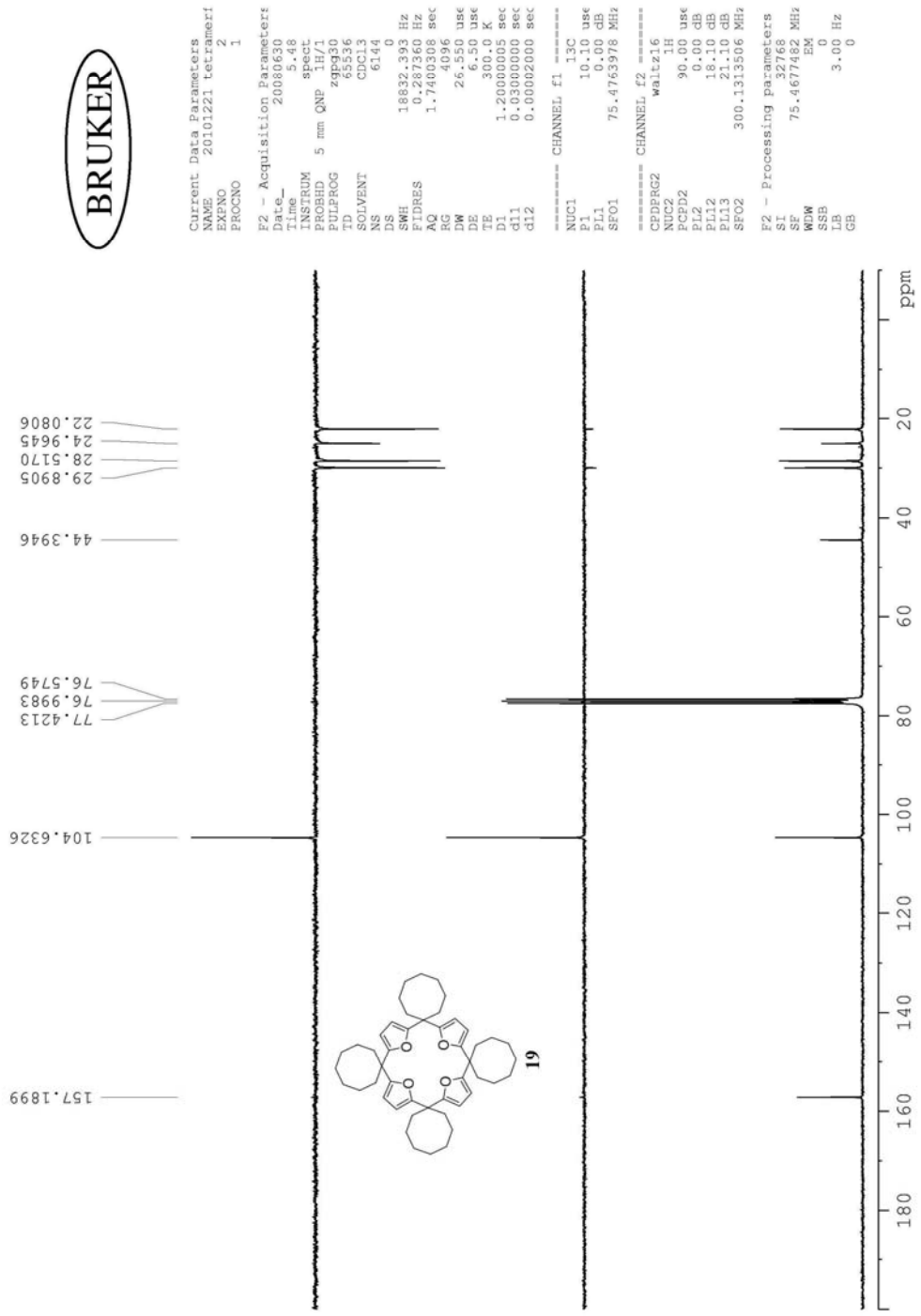

Current Data Parameters
NAME 20101221 ThioNon
EXPNO 1
PROCNO 1
F2 - Acquisition Parameter
Date_ 20090820
Time 6.42
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 se
RG 645.1
DW 111.200 us
DE 6.50 us
TE 300.0 K
D1 1.50000000 se
======== CHANNEL f1 ======
NUC1 1H
P1 10.70 us
PL1 0.00 dB
SFO1 300.1319508 MH
F2 - Processing parameters
SI 16384
SF 300.1300060 MH
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
0.7859
0.8104
0.8342
1.0157
1.0386
1.0643
1.0772
1.0891
1.1142
1.1926
1.2149
1.2387
1.2627
1.2868
1.3115
1.5413
1.9108
1.9265
1.9377
1.9639
6.5538
7.2595
17
8.000
15.835
15.514
15.263
24.589
ppm

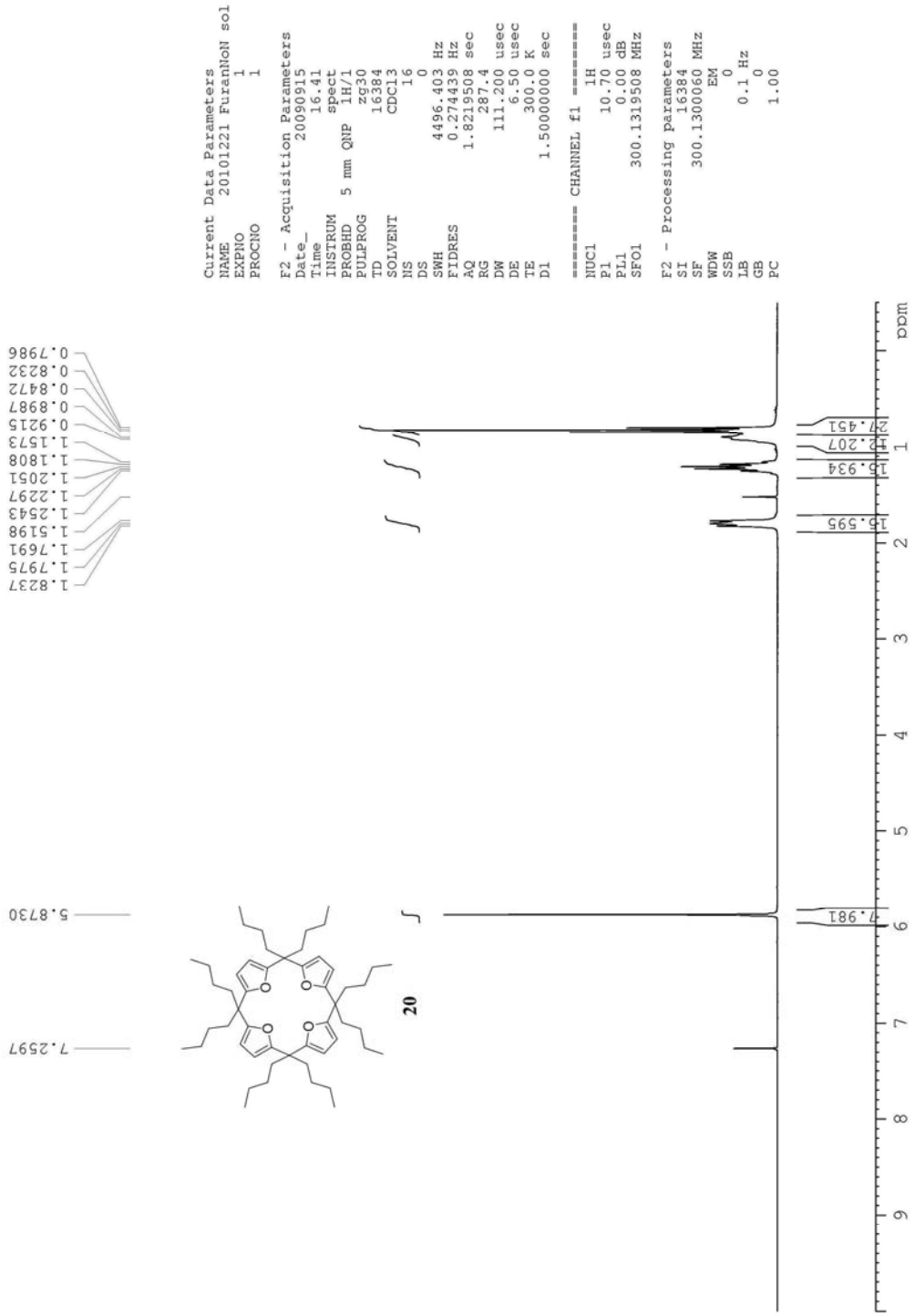

BRUKER
13.8885
22.9368
25.8790
37.5192
46.7241
76.5746
76.9979
77.1987
77.4210
119.9894
155.0613
ppm
17

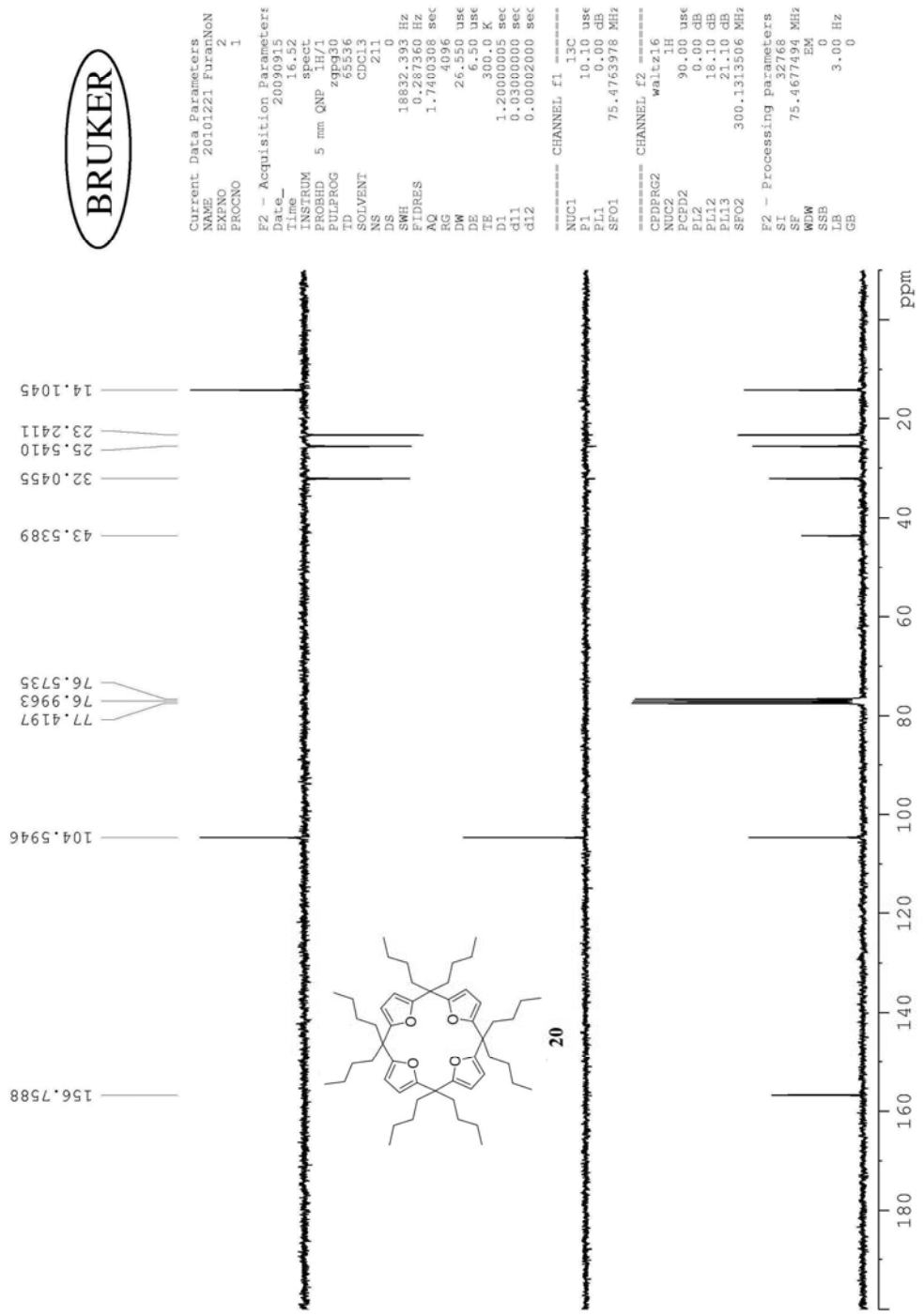

Current Data Parameters
NAME 20101221 Furan7 t
EXPNO 1
PROCNO 1
F2 - Acquisition Parameter
Date_ 20091015
Time 4.30
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 16384
SOLVENT CDC13
NS 16
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 se
RG 362
DW 111.200 us
DE 6.50 us
TE 300.0 K
D1 1.50000000 se
======== CHANNEL f1 ======
NUC1 1H
P1 10.70 us
PL1 0.00 dB
SFO1 300.1319508 MH
F2 - Processing parameters
SI 16384
SF 300.1300060 MH
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1.4989
2.0463
2.0759
5.8693
7.2605
18
38.471
15.345
8.000
ppm


BRUKER
Current Data Parameters
NAME 20101221 furan7ene
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20091015
Time 4.31
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 0
SWH 18832.393 Hz
FIDRES 0.287360 Hz
AQ 1.7400308 sec
RG 4096
DW 26.550 use
DE 6.50 use
TE 300.0 K
D1 1.20000005 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.10 use
PL1 0.00 dB
SFO1 75.4763978 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 use
PL2 0.00 dB
PL12 18.10 dB
PL13 21.10 dB
SFO2 300.1313506 MHz
F2 - Processing parameters
SI 32768
SF 75.4677500 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
22.6178
29.6807
35.6432
44.3258
76.5752
76.9983
77.4216
103.7118
158.2545
18
ppm
20
40
60
80
100
120
140
160
180


Current Data Parameters
NAME 20101221 Furan8 t
EXPNO 6
PROCNO 1
F2 - Acquisition Parameter
Date_ 20091015
Time 5.40
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 se
RG 912.3
DW 111.200 us
DE 6.50 us
TE 300.0 K
D1 1.50000000 se
======== CHANNEL f1 ======
NUC1 1H
P1 10.70 us
PL1 0.00 dB
SFO1 300.1319508 MH
F2 - Processing parameters
SI 16384
SF 300.1300060 MH
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00
1.3164
1.5042
1.5339
2.0252
2.0433
2.0610
5.9079
7.2604
8.000
15.339
25.468
15.766
19
ppm


BRUKER
Current Data Parameters
NAME 20101221 tetramerf
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080630
Time 5.48
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 6144
DS 0
SWH 18832.393 Hz
FIDRES 0.287360 Hz
AQ 1.7400308 sec
RG 4096
DW 26.550 use
DE 6.50 use
TE 300.0 K
D1 1.20000005 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.10 use
PL1 0.00 dB
SFO1 75.4763978 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 use
PL2 0.00 dB
PL12 18.10 dB
PL13 21.10 dB
SFO2 300.1313506 MHz
F2 - Processing parameters
SI 32768
SF 75.4677482 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
22.0806
24.9645
28.5170
29.8905
44.3946
76.5749
76.9983
77.4213
104.6326
157.1899
19
ppm
180
160
140
120
100
80
60
40
20


Current Data Parameters
NAME 20101221 FuranNoN sol
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090915
Time 16.41
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 287.4
DW 111.200 usec
DE 6.50 usec
TE 300.0 K
D1 1.50000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.70 usec
PL1 0.00 dB
SFO1 300.1319508 MHz
F2 - Processing parameters
SI 16384
SF 300.1300060 MHz
WDW EM
SSB 0
LB 0.1 Hz
GB 0
PC 1.00
7.981
15.934
15.595
12.207
27.451
ppm
20
5.8730
7.2597
1.8237
1.7975
1.7691
1.5198
1.2543
1.2297
1.2051
1.1808
1.1573
0.9215
0.8987
0.8472
0.8232
0.7986


BRUKER
Current Data Parameters
NAME 20101221 FuranNoN
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090915
Time 16.52
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 211
DS 0
SWH 18832.393 Hz
FIDRES 0.287360 Hz
AQ 1.7400308 sec
RG 4096
DW 26.550 use
DE 6.50 use
TE 300.0 K
D1 1.20000005 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.10 use
PL1 0.00 dB
SFO1 75.4763978 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 use
PL2 0.00 dB
PL12 18.10 dB
PL13 21.10 dB
SFO2 300.1313506 MHz
F2 - Processing parameters
SI 32768
SF 75.4677494 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
14.1045
23.2411
25.5410
32.0455
43.5389
76.5735
76.9963
77.4197
104.5946
156.7588
20
ppm