KHMDS Mediated Synthesis of 9-Arylfluorenes from Dibenzothiophene dioxides and Arylacetonitriles by Tandem S_NAr-Decyanation-Based Arylation

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Instrumentation and Chemicals

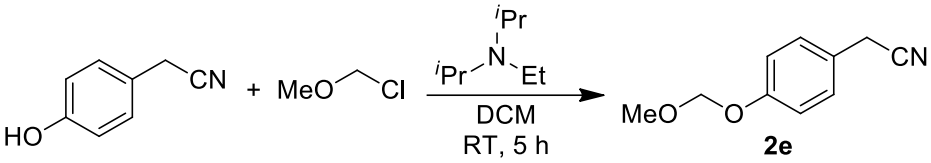
^{1}H NMR (500 MHz) and ^{13}C NMR (125 MHz) spectra were taken on a Bruker Avance 500 MHz NMR spectrometer. Chemical shifts (δ) are reported in parts per million, relative to chloroform at 7.26 ppm for ^{1}H and relative to $CDCl_3$ at 77.0 ppm for ^{13}C in $CDCl_3$. Data for ^{1}H NMR are reported as follows: chemical shift (ppm), multiplicity (s = singlet; bs = broad singlet; d = doublet; bd = broad doublet, t = triplet; bt = broad triplet; q = quartet; m = multiplet), coupling constants, *J*, in (Hz), and integration. Data for ^{13}C NMR was reported in terms of chemical shift (ppm). High-resolution mass spectra (HRMS) were obtained by using a TOF analyzer in ESI mode. X-ray data were taken at 273K with a Bruker APEX-II CCD single crystal diffractometer by using graphite monochromated Mo-Kα radiation (0.71073 Å). Data integration was done using SAINT.[1] Intensities for absorption were corrected using SADABS.[2] Structure solution and refinement were carried out using Bruker SHELX-TL.[3] TLC analyses were performed on commercial Aluminum TLC plate, silica gel coated with flourescent indicator F254. Silica gel (Merk 100-200 mesh) was used for column chromatography. Unless otherwise noted, materials obtained from commercial suppliers were used without further purification. Dibenzothiophene 5,5-dioxide (**1a**), benzyl cyanide (**2a**), and Potassium hexamethyldisilazide (0.5 M toluene solution) were purchased from Sigma-Aldrich. Pet ether was distilled over CaH_2 and stored under nitrogen. All the reactions were performed in an oven-dried glass reaction tubes.

Preparation of Starting Materials

The dibenzothiophene dioxide derivatives such as 2-phenyldibenzo[*b,d*]thiophene 5,5-dioxide (**1b**),[4] 2,8-diphenyldibenzo[*b,d*]thiophene 5,5-dioxide (**1c**),[5] 3-phenyldibenzo[*b,d*]thiophene 5,5-dioxide (**1e**),[5] and 2,3-diphenylbenzo[*b*]thiophene 1,1-dioxide (**5**)[5] were prepared according to the literature procedure.

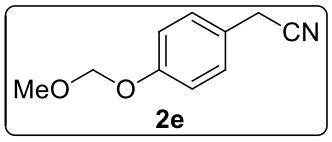
Experimental Procedures and Characterization Data:

Synthesis of 2-(4-(methoxymethoxy)phenyl)acetonitrile (2e):



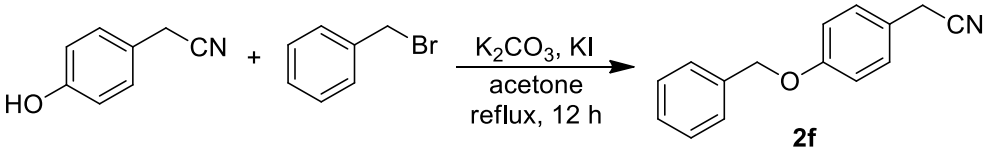
To a solution of 4-Hydroxyphenylacetonitrile (800 mg, 6.0 mmol) in dichloromethane was added N,N-Diisopropylethylamine (900 mg, 7.2 mmol) at room temperature. To this reaction mixture chloromethyl methyl ether (720 mg, 9.0 mmol) was added. The resulting solution was stirred for 5 h at room temperature. The reaction mixture was quenched with sat. $NaHCO_3$ solution and extracted with DCM. The organic layer was separated, dried over Na_2SO_4, and concentrated under vacuum. The crude residue was purified using column chromatography on silica gel (Hexane/EtOAc = 20/1) to provide 2-(4-(methoxymethoxy)phenyl)acetonitrile (**2e**) (710 mg, 4.0 mmol, 67% yield).

2-(4-(methoxymethoxy)phenyl)acetonitrile (2e):



Colorless liquid; ^{1}H NMR ($CDCl_3$)δ 7.23 (d, J = 8.0 Hz, 2H), 7.04 (d, J = 8.5 Hz, 2H), 5.17 (d, J = 5.0 Hz, 2H), 3.68 (s, 2H), 3.47 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$)δ 157.1, 129.2, 123.2, 118.2, 116.9, 94.4, 56.1, 22.9 ppm; HRMS (ESI-TOF) (*m/z*): $[M+Na]^+$ Calcd for $C_{10}H_{11}NO_2Na$, 200.0682; found, 200.0686.

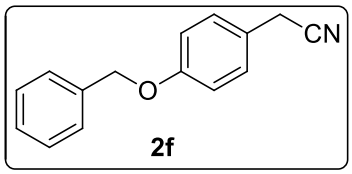
Synthesis of 2-(4-(benzyloxy)phenyl)acetonitrile (2f):



4-Hydroxyphenylacetonitrile (500 mg, 3.75 mmol), benzyl bromide (770 mg, 4.5 mmol), K_2CO_3 (1.03 g, 7.5 mmol), and KI (90 mg, 0.56 mmol) were taken in an oven-dried round bottom flask. Acetone (10 mL) was added to this mixture. The resulting solution was refluxed

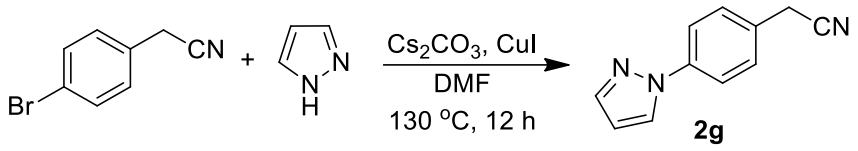
for 12 h. The reaction mixture was quenched with water and extracted with ethyl acetate. The organic layer was separated, dried over Na_2SO_4, and concentrated under vacuum. The crude residue was purified using column chromatography on silica gel (Hexane/EtOAc = 20/1) to provide 2-(4-(benzyloxy)phenyl)acetonitrile (**2f**) (710 mg, 3.18 mmol, 85% yield).

2-(4-(benzyloxy)phenyl)acetonitrile (2f):



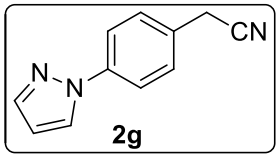
Colorless solid. mp = 65–67 °C; ^{1}H NMR ($CDCl_3$)δ 7.48–7.38 (m, 4H), 7.35 (t, J = 7.0 Hz, 1H), 7.25 (d, J = 8.5 Hz, 2H), 6.98 (d, J = 8.5 Hz, 2H), 5.08 (s, 2H), 3.69 (s, 2H) ppm; ^{13}C NMR ($CDCl_3$)δ158.5, 136.6, 129.1, 128.6, 128.1, 127.4, 122.0, 118.2, 154.4, 70.0, 22.8 ppm; HRMS (ESI-TOF) (*m/z*): $[M+H]^+$ Calcd for $C_{15}H_{14}NO$, 224.1070; found, 224.1080.

Synthesis of 2-(4-(1*H*-pyrazol-1-yl)phenyl)acetonitrile (2g):



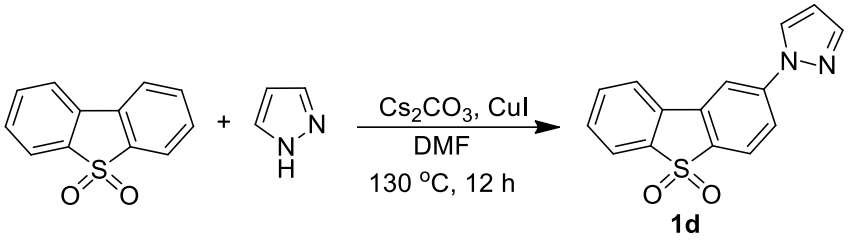
4-Bromophenylacetonitrile (500 mg, 2.55 mmol), pyrazole (260 mg, 3.8 mmol), Cs_2CO_3 (2.6 g, 8.0 mmol), and CuI (90 mg, 0.56 mmol) were taken in an oven-dried round bottom flask under N_2 atmosphere. DMF (5 mL) was added to this mixture. The resulting solution was stirred at 130 °C for 12 h. The reaction mixture was diluted with water and extracted with ethyl acetate. The organic layer was separated, dried over Na_2SO_4, and concentrated under vacuum. The crude residue was purified using column chromatography on silica gel (Hexane/EtOAc = 9/1) to provide 2-(4-(benzyloxy)phenyl)acetonitrile (**2f**) (112 mg, 0.61 mmol, 24% yield).

2-(4-(1*H*-pyrazol-1-yl)phenyl)acetonitrile (2g):



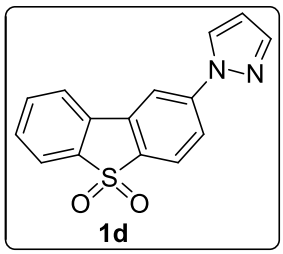
Light orange solid. mp = 127–128 °C; ^{1}H NMR ($CDCl_3$)δ 7.94 (bd, J = 2.5 Hz, 1H), 7.76-7.70 (m, 3H), 7.42 (d, J = 8.0 Hz, 2H), 6.49 (bt, J = 1.5 Hz, 1H), 3.80 (s, 2H) ppm; ^{13}C NMR ($CDCl_3$)δ 141.4, 140.0, 129.0, 127.8, 126.7, 119.6, 117.6, 107.9, 23.11 ppm; HRMS (ESI-TOF) (*m/z*): $[M+H]^+$ Calcd for $C_{11}H_{10}N_3$, 184.0869; found, 184.0869.

Synthesis of 2-(1*H*-pyrazol-1-yl)dibenzo[*b*,*d*]thiophene 5,5-dioxide (1d):



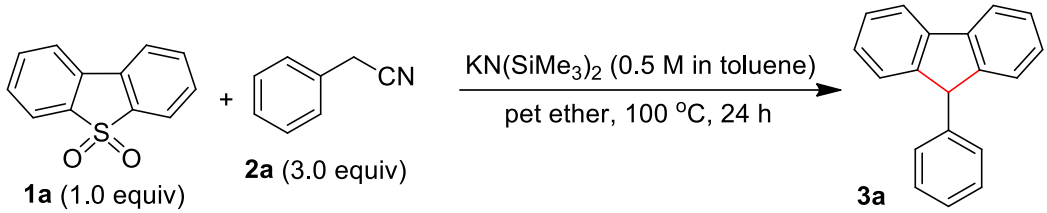
2-Bromodibenzothiophene 5,5-dioxide (200 mg, 0.67 mmol), pyrazole (900 mg, 1.4 mmol), Cs_2CO_3 (848 g, 2.6 mmol), and CuI (50 mg, 0.26 mmol) were taken in an oven-dried Schlenk flask under an argon atmosphere. DME (3 mL) was added to this mixture. The resulting solution was stirred at 130 °C for 12 h. The reaction mixture was diluted with water and extracted with ethyl acetate. The organic layer was separated, dried over Na_2SO_4, and concentrated under vacuum. The crude residue was purified using column chromatography on silica gel (Hexane/EtOAc = 5/1) to provide 2-(1*H*-pyrazol-1-yl)dibenzo[*b*,*d*]thiophene 5,5-dioxide **(1d)** (121 mg, 0.43 mmol, 64% yield).

2-(1*H*-pyrazol-1-yl)dibenzo[*b*,*d*]thiophene 5,5-dioxide (1d):



Colorless solid. mp = 224–225 °C; ^{1}H NMR ($CDCl_3$)δ 8.26 (bd, J = 1.5 Hz, 1H), 8.06 (bd, J = 2.5 Hz, 1H), 7.89 (d, J = 8.5 Hz, 2H), 7.86 (d, J = 8.0 Hz, 1H), 7.82 (s, 1H), 7.77 (dd, J = 8.0, 1.5 Hz, 1H), 7.69 (t, J = 7.5 Hz, 1H), 7.59 (t, J = 7.5 Hz, 1H), 6.58 (bt, J = 2.5 Hz, 1H) ppm; ^{13}C NMR ($CDCl_3$)δ 144.3, 142.4, 138.3, 134.6, 134.0, 133.7, 130.9, 130.8, 127.0, 123.5, 122.1, 121.9, 119.4, 111.9, 109.1 ppm; HRMS (ESI-TOF) (*m/z*): $[M+H]^+$ Calcd for $C_{15}H_{11}N_2O_2S$, 283.0536; found, 283.0536.

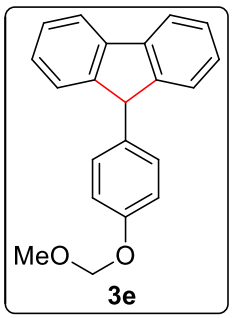
General Procedure for the Preparation of 9-Arylfluorenes:



Preparation of **3a** is representative. To a solution of dibenzo[*b*,*d*]thiophene 5,5-dioxide (**1a**) (108 mg, 0.5 mmol) and benzyl cyanide (**2a**) (351 mg, 1.5 mmol) in pet ether (1.0 mL) was added dropwise a solution of KHMDS (0.5 M in toluene, 3.0 mL, 1.5 mmol) under argon atmosphere at ambient temperature. The resulting solution was stirred at 100 °C for 24 h. The reaction mixture was quenched with sat. NH_4Cl solution (3 mL) and extracted with ethyl acetate (3 x 20 mL). The organic layer was separated, dried over Na_2SO_4, and concentrated under vacuum. The crude residue was purified by using column chromatography on silica gel (Hexane only) to provide 9-phenyl-9*H*-fluorene (**3a**) (99 mg, 0.41 mmol, 81% yield) as a colourless solid.

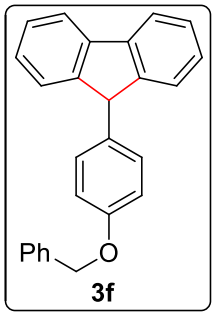
Compounds **3a**,[6] **3b**,[7] **3c**,[8] **3d**,[7] **3h**,[9] **3i-k**,[10] and **6**[11] are known compounds and showed the identical spectra according to the literature.

9-(4-(methoxymethoxy)phenyl)-9*H*-fluorene (3e):



Colorless solid. mp = 99–100 °C; ^{1}H NMR ($CDCl_3$) δ 7.80 (d, J = 7.5 Hz, 2H), 7.38 (t, J = 7.5 Hz, 2H), 7.32 (d, J = 7.5 Hz, 2H), 7.27 (t, J = 7.5 Hz, 2H), 7.02 (d, J = 8.5 Hz, 2H), 6.95 (d, J = 8.5 Hz, 2H), 5.15 (s, 2H), 5.02 (s, 1H), 3.48 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$) δ 156.2, 148.1, 140.9, 134.9, 129.3, 127.3, 125.3, 119.8, 116.5, 94.6, 56.0, 53.7 ppm; HRMS (ESI-TOF) (*m/z*): $[M+H]^+$ Calcd for $C_{21}H_{19}O_2$, 303.1380; found, 303.1387.

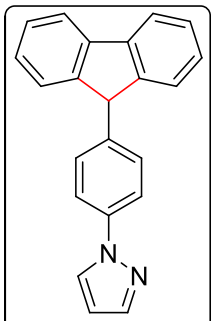
9-(4-(benzyloxy)phenyl)-9*H*-fluorene (3f):



3f

Colorless solid. mp = 144–146 °C; ^{1}H NMR ($CDCl_3$)δ 7.80 (d, J = 7.5 Hz, 2H), 7.42 (d, J = 7.0 Hz, 2H), 7.38 (t, J = 7.5 Hz, 4H), 7.32 (d, J = 7.5 Hz, 3H), 7.27 (t, J = 7.5 Hz, 2H), 7.02 (d, J = 9.0 Hz, 2H), 6.89 (d, J = 9.0 Hz, 2H), 5.03 (s, 2H), 5.01 (s, 1H) ppm; ^{13}C NMR ($CDCl_3$)δ 151.9, 148.2, 140.9, 137.1, 133.9, 129.3, 128.6, 127.9, 127.5, 127.3, 127.2, 125.3, 119.8, 115.1, 70.1, 53.7 ppm; HRMS (ESI-TOF) (*m/z*): $[M+H]^+$ Calcd for $C_{26}H_{21}O$, 349.1587; found, 349.1593.

1-(4-(9*H*-fluoren-9-yl)phenyl)-1*H*-pyrazole (3g):

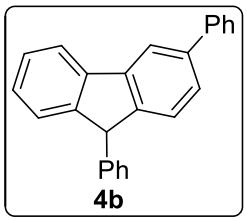


3g

Colorless solid. mp = 191–192 °C; ^{1}H NMR ($CDCl_3$)δ 7.87 (bd, J = 2.0 Hz, 1H), 7.83 (d, J = 8.0 Hz, 2H), 7.71 (s, 1H), 7.60 (d, J = 8.5 Hz, 2H), 7.41 (t, J = 7.5 Hz, 2H), 7.33 (d, J = 7.5 Hz, 2H), 7.29 (d, J = 7.0 Hz, 2H), 7.18 (d, J = 8.5, 2H), 6.45 (bt, J = 2.0 Hz, 1H), 5.10 (s, 1H), ppm; ^{13}C NMR ($CDCl_3$)δ 147.6, 141.0, 140.9, 140.0, 139.1, 129.3, 127.5, 127.4, 126.7, 125.3,

120.0, 119.6, 107.5, 53.8 ppm; HRMS (ESI-TOF) (*m/z*): $[M+H]^+$ Calcd for $C_{22}H_{17}N_2$, 309.1386; found, 309.1386.

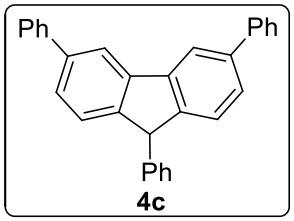
3,9-diphenyl-9*H*-fluorene (4b):



4b

Colorless solid; mp = 118–120 °C; ^{1}H NMR ($CDCl_3$)δ 8.00 (s, 1H), 7.87 (d, J = 7.5, 1H), 7.68 (d, J = 7.5 Hz, 2H), 7.49 (t, J = 8.0 Hz, 3H), 7.45–7.33 (m, 4H), 7.33 – 7.23 (m, 4H), 7.14 (d, J = 7.0 Hz, 2H), 5.11 (s, 1H) ppm; ^{13}C NMR ($CDCl_3$)δ 148.3, 147.0, 141.6, 141.52, 141.48, 140.8, 140.7, 128.8, 128.7, 128.3, 127.5, 127.4, 127.3, 127.2, 126.9, 126.6, 125.5, 125.4, 119.9, 118.6, 54.2 ppm; HRMS (ESI-TOF) (*m/z*): $[M]^+$ Calcd for $C_{25}H_{18}$, 318.1409; found, 318.1409.

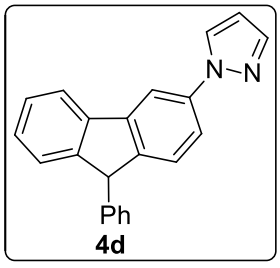
3,6,9-triphenyl-9H-fluorene (4c):



4c

Colorless solid. mp = 166–167 °C; ^{1}H NMR ($CDCl_3$)δ 8.08 (s, 2H), 7.70 (bd, J = 7.0 Hz, 4H), 7.55–7.46 (m, 6H), 7.44–7.37 (m, 4H), 7.34–7.30 (m, 2H), 7.29–7.24 (m, 1H), 7.19 (bd, J = 7.5 Hz, 2H), 5.16 (s, 1H) ppm; ^{13}C NMR ($CDCl_3$)δ 147.3, 141.45, 141.43, 141.4, 140.7, 128.8 (2C), 128.3, 127.30, 127.26, 126.9, 126.7, 125.6, 118.6, 54.0 ppm; HRMS (ESI-TOF) (*m/z*): $[M+H]^+$ Calcd for $C_{31}H_{23}$, 395.1799; found, 395.1809.

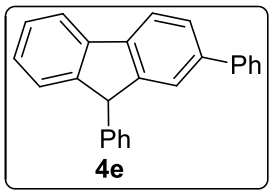
1-(9-phenyl-9*H*-fluoren-3-yl)-1*H*-pyrazole (4d):



4d

Colorless solid. mp = 93–94 °C; ^{1}H NMR ($CDCl_3$)δ 8.16 (bd, J = 2.0 Hz, 1H), 8.01 (bd, J = 2.5 Hz, 1H), 7.86 (d, J = 7.5 Hz, 1H), 7.78 (d, J = 1.5 Hz, 1H), 7.54 (dd, J = 8.5, 2.0 Hz, 1H), 7.42 (t, J = 7.5 Hz, 1H), 7.38 (d, J = 8.0 Hz, 1H), 7.36–7.23 (m, 5H), 7.11 (d, J = 7.5 Hz, 2H), 6.51 (t, J = 2.0 Hz, 1H), 5.10 (s, 1H) ppm; ^{13}C NMR ($CDCl_3$)δ 148.5, 146.0, 142.5, 141.1, 141.0, 140.2, 139.9, 128.8, 128.3, 127.9, 127.5, 127.03, 126.98, 126.0, 125.4, 120.2, 118.3, 111.2, 107.6, 54.1 ppm; HRMS (ESI-TOF) (*m/z*): $[M+H]^+$ Calcd for $C_{22}H_{17}N_2$, 309.1386; found, 309.1386.

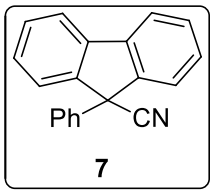
2,9-Diphenyl-9*H*-fluorene (4e):



4e

Colorless solid. mp = 178–189 °C; ^{1}H NMR ($CDCl_3$)δ 7.87 (d, J = 8.0 Hz, 1H), 7.84 (d, J = 8.0 Hz, 1H), 7.64 (d, J = 7.5 Hz, 1H), 7.59 (d, J = 7.0 Hz, 2H), 7.54 (s, 1H), 7.42 (t, J = 7.5 Hz, 3H), 7.36–7.23 (m, 6H), 7.14 (d, J = 7.0 Hz, 2H), 5.12 (s, 1H) ppm; ^{13}C NMR ($CDCl_3$)δ 148.5, 148.1, 141.4, 141.2, 140.6, 140.4, 140.2, 128.71, 128.68, 128.4, 127.4, 127.3, 127.1 (2C), 126.9, 126.5, 125.3, 124.1, 120.1, 119.9, 54.5 ppm; HRMS (ESI-TOF) (*m/z*): $[M+H]^+$ Calcd for $C_{22}H_{19}$, 319.1481; found, 319.1478.

9-phenyl-9*H*-fluorene-9-carbonitrile (7):



7

Colorless solid.mp = 150–151 °C; ^{1}H NMR ($CDCl_3$)δ 7.81 (d, J = 7.5 Hz, 2H), 7.49 (t, J = 7.5 Hz, 2H), 7.45 (d, J = 7.5 Hz, 2H), 7.35 (t, J = 7.5 Hz, 2H), 7.33–7.25 (m, 5H) ppm; ^{13}C NMR ($CDCl_3$)δ 145.1, 140.2, 137.8, 129.6, 129.2, 128.9, 128.4, 126.2, 125.4, 120.7, 120.3, 54.0 ppm; HRMS (ESI-TOF) (*m/z*): $[M+H]^+$ Calcd for $C_{20}H_{14}N$, 268.1121; found, 268.1119.

X-ray Crystallographic Analysis

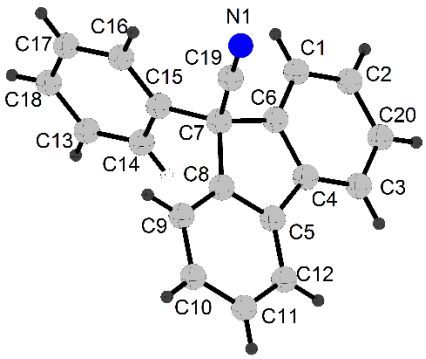


Figure S1. Molecular structures of compounds **7** thermal ellipsoids are set at 30% probability and hydrogen atoms omitted for clarity.

Table S1. Crystal data and structure refinements for **7**.

Compound	**7**
Formula	$C_{20}H_{13}N$
Fw	267.31
Crystal System	Monoclinic
Space Group	$P2_1/c$
a	11.6754(5) Å
b	11.0520(4) Å
c	11.6067(4) Å
α	90°
β	108.497(1)°
γ	90°
Volume	1420.32(9) $Å^3$
Z	4
Density (calcd.)	1.250 g / cm^3
Completeness	0.992
GOF (all data)	1.043
R_1 ($I>2\sigma(I)$)	0.0477
wR_2 (all data)	0.1437
CCDC No.	1866811

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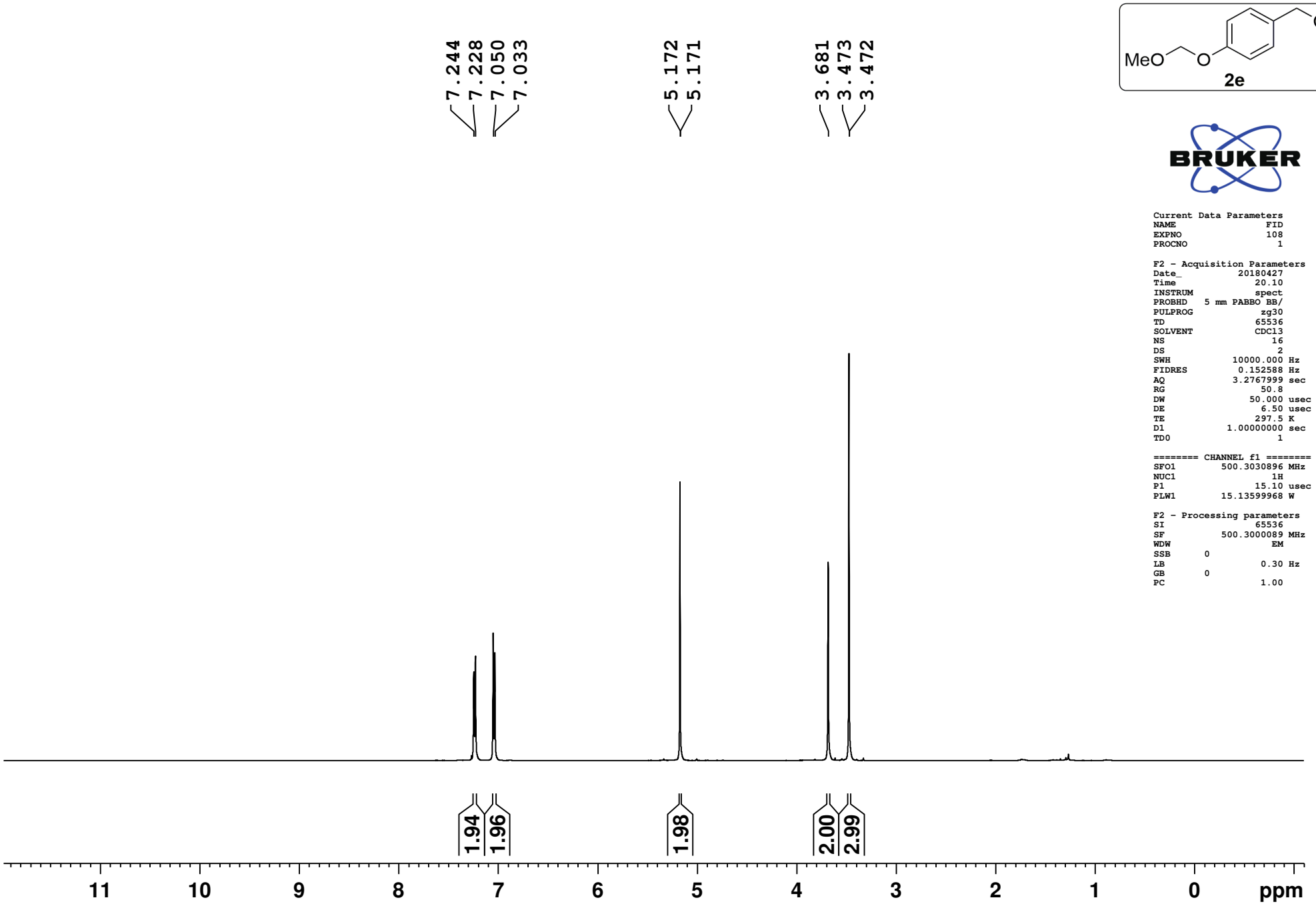
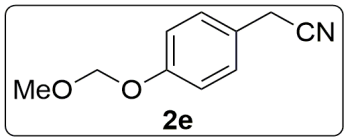




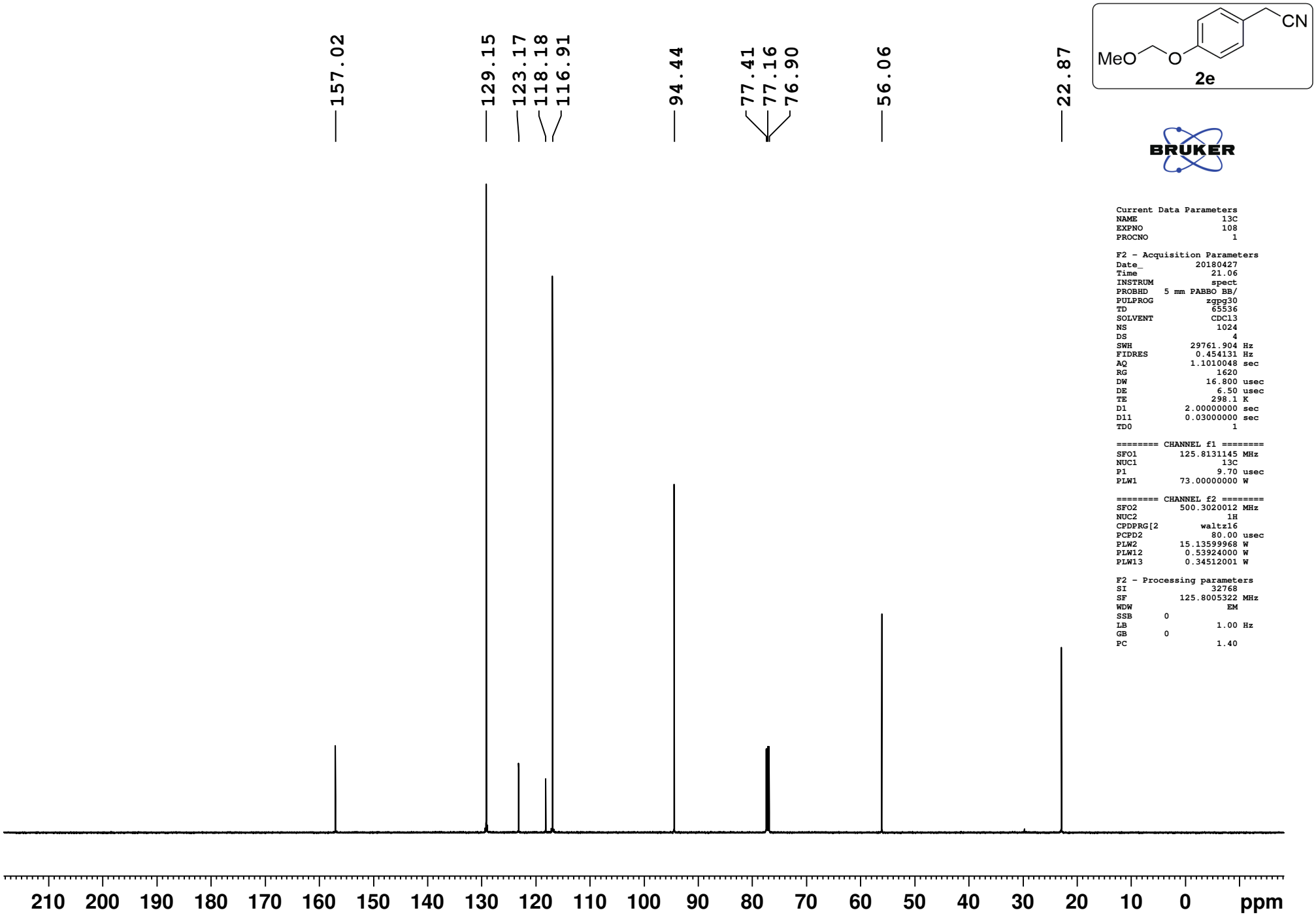
BRUKER

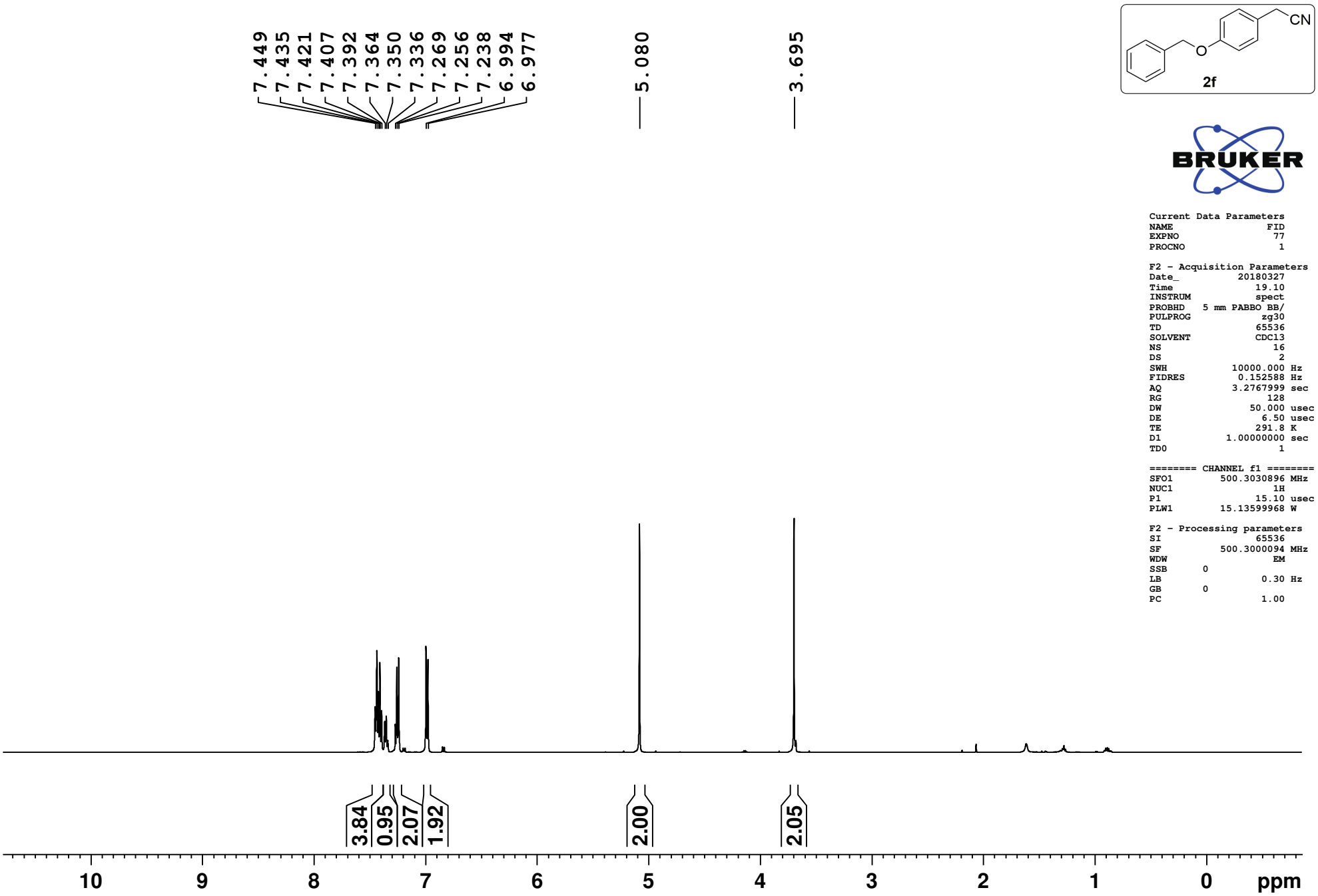
Current Data Parameters
NAME FID
EXPNO 108
PROCNO 1

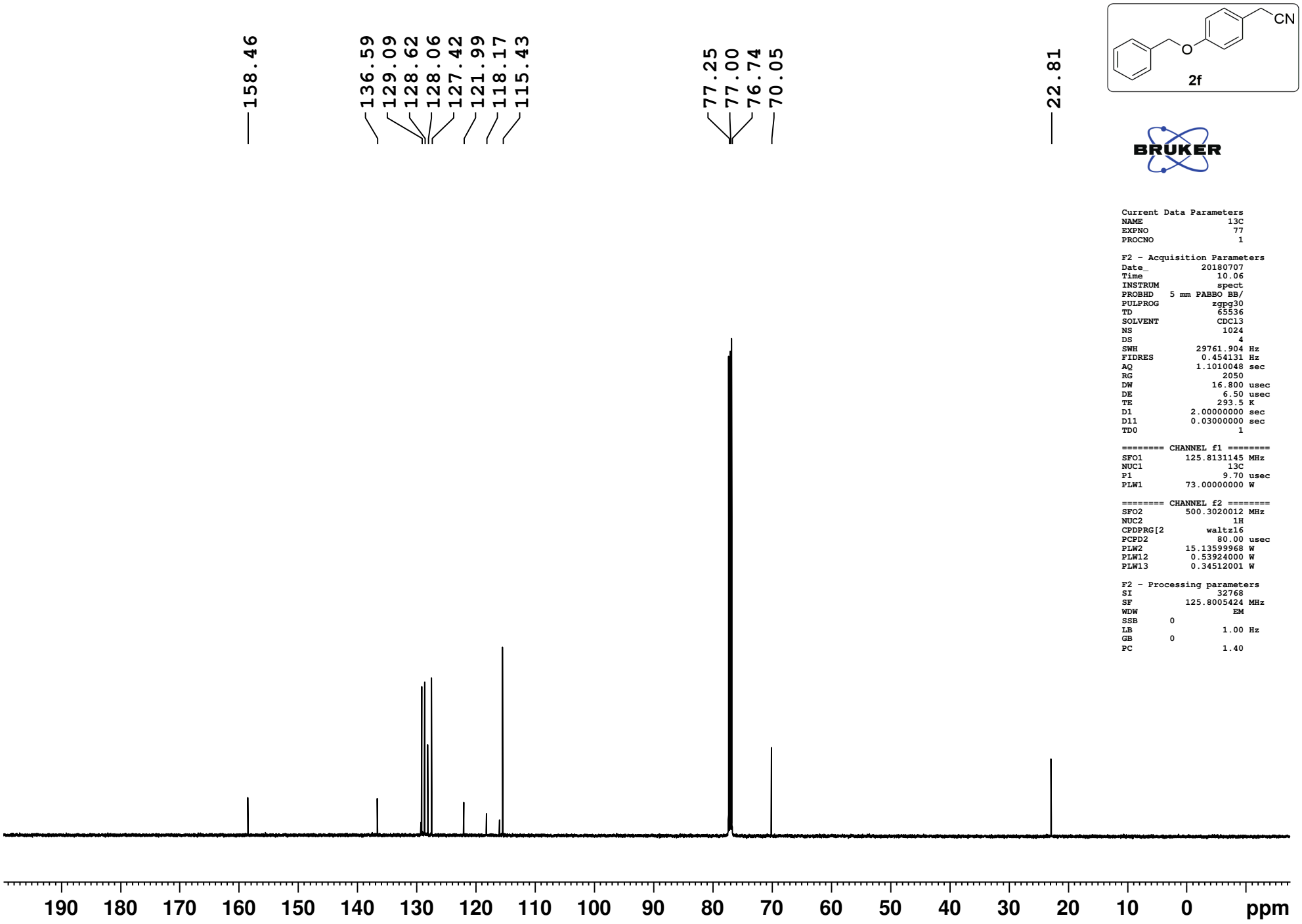
F2 - Acquisition Parameters
Date_ 20180427
Time 20.10
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 50.8
DW 50.000 usec
DE 6.50 usec
TE 297.5 K
D1 1.00000000 sec
TD0 1

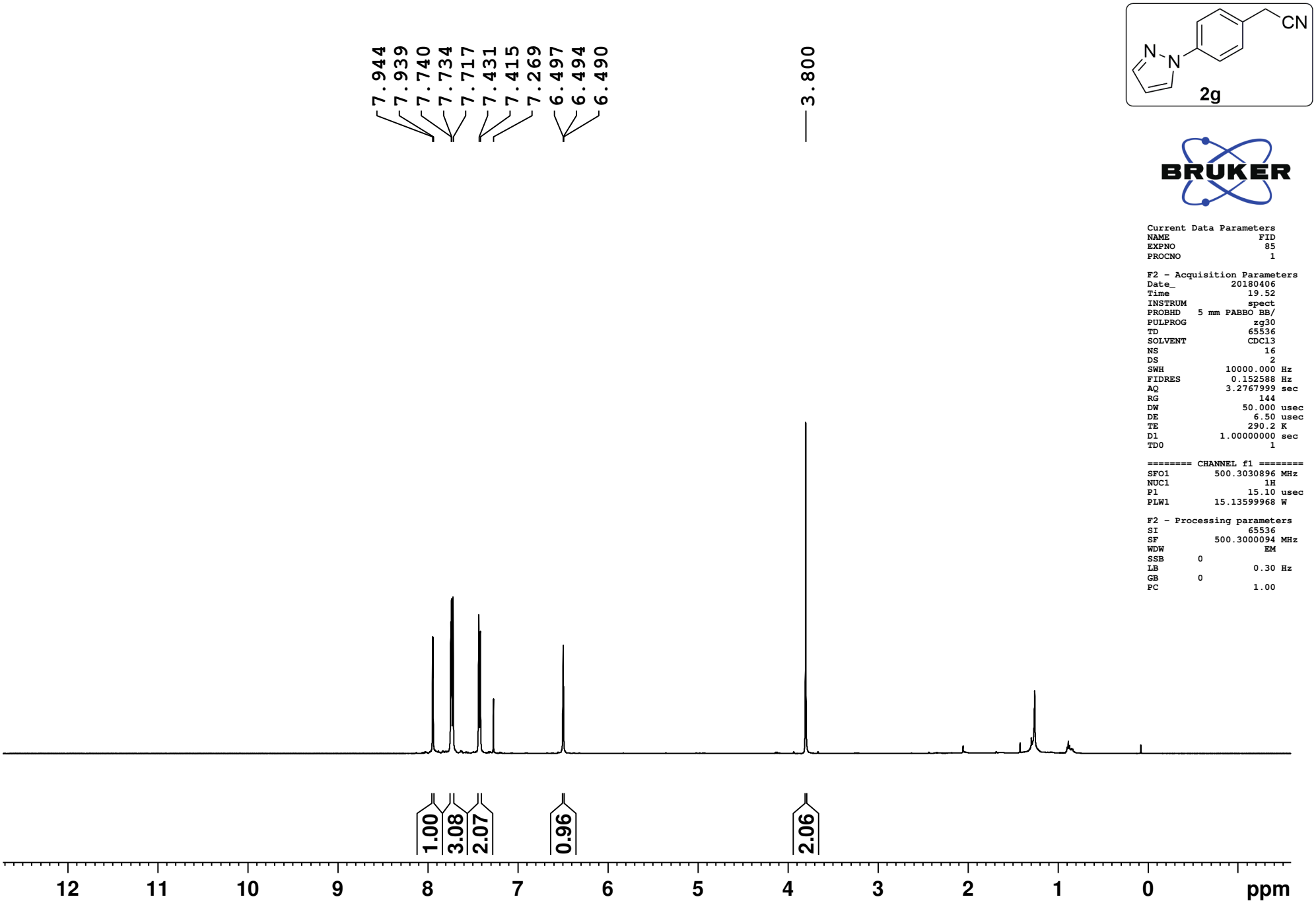
======== CHANNEL f1 ========
SFO1 500.3030896 MHz
NUC1 1H
P1 15.10 usec
PLW1 15.13599968 W

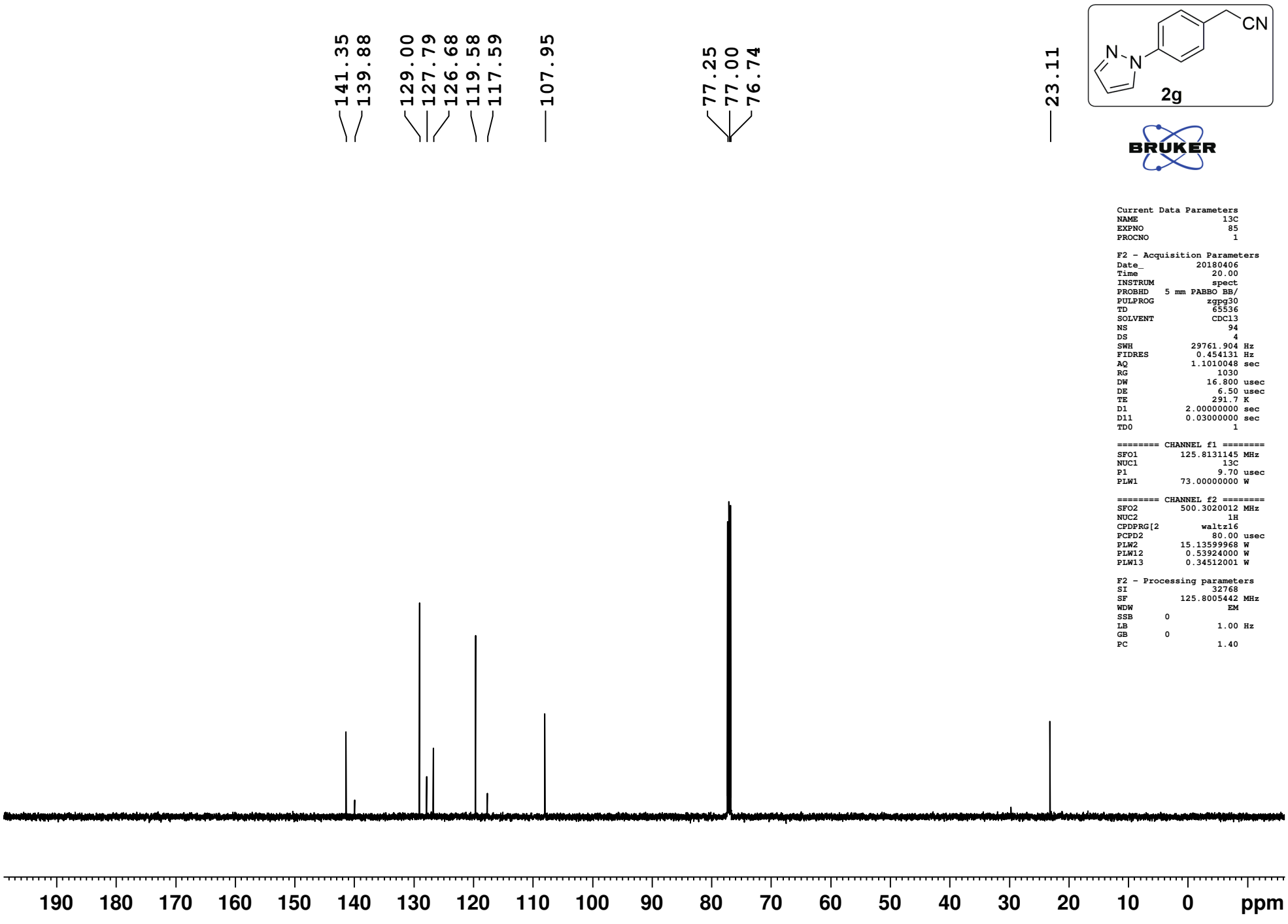
F2 - Processing parameters
SI 65536
SF 500.3000089 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

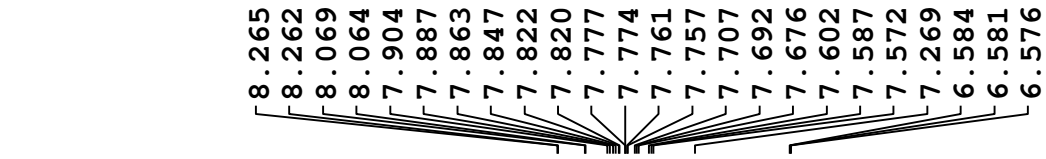

157.02
129.15
123.17
118.18
116.91
94.44
77.41
77.16
76.90
56.06
22.87
MeO
O
CN
2e
BRUKER
Current Data Parameters
NAME 13C
EXPNO 108
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180427
Time 21.06
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 1620
DW 16.800 usec
DE 6.50 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.8131145 MHz
NUC1 13C
P1 9.70 usec
PLW1 73.00000000 W
======== CHANNEL f2 ========
SFO2 500.3020012 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.13599968 W
PLW12 0.53924000 W
PLW13 0.34512001 W
F2 - Processing parameters
SI 32768
SF 125.8005322 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

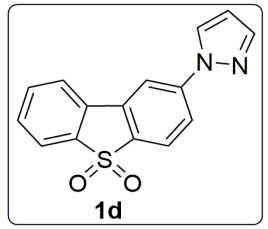

7.449
7.435
7.421
7.407
7.392
7.364
7.350
7.336
7.269
7.256
7.238
6.994
6.977
5.080
3.695
CN
O
2f
BRUKER
Current Data Parameters
NAME FID
EXPNO 77
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180327
Time 19.10
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 128
DW 50.000 usec
DE 6.50 usec
TE 291.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.3030896 MHz
NUC1 1H
P1 15.10 usec
PLW1 15.13599968 W
F2 - Processing parameters
SI 65536
SF 500.3000094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
3.84
0.95
2.07
1.92
2.00
2.05
10
9
8
7
6
5
4
3
2
1
0
ppm


158.46
136.59
129.09
128.62
128.06
127.42
121.99
118.17
115.43
77.25
77.00
76.74
70.05
22.81
CN
O
2f
BRUKER
Current Data Parameters
NAME 13C
EXPNO 77
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180707
Time 10.06
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 293.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.8131145 MHz
NUC1 13C
P1 9.70 usec
PLW1 73.00000000 W
======== CHANNEL f2 ========
SFO2 500.3020012 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.13599968 W
PLW12 0.53924000 W
PLW13 0.34512001 W
F2 - Processing parameters
SI 32768
SF 125.8005424 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.944
7.939
7.740
7.734
7.717
7.431
7.415
7.269
6.497
6.494
6.490
3.800
CN
N
N
2g
BRUKER
Current Data Parameters
NAME FID
EXPNO 85
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180406
Time 19.52
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 144
DW 50.000 usec
DE 6.50 usec
TE 290.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.3030896 MHz
NUC1 1H
P1 15.10 usec
PLW1 15.13599968 W
F2 - Processing parameters
SI 65536
SF 500.3000094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.00
3.08
2.07
0.96
2.06
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm

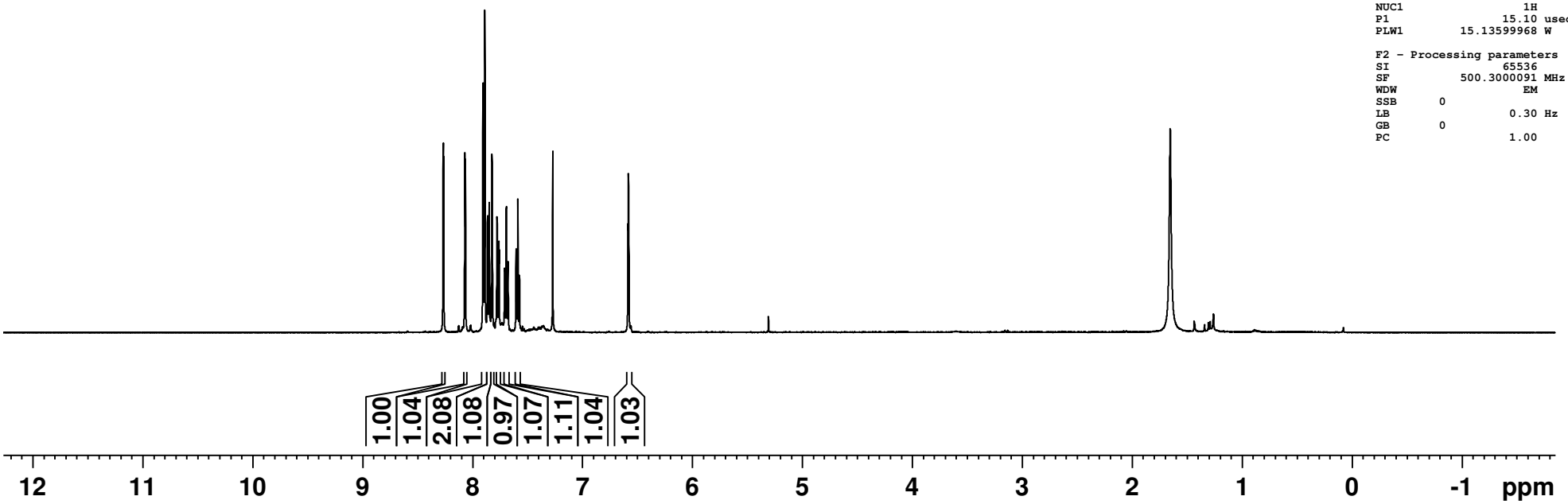

141.35
139.88
129.00
127.79
126.68
119.58
117.59
107.95
77.25
77.00
76.74
23.11
CN
N
N
2g
BRUKER
Current Data Parameters
NAME 13C
EXPNO 85
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180406
Time 20.00
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 94
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 1030
DW 16.800 usec
DE 6.50 usec
TE 291.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.8131145 MHz
NUC1 13C
P1 9.70 usec
PLW1 73.00000000 W
======== CHANNEL f2 ========
SFO2 500.3020012 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.13599968 W
PLW12 0.53924000 W
PLW13 0.34512001 W
F2 - Processing parameters
SI 32768
SF 125.8005442 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

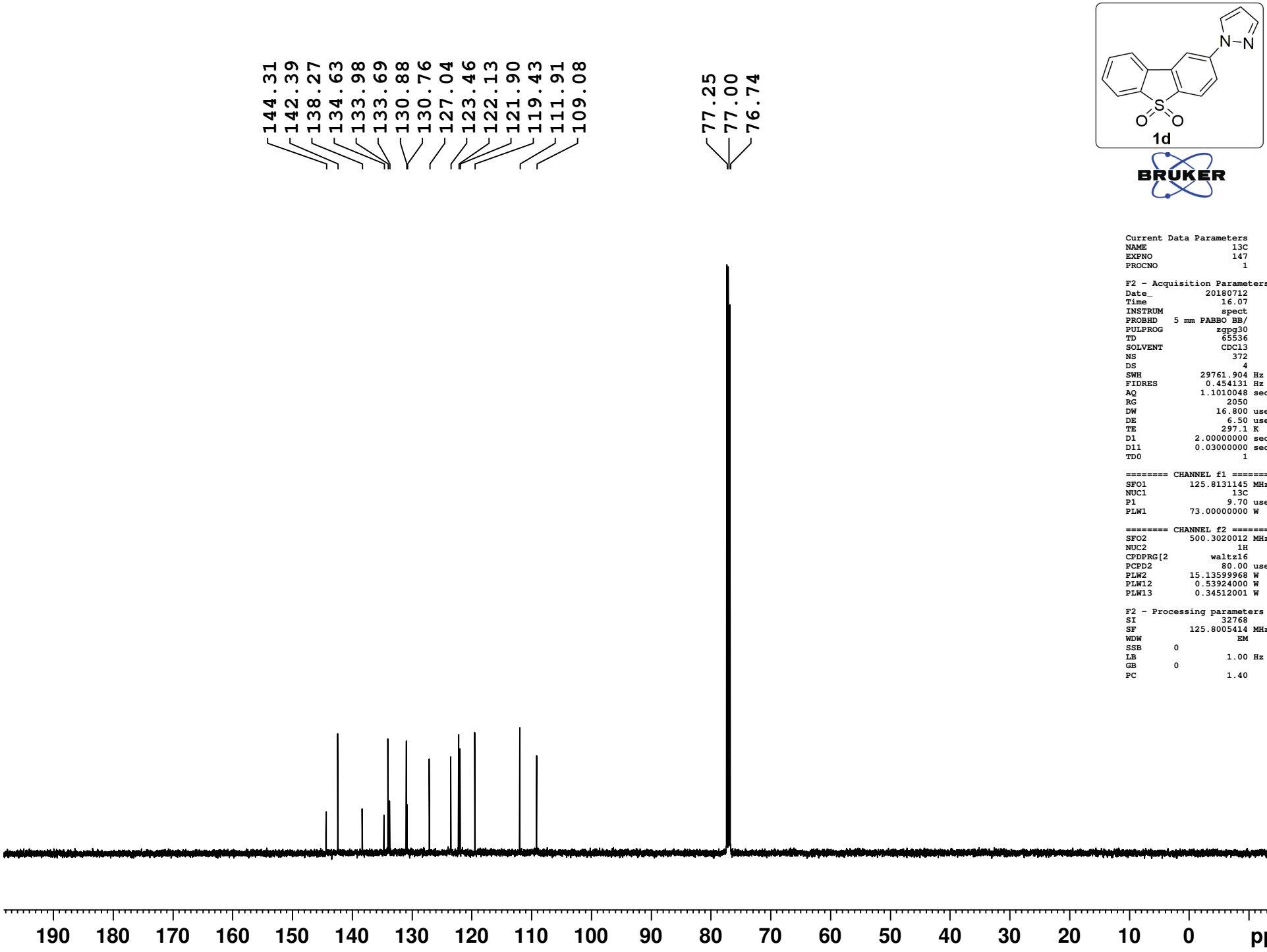


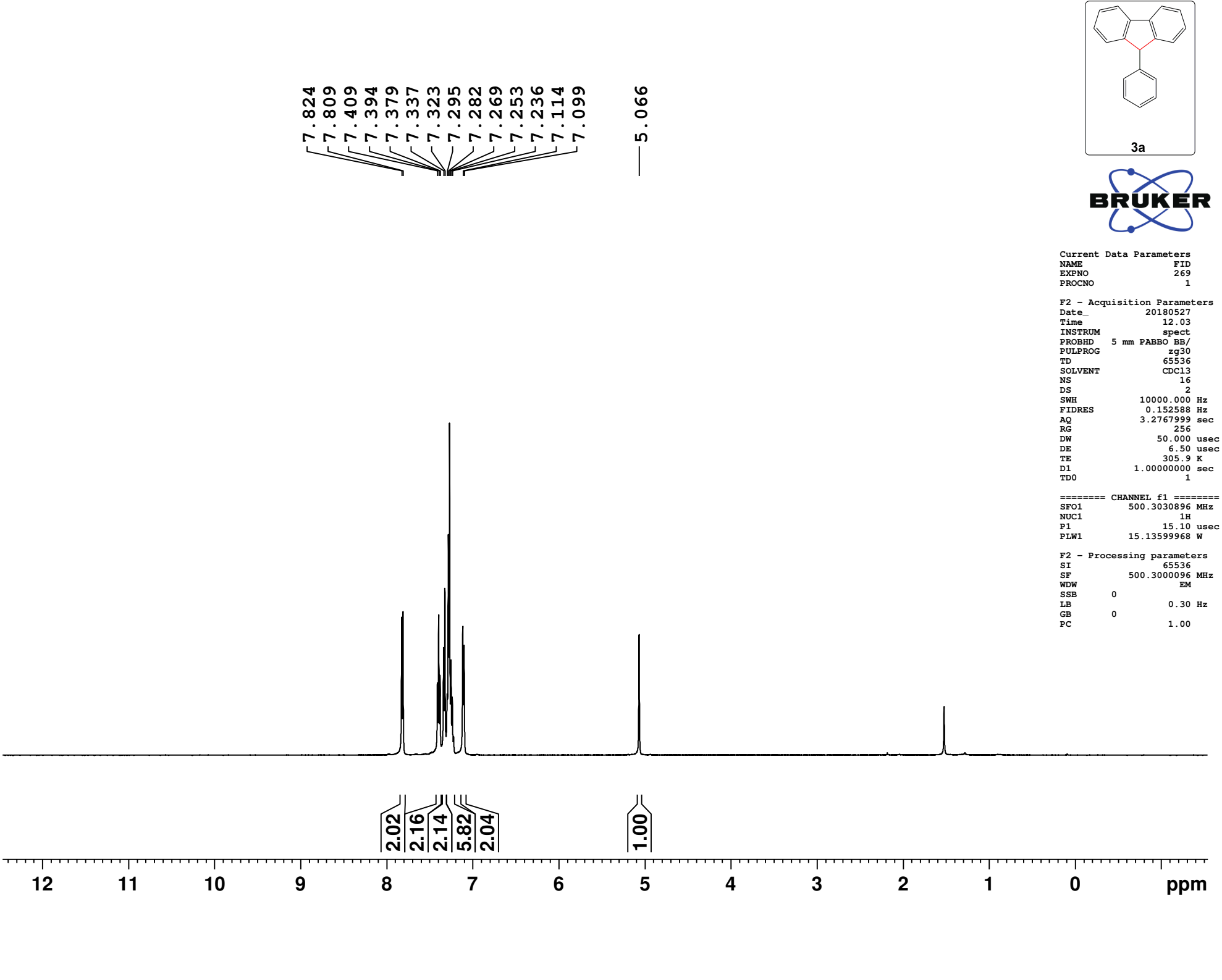


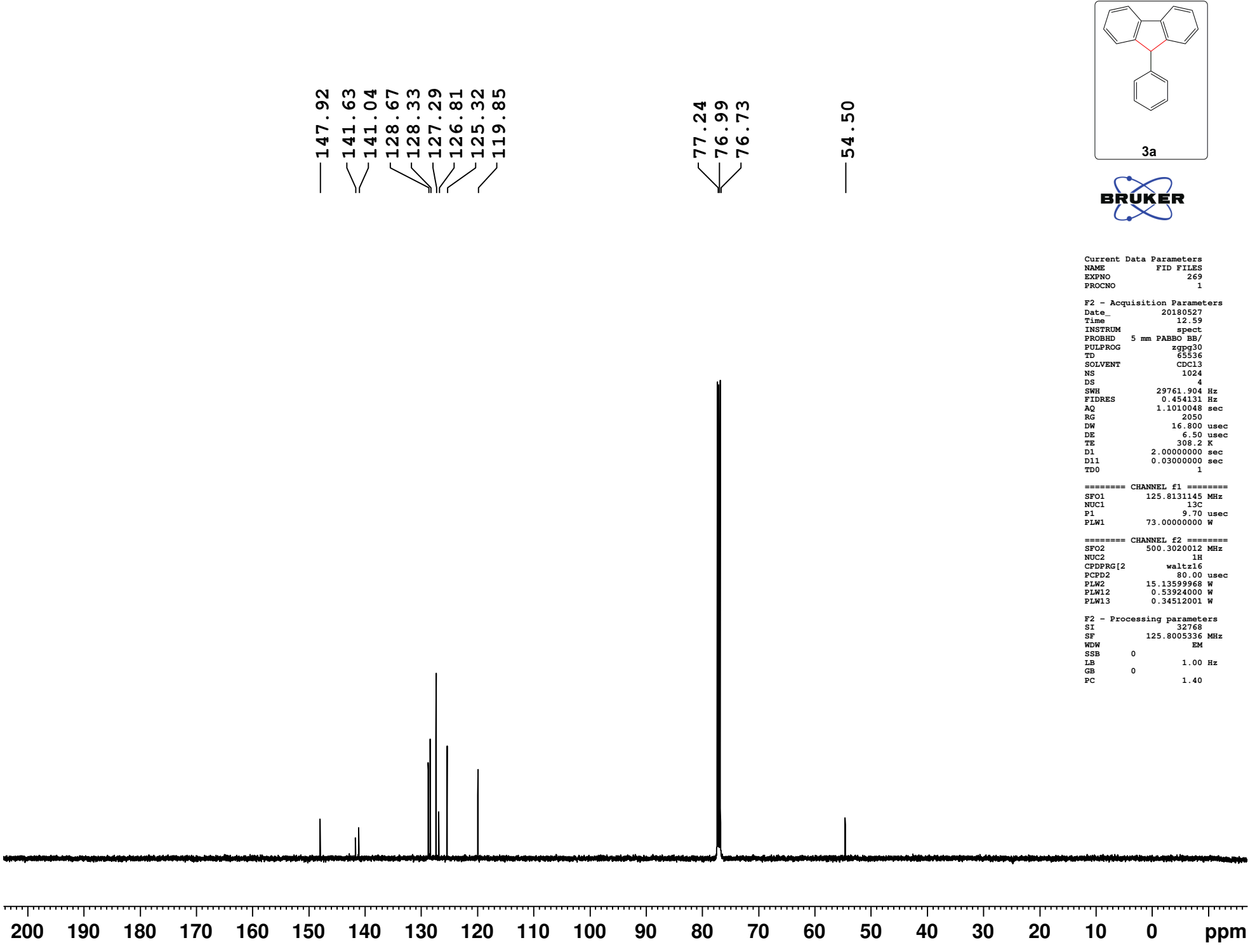
BRUKER

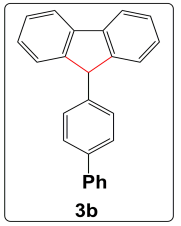
```
Current Data Parameters
NAME          12-07-2018
EXPNO                 20
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180712
Time               15.20
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            10000.000 Hz
FIDRES             0.152588 Hz
AQ              3.2767999 sec
RG                   256
DW                50.000 usec
DE                 6.50 usec
TE                294.5 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          500.3030896 MHz
NUC1                  1H
P1                 15.10 usec
PLW1          15.13599968 W

F2 - Processing parameters
SI                 65536
SF            500.3000091 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```




144.31
142.39
138.27
134.63
133.98
133.69
130.88
130.76
127.04
123.46
122.13
121.90
119.43
111.91
109.08
77.25
77.00
76.74
1d
BRUKER
Current Data Parameters
NAME 13C
EXPNO 147
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180712
Time 16.07
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 372
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 297.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.8131145 MHz
NUC1 13C
P1 9.70 usec
PLW1 73.00000000 W
======== CHANNEL f2 ========
SFO2 500.3020012 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.13599968 W
PLW12 0.53924000 W
PLW13 0.34512001 W
F2 - Processing parameters
SI 32768
SF 125.8005414 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.824
7.809
7.409
7.394
7.379
7.337
7.323
7.295
7.282
7.269
7.253
7.236
7.114
7.099
5.066
3a
BRUKER
Current Data Parameters
NAME FID
EXPNO 269
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180527
Time 12.03
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 256
DW 50.000 usec
DE 6.50 usec
TE 305.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.3030896 MHz
NUC1 1H
P1 15.10 usec
PLW1 15.13599968 W
F2 - Processing parameters
SI 65536
SF 500.3000096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
2.02
2.16
2.14
5.82
2.04
1.00
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm


147.92
141.63
141.04
128.67
128.33
127.29
126.81
125.32
119.85
77.24
76.99
76.73
54.50
3a
BRUKER
Current Data Parameters
NAME FID FILES
EXPNO 269
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180527
Time 12.59
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 308.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.8131145 MHz
NUC1 13C
P1 9.70 usec
PLW1 73.00000000 W
======== CHANNEL f2 ========
SFO2 500.3020012 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.13599968 W
PLW12 0.53924000 W
PLW13 0.34512001 W
F2 - Processing parameters
SI 32768
SF 125.8005336 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



7.841
7.826
7.580
7.578
7.564
7.517
7.500
7.440
7.425
7.395
7.385
7.370
7.345
7.331
7.316
7.306
7.291
7.276
7.275
7.269
7.185
7.169
5.115

BRUKER

Current Data Parameters
NAME FID
EXPNO 295
PROCNO 1

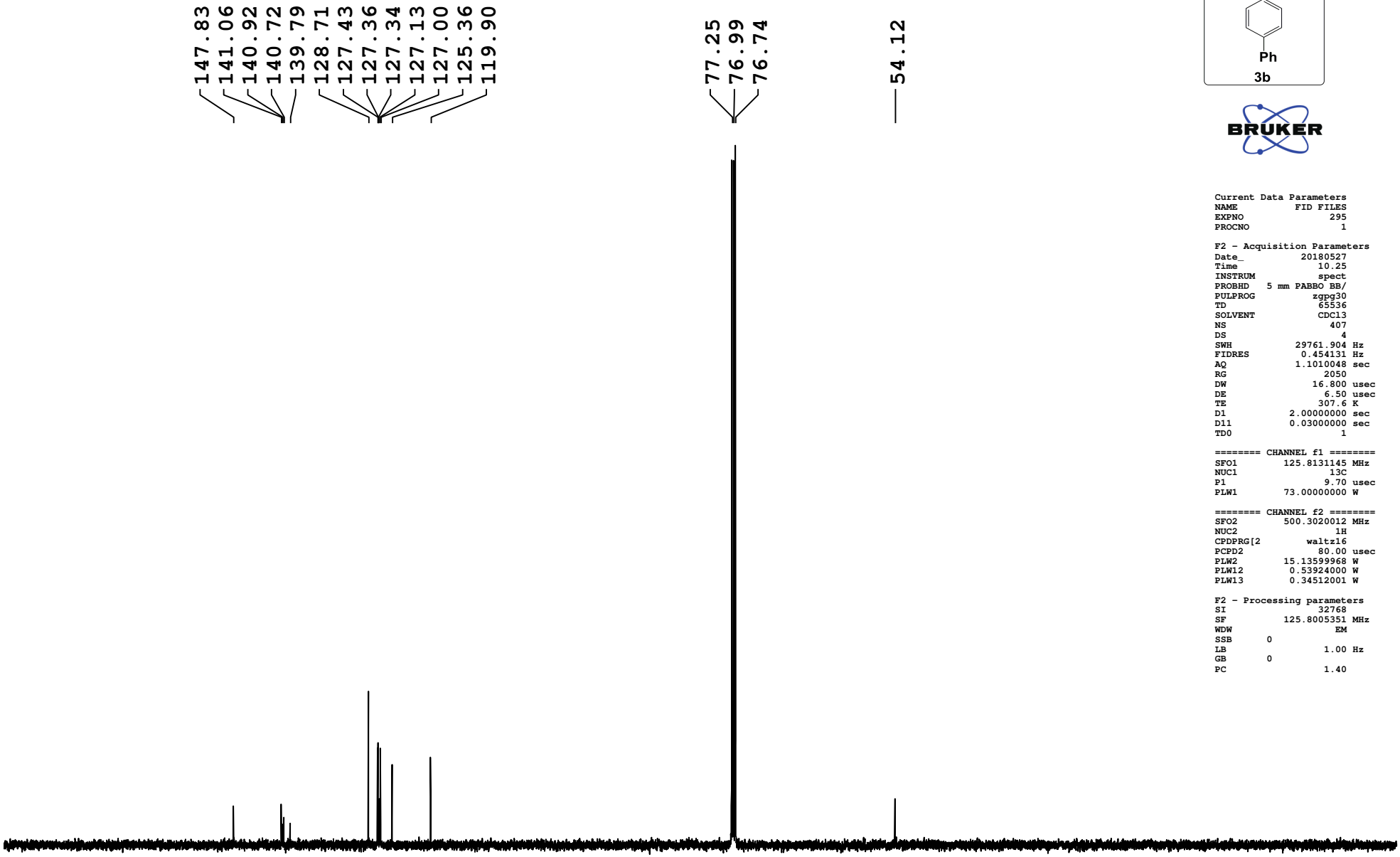
F2 - Acquisition Parameters
Date_ 20180527
Time 10.04
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 287
DW 50.000 usec
DE 6.50 usec
TE 305.3 K
D1 1.00000000 sec
TD0 1

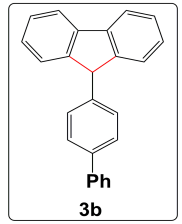
======== CHANNEL f1 ========
SFO1 500.3030896 MHz
NUC1 1H
P1 15.10 usec
PLW1 15.13599968 W

F2 - Processing parameters
SI 65536
SF 500.3000086 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1.00
1.04
1.04
3.07
2.02
1.03
0.51

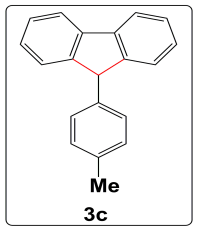
11 10 9 8 7 6 5 4 3 2 1 0 ppm





BRUKER

```
Current Data Parameters
NAME                  FID FILES
EXPNO                       295
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20180527
Time                      10.25
INSTRUM                   spect
PROBHD    5 mm PABBO BB/
PULPROG                 zgpg30
TD                         65536
SOLVENT                   CDC13
NS                          407
DS                            4
SWH                   29761.904 Hz
FIDRES                 0.454131 Hz
AQ                     1.1010048 sec
RG                         2050
DW                       16.800 usec
DE                        6.50 usec
TE                       307.6 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                125.8131145 MHz
NUC1                        13C
P1                        9.70 usec
PLW1                73.00000000 W

======== CHANNEL f2 ========
SFO2                500.3020012 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                    80.00 usec
PLW2                15.13599968 W
PLW12                0.53924000 W
PLW13                0.34512001 W

F2 - Processing parameters
SI                         32768
SF                  125.8005351 MHz
WDW                           EM
SSB       0
LB                        1.00 Hz
GB        0
PC                        1.40
```



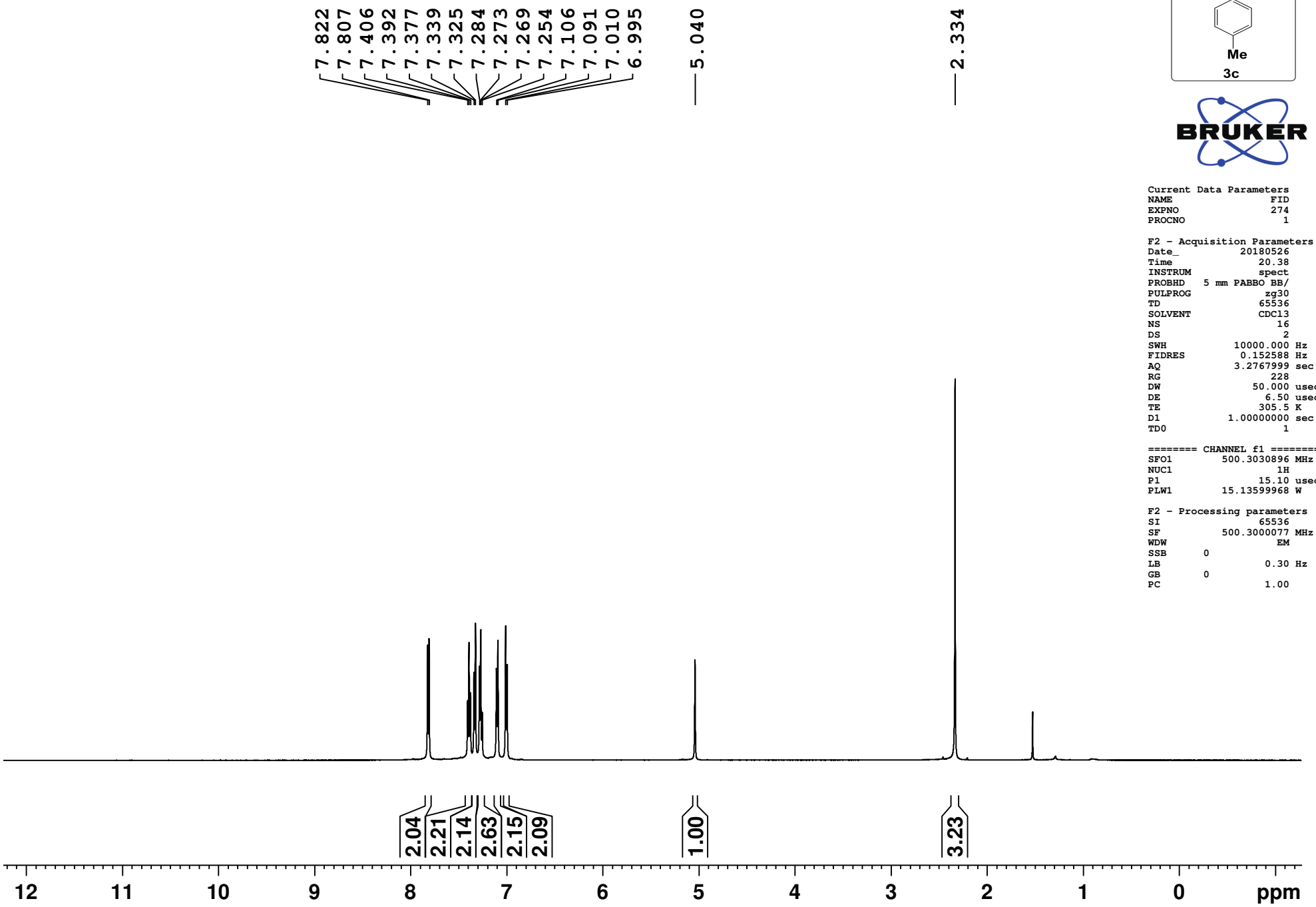
BRUKER

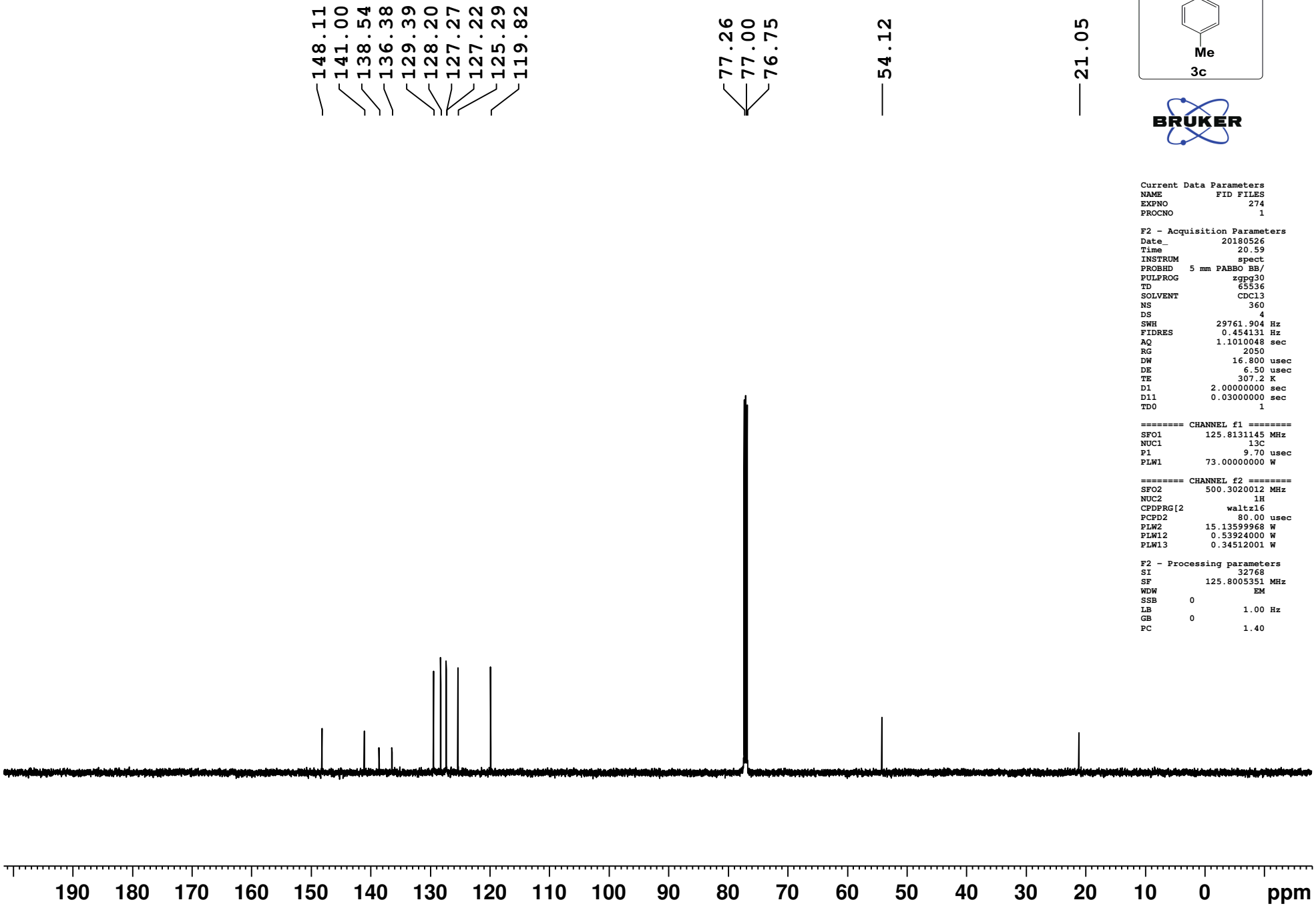
Current Data Parameters
NAME FID
EXPNO 274
PROCNO 1

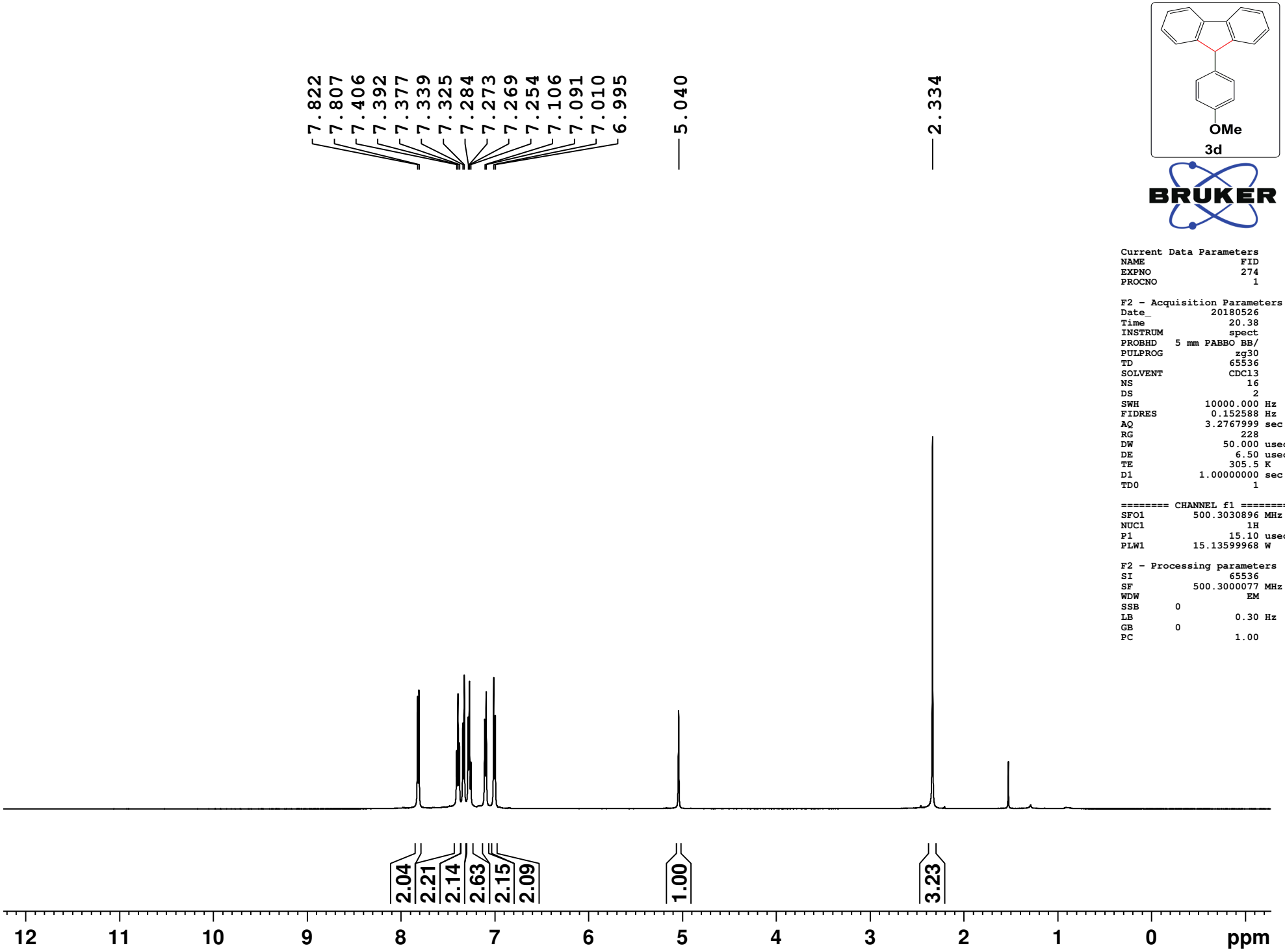
F2 - Acquisition Parameters
Date_ 20180526
Time 20.38
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 228
DW 50.000 usec
DE 6.50 usec
TE 305.5 K
D1 1.00000000 sec
TD0 1

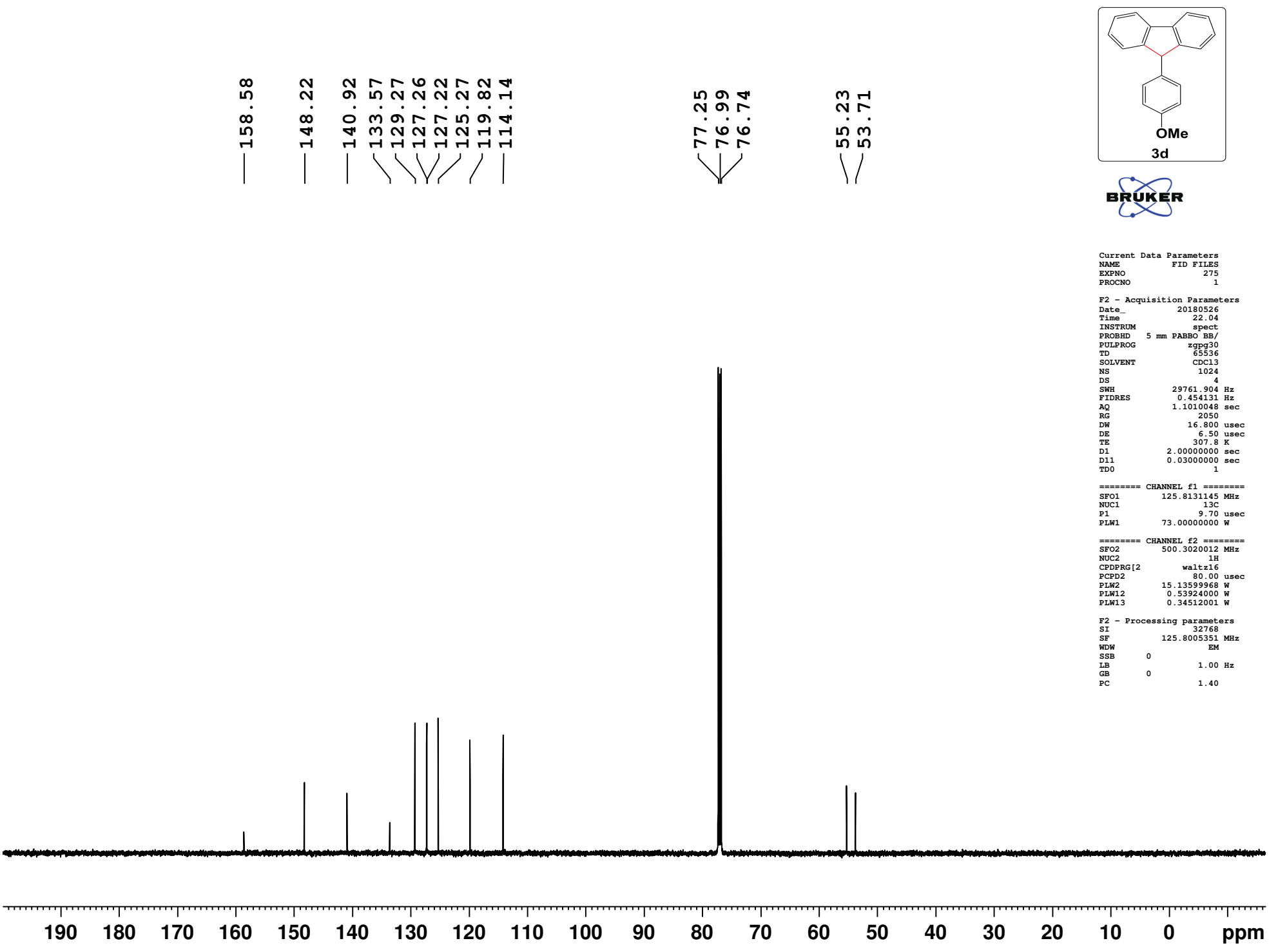
======== CHANNEL f1 ========
SFO1 500.3030896 MHz
NUC1 1H
P1 15.10 usec
PLW1 15.13599968 W

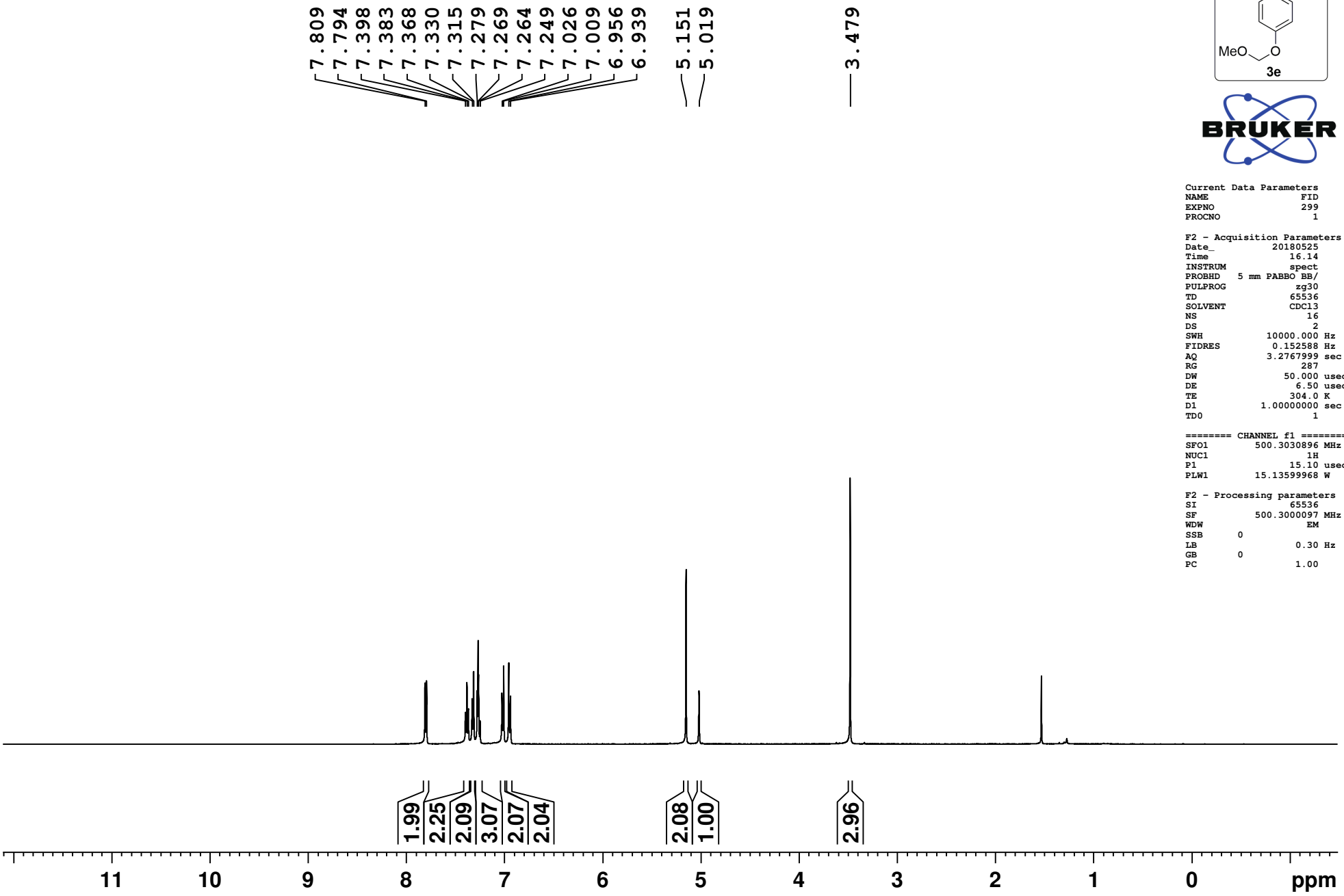
F2 - Processing parameters
SI 65536
SF 500.3000077 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

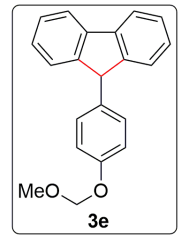



148.11
141.00
138.54
136.38
129.39
128.20
127.27
127.22
125.29
119.82
77.26
77.00
76.75
54.12
21.05
Me
3c
BRUKER
Current Data Parameters
NAME FID FILES
EXPNO 274
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180526
Time 20.59
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 360
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 307.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.8131145 MHz
NUC1 13C
P1 9.70 usec
PLW1 73.00000000 W
======== CHANNEL f2 ========
SFO2 500.3020012 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.13599968 W
PLW12 0.53924000 W
PLW13 0.34512001 W
F2 - Processing parameters
SI 32768
SF 125.8005351 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.822
7.807
7.406
7.392
7.377
7.339
7.325
7.284
7.273
7.269
7.254
7.106
7.091
7.010
6.995
5.040
2.334
OMe
3d
BRUKER
Current Data Parameters
NAME FID
EXPNO 274
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180526
Time 20.38
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 228
DW 50.000 usec
DE 6.50 usec
TE 305.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.3030896 MHz
NUC1 1H
P1 15.10 usec
PLW1 15.13599968 W
F2 - Processing parameters
SI 65536
SF 500.3000077 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
2.04
2.21
2.14
2.63
2.15
2.09
1.00
3.23
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm


158.58
148.22
140.92
133.57
129.27
127.26
127.22
125.27
119.82
114.14
77.25
76.99
76.74
55.23
53.71
OMe
3d
BRUKER
Current Data Parameters
NAME FID FILES
EXPNO 275
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180526
Time 22.04
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 307.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.8131145 MHz
NUC1 13C
P1 9.70 usec
PLW1 73.00000000 W
======== CHANNEL f2 ========
SFO2 500.3020012 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.13599968 W
PLW12 0.53924000 W
PLW13 0.34512001 W
F2 - Processing parameters
SI 32768
SF 125.8005351 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm





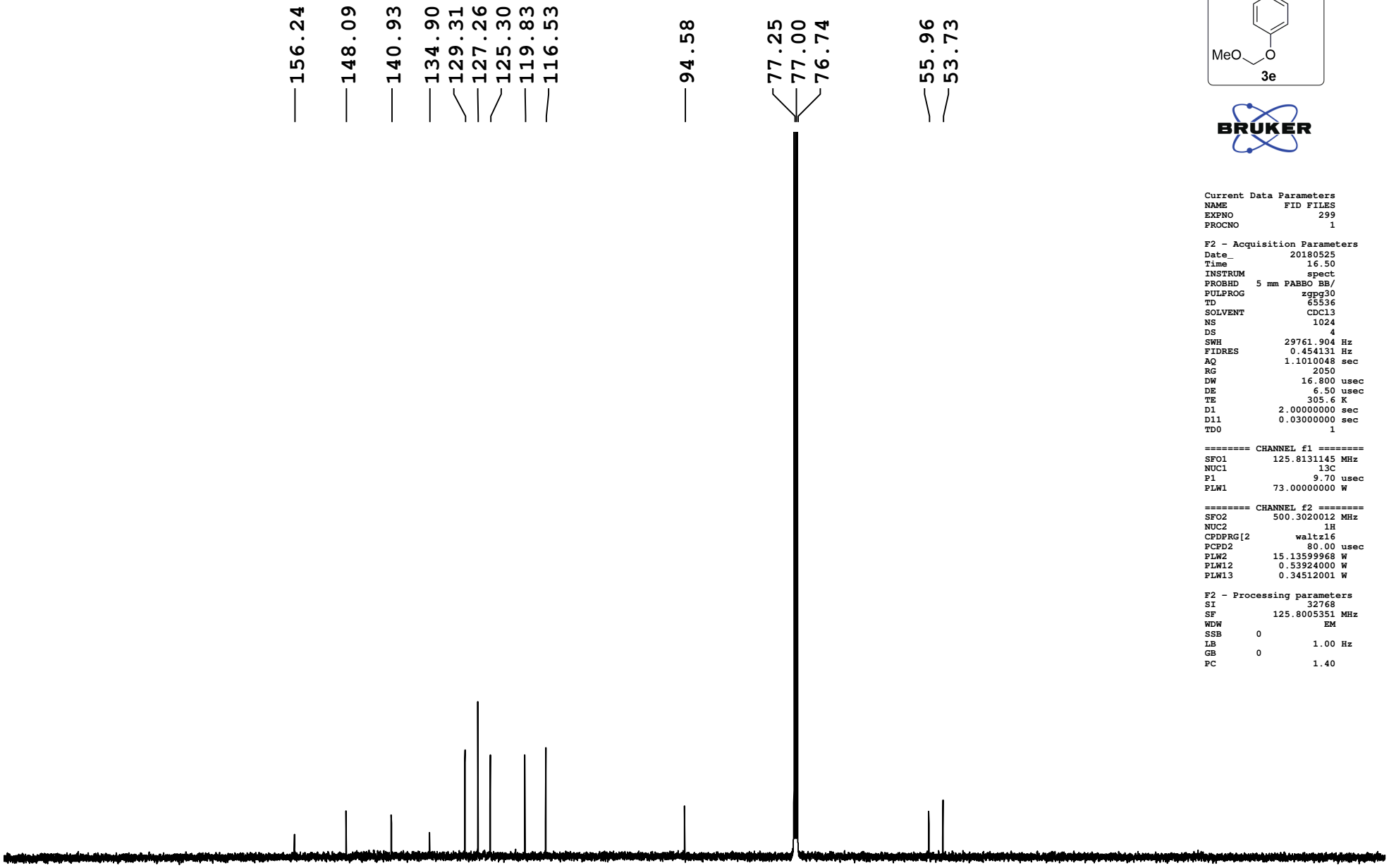
BRUKER

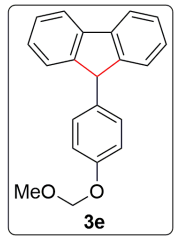
Current Data Parameters
NAME FID
EXPNO 299
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180525
Time 16.14
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 287
DW 50.000 usec
DE 6.50 usec
TE 304.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 500.3030896 MHz
NUC1 1H
P1 15.10 usec
PLW1 15.13599968 W

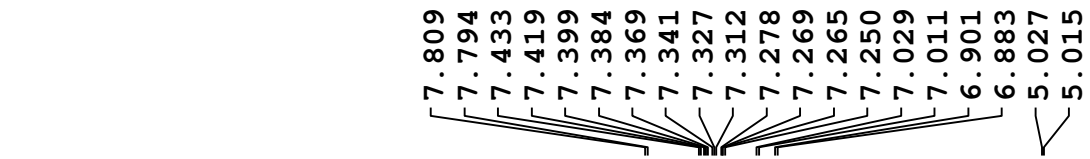
F2 - Processing parameters
SI 65536
SF 500.3000097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

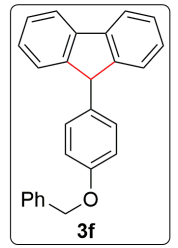




BRUKER

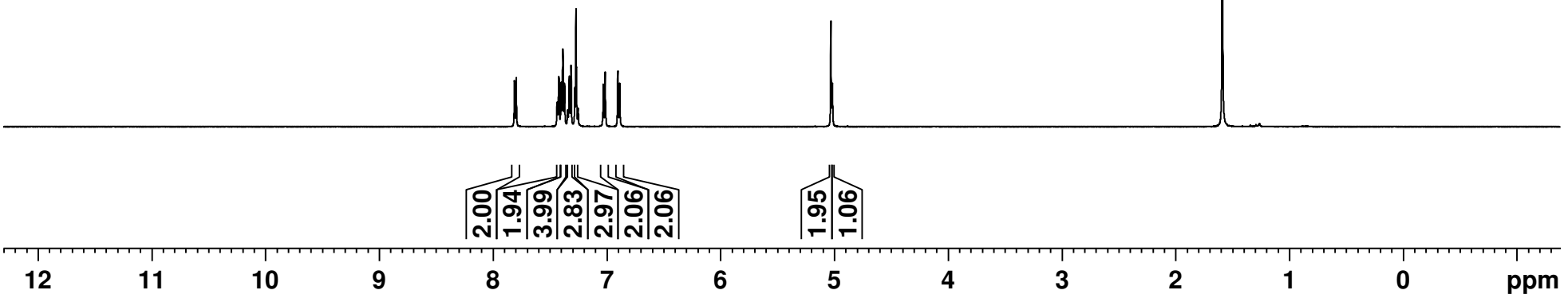
```
Current Data Parameters
NAME                  FID FILES
EXPNO                       299
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20180525
Time                      16.50
INSTRUM                   spect
PROBHD    5 mm PABBO BB/
PULPROG                 zgpg30
TD                         65536
SOLVENT                   CDC13
NS                         1024
DS                            4
SWH                   29761.904 Hz
FIDRES                0.454131 Hz
AQ                    1.1010048 sec
RG                         2050
DW                       16.800 usec
DE                        6.50 usec
TE                       305.6 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                125.8131145 MHz
NUC1                        13C
P1                        9.70 usec
PLW1                73.00000000 W

======== CHANNEL f2 ========
SFO2                500.3020012 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                    80.00 usec
PLW2                15.13599968 W
PLW12                0.53924000 W
PLW13                0.34512001 W

F2 - Processing parameters
SI                         32768
SF                  125.8005351 MHz
WDW                           EM
SSB       0
LB                        1.00 Hz
GB        0
PC                        1.40
```

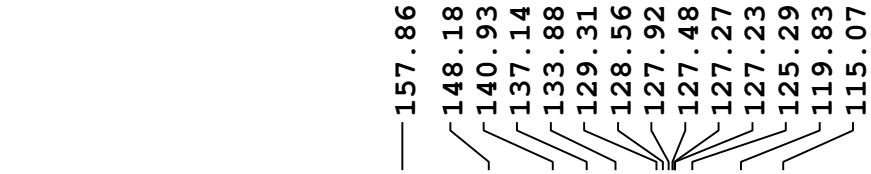


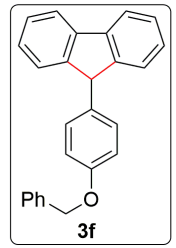


BRUKER

```
Current Data Parameters
NAME                  FID
EXPNO                 292
PROCNO                  1

F2 - Acquisition Parameters
Date_           20180704
Time               11.11
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            10000.000 Hz
FIDRES             0.152588 Hz
AQ             3.2767999 sec
RG                   228
DW                50.000 usec
DE                 6.50 usec
TE                 291.3 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         500.3030896 MHz
NUC1                 1H
P1                15.10 usec
PLW1         15.13599968 W

F2 - Processing parameters
SI                 65536
SF           500.3000097 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

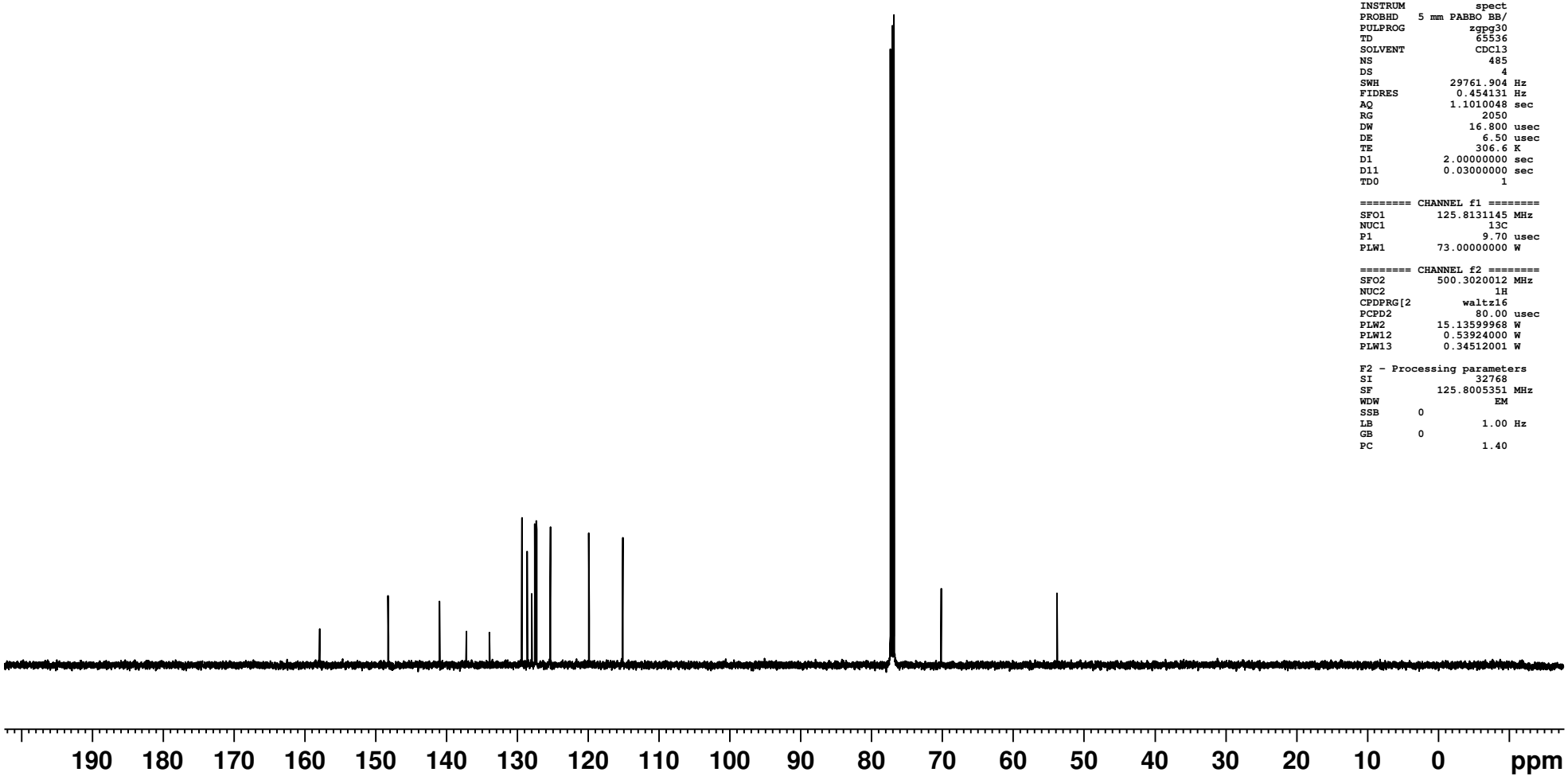


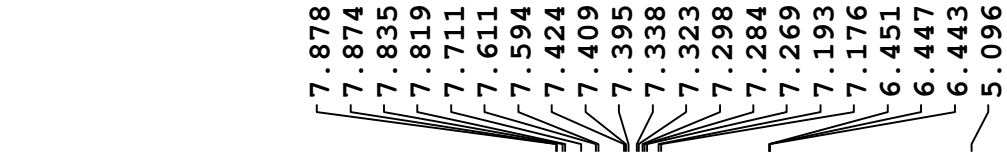


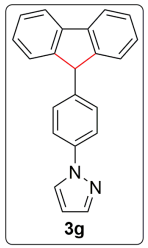


BRUKER

```
Current Data Parameters
NAME                  FID FILES
EXPNO                       292
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20180526
Time                      20.06
INSTRUM                   spect
PROBHD    5 mm PABBO BB/
PULPROG                 zgpg30
TD                         65536
SOLVENT                   CDC13
NS                          485
DS                            4
SWH                   29761.904 Hz
FIDRES                 0.454131 Hz
AQ                     1.1010048 sec
RG                         2050
DW                       16.800 usec
DE                        6.50 usec
TE                       306.6 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                125.8131145 MHz
NUC1                        13C
P1                        9.70 usec
PLW1                73.00000000 W

======== CHANNEL f2 ========
SFO2                500.3020012 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                    80.00 usec
PLW2                15.13599968 W
PLW12                0.53924000 W
PLW13                0.34512001 W

F2 - Processing parameters
SI                         32768
SF                  125.8005351 MHz
WDW                           EM
SSB       0
LB                        1.00 Hz
GB        0
PC                        1.40
```







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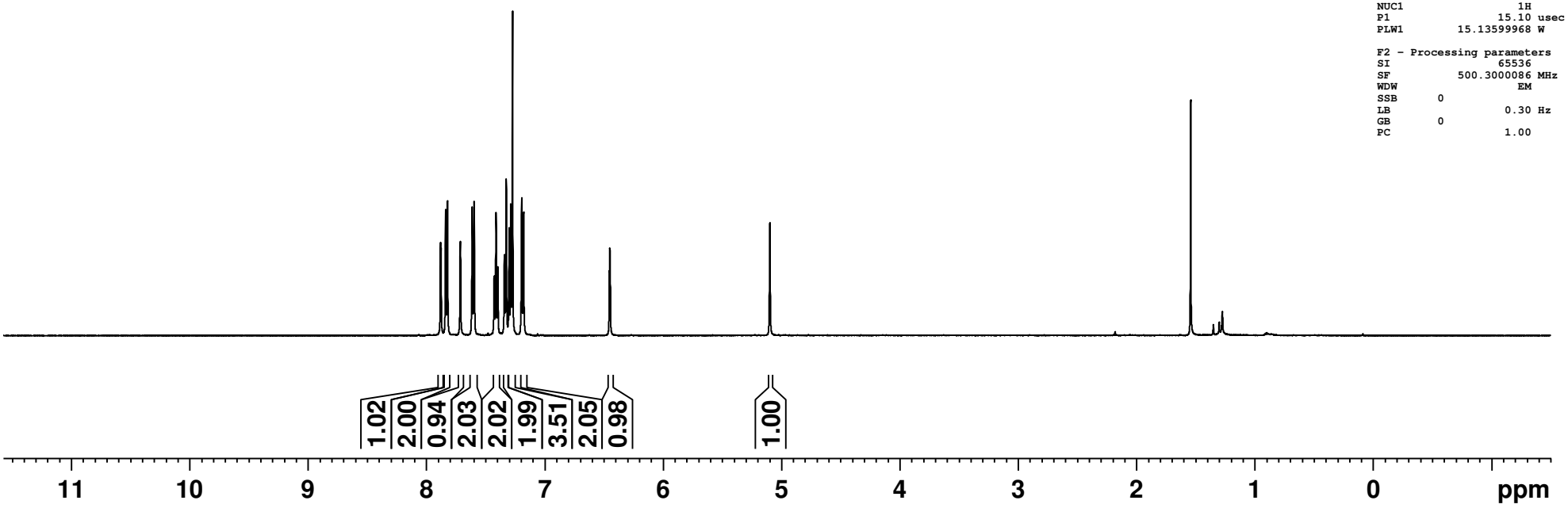
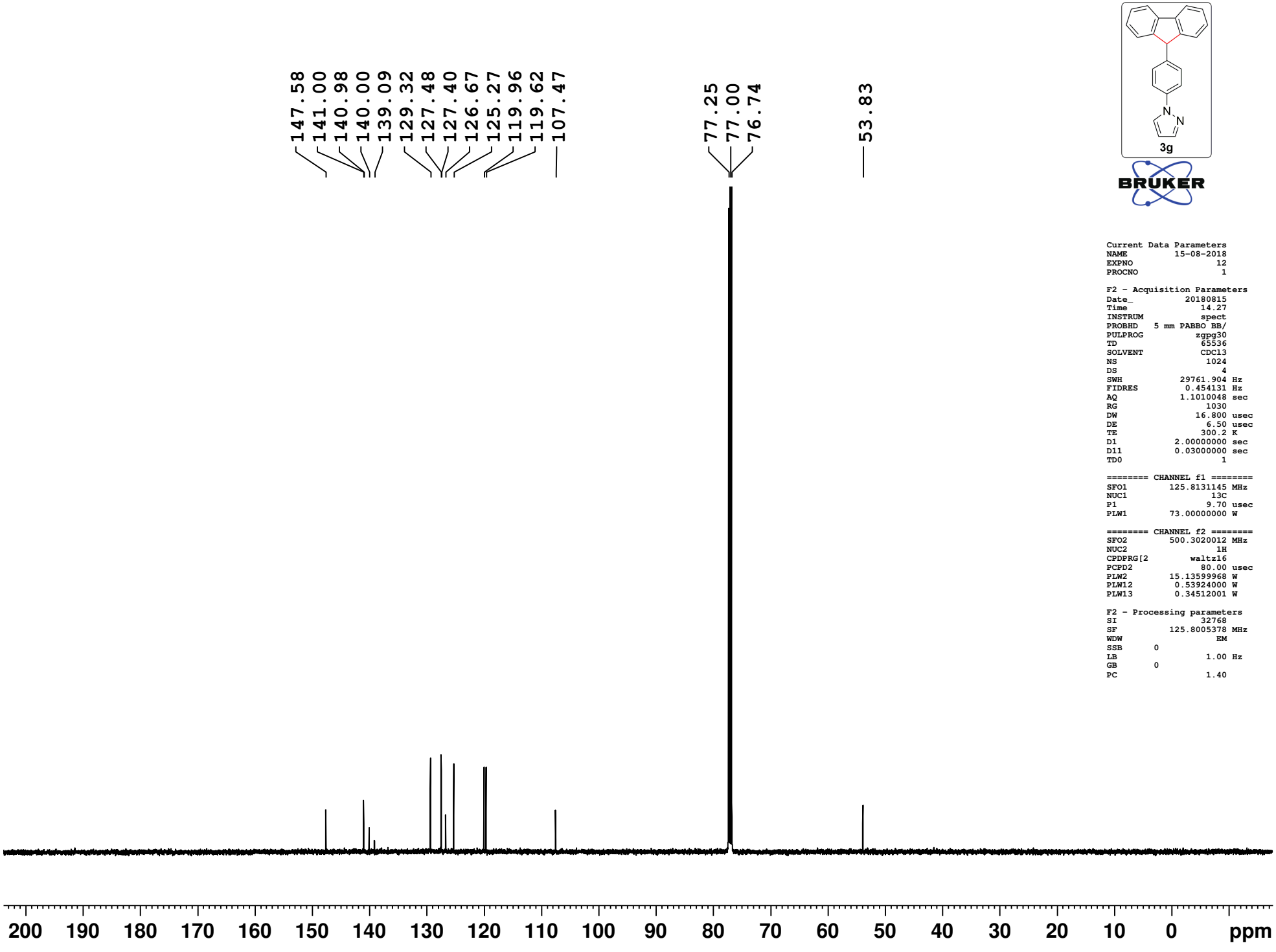
Current Data Parameters
NAME FID
EXPNO 300
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180526
Time 18.35
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 287
DW 50.000 usec
DE 6.50 usec
TE 304.9 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 500.3030896 MHz
NUC1 1H
P1 15.10 usec
PLW1 15.13599968 W

F2 - Processing parameters
SI 65536
SF 500.3000086 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00





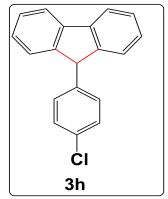

```
Current Data Parameters
NAME          15-08-2018
EXPNO                 12
PROCNO                 1

F2 - Acquisition Parameters
Date_           20180815
Time               14.27
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDC13
NS                 1024
DS                    4
SWH           29761.904 Hz
FIDRES        0.454131 Hz
AQ            1.1010048 sec
RG                 1030
DW               16.800 usec
DE                6.50 usec
TE                300.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         125.8131145 MHz
NUC1                 13C
P1                  9.70 usec
PLW1         73.00000000 W

======== CHANNEL f2 ========
SFO2         500.3020012 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2              80.00 usec
PLW2         15.13599968 W
PLW12         0.53924000 W
PLW13         0.34512001 W

F2 - Processing parameters
SI                 32768
SF           125.8005378 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



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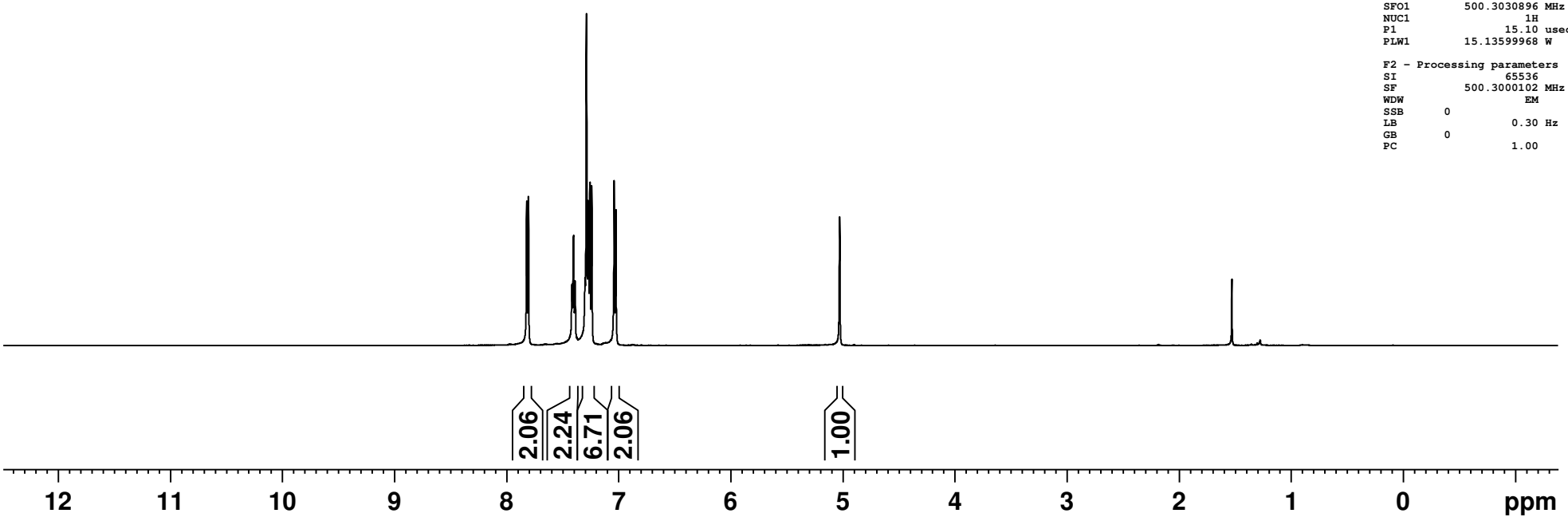
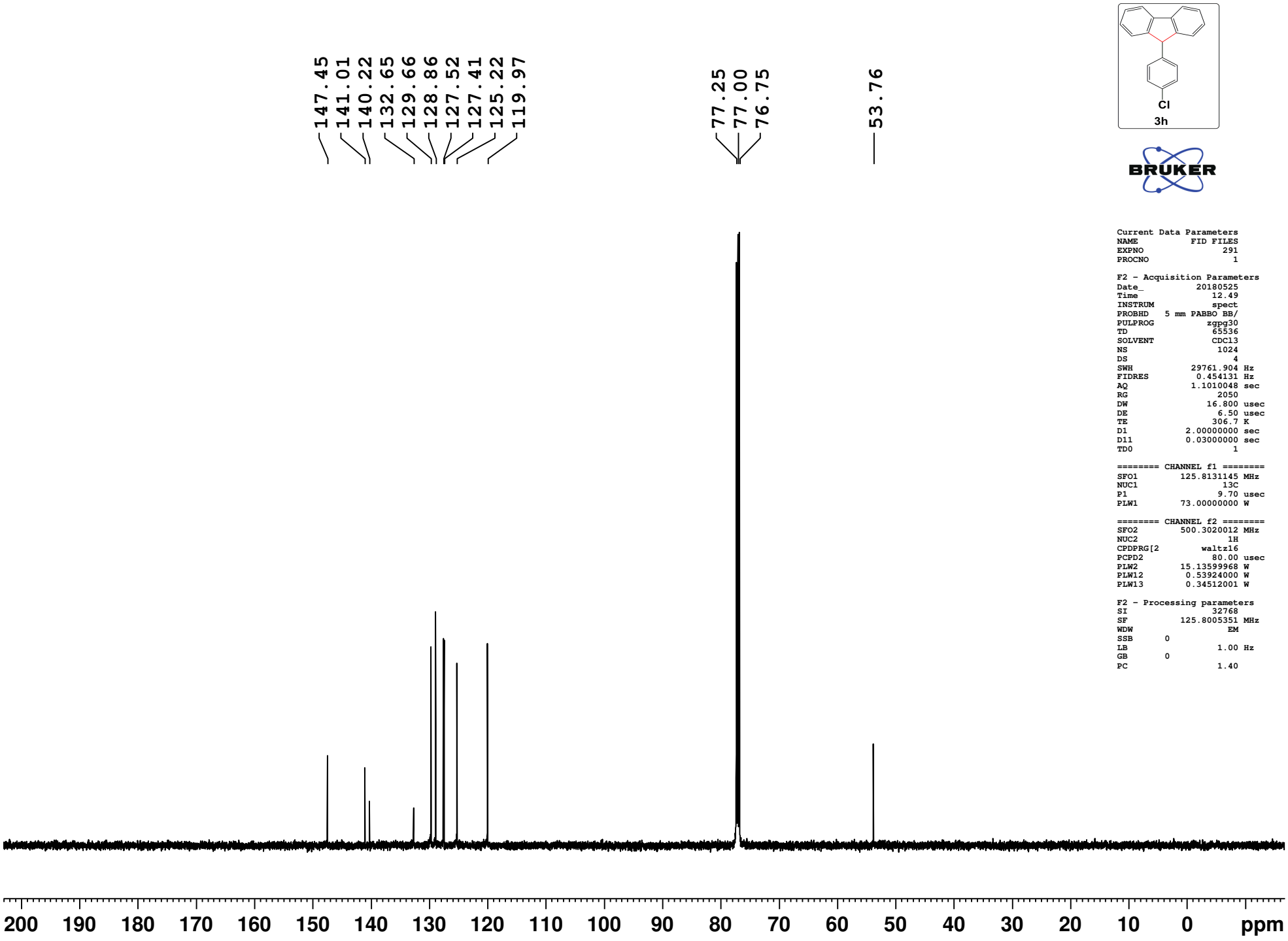
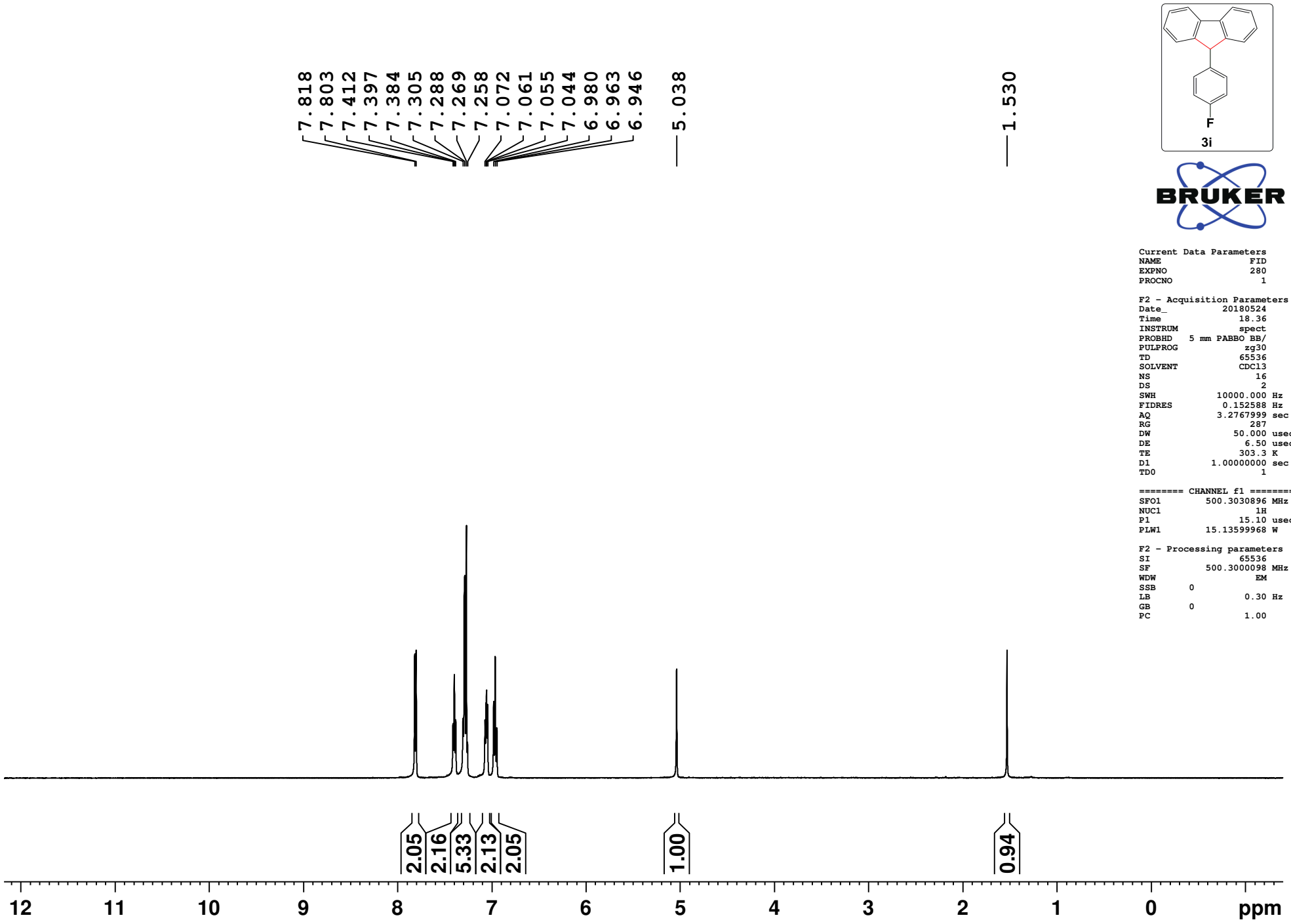
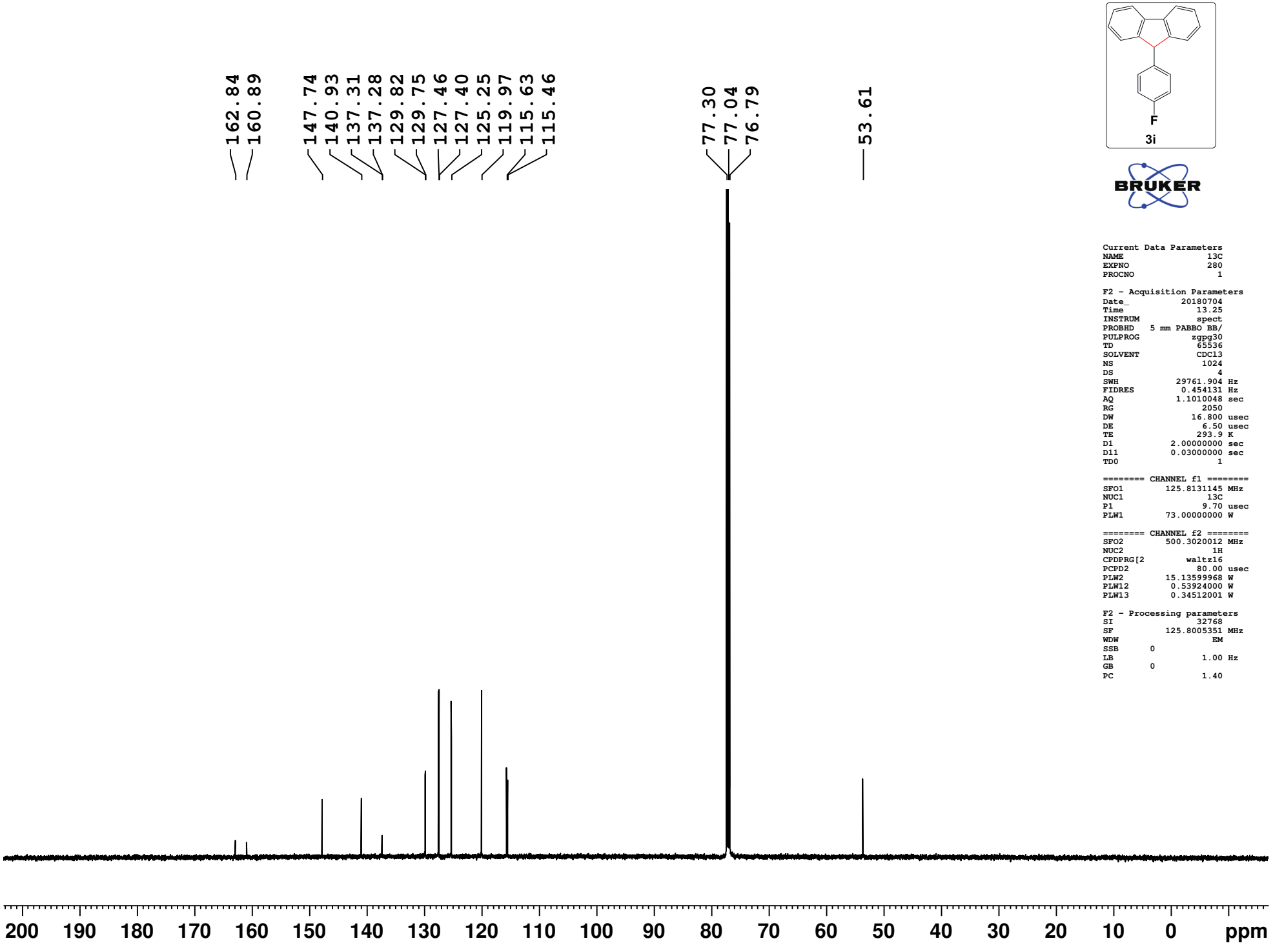
```
Current Data Parameters
NAME                  FID
EXPNO                 291
PROCNO                  1

F2 - Acquisition Parameters
Date_          20180524
Time              18.53
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT           CDC13
NS                   16
DS                    2
SWH           10000.000 Hz
FIDRES            0.152588 Hz
AQ             3.2767999 sec
RG                  228
DW                50.000 usec
DE                 6.50 usec
TE                303.4 K
D1             1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1          500.3030896 MHz
NUC1                  1H
P1                15.10 usec
PLW1          15.13599968 W

F2 - Processing parameters
SI                65536
SF            500.3000102 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

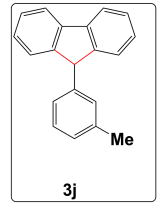



147.45
141.01
140.22
132.65
129.66
128.86
127.52
127.41
125.22
119.97
77.25
77.00
76.75
53.76
Cl
3h
BRUKER
Current Data Parameters
NAME FID FILES
EXPNO 291
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180525
Time 12.49
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 306.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.8131145 MHz
NUC1 13C
P1 9.70 usec
PLW1 73.00000000 W
======== CHANNEL f2 ========
SFO2 500.3020012 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.13599968 W
PLW12 0.53924000 W
PLW13 0.34512001 W
F2 - Processing parameters
SI 32768
SF 125.8005351 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.818
7.803
7.412
7.397
7.384
7.305
7.288
7.269
7.258
7.072
7.061
7.055
7.044
6.980
6.963
6.946
5.038
1.530
F
3i
BRUKER
Current Data Parameters
NAME FID
EXPNO 280
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180524
Time 18.36
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 287
DW 50.000 usec
DE 6.50 usec
TE 303.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.3030896 MHz
NUC1 1H
P1 15.10 usec
PLW1 15.13599968 W
F2 - Processing parameters
SI 65536
SF 500.3000098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
2.05
2.16
5.33
2.13
2.05
1.00
0.94
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm




```
Current Data Parameters
NAME                  13C
EXPNO                 280
PROCNO                  1

F2 - Acquisition Parameters
Date_              20180704
Time                 13.25
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG            zgpg30
TD                  65536
SOLVENT             CDC13
NS                   1024
DS                      4
SWH              29761.904 Hz
FIDRES           0.454131 Hz
AQ               1.1010048 sec
RG                   2050
DW                 16.800 usec
DE                  6.50 usec
TE                 293.9 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1           125.8131145 MHz
NUC1                   13C
P1                    9.70 usec
PLW1           73.00000000 W

======== CHANNEL f2 ========
SFO2           500.3020012 MHz
NUC2                   1H
CPDPRG[2           waltz16
PCPD2                80.00 usec
PLW2           15.13599968 W
PLW12           0.53924000 W
PLW13           0.34512001 W

F2 - Processing parameters
SI                    32768
SF             125.8005351 MHz
WDW                     EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```



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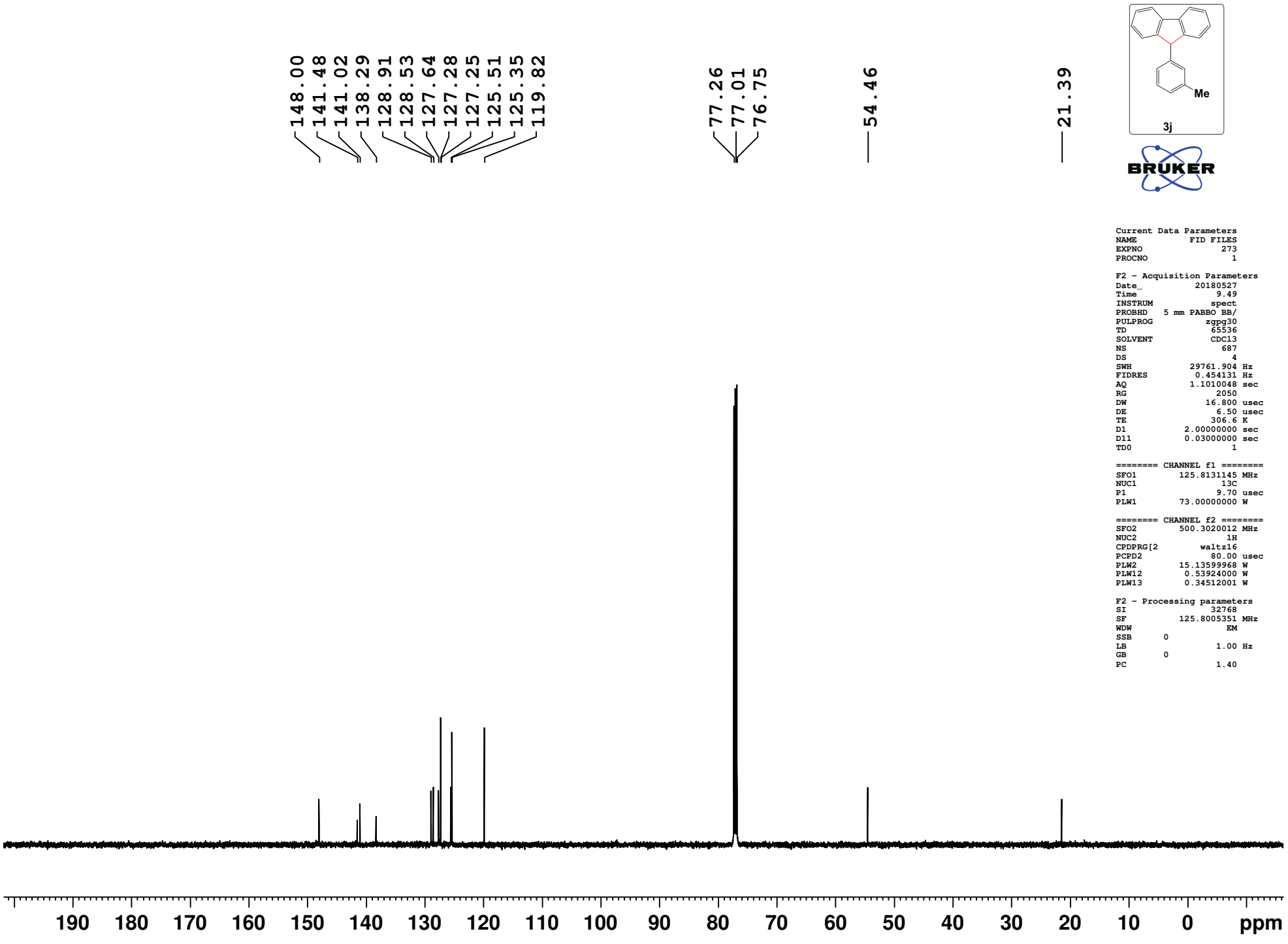
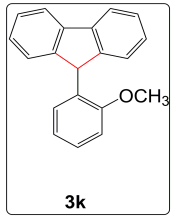
7.824
7.809
7.409
7.394
7.379
7.343
7.328
7.285
7.269
7.256
7.193
7.178
7.162
7.061
7.046
6.936
6.921
6.893
5.029
2.288

```
Current Data Parameters
NAME                  FID
EXPNO                 273
PROCNO                  1

F2 - Acquisition Parameters
Date_              20180527
Time                  9.12
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH             10000.000 Hz
FIDRES              0.152588 Hz
AQ                3.2767999 sec
RG                     228
DW                  50.000 usec
DE                   6.50 usec
TE                  304.7 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1            500.3030896 MHz
NUC1                    1H
P1                   15.10 usec
PLW1            15.13599968 W

F2 - Processing parameters
SI                   65536
SF              500.3000109 MHz
WDW                    EM
SSB       0
LB                   0.30 Hz
GB        0
PC                   1.00
```

2.05 2.24 2.09 2.53 1.08 0.99 1.07 0.95 1.00 3.13

11 10 9 8 7 6 5 4 3 2 1 0 ppm


148.00
141.48
141.02
138.29
128.91
128.53
127.64
127.28
127.25
125.51
125.35
119.82
77.26
77.01
76.75
54.46
21.39
Me
3j
BRUKER
Current Data Parameters
NAME FID FILES
EXPNO 273
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180527
Time 9.49
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 687
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 306.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.8131145 MHz
NUC1 13C
P1 9.70 usec
PLW1 73.00000000 W
======== CHANNEL f2 ========
SFO2 500.3020012 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.13599968 W
PLW12 0.53924000 W
PLW13 0.34512001 W
F2 - Processing parameters
SI 32768
SF 125.8005351 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



3k

7.815
7.800
7.392
7.377
7.361
7.269
7.257
7.242
7.228
7.212
7.197
7.024
7.008
6.754
6.740
6.726
6.595
5.698

3.984

BRUKER

Current Data Parameters
NAME FID
EXPNO 283
PROCNO 1

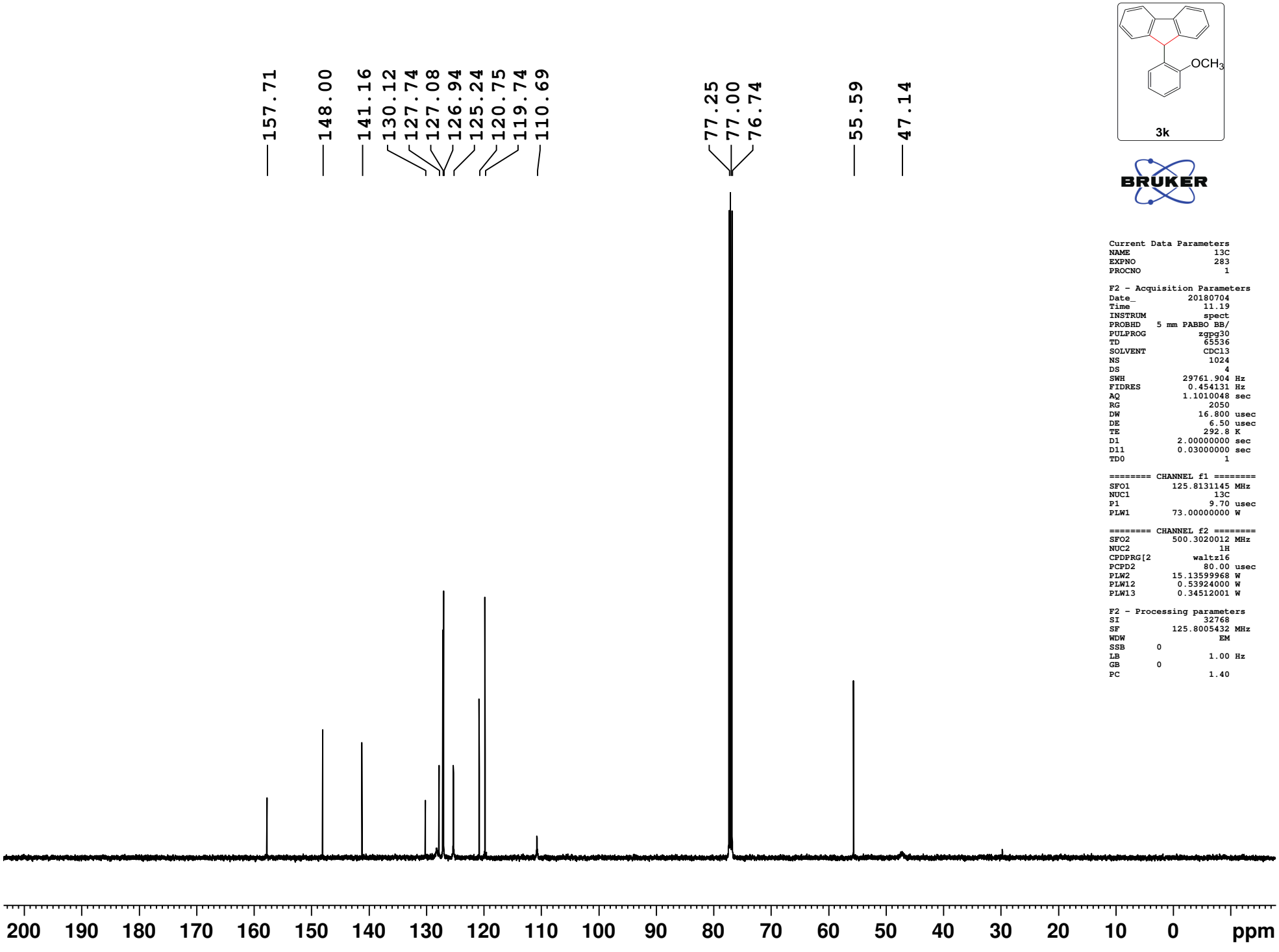
F2 - Acquisition Parameters
Date_ 20180527
Time 10.35
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 228
DW 50.000 usec
DE 6.50 usec
TE 305.5 K
D1 1.00000000 sec
TD0 1

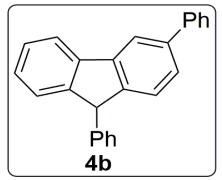
======== CHANNEL f1 ========
SFO1 500.3030896 MHz
NUC1 1H
P1 15.10 usec
PLW1 15.13599968 W

F2 - Processing parameters
SI 65536
SF 500.3000090 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

2.19
4.42
3.87
1.11
1.07
0.85
0.88
3.00

12 11 10 9 8 7 6 5 4 3 2 1 0 ppm


157.71
148.00
141.16
130.12
127.74
127.08
126.94
125.24
120.75
119.74
110.69
77.25
77.00
76.74
55.59
47.14
OCH3
3k
BRUKER
Current Data Parameters
NAME 13C
EXPNO 283
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180704
Time 11.19
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 292.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.8131145 MHz
NUC1 13C
P1 9.70 usec
PLW1 73.00000000 W
======== CHANNEL f2 ========
SFO2 500.3020012 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.13599968 W
PLW12 0.53924000 W
PLW13 0.34512001 W
F2 - Processing parameters
SI 32768
SF 125.8005432 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



4b

8.012
7.877
7.862
7.691
7.676
7.501
7.485
7.469
7.417
7.394
7.381
7.340
7.299
7.291
7.284
7.269
7.149
7.135
5.107

BRUKER

Current Data Parameters
NAME FID
EXPNO 313
PROCNO 1

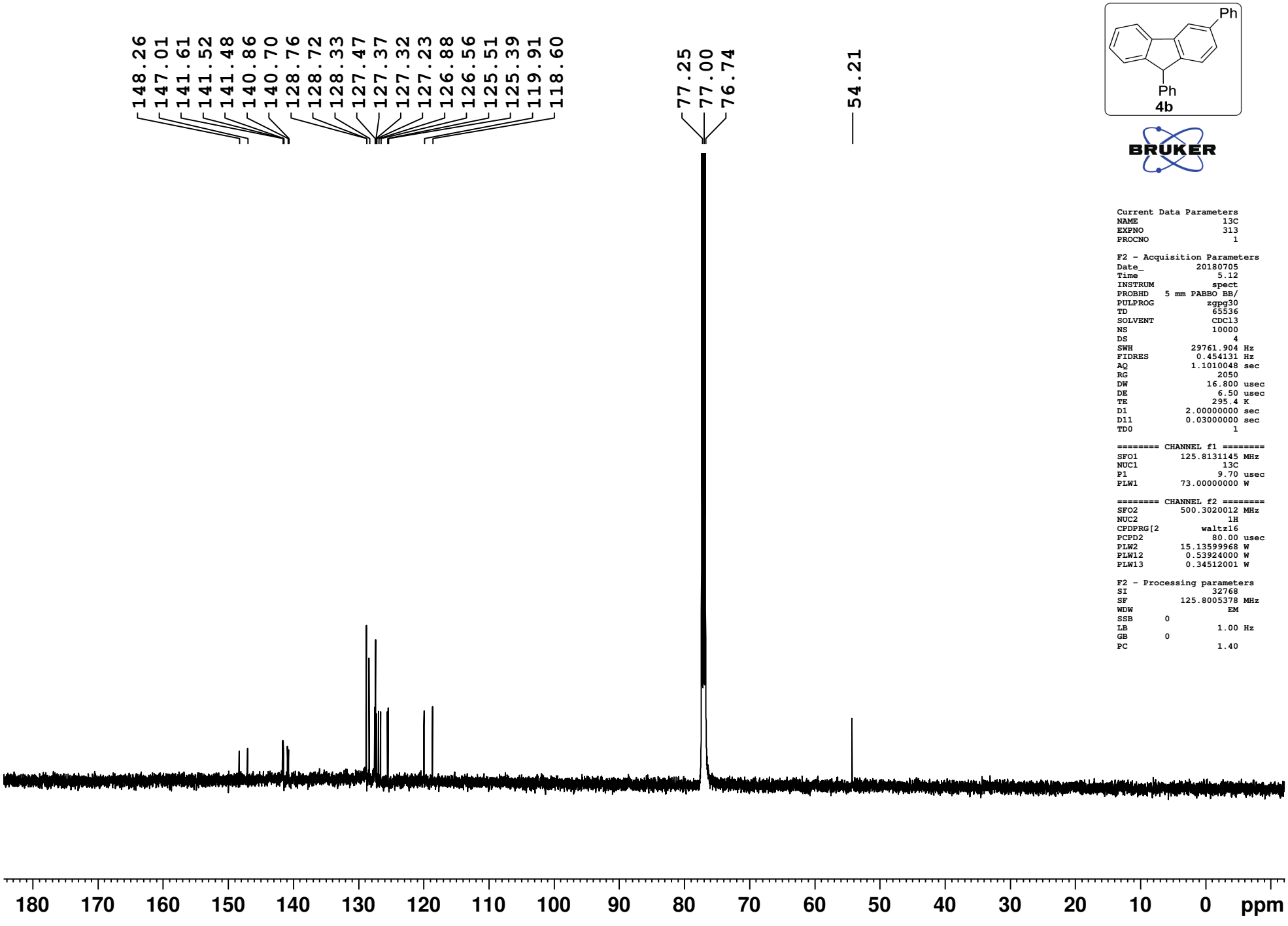
F2 - Acquisition Parameters
Date_ 20180704
Time 19.17
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 300
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 256
DW 50.000 usec
DE 6.50 usec
TE 292.6 K
D1 1.00000000 sec
TD0 1

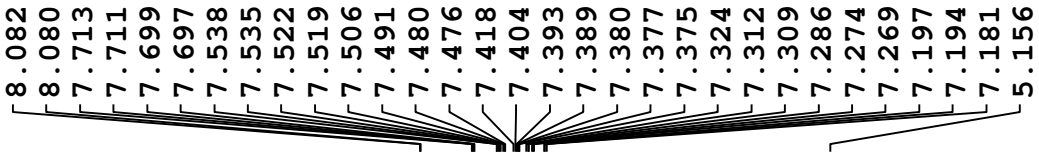
======== CHANNEL f1 ========
SFO1 500.3030896 MHz
NUC1 1H
P1 15.10 usec
PLW1 15.13599968 W

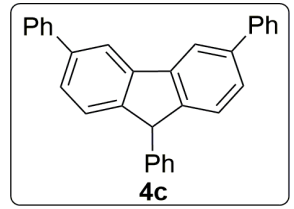
F2 - Processing parameters
SI 65536
SF 500.3000094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1.01
1.01
1.87
2.82
3.79
5.72
1.86
1.07

12 11 10 9 8 7 6 5 4 3 2 1 0 ppm

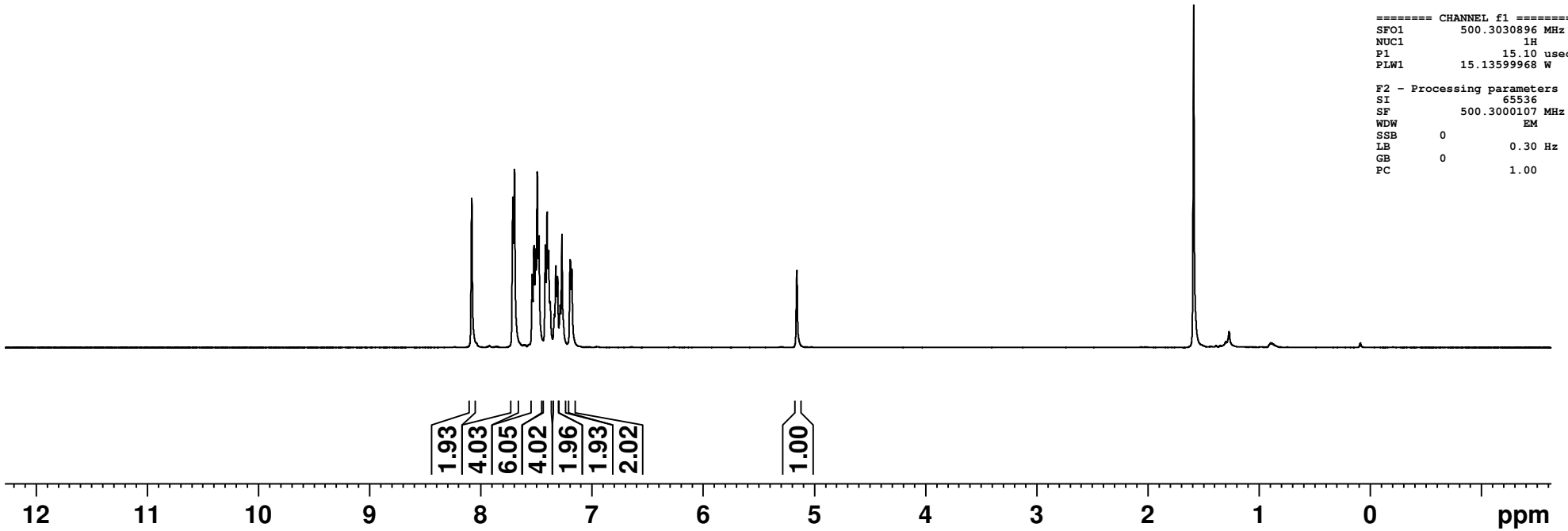

148.26
147.01
141.61
141.52
141.48
140.86
140.70
128.76
128.72
128.33
127.47
127.37
127.32
127.23
126.88
126.56
125.51
125.39
119.91
118.60
77.25
77.00
76.74
54.21
Ph
Ph
4b
BRUKER
Current Data Parameters
NAME 13C
EXPNO 313
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180705
Time 5.12
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 10000
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 295.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.8131145 MHz
NUC1 13C
P1 9.70 usec
PLW1 73.00000000 W
======== CHANNEL f2 ========
SFO2 500.3020012 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.13599968 W
PLW12 0.53924000 W
PLW13 0.34512001 W
F2 - Processing parameters
SI 32768
SF 125.8005378 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

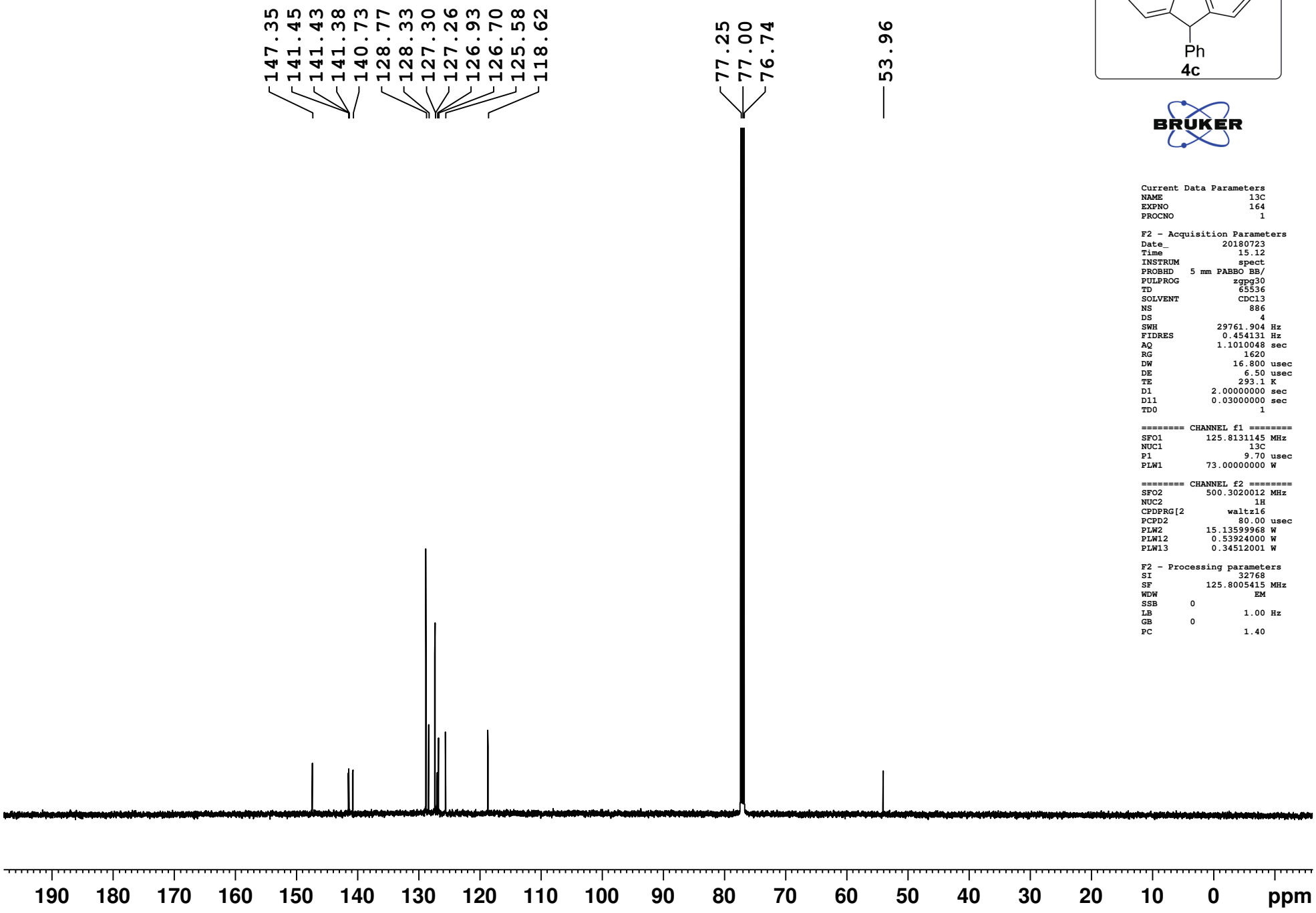


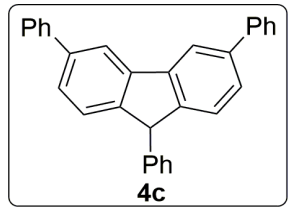


BRUKER

```
Current Data Parameters
NAME                  FID
EXPNO                 164
PROCNO                  1

F2 - Acquisition Parameters
Date_              20180723
Time                 16.01
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH              10000.000 Hz
FIDRES               0.152588 Hz
AQ                3.2767999 sec
RG                     203
DW                  50.000 usec
DE                   6.50 usec
TE                  292.4 K
D1               1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1            500.3030896 MHz
NUC1                    1H
P1                   15.10 usec
PLW1            15.13599968 W

F2 - Processing parameters
SI                   65536
SF              500.3000107 MHz
WDW                     EM
SSB          0
LB                   0.30 Hz
GB           0
PC                   1.00
```

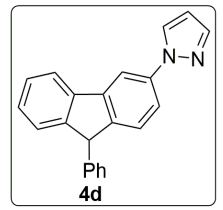






BRUKER

```
Current Data Parameters
NAME                  13C
EXPNO                 164
PROCNO                  1

F2 - Acquisition Parameters
Date_              20180723
Time                  15.12
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDC13
NS                     886
DS                       4
SWH               29761.904 Hz
FIDRES             0.454131 Hz
AQ                1.1010048 sec
RG                     1620
DW                   16.800 usec
DE                    6.50 usec
TE                   293.1 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1           125.8131145 MHz
NUC1                    13C
P1                    9.70 usec
PLW1           73.00000000 W

======== CHANNEL f2 ========
SFO2           500.3020012 MHz
NUC2                    1H
CPDPRG[2            waltz16
PCPD2                80.00 usec
PLW2           15.13599968 W
PLW12           0.53924000 W
PLW13           0.34512001 W

F2 - Processing parameters
SI                    32768
SF             125.8005415 MHz
WDW                      EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```



8.162
8.158
8.010
8.005
7.874
7.859
7.780
7.777
7.555
7.551
7.538
7.534
7.438
7.423
7.409
7.383
7.367
7.349
7.335
7.323
7.308
7.294
7.279
7.269
7.256
7.251
7.122
7.119
7.105
6.516
6.512
6.508
5.101

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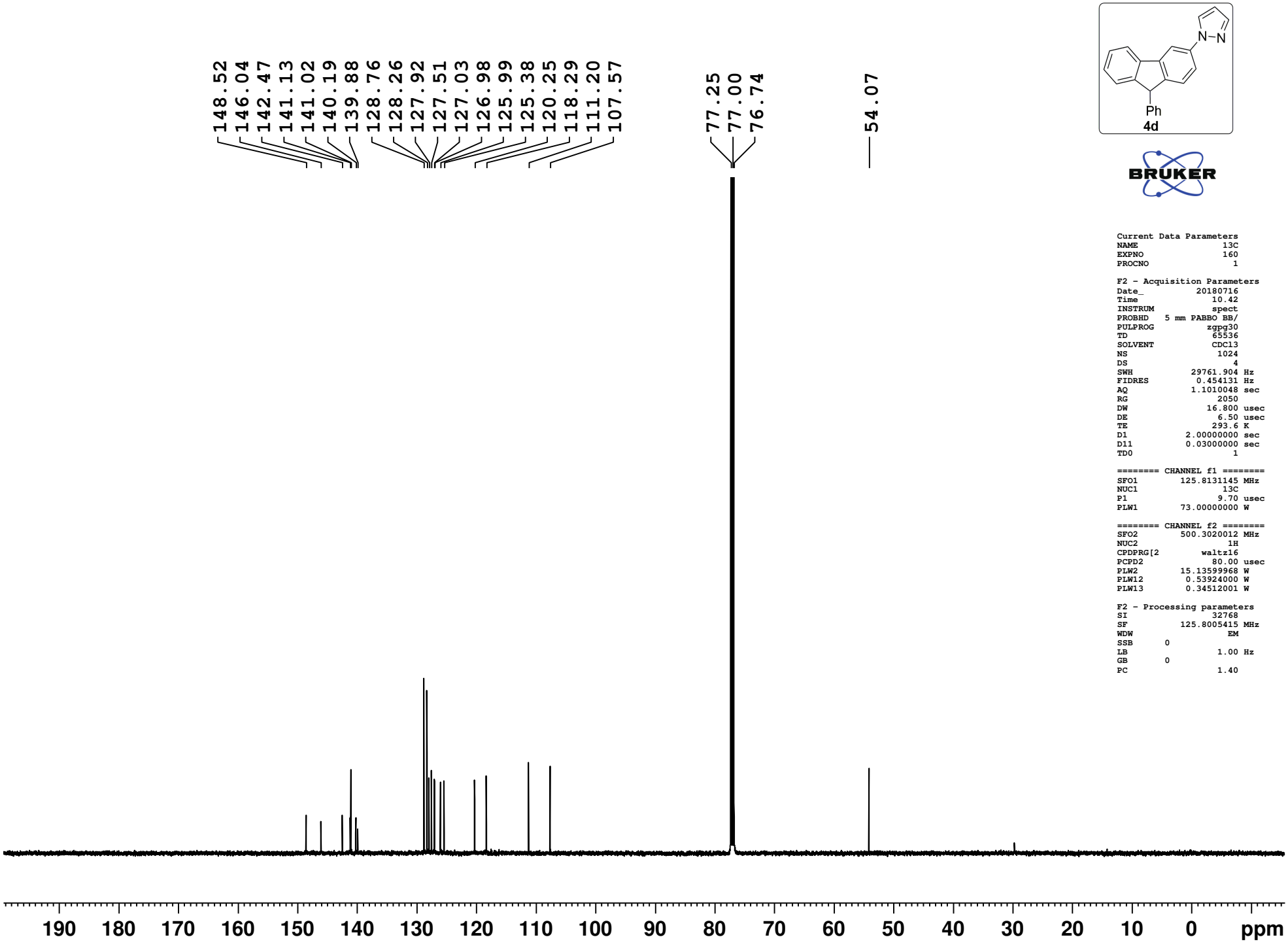
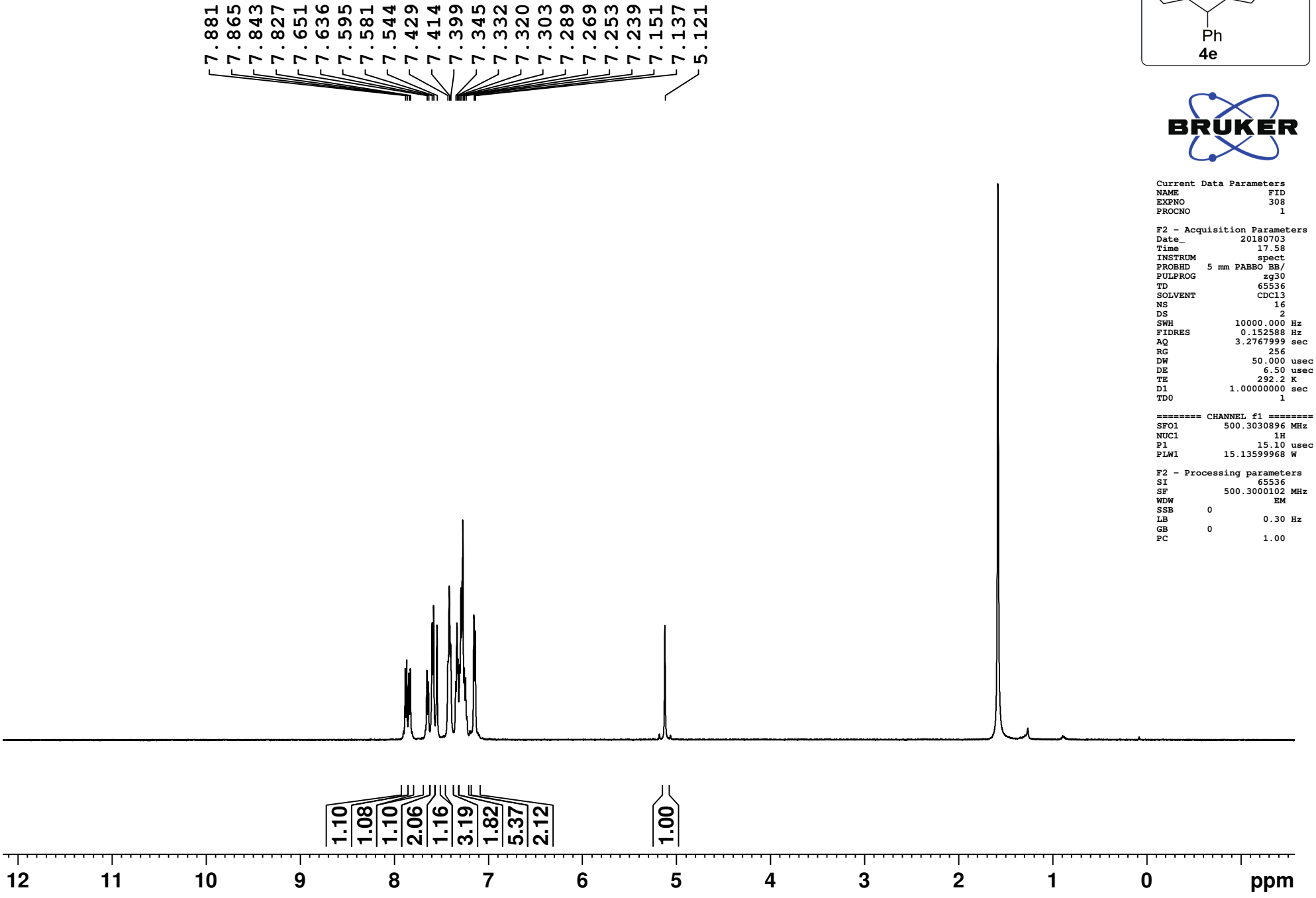
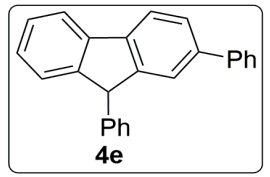
```
Current Data Parameters
NAME                  FID
EXPNO                 160
PROCNO                1

F2 - Acquisition Parameters
Date_              20180716
Time                  9.24
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH             10000.000 Hz
FIDRES              0.152588 Hz
AQ                3.2767999 sec
RG                    256
DW                 50.000 usec
DE                  6.50 usec
TE                 290.9 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          500.3030896 MHz
NUC1                  1H
P1                 15.10 usec
PLW1          15.13599968 W

F2 - Processing parameters
SI                  65536
SF            500.3000094 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

1.03 1.07 1.09 1.00 1.10 1.11 1.06 6.63 2.14 1.03 1.08

12 11 10 9 8 7 6 5 4 3 2 1 0 ppm


148.52
146.04
142.47
141.13
141.02
140.19
139.88
128.76
128.26
127.92
127.51
127.03
126.98
125.99
125.38
120.25
118.29
111.20
107.57
77.25
77.00
76.74
54.07
N–N
Ph
4d
BRUKER
Current Data Parameters
NAME 13C
EXPNO 160
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180716
Time 10.42
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 293.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.8131145 MHz
NUC1 13C
P1 9.70 usec
PLW1 73.00000000 W
======== CHANNEL f2 ========
SFO2 500.3020012 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.13599968 W
PLW12 0.53924000 W
PLW13 0.34512001 W
F2 - Processing parameters
SI 32768
SF 125.8005415 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.881
7.865
7.843
7.827
7.651
7.636
7.595
7.581
7.544
7.429
7.414
7.399
7.345
7.332
7.320
7.303
7.289
7.269
7.253
7.239
7.151
7.137
5.121
Ph
Ph
4e
BRUKER
Current Data Parameters
NAME FID
EXPNO 308
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180703
Time 17.58
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 256
DW 50.000 usec
DE 6.50 usec
TE 292.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.3030896 MHz
NUC1 1H
P1 15.10 usec
PLW1 15.13599968 W
F2 - Processing parameters
SI 65536
SF 500.3000102 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.10
1.08
1.10
2.06
1.16
3.19
1.82
5.37
2.12
1.00
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm



148.53
148.15
141.44
141.23
140.62
140.38
140.22
128.71
128.68
128.37
127.37
127.30
127.12
126.86
126.49
125.32
124.06
120.12
119.93

77.25
77.00
76.74

54.48

BRUKER

Current Data Parameters
NAME 13C
EXPNO 308
PROCNO 1

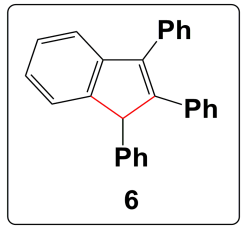
F2 - Acquisition Parameters
Date_ 20180703
Time 18.13
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 972
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 293.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 125.8131145 MHz
NUC1 13C
P1 9.70 usec
PLW1 73.00000000 W

======== CHANNEL f2 ========
SFO2 500.3020012 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.13599968 W
PLW12 0.53924000 W
PLW13 0.34512001 W

F2 - Processing parameters
SI 32768
SF 125.8005435 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

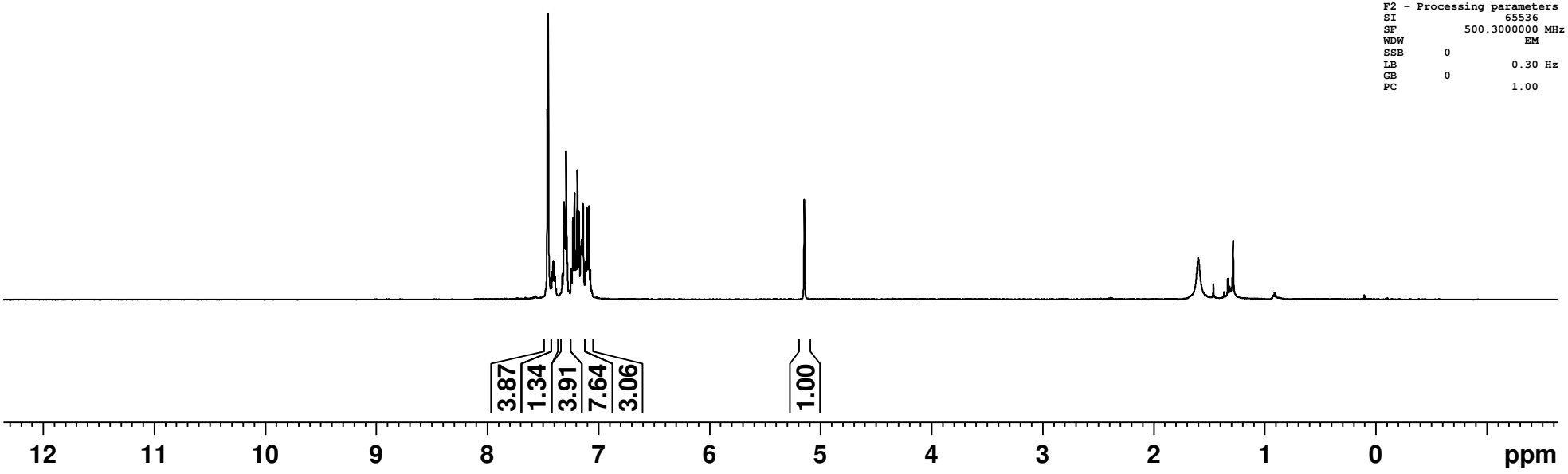
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



6

BRUKER

7.457
7.449
7.413
7.404
7.395
7.386
7.305
7.298
7.288
7.277
7.226
7.212
7.198
7.187
7.173
7.157
7.152
7.148
7.135
7.111
7.099
7.083
5.143

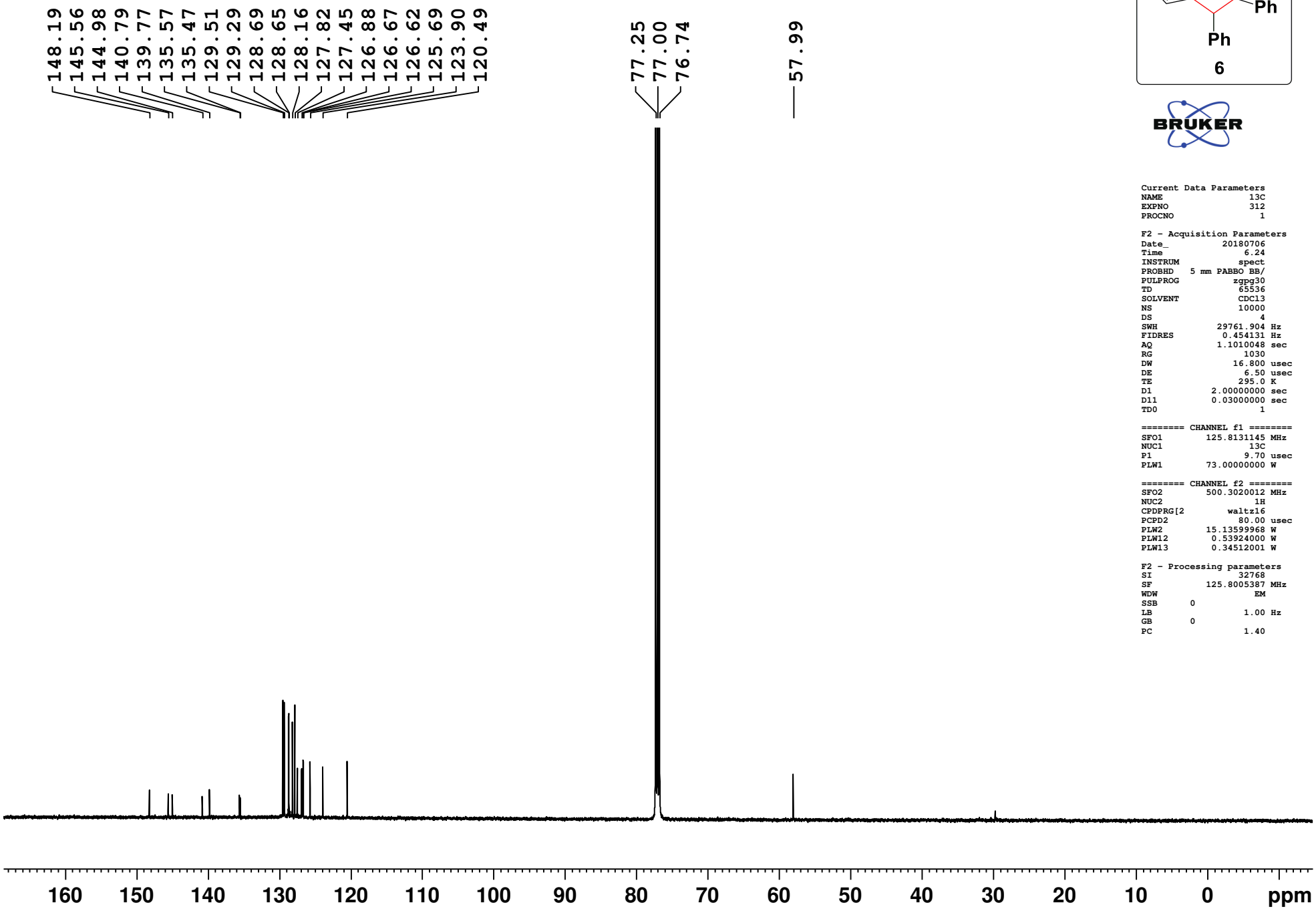


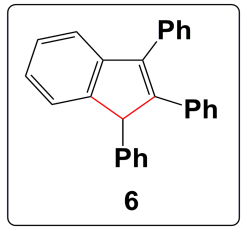
Current Data Parameters
NAME FID
EXPNO 312
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180706
Time 9.07
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 203
DW 50.000 usec
DE 6.50 usec
TE 291.2 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 500.3030896 MHz
NUC1 1H
P1 15.10 usec
PLW1 15.13599968 W

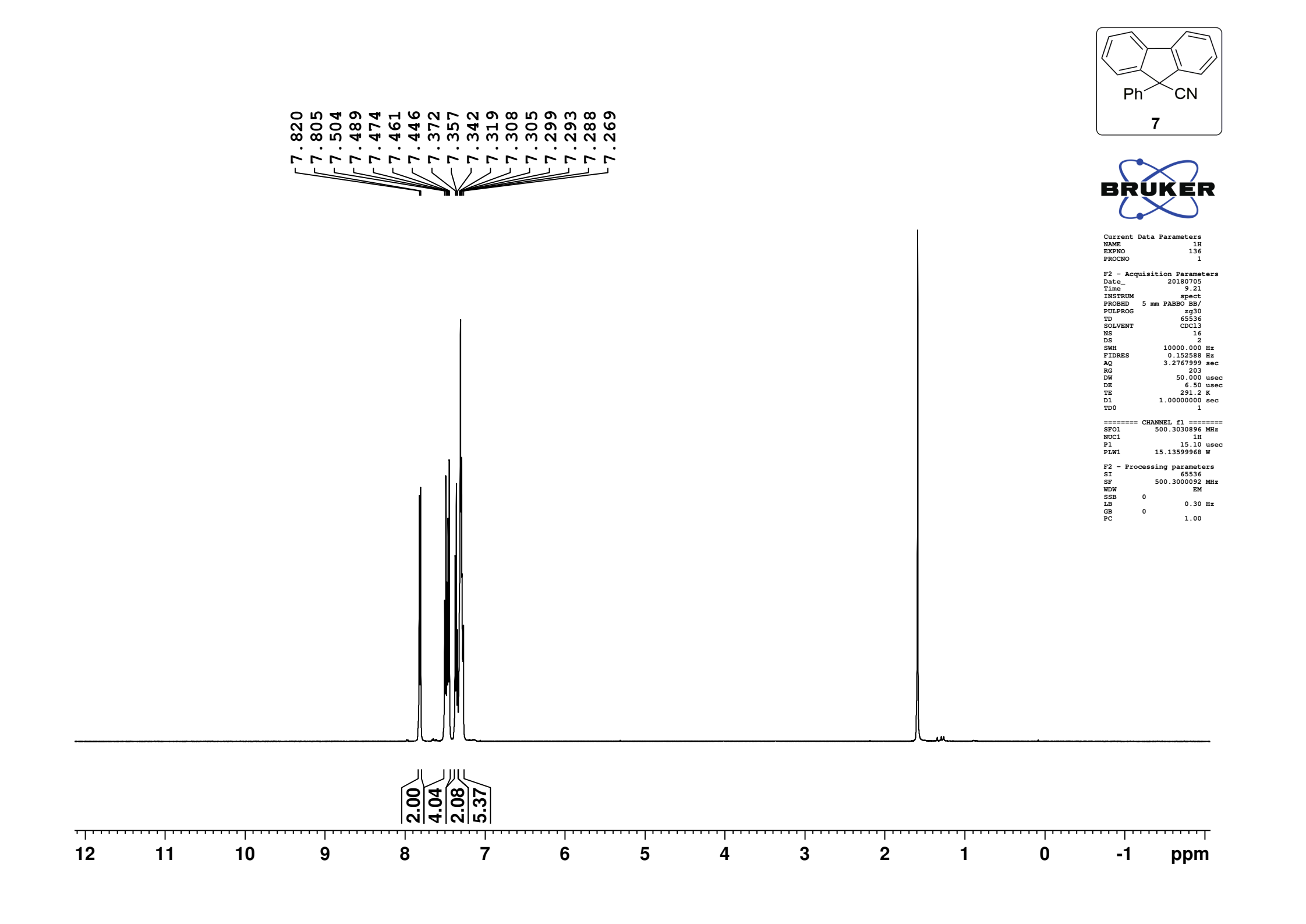
F2 - Processing parameters
SI 65536
SF 500.3000000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

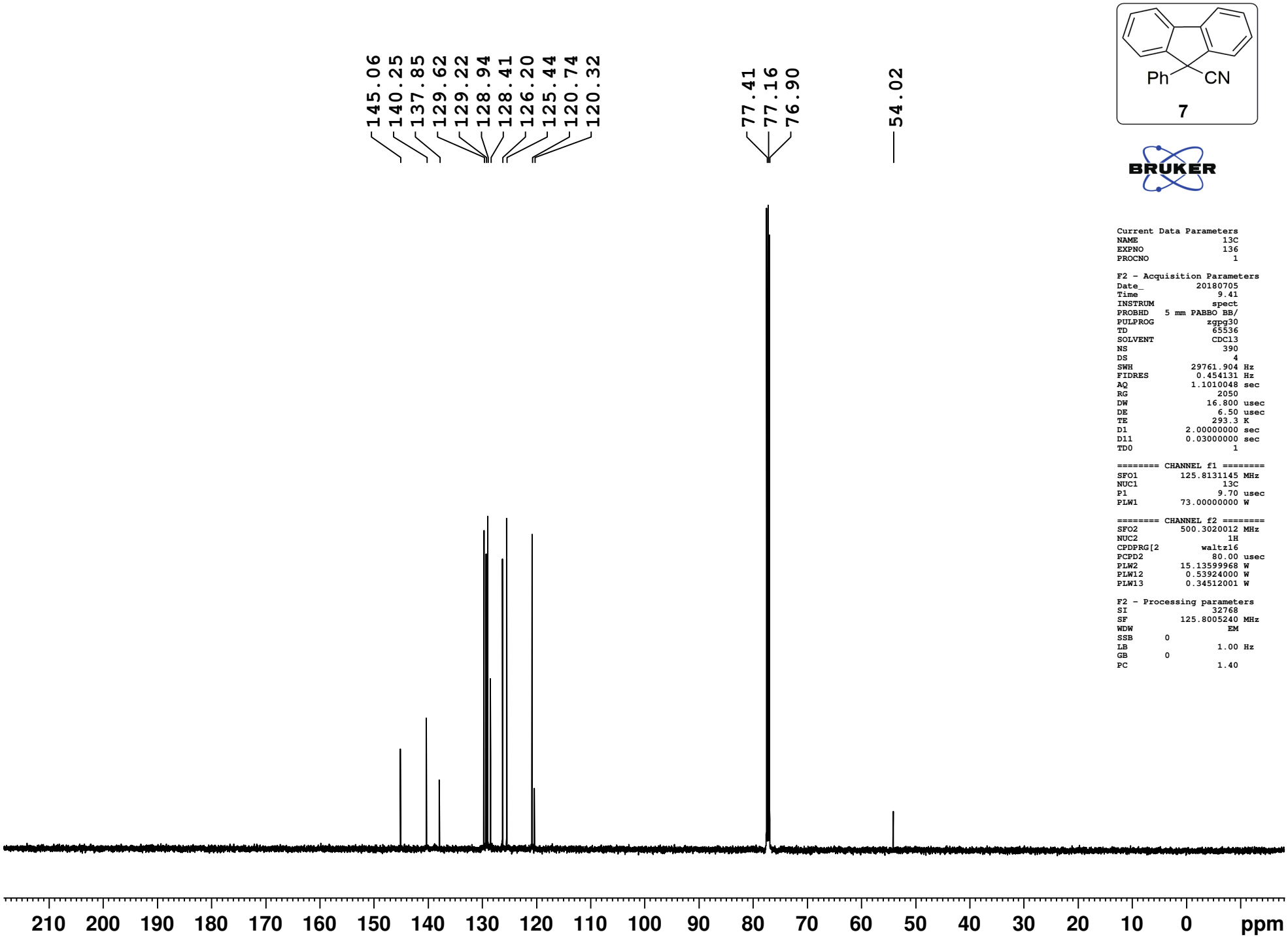


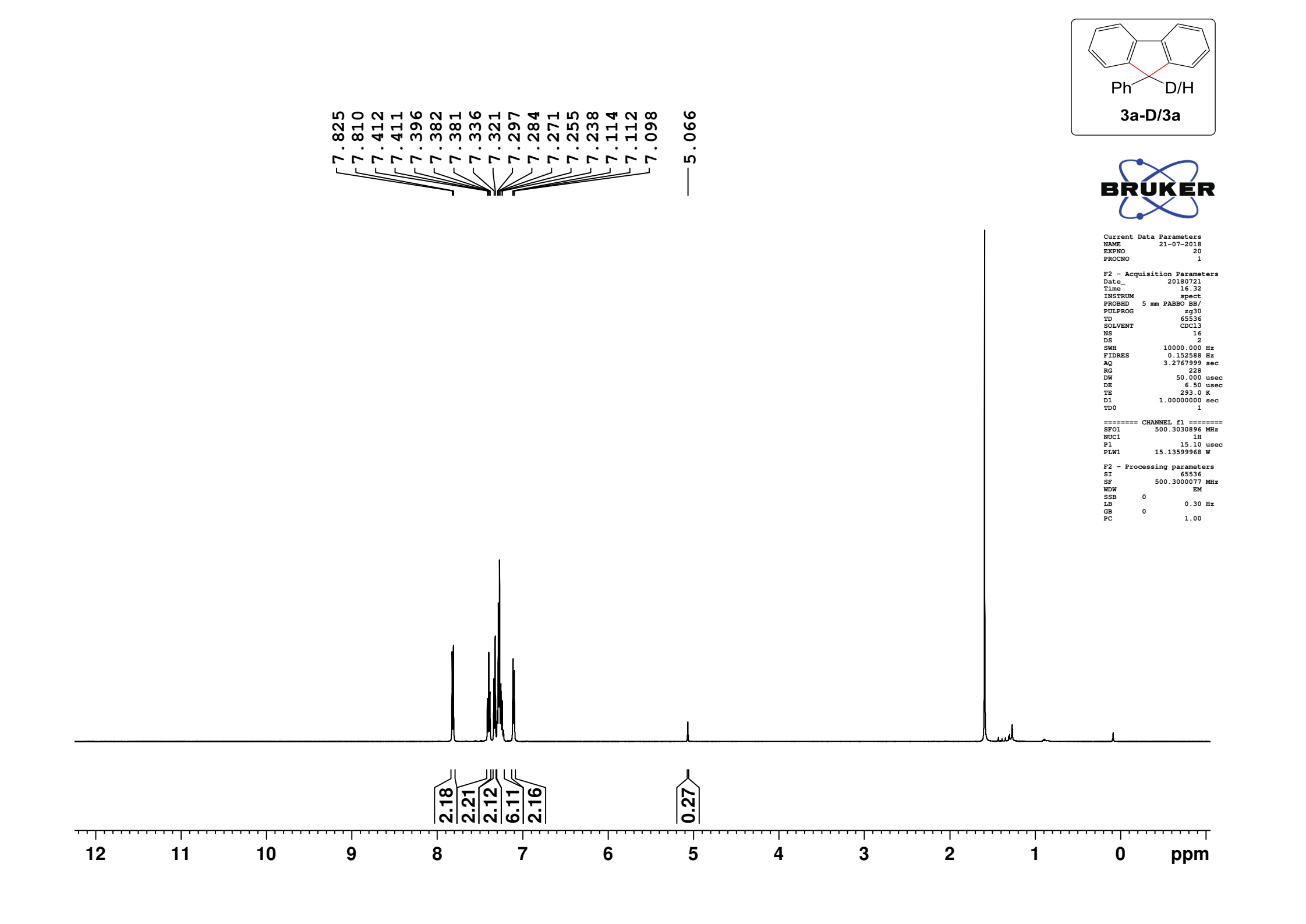


BRUKER

```
Current Data Parameters
NAME                  13C
EXPNO                 312
PROCNO                  1

F2 - Acquisition Parameters
Date_              20180706
Time                   6.24
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                   10000
DS                       4
SWH              29761.904 Hz
FIDRES           0.454131 Hz
AQ               1.1010048 sec
RG                    1030
DW                 16.800 usec
DE                  6.50 usec
TE                  295.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1           125.8131145 MHz
NUC1                   13C
P1                    9.70 usec
PLW1           73.00000000 W

======== CHANNEL f2 ========
SFO2           500.3020012 MHz
NUC2                   1H
CPDPRG[2            waltz16
PCPD2                80.00 usec
PLW2           15.13599968 W
PLW12           0.53924000 W
PLW13           0.34512001 W

F2 - Processing parameters
SI                    32768
SF             125.8005387 MHz
WDW                      EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```


7.820
7.805
7.504
7.489
7.474
7.461
7.446
7.372
7.357
7.342
7.319
7.308
7.305
7.299
7.293
7.288
7.269
Ph
CN
7
BRUKER
Current Data Parameters
NAME 1H
EXPNO 136
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180705
Time 9.21
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 203
DW 50.000 usec
DE 6.50 usec
TE 291.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.3030896 MHz
NUC1 1H
P1 15.10 usec
PLW1 15.13599968 W
F2 - Processing parameters
SI 65536
SF 500.3000092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
2.00
4.04
2.08
5.37
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm


145.06
140.25
137.85
129.62
129.22
128.94
128.41
126.20
125.44
120.74
120.32
77.41
77.16
76.90
54.02
Ph
CN
7
BRUKER
Current Data Parameters
NAME 13C
EXPNO 136
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180705
Time 9.41
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 390
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 293.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.8131145 MHz
NUC1 13C
P1 9.70 usec
PLW1 73.00000000 W
======== CHANNEL f2 ========
SFO2 500.3020012 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 15.13599968 W
PLW12 0.53924000 W
PLW13 0.34512001 W
F2 - Processing parameters
SI 32768
SF 125.8005240 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.825
7.810
7.412
7.411
7.396
7.382
7.381
7.336
7.321
7.297
7.284
7.271
7.255
7.238
7.114
7.112
7.098
5.066
Ph
D/H
3a-D/3a
BRUKER
Current Data Parameters
NAME 21-07-2018
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180721
Time 16.32
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 228
DW 50.000 usec
DE 6.50 usec
TE 293.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.3030896 MHz
NUC1 1H
P1 15.10 usec
PLW1 15.13599968 W
F2 - Processing parameters
SI 65536
SF 500.3000077 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
2.18
2.21
2.12
6.11
2.16
0.27
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm