Electronic Supplementary Material (ESI) for Nanoscale Advances.

Electronic Supporting Information

Direct aerobic oxidation of alcohols into esters catalyzed by carbon nanotube-gold nanohybrids

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1) General

Unless otherwise specified, chemicals were purchased from Sigma–Aldrich and used without further purification. Multi-walled carbon nanotubes were prepared by catalytic decomposition of methane on a Ni-MgO catalyst at the University of Xiamen (China).[1] Raw MWCNTs were purified by treatment with 8N HNO_3 for 12 h under refluxing conditions. Flash chromatography was carried out on Kieselgel 60 (230–240 mesh, Merck) and analytical TLC was performed on Merck precoated silica gel (60 F254). NMR spectra were recorded on a Bruker AVANCE DPX 400 spectrometer. ^{1}H NMR spectra were recorded at 400 MHz.

2) Catalyst preparation

The AuCNTs hybrid was prepared according to our previously described procedure.[2] The catalyst assembly was obtained as an aqueous suspension with a Au concentration of 5.4 mM as determined by inductively coupled plasma MS.

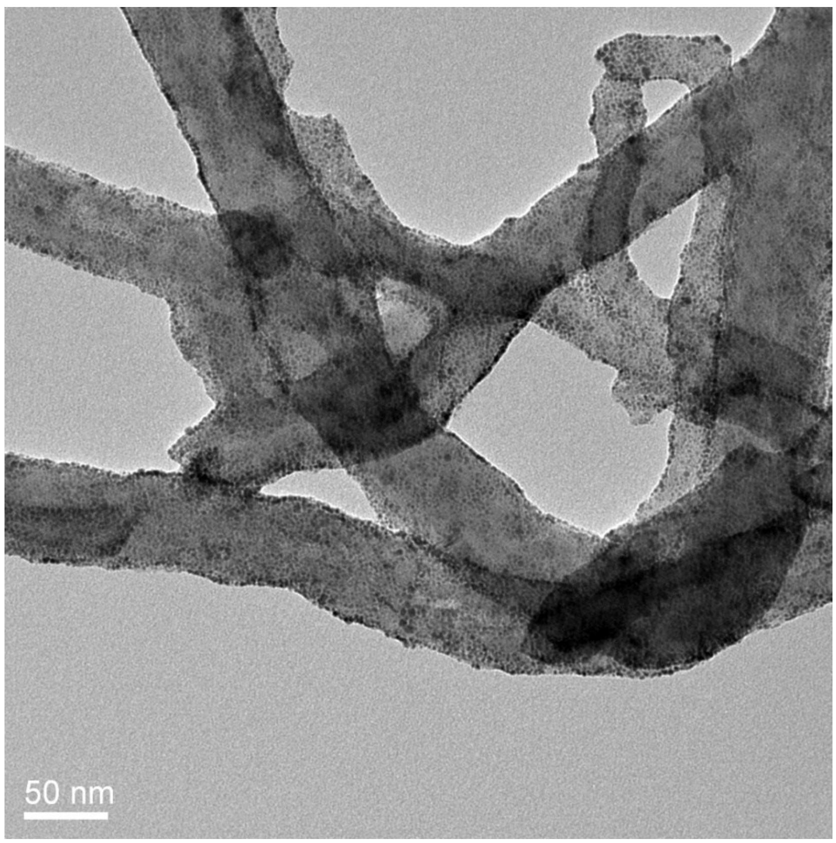


Figure S1: TEM image of the gold-CNT nanohybrid (AuCNT) catalyst.

3) General procedure for synthesis of methyl esters 2

100 μL of a 5.4 mM aqueous suspension of AuCNTs (0.54 mol%) was first washed twice with dry methanol, centrifuged and the supernatant was discarded. The pellet was redispersed in 1 mL of dry methanol and added to a stirred solution of either alcohol **1** or aldehyde **3** (0.1 mmol, 1 equiv.) and NaOH (1.5 equiv). The reaction mixture was stirred at room temperature under air until complete consumption of the starting material (as monitored by TLC). Solvent was then evaporated and the crude product was directly purified by silica gel chromatography using gradual elution of ethyl acetate and hexane as eluent. The structure of products **2a-j** was confirmed by ^{1}H NMR analysis.

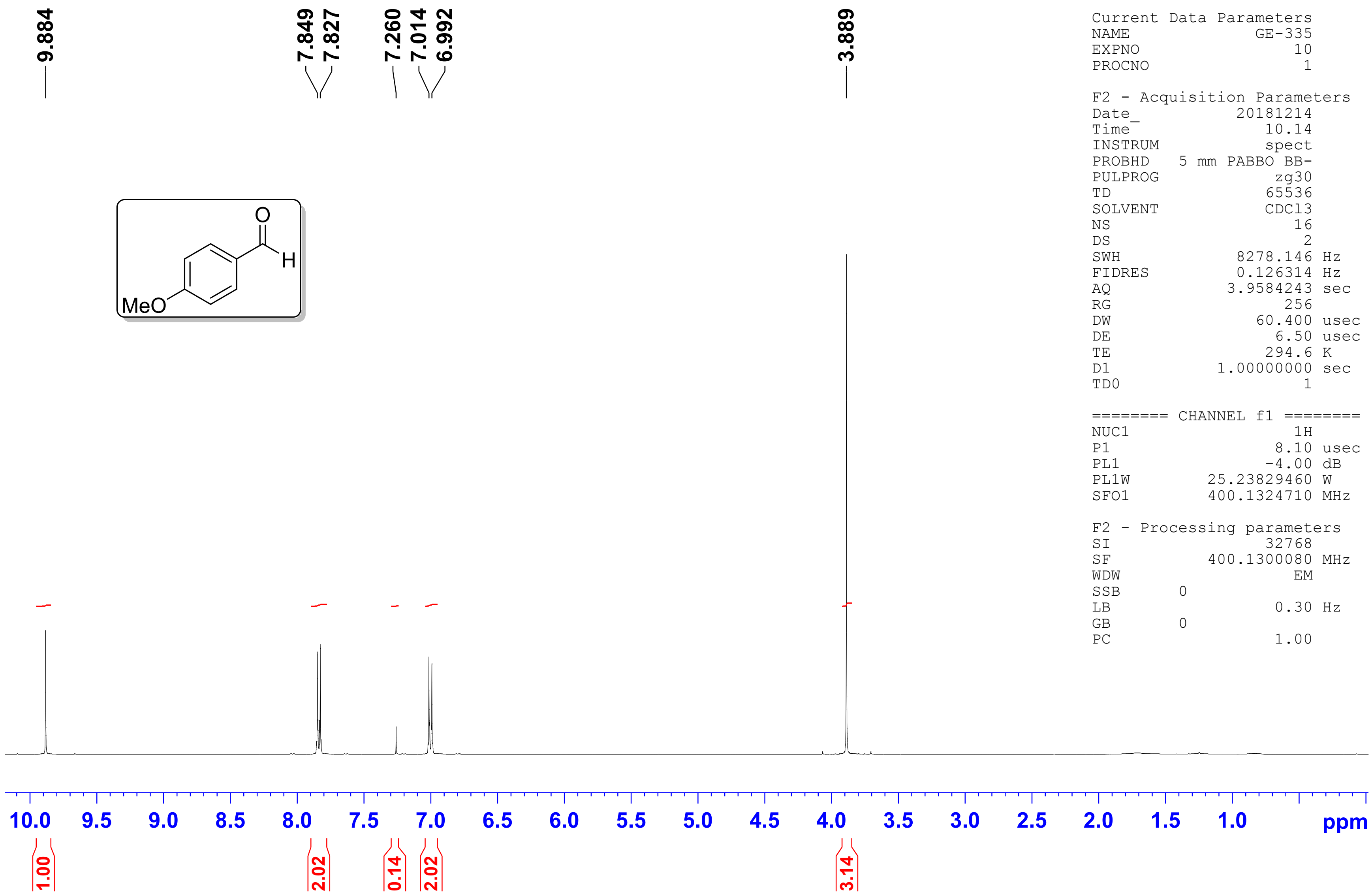
4) General Procedure for recycling experiments

100 μL of a 5.4 mM aqueous suspension of AuCNTs (0.54 mol%) was first washed twice with dry methanol, centrifuged and the supernatant was discarded. The pellet was redispersed in 1 mL of dry methanol and added to a stirred solution of anisyl alcohol **1a** (13.8 mg, 0.1 mmol, 1 equiv.) and NaOH (6 mg, 1.5 equiv.). The reaction mixture was stirred at room temperature under air until complete consumption of the starting material (as monitored by TLC). After completion of the reaction, the catalyst was separated from the reaction mixture by bench centrifugation for 5 min at 5000 rpm. The methanolic layer was collected and the nanohybrid was washed again with methanol by dispersion/centrifugation cycles. The combined methanolic phases were evaporated and the product purified by silica gel chromatography. The recovered catalyst was reused as is in subsequent experiments.

5) Procedure for the recovery of the aldehyde intermediate from the esterification reaction of 4-methoxy benzyl alcohol 2a

100 μL of a 5.4 mM aqueous suspension of AuCNTs (0.54 mol%) was first washed twice with dry methanol, centrifuged and the supernatant was discarded. The pellet was redispersed in 1 mL of dry methanol and added to a stirred solution of 4-methoxy benzyl alcohol **2a** (0.1 mmol, 1 equiv.) and NaOH (1.5 equiv.). The reaction mixture was stirred at room temperature under air for 18 h. Solvent was then evaporated and the crude product was directly purified by silica gel chromatography using gradual elution of ethyl acetate and hexane as eluent to yield 4-methoxy benzalhedyde in 35% yield.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.88 (s, 1H), 7.84 (d, J = 8.8 Hz, 2H), 7.00 (d, J = 8.8 Hz, 2H), 3.89 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 191.0, 164.7, 132.1, 130.1, 114.4, 55.7 ppm. NMR data are consistent with the literature.[3]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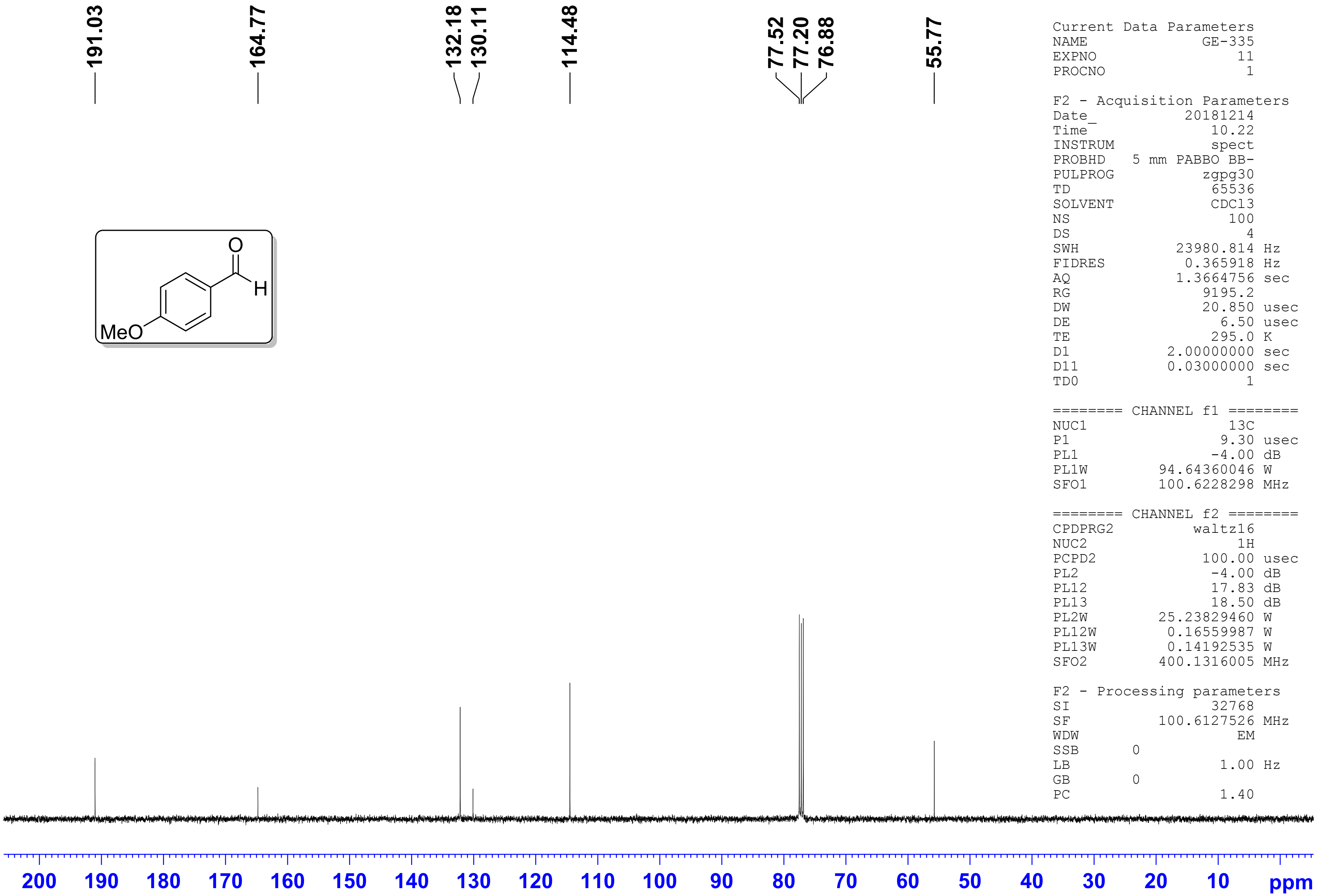
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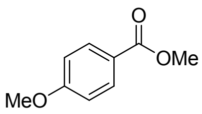
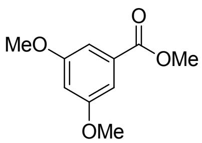
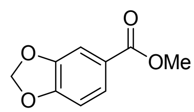
6) ^{1}H NMR Spectral data of synthesized methyl esters



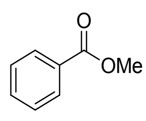
Methyl 4-methoxybenzoate (2a): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (d, J = 9.0 Hz, 2H), 6.92 (d, J = 9.0 Hz, 2H), 3.88 (s, 3H), 3.86 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.0, 163.4, 131.7, 122.7, 113.7, 55.5, 52.0 ppm. NMR data are consistent with the literature.[4]



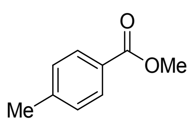
Methyl 3,5-dimethoxybenzoate (2b): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.19 (d, J = 2.4 Hz, 2H), 6.65 (t, J = 2.4 Hz, 1H), 3.91 (s, 3H), 3.83 (s, 6H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.0, 160.7, 132.1, 107.2, 105.9, 55.7, 52.4 ppm. NMR data are consistent with the literature.[5]



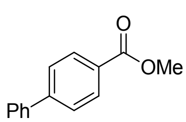
Methyl benzo[d] [1,3] dioxole-5-carboxylate (2c): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 (dd, J = 8.2, 1.6 Hz, 1H), 7.47 (d, J = 1.6 Hz, 1H), 6.84 (d, J = 8.2 Hz, 1H), 6.04 (s, 2H), 3.88 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.6, 151.7, 147.8, 125.4, 124.3, 109.6, 108.1, 101.9, 52.2 ppm. NMR data are consistent with the literature.[6]



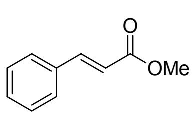
Methyl benzoate (2d): ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06-8.03 (m, 2H), 7.58 – 7.54 (m, 1H), 7.46–7.42 (m, 2H), 3.92 (s, 3H) ppm.^{13}C NMR (100 MHz, $CDCl_3$) δ 167.3, 133.0, 130.3, 129.7, 128.5, 52.2 ppm. NMR data are consistent with the literature.[5]



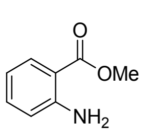
Methyl 4-methylbenzoate (2e): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 (d, J = 8.1 Hz, 2H), 7.24 (d, J = 8.1 Hz, 2H), 3.90 (s, 3H), 2.41 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.3, 143.7, 129.7, 129.2, 127.5, 52.1, 21.8 ppm. NMR data are consistent with the literature.[6]



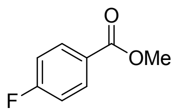
Methyl [1,1'-biphenyl]-4-carboxylate (2f): ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 – 8.09 (m, 2H), 7.68 – 7.65 (m, 2H), 7.64 – 7.62 (m, 2H), 7.49 – 7.45 (m, 2H), 7.42 – 7.37 (m, 1H), 3.94 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.1, 145.8, 140.1, 130.2, 129.1, 129.0, 128.3, 127.4, 127.2, 52.3 ppm. NMR data are consistent with the literature.[7]



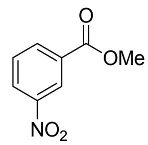
Methyl cinnamate (2g): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 (d, J = 16.0 Hz, 1H), 7.54 – 7.52 (m, 2H), 7.40 – 7.38 (m, 3H), 6.45 (d, J = 16.0 Hz, 1H), 3.81 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.5, 145.0, 134.5, 130.4, 129.0, 128.2, 117.9, 51.8 ppm. NMR data are consistent with the literature.[6]



Methyl 2-aminobenzoate (2h): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (dd, J = 8.0, 1.6 Hz, 1H), 7.29 – 7.25 (m, 1H), 6.70 – 6.64 (m, 2H), 5.52 (br s, 1H), 3.87 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.7, 150.1, 134.2, 131.3, 117.0, 116.7, 111.2, 51.7 ppm. NMR data are consistent with the literature.[8]



Methyl 4-fluorobenzoate (2i): ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08-8.03 (m, 2H), 7.14–7.08 (m, 2H), 3.91 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.1, 166.3, 164.6, 132.3, 132.2, 126.5, 115.7, 115.5, 52.3 ppm. NMR data are consistent with the literature.[9]



Methyl 3-nitrobenzoate (2j): ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.87 (t, *J* = 1.9, 1H), 8.42 (ddd, *J* = 8.2, 2.3, 1.1 Hz, 1H), 8.39–8.35 (m, *J* = 7.8, 1.3 Hz, 1H), 7.66 (t, *J* = 8.0 Hz, 1H), 3.99 (s, 4H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.1, 148.4, 135.4, 132.0, 129.8, 127.5, 124.7, 52.9 ppm. NMR data are consistent with the literature.[10]

7) References

1. P. Chen, H. B. Zhang, G. D. Lin, Q. Hong and K. R. Tsai. *Carbon* 1997, **35**, 1495.
2. J. John, E. Gravel, A. Hagege, H. Li, T. Gacoin and E. Doris. *Angew. Chem. int. Ed.* 2011, **50**, 7533.
3. S. Velusamy, M. Ahamed and T. Punniyamurthy. *Org. Lett.* 2004, **6**, 4821.
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5. J. Xia, A. Shao, S. Tang, X. Gao, M. Gao and A. Lei. *Org. Biomol. Chem.*, 2015, **13**, 6154.
6. F. Mao, Z. Qi, H. Fan, D. Sui, R. Chen and J. Huang. *RSC Adv.*, 2017, **7**, 1498.
7. X.-Y. Wang, H.-X. Song, S.-M. Wang, J. Yang, H.-L. Qin, X. Jiang and C.-P. Zhang. *Tetrahedron* 2016, **72**, 7606.
8. M. Baron, E. Métay, M. Lemaire and F. Popowycz. *Green Chem.*, 2013, **15**, 1006.
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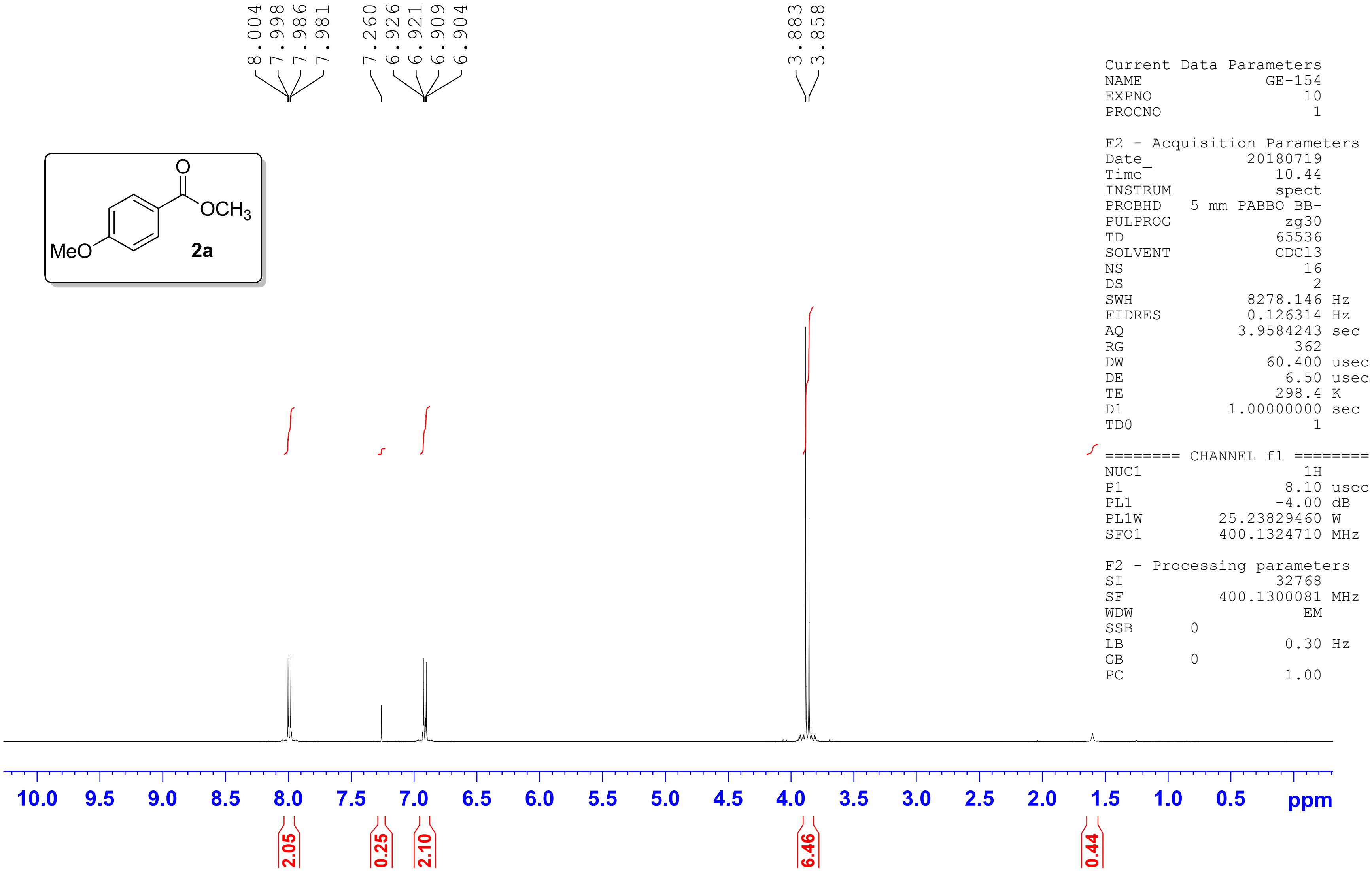
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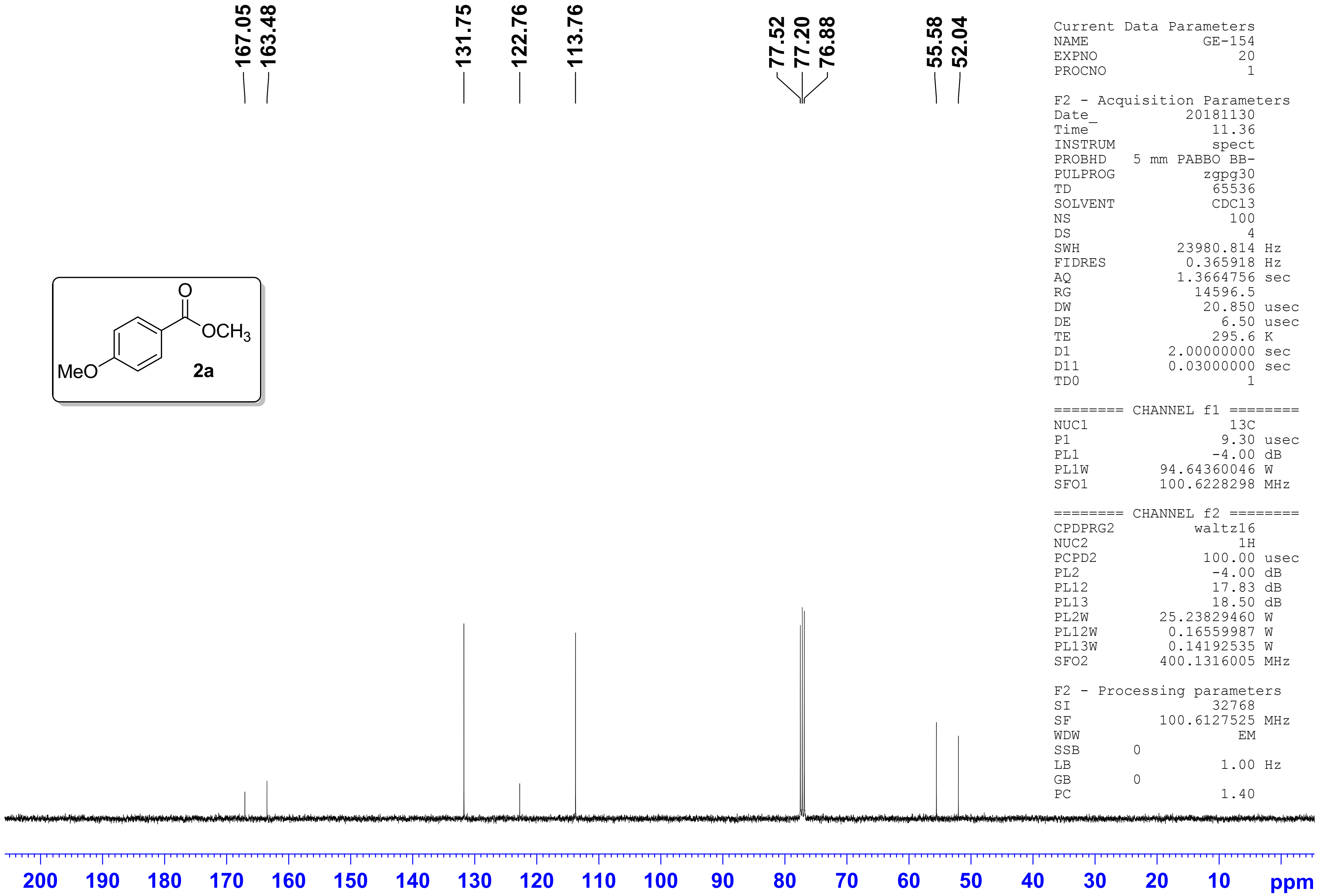


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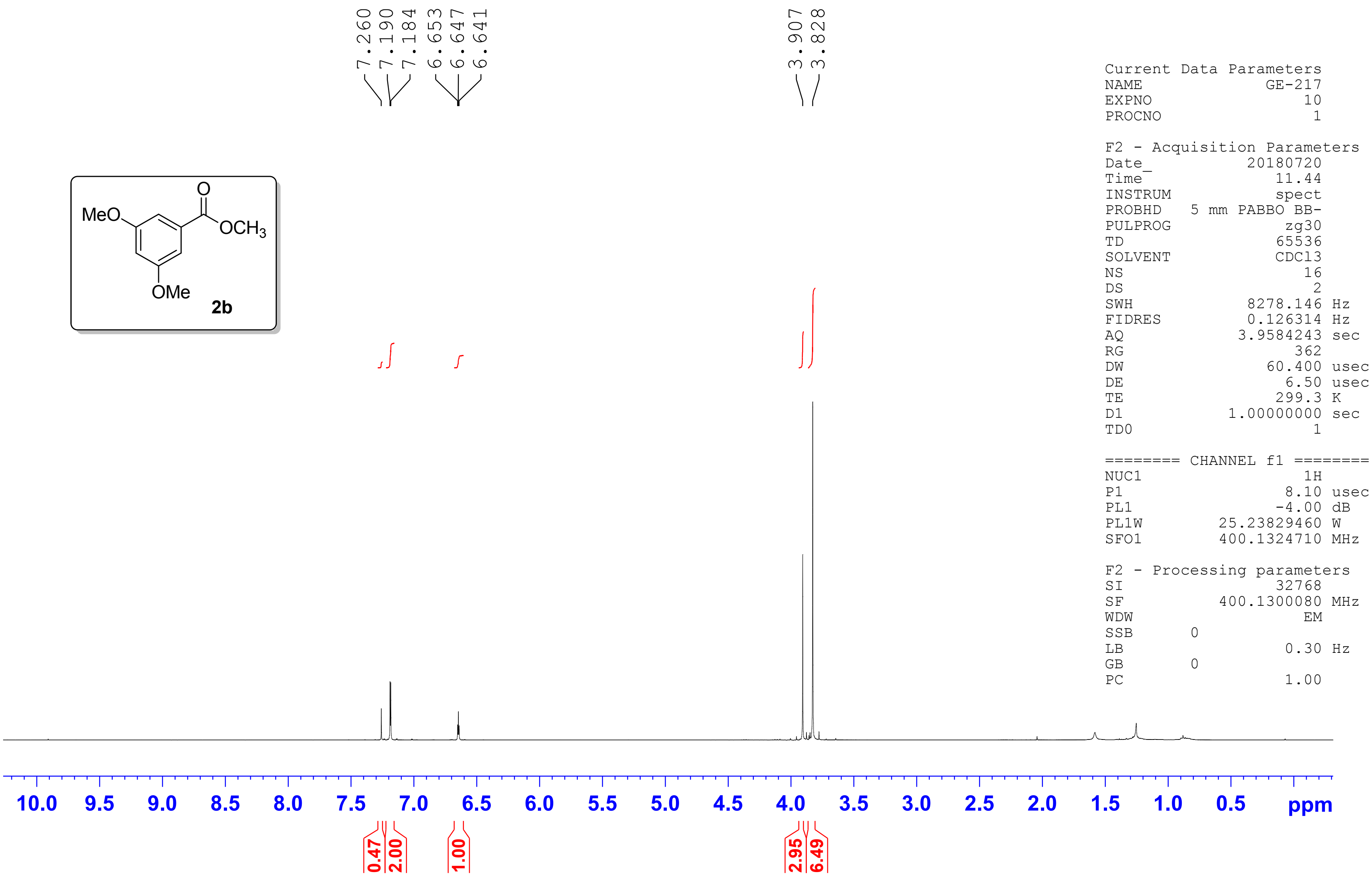
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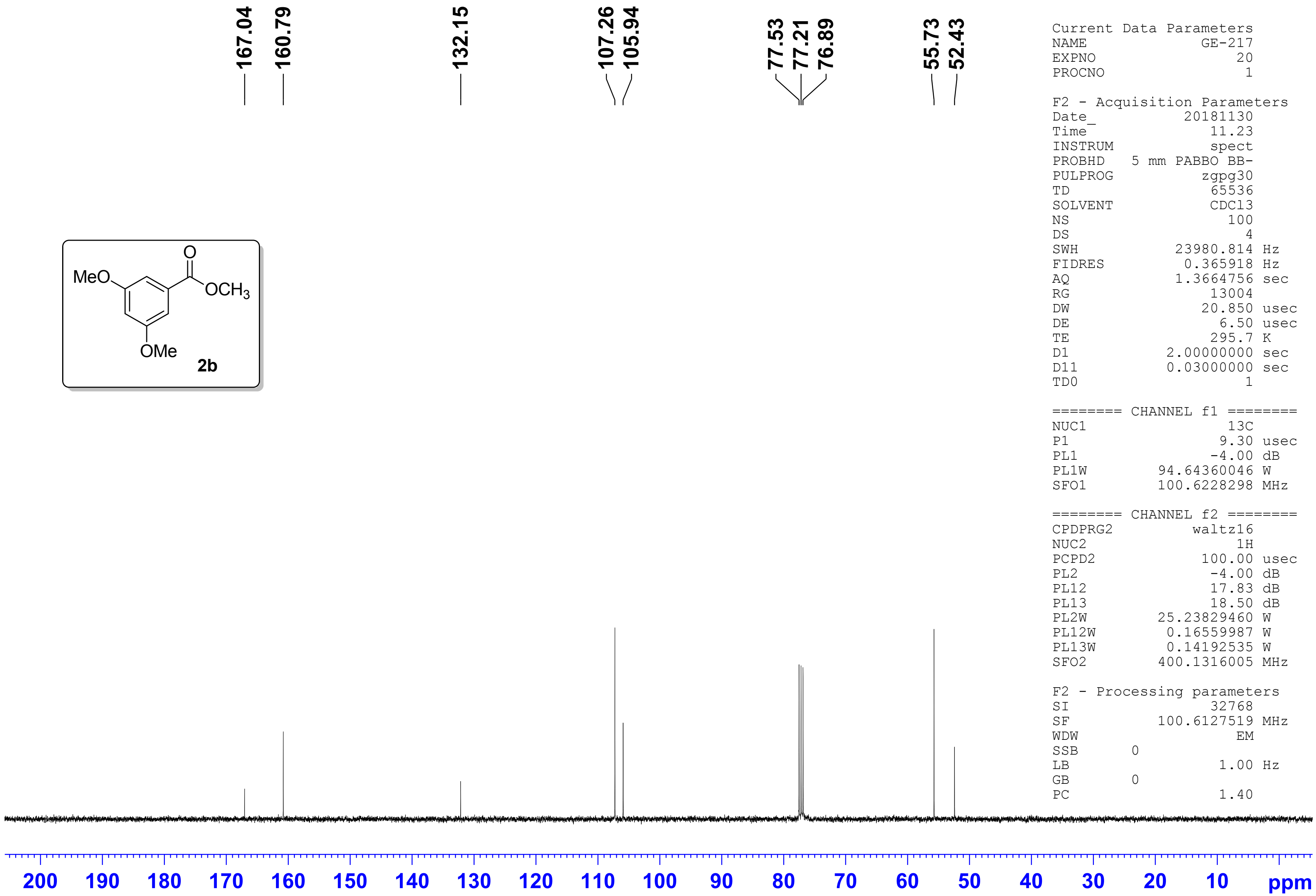


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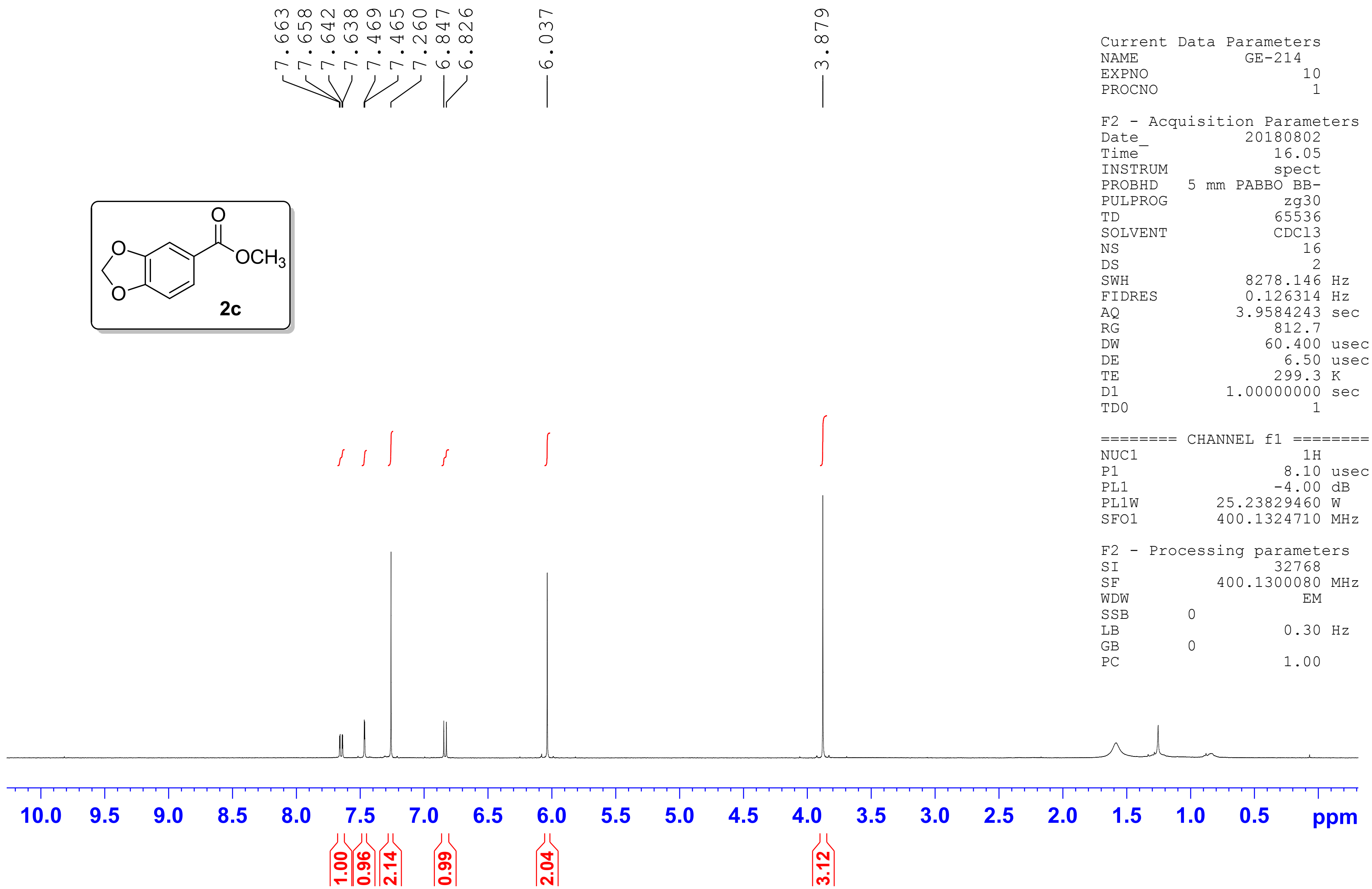
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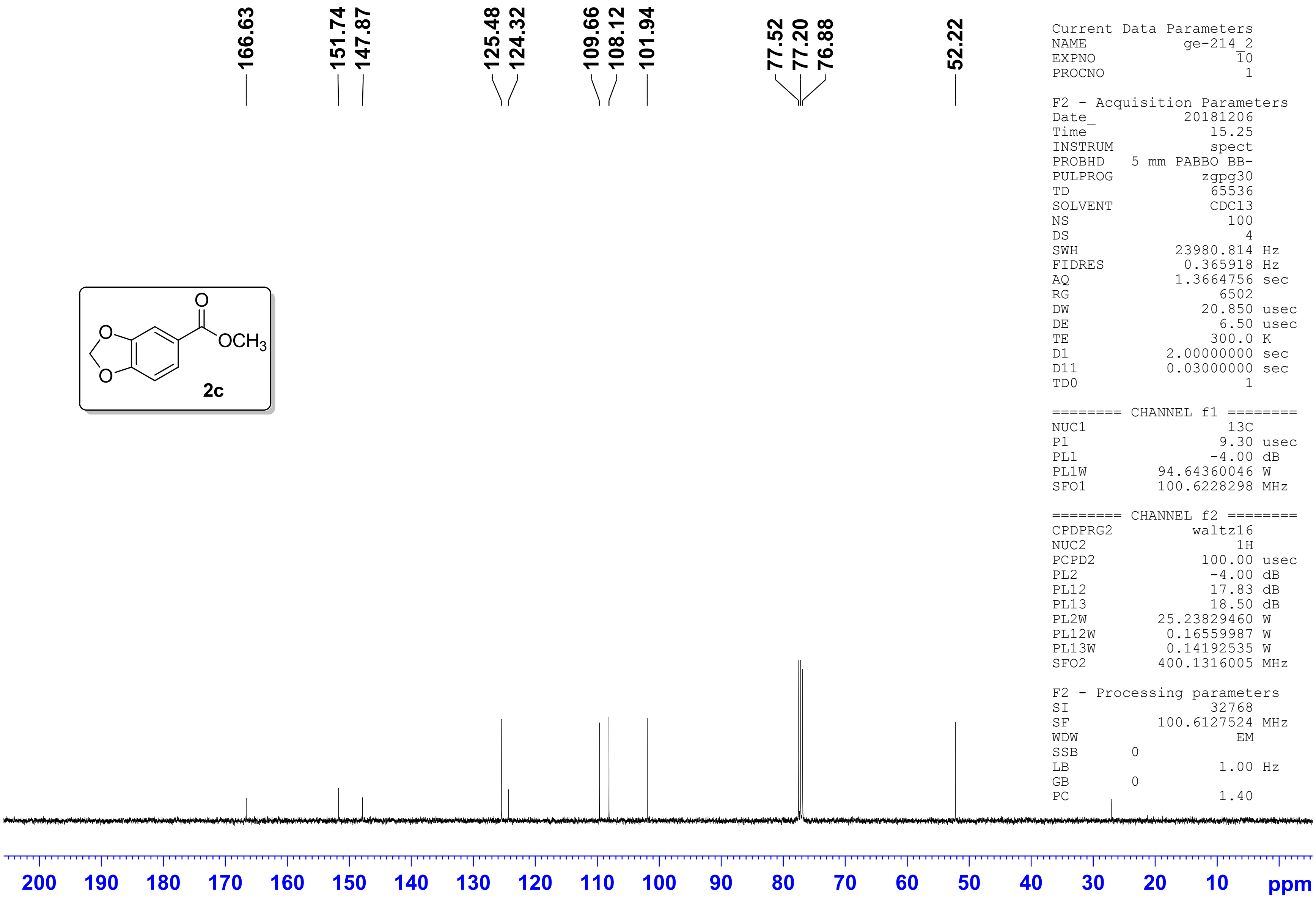
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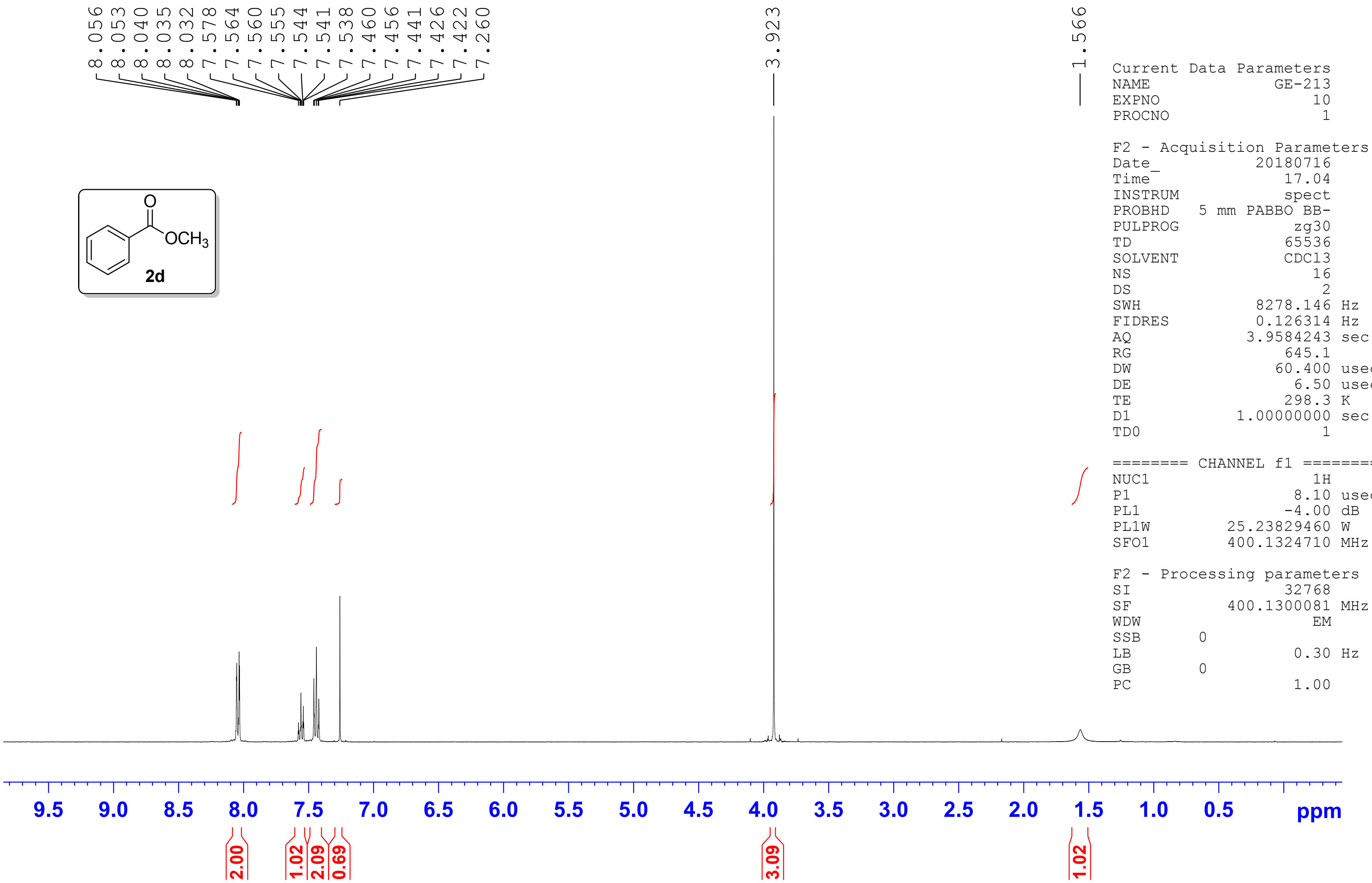
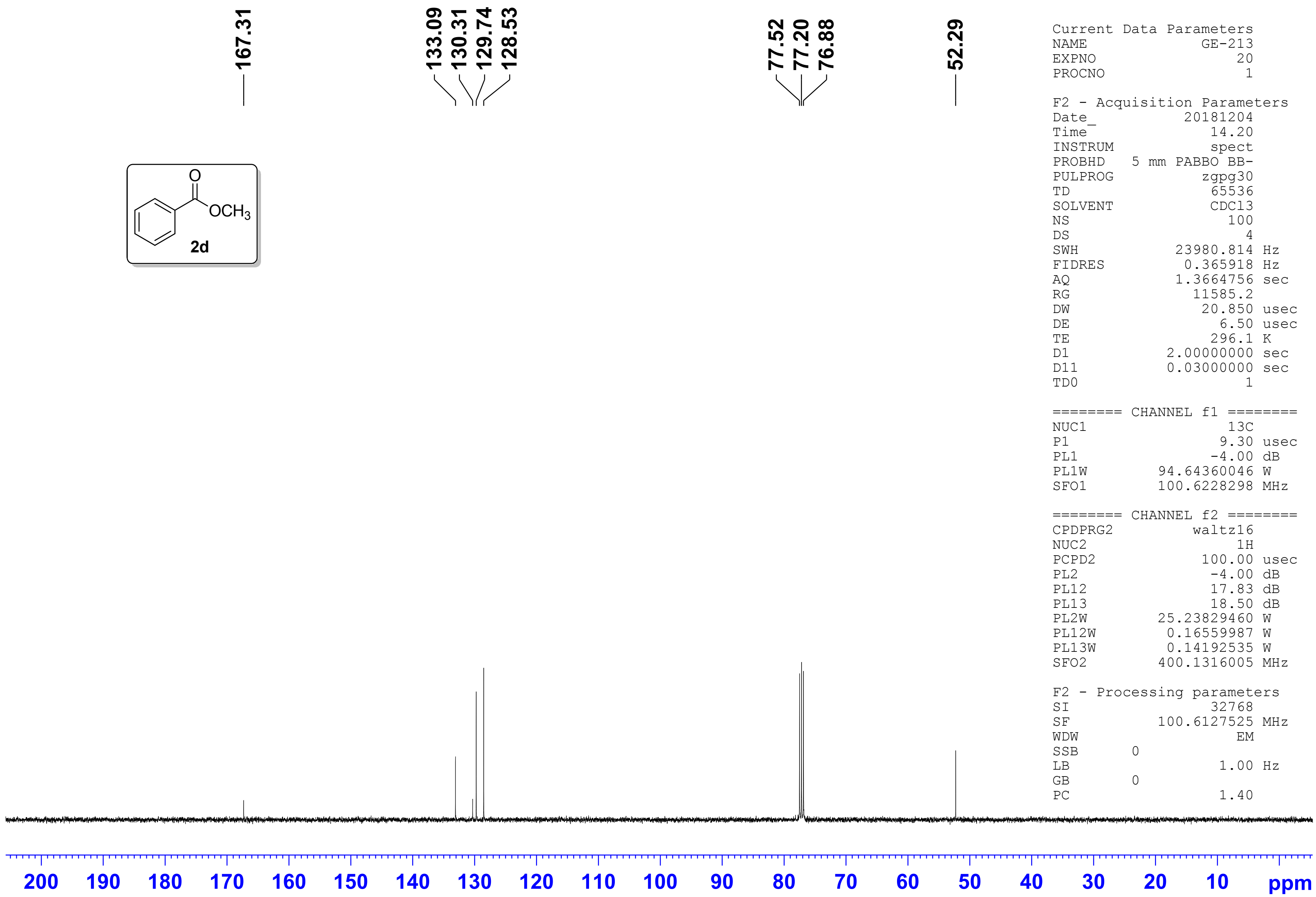
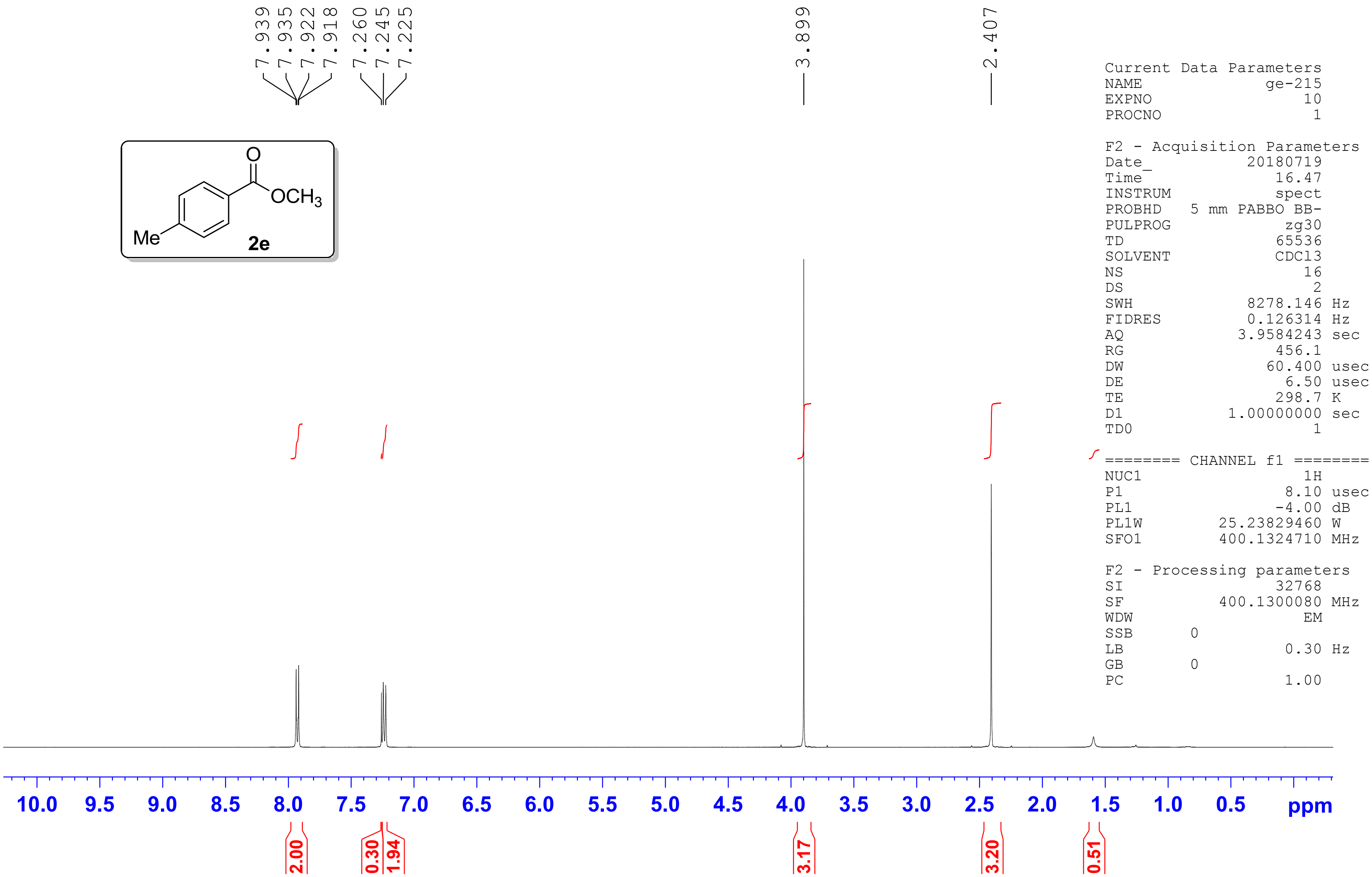
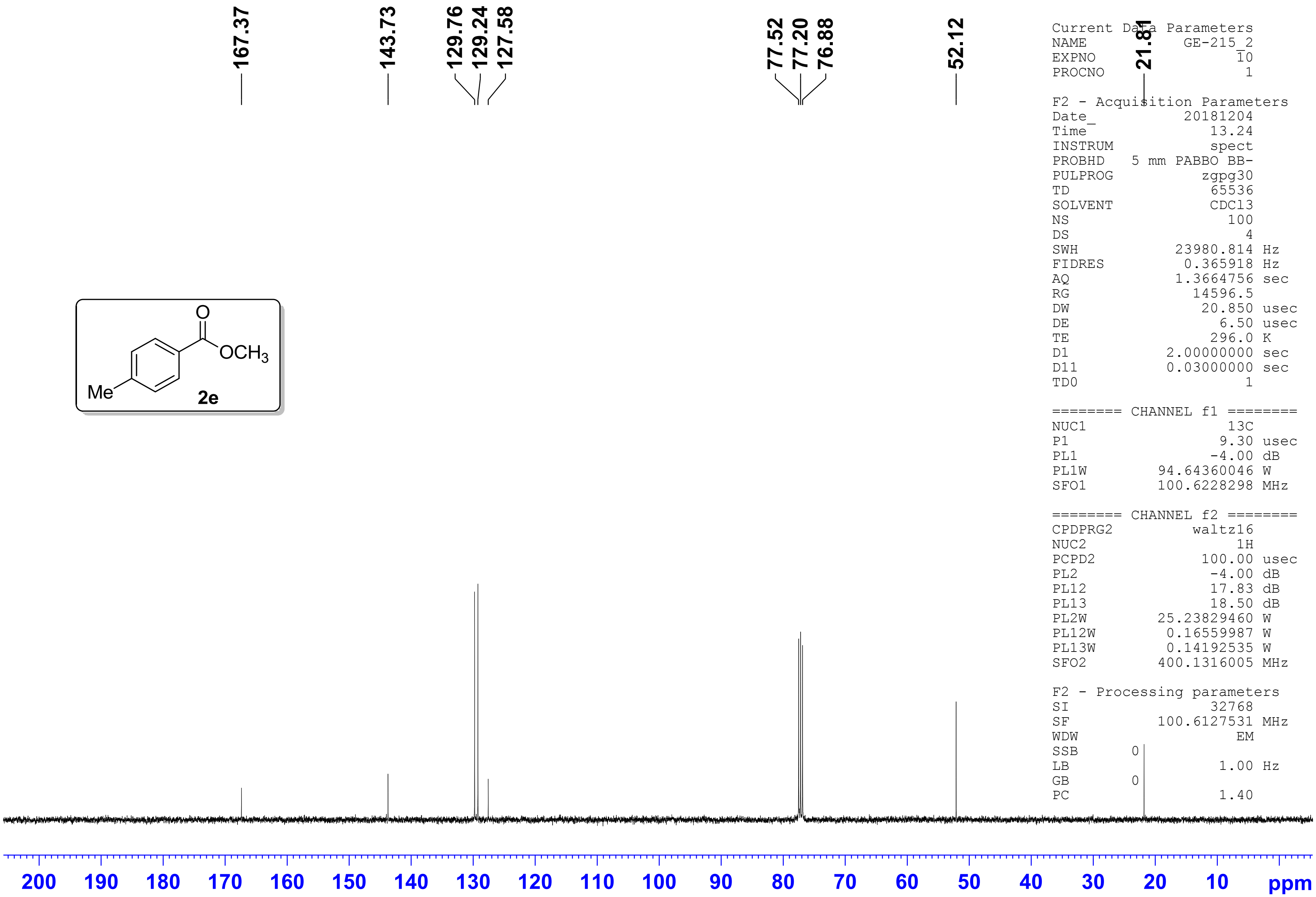
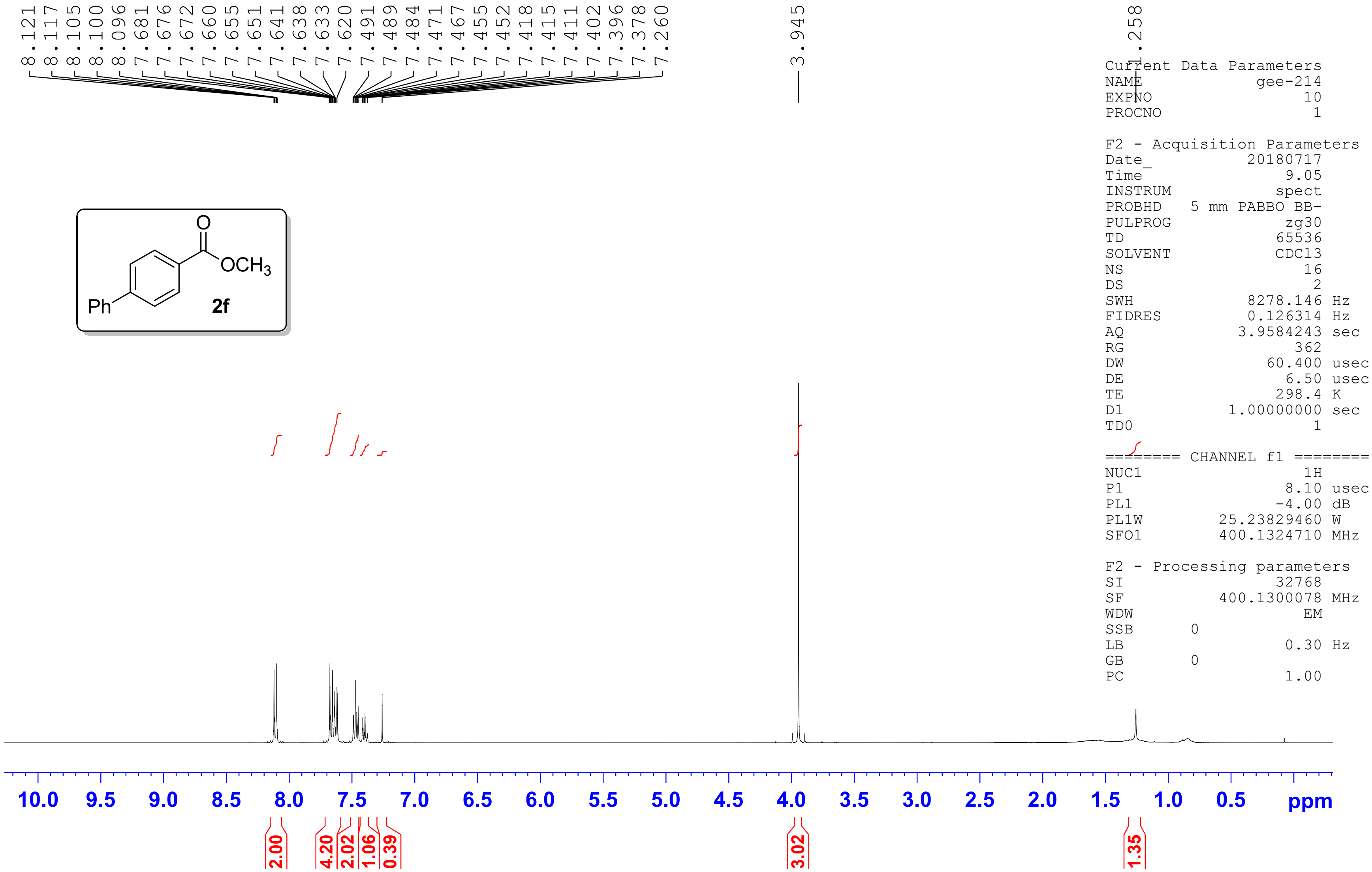
NS 100
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 6502
DW 20.850 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 -4.00 dB
PL1W 94.64360046 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.83 dB
PL13 18.50 dB
PL2W 25.23829460 W
PL12W 0.16559987 W
PL13W 0.14192535 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127524 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 ppm

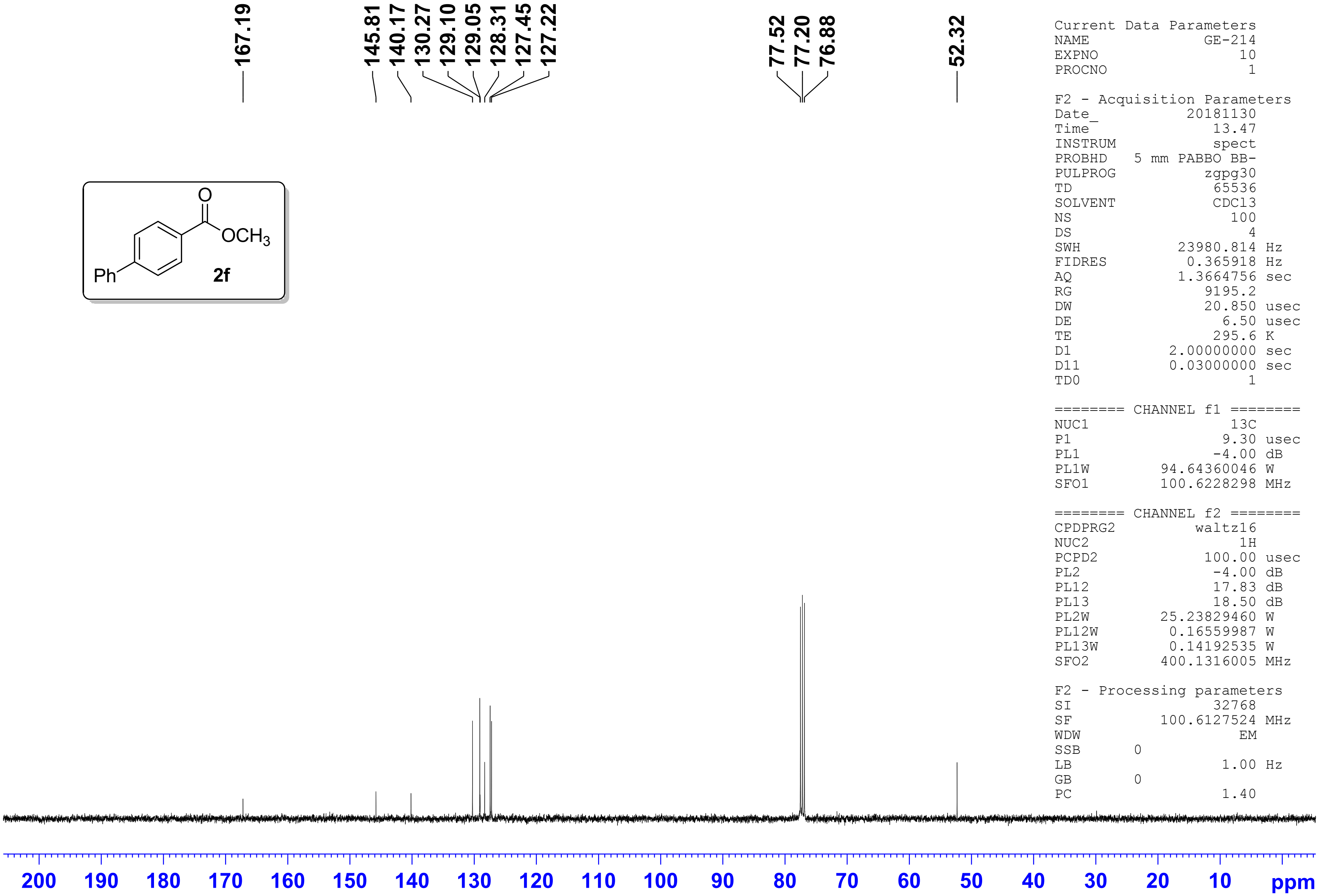

8.056 8.053 8.040 8.035 8.032 7.578 7.564 7.560 7.555 7.544 7.541 7.538 7.460 7.456 7.441 7.426 7.422 7.260
3.923
1.566
O
OCH3
2d
Current Data Parameters
NAME GE-213
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180716
Time 17.04
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 645.1
DW 60.400 usec
DE 6.50 usec
TE 298.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 8.10 usec
PL1 -4.00 dB
PL1W 25.23829460 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300081 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
2.00 1.02 2.09 0.69 3.09 1.02

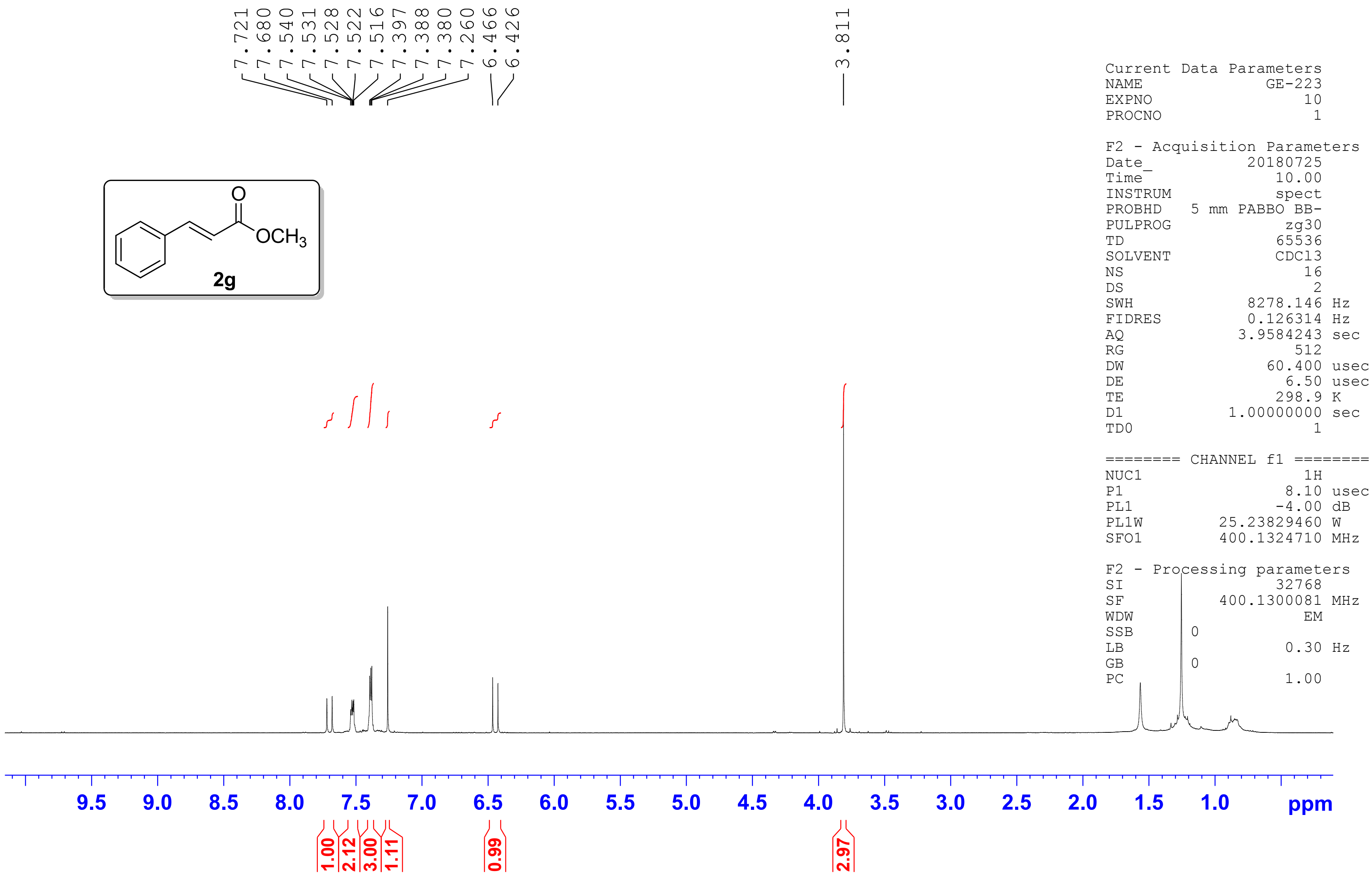

167.31
133.09
130.31
129.74
128.53
77.52
77.20
76.88
52.29
O
OCH3
2d
Current Data Parameters
NAME GE-213
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20181204
Time 14.20
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 100
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 11585.2
DW 20.850 usec
DE 6.50 usec
TE 296.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 -4.00 dB
PL1W 94.64360046 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.83 dB
PL13 18.50 dB
PL2W 25.23829460 W
PL12W 0.16559987 W
PL13W 0.14192535 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127525 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 ppm

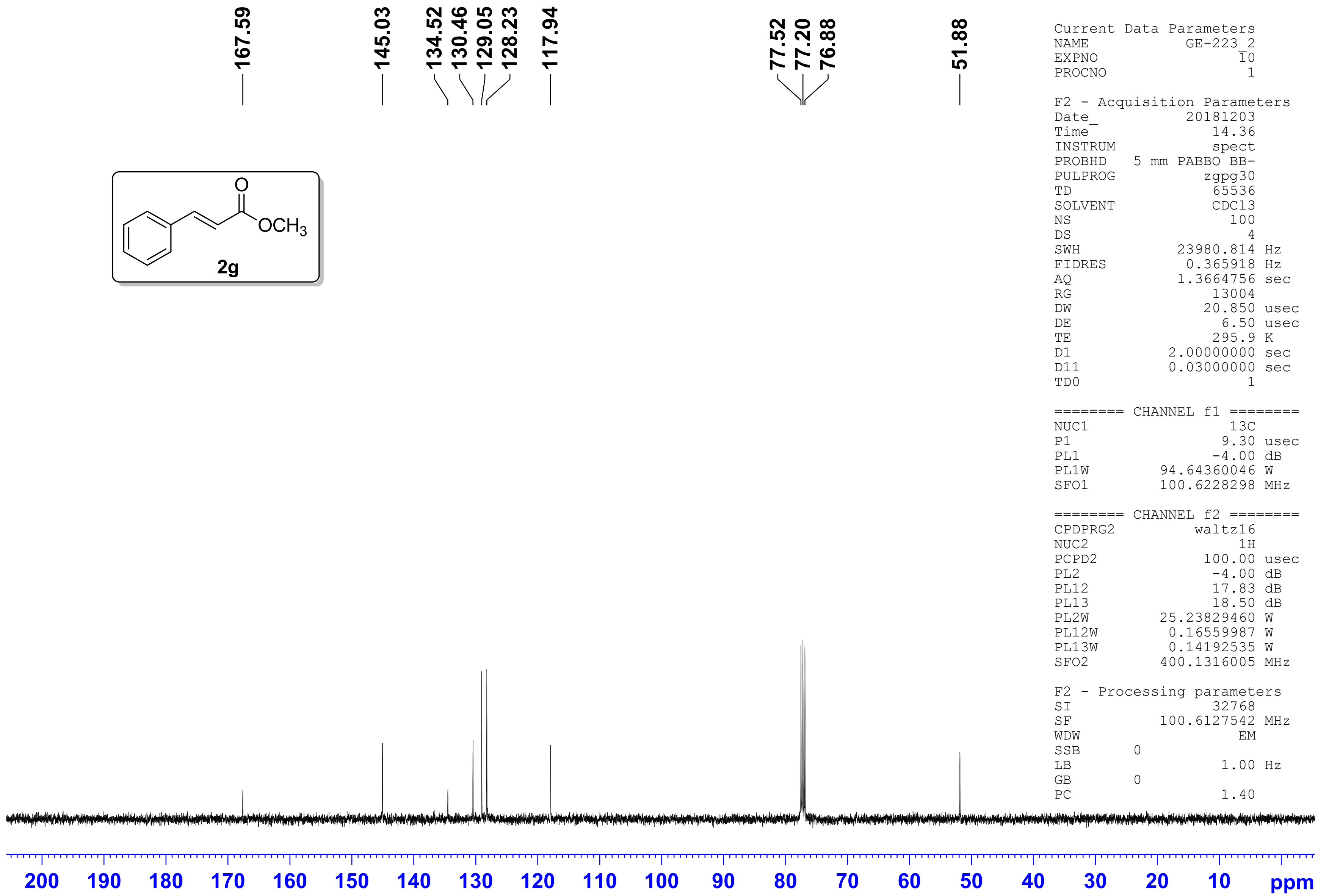

7.939
7.935
7.922
7.918
7.260
7.245
7.225
3.899
2.407
O
OCH3
Me
2e
Current Data Parameters
NAME ge-215
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180719
Time 16.47
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 456.1
DW 60.400 usec
DE 6.50 usec
TE 298.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 8.10 usec
PL1 -4.00 dB
PL1W 25.23829460 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300080 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
2.00
0.30
1.94
3.17
3.20
0.51


167.37
143.73
129.76
129.24
127.58
77.52
77.20
76.88
52.12
21.81
O
OCH3
Me
2e
Current Data Parameters
NAME GE-215_2
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20181204
Time_ 13.24
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 100
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 14596.5
DW 20.850 usec
DE 6.50 usec
TE 296.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 -4.00 dB
PL1W 94.64360046 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.83 dB
PL13 18.50 dB
PL2W 25.23829460 W
PL12W 0.16559987 W
PL13W 0.14192535 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127531 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 ppm

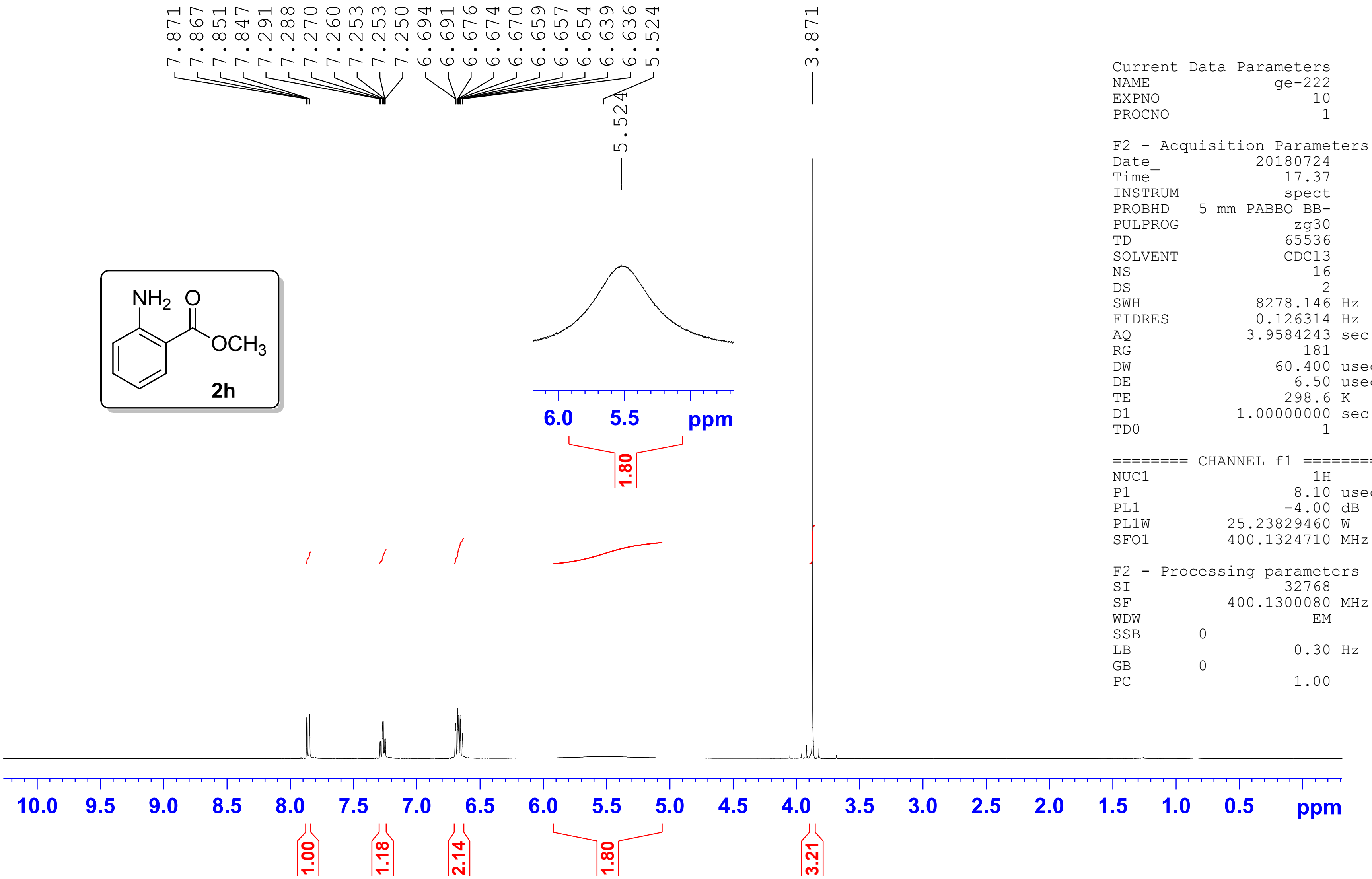

8.121
8.117
8.105
8.100
8.096
7.681
7.676
7.672
7.660
7.655
7.651
7.641
7.638
7.633
7.620
7.491
7.489
7.484
7.471
7.467
7.455
7.452
7.418
7.415
7.411
7.402
7.396
7.378
7.260
3.945
1.258
O
OCH3
Ph
2f
Current Data Parameters
NAME gee-214
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180717
Time 9.05
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DW 60.400 usec
DE 6.50 usec
TE 298.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 8.10 usec
PL1 -4.00 dB
PL1W 25.23829460 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300078 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.00
4.20
2.02
1.06
0.39
3.02
1.35


167.19
145.81
140.17
130.27
129.10
129.05
128.31
127.45
127.22
77.52
77.20
76.88
52.32
O
OCH3
Ph
2f
Current Data Parameters
NAME GE-214
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20181130
Time 13.47
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 100
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 9195.2
DW 20.850 usec
DE 6.50 usec
TE 295.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 -4.00 dB
PL1W 94.64360046 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.83 dB
PL13 18.50 dB
PL2W 25.23829460 W
PL12W 0.16559987 W
PL13W 0.14192535 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127524 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 ppm

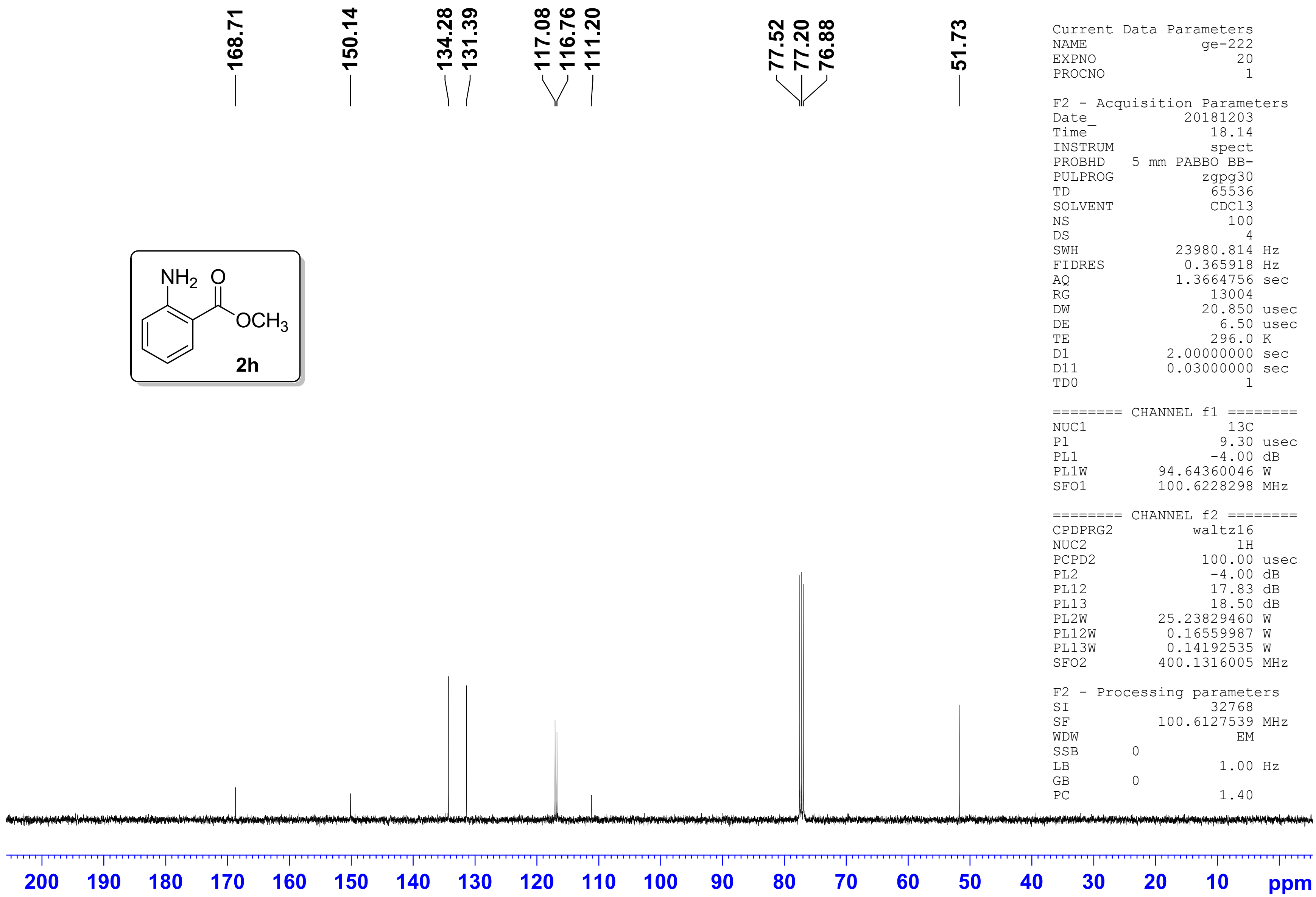

7.721
7.680
7.540
7.531
7.528
7.522
7.516
7.397
7.388
7.380
7.260
6.466
6.426
3.811
OCH3
2g
Current Data Parameters
NAME GE-223
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180725
Time 10.00
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 512
DW 60.400 usec
DE 6.50 usec
TE 298.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 8.10 usec
PL1 -4.00 dB
PL1W 25.23829460 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300081 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
1.00
2.12
3.00
1.11
0.99
2.97




```
Current Data Parameters
NAME            GE-223_2
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20181203
Time               14.36
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   100
DS                     4
SWH            23980.814 Hz
FIDRES          0.365918 Hz
AQ             1.3664756 sec
RG                 13004
DW                20.850 usec
DE                  6.50 usec
TE                 295.9 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.30 usec
PL1                -4.00 dB
PL1W         94.64360046 W
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                -4.00 dB
PL12               17.83 dB
PL13               18.50 dB
PL2W         25.23829460 W
PL12W         0.16559987 W
PL13W         0.14192535 W
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127542 MHz
WDW                   EM
SSB      0
LB                  1.00 Hz
GB       0
PC                  1.40
```

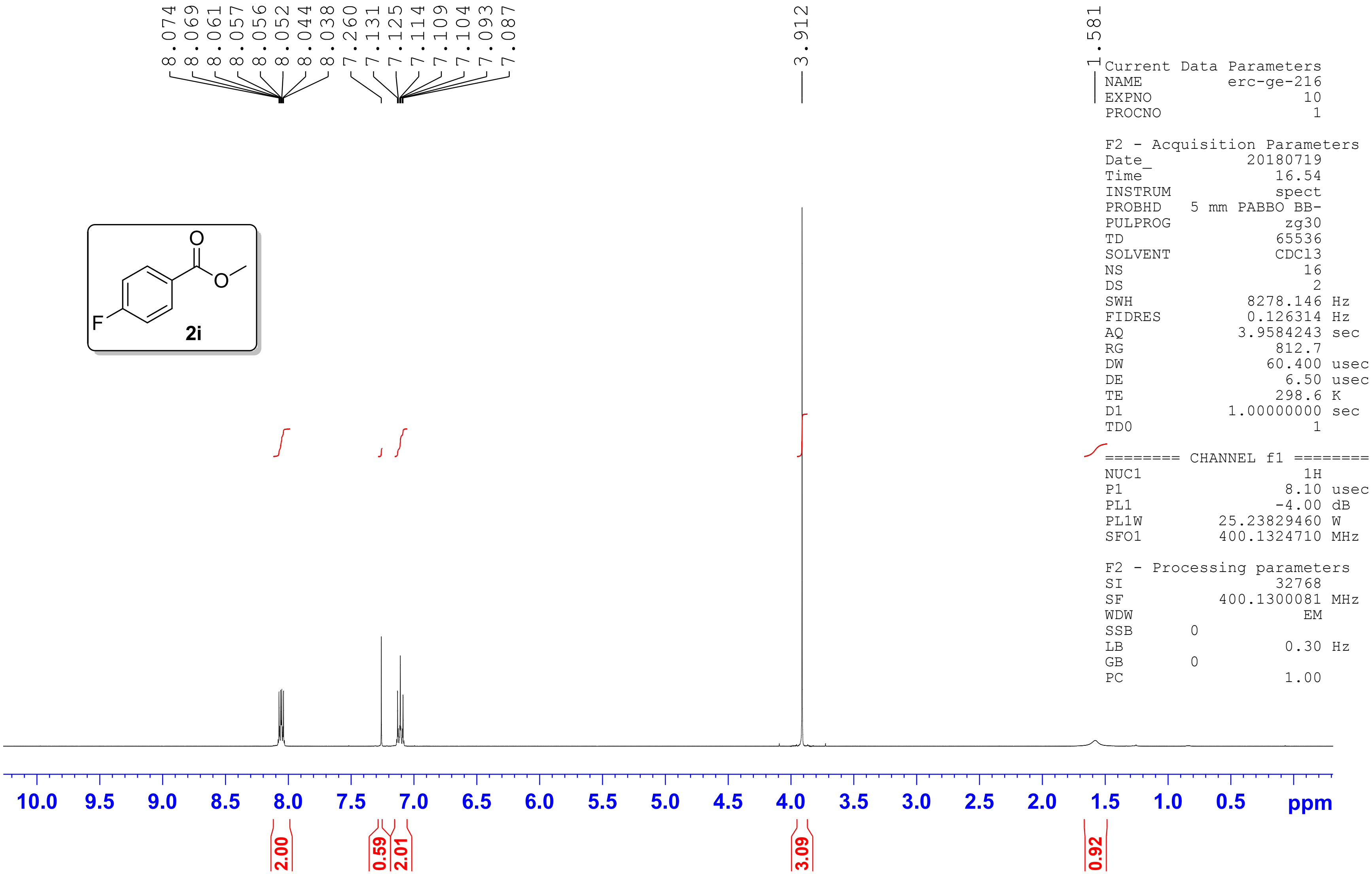


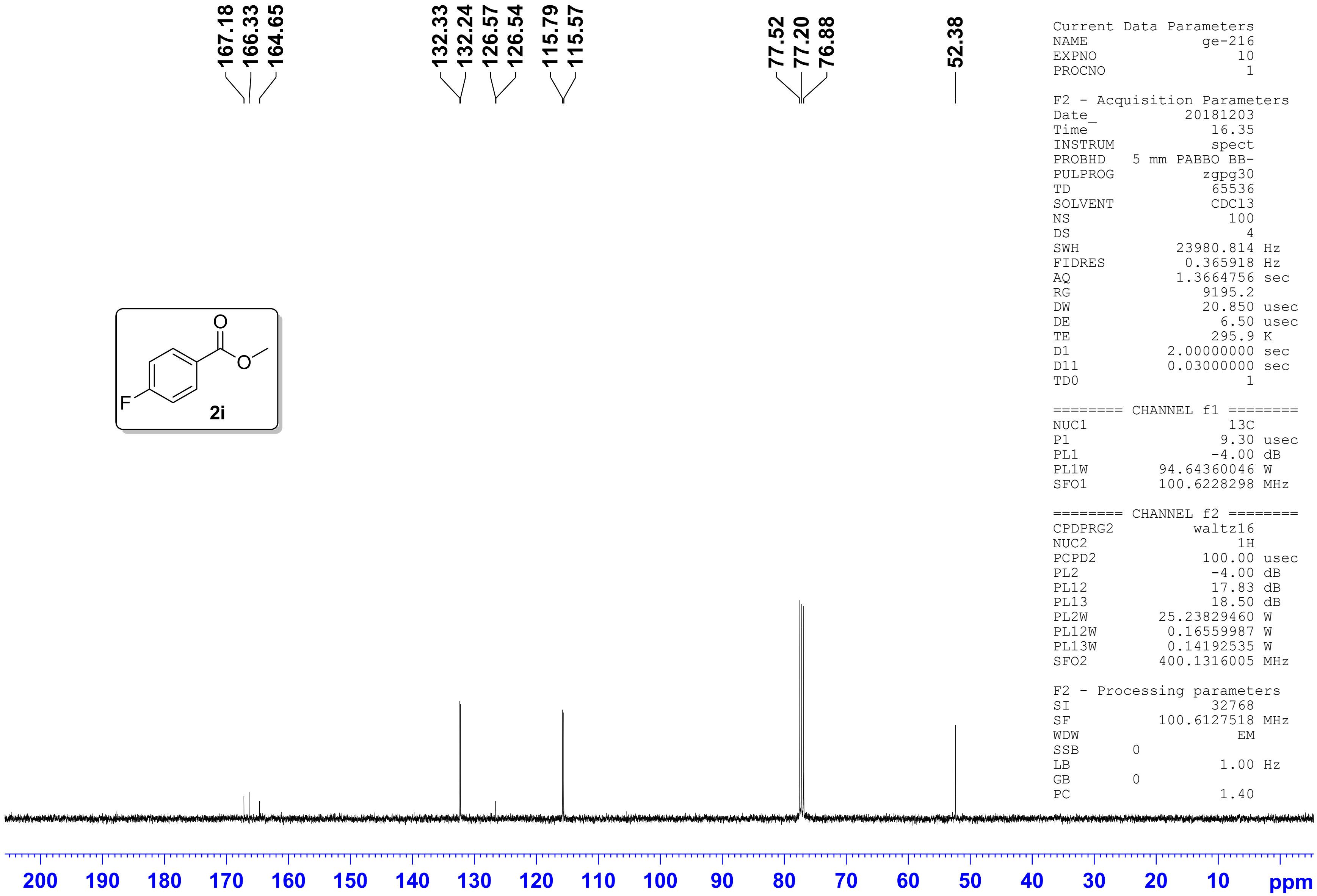
7.871 7.867 7.851 7.847 7.291 7.288 7.270 7.260 7.253 7.253 7.250 6.694 6.691 6.676 6.674 6.670 6.659 6.657 6.654 6.639 6.636 5.524
3.871
5.524
NH2 O
OCH3
2h
6.0 5.5 ppm
1.80
Current Data Parameters
NAME ge-222
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180724
Time 17.37
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 181
DW 60.400 usec
DE 6.50 usec
TE 298.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 8.10 usec
PL1 -4.00 dB
PL1W 25.23829460 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300080 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00 1.18 2.14 1.80 3.21



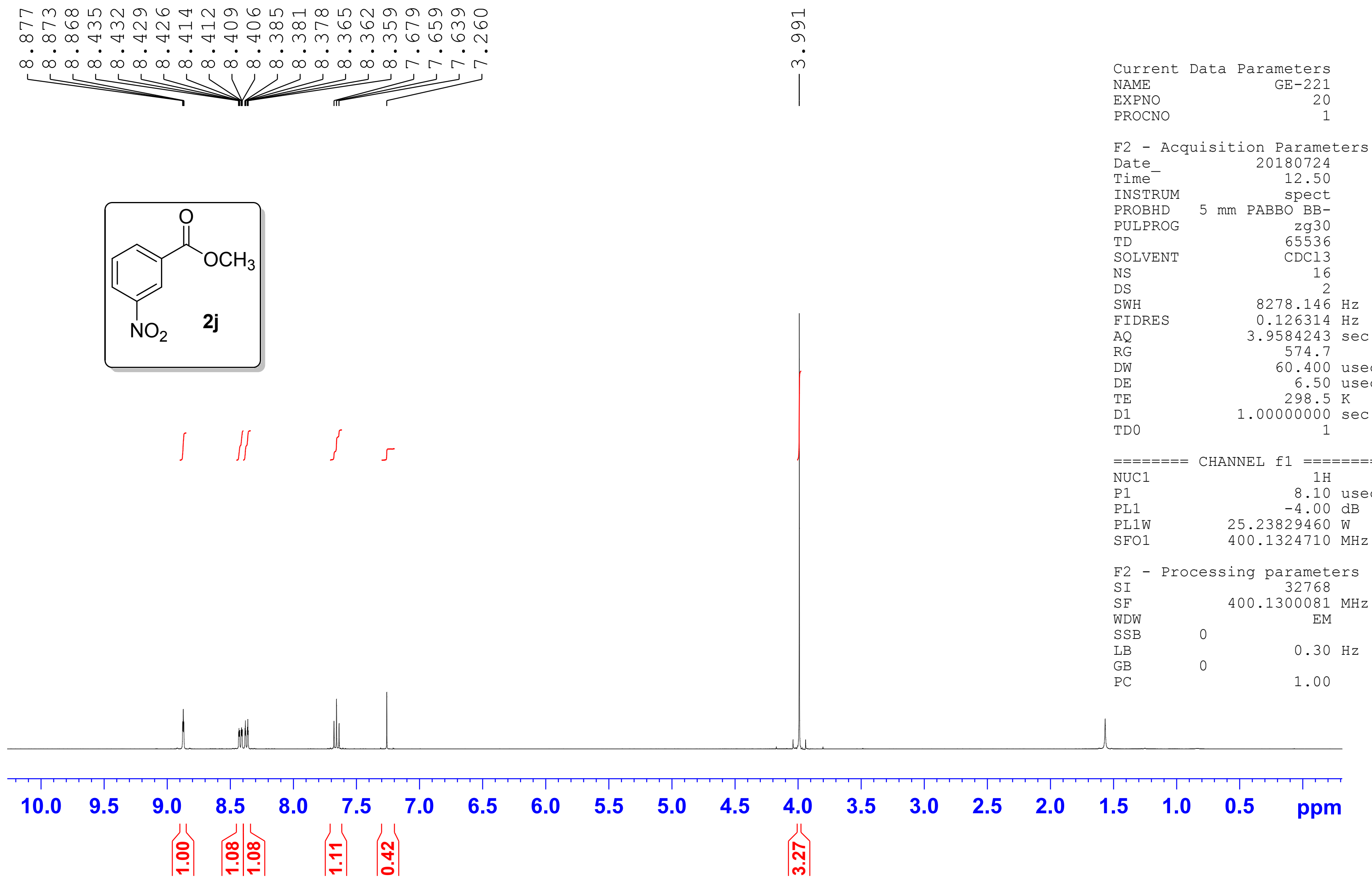
2h

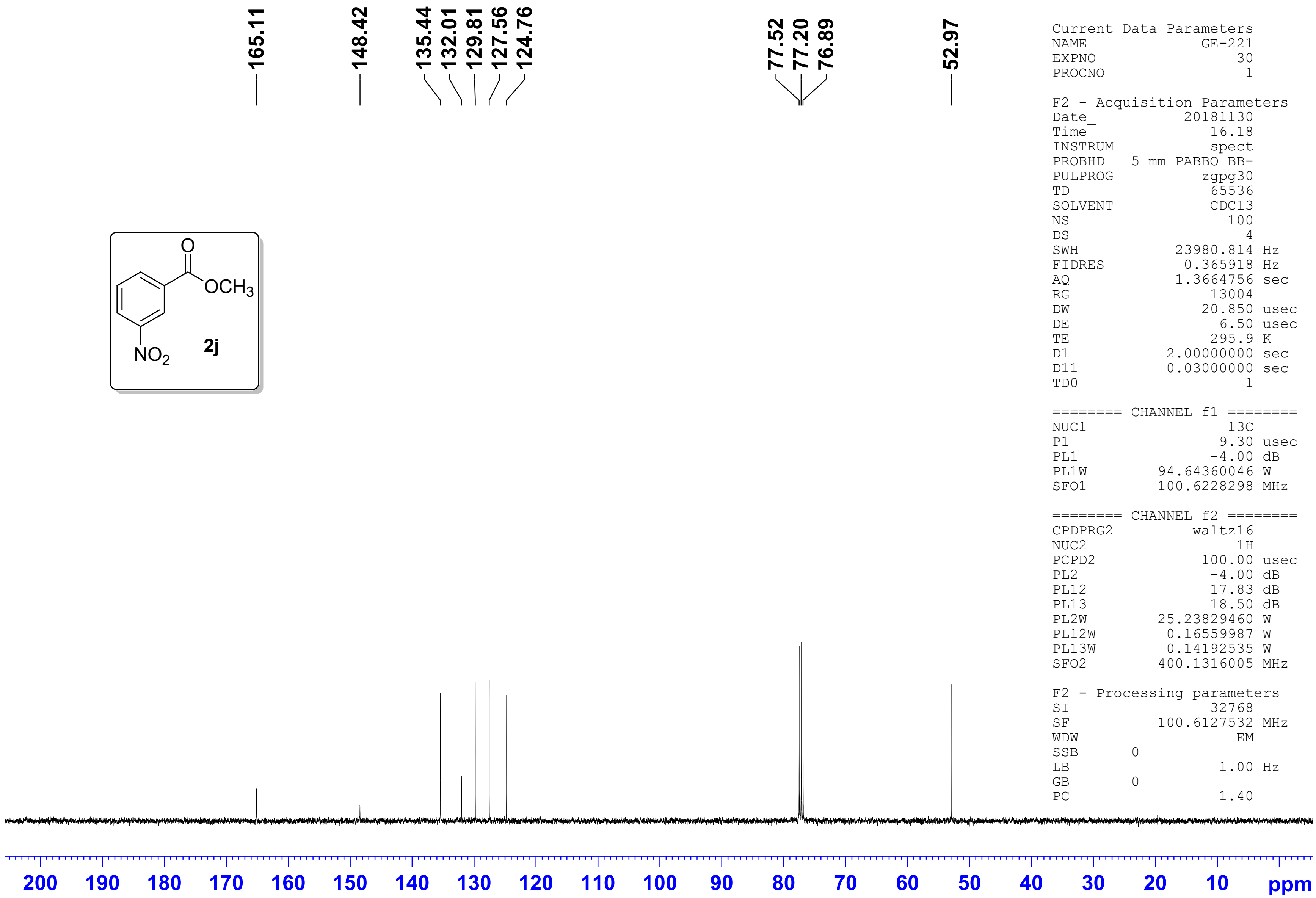
```
Current Data Parameters
NAME              ge-222
EXPNO                 20
PROCNO                 1

F2 - Acquisition Parameters
Date_           20181203
Time               18.14
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   100
DS                     4
SWH            23980.814 Hz
FIDRES          0.365918 Hz
AQ             1.3664756 sec
RG                 13004
DW                20.850 usec
DE                  6.50 usec
TE                 296.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.30 usec
PL1                -4.00 dB
PL1W         94.64360046 W
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                -4.00 dB
PL12               17.83 dB
PL13               18.50 dB
PL2W         25.23829460 W
PL12W         0.16559987 W
PL13W         0.14192535 W
SFO2         400.1316005 MHz

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


8.074
8.069
8.061
8.057
8.056
8.052
8.044
8.038
7.260
7.131
7.125
7.114
7.109
7.104
7.093
7.087
3.912
1.581
O
O
F
2i
Current Data Parameters
NAME erc-ge-216
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180719
Time 16.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 812.7
DW 60.400 usec
DE 6.50 usec
TE 298.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 8.10 usec
PL1 -4.00 dB
PL1W 25.23829460 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300081 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
2.00
0.59
2.01
3.09
0.92




```
Current Data Parameters
NAME             ge-216
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20181203
Time              16.35
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                  100
DS                    4
SWH          23980.814 Hz
FIDRES        0.365918 Hz
AQ           1.3664756 sec
RG               9195.2
DW               20.850 usec
DE                 6.50 usec
TE                295.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                 9.30 usec
PL1               -4.00 dB
PL1W        94.64360046 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2               -4.00 dB
PL12              17.83 dB
PL13              18.50 dB
PL2W        25.23829460 W
PL12W        0.16559987 W
PL13W        0.14192535 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127518 MHz
WDW                  EM
SSB      0
LB                 1.00 Hz
GB       0
PC                 1.40
```


8.877
8.873
8.868
8.435
8.432
8.429
8.426
8.414
8.412
8.409
8.406
8.385
8.381
8.378
8.365
8.362
8.359
7.679
7.659
7.639
7.260
3.991
O
OCH3
NO2
2j
Current Data Parameters
NAME GE-221
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180724
Time 12.50
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 574.7
DW 60.400 usec
DE 6.50 usec
TE 298.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 8.10 usec
PL1 -4.00 dB
PL1W 25.23829460 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300081 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
1.08
1.08
1.11
0.42
3.27


O
OCH3
NO2
2j
165.11
148.42
135.44
132.01
129.81
127.56
124.76
77.52
77.20
76.89
52.97
Current Data Parameters
NAME GE-221
EXPNO 30
PROCNO 1
F2 - Acquisition Parameters
Date_ 20181130
Time 16.18
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 100
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 13004
DW 20.850 usec
DE 6.50 usec
TE 295.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.30 usec
PL1 -4.00 dB
PL1W 94.64360046 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.83 dB
PL13 18.50 dB
PL2W 25.23829460 W
PL12W 0.16559987 W
PL13W 0.14192535 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127532 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 ppm