Supporting Information

Highly Efficient One-Pot Tandem Friedlander Annulation and Chemo-selective C_{sp3}-H functionalization under Calcium Catalysis

Garima Singh and Srinivasarao Yaragorla[*a]

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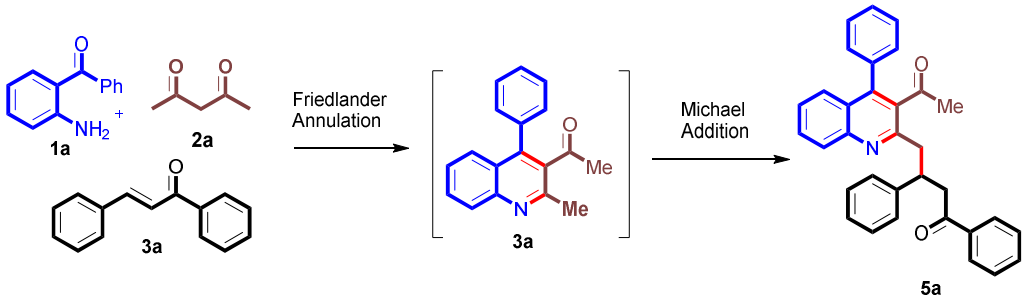
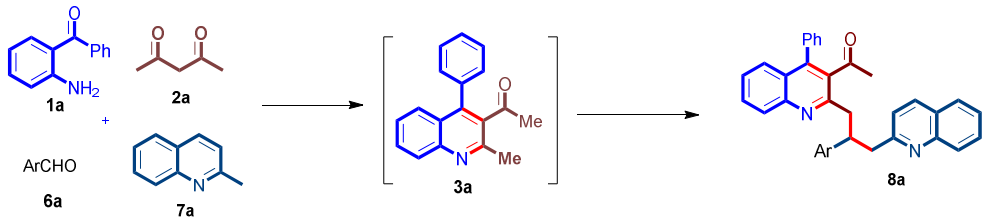
1. Optimization table for the synthesis of **5a**



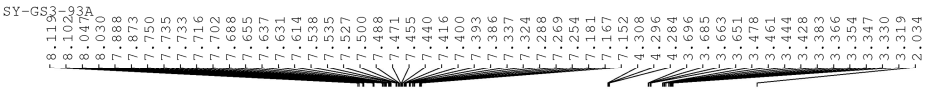
S.No	Catalyst (mol %)	Reaction conditions	Yield %(3a)	Reaction conditions	Yield %(5a)
1	Ca $(OTf)_2$ (10) + Bu_4NPF_6 (10)	Water,6 h,110 °C	61	Water,15 h,110 °C	43
2	Ca $(OTf)_2$ (10) + Bu_4NPF_6 (10)	Toluene,6 h, 120 °C	73	Toluene,10 h, 120 °C	65
3	Ca $(OTf)_2$ (10) + Bu_4NPF_6 (10)	DCE,24 h, 80 °C	45	-	-
4	**Ca$(OTf)_2$ (10) + Bu_4NPF_6 (10)**	**Neat,5, 120 °C**	**97**	**Neat,18 h, 120** °C	**72**
5	Ca $(OTf)_2$ (10) + Bu_4NPF_6 (10)	Neat,9 h,100 °C	80	Neat,23 h,100 °C	70
6	Ca $(OTf)_2$ (5) + Bu_4NPF_6 (5)	Neat,11 h,120 °C	93	Neat,20 h,120 °C	60
7	Ca $(OTf)_2$ (5) + Bu_4NPF_6 (10)	Neat,10 h,120 °C	95	Neat,20 h,120 °C	67

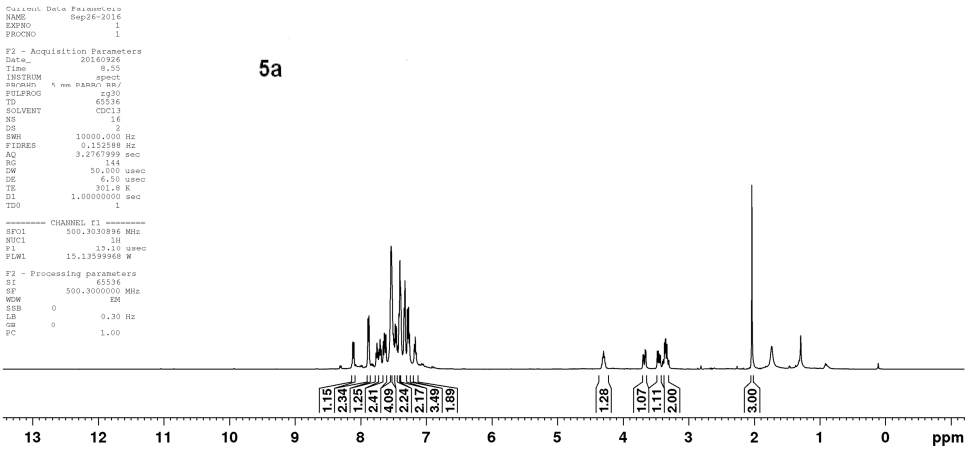
2. Optimization table for the synthesis of **8a**

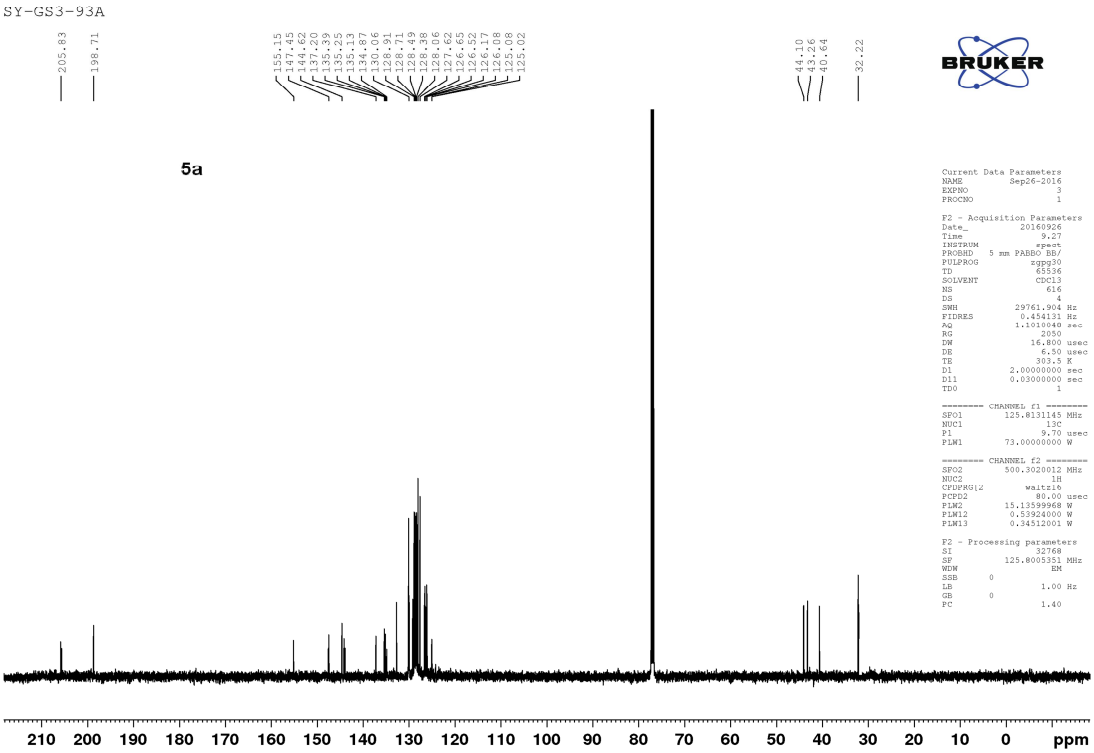


S.No	Catalyst (mol %)	Reaction conditions	Yield %(3a)	Reaction conditions	Yield(%) (8a)
1.	Ca$(OTf)_2$ (10) + Bu_4NPF_6 (10)	Neat,5 h, 120 °C	98	Water,15 h,110 °C	72
2.	Ca$(OTf)_2$ (10) + Bu_4NPF_6 (10)	Neat,5 h, 120 °C	98	Toluene,12 h, 120°C	80
3	**Ca$(OTf)_2$ (10) + Bu_4NPF_6 (10)**	**Neat,5 h, 120 °C**	**98**	**Neat,8 h, 120 °C**	**80**
4	Ca$(OTf)_2$ (10) + Bu_4NPF_6 (10)	Neat,5 h, 120 °C	98	Neat,12 h,100 °C	78

3. Spectral copies Spectra







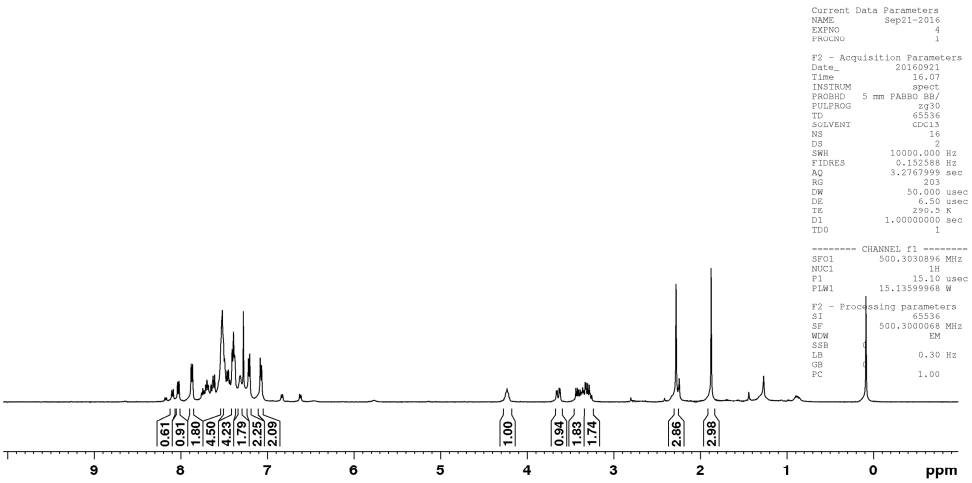
SY-GS3-91

8.134 8.038 8.037 8.020 7.830 7.855 7.531 7.527 7.520 7.407 7.392 7.378 7.319 7.313 7.219 7.203 7.032 7.056

4.242 4.231 3.652 3.651 3.629 3.618 3.437 3.419 3.403 3.386 3.371 3.358 3.346 3.329 3.317 3.300 3.232 3.272 3.253 2.279 1.873

BRUKER

5b



SY-GS3-91

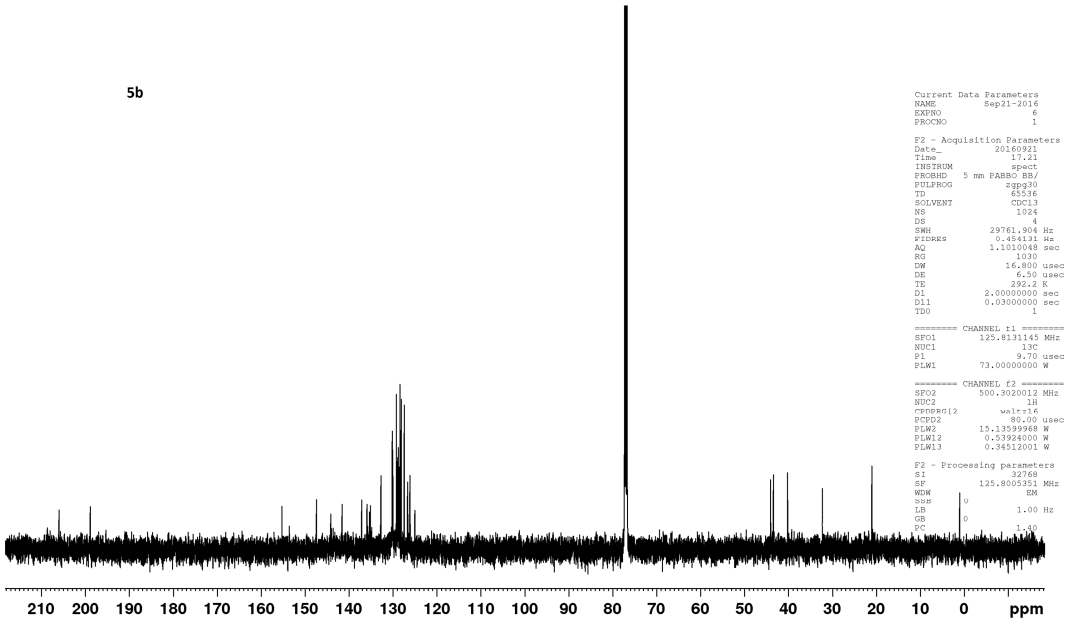
205.99 198.86

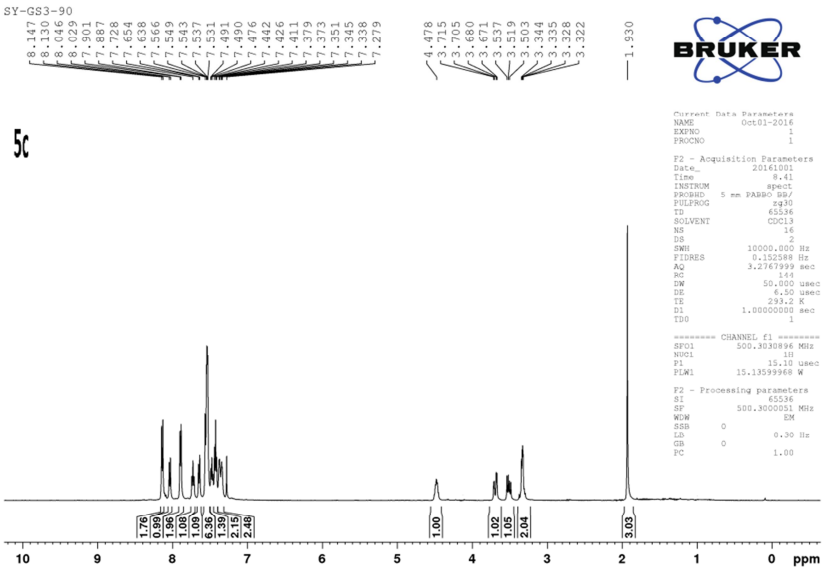
155.25 147.43 144.17 141.56 137.10 135.94 135.55 135.32 132.76 130.18 130.11 130.06 129.22 129.22 129.17 128.92 128.88 128.76 128.59 128.39 128.11 127.44 126.68 126.58 126.21 126.12 125.01

44.11 43.43 40.22 32.27 23.94 21.04

BRUKER

5b

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5c
BRUKER
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EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161001
Time 8.41
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
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FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 144
DW 50.000 usec
DE 6.50 usec
TE 293.2 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 500.3000051 MHz
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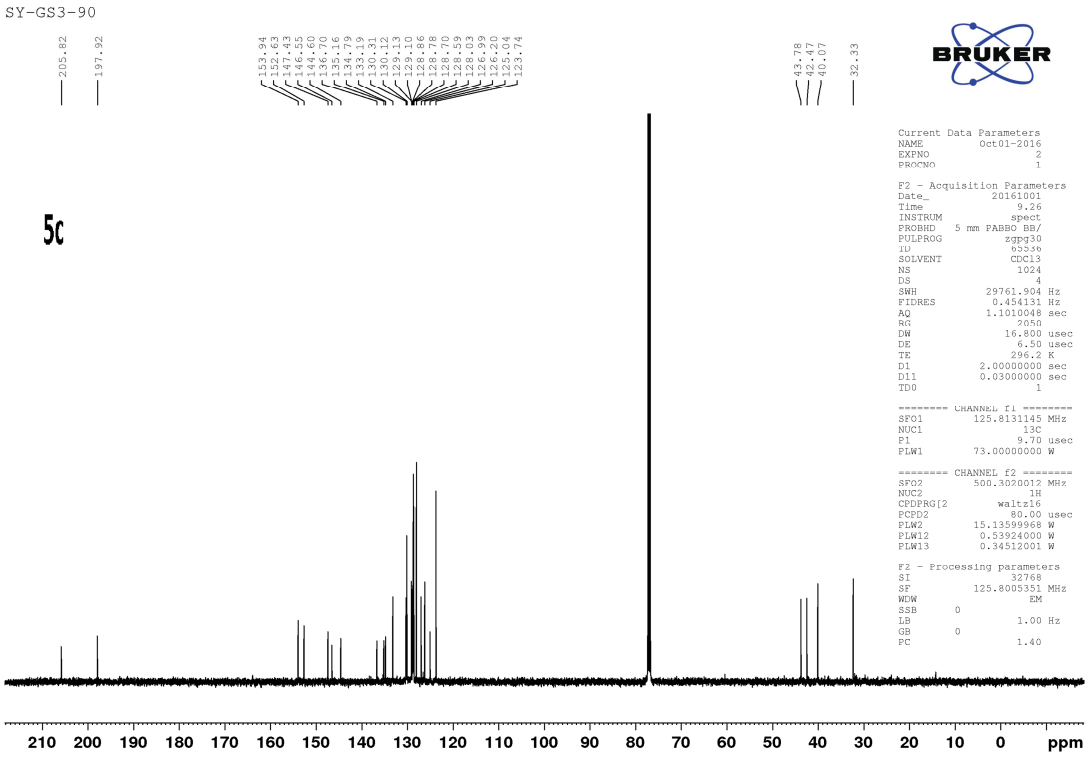
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm


SY-GS3-90
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PROCNO 1
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PULPROG zgpg30
TD 65536
SOLVENT CDCl3
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 296.2 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
ppm

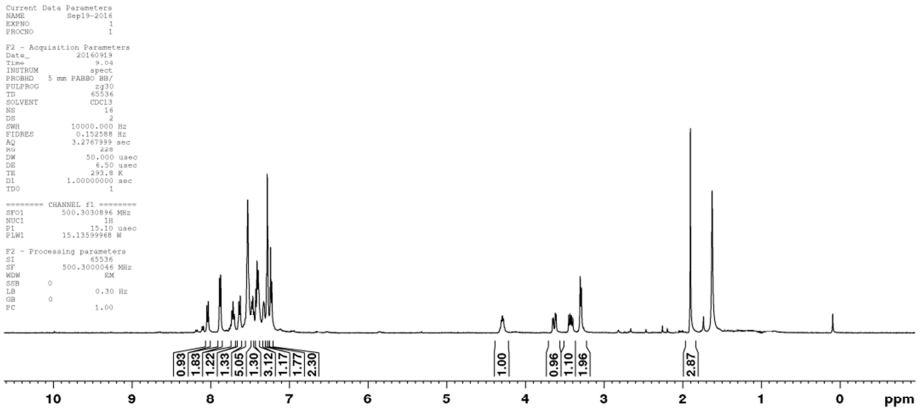
SY-GS3-86A

8.047 8.031 7.888 7.873 7.730 7.716 7.701 7.640 7.624 7.530 7.478 7.464 7.427 7.411 7.396 7.326 7.319 7.290 7.280 7.274 7.239 7.222

4.303 4.291 4.278 3.651 3.641 3.618 3.607 3.301 3.288

1.899

5d



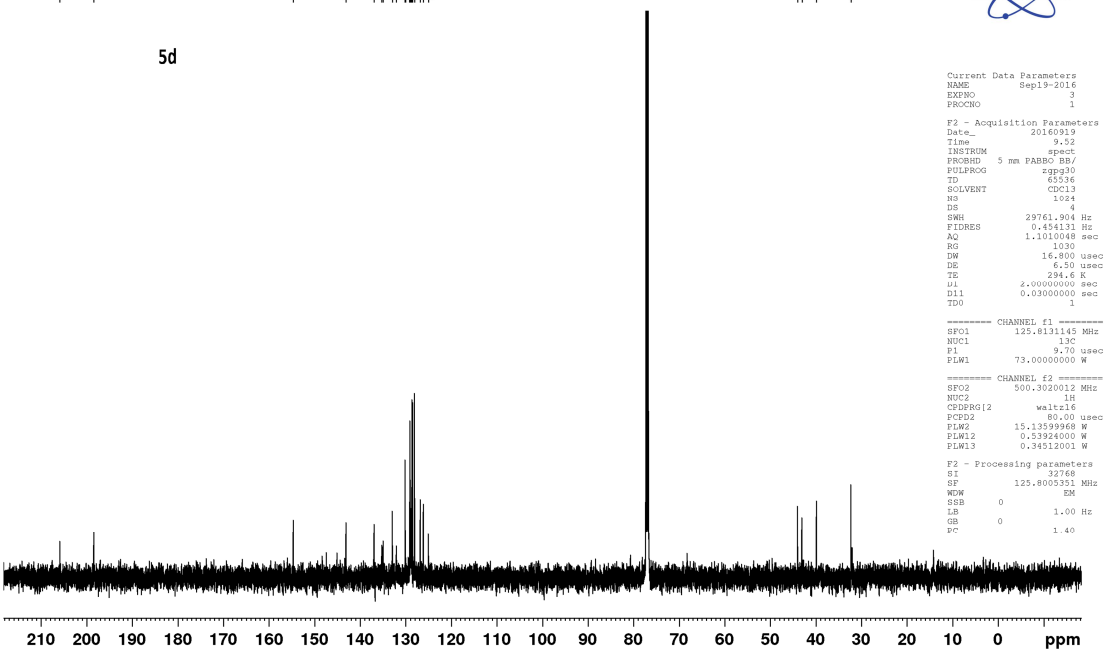
SY-GS3-86A

205.89 198.46

154.68 143.12 136.96 135.27 133.93 132.05 130.15 130.06 129.16 129.05 128.89 128.85 128.75 128.67 128.60 128.48 128.06 126.81 126.15 125.02

44.61 43.05 39.87 32.30

5d

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PROCNO                     1

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Time                   9.52
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PROBHD   5 mm PABBO BB/
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
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                 294.6 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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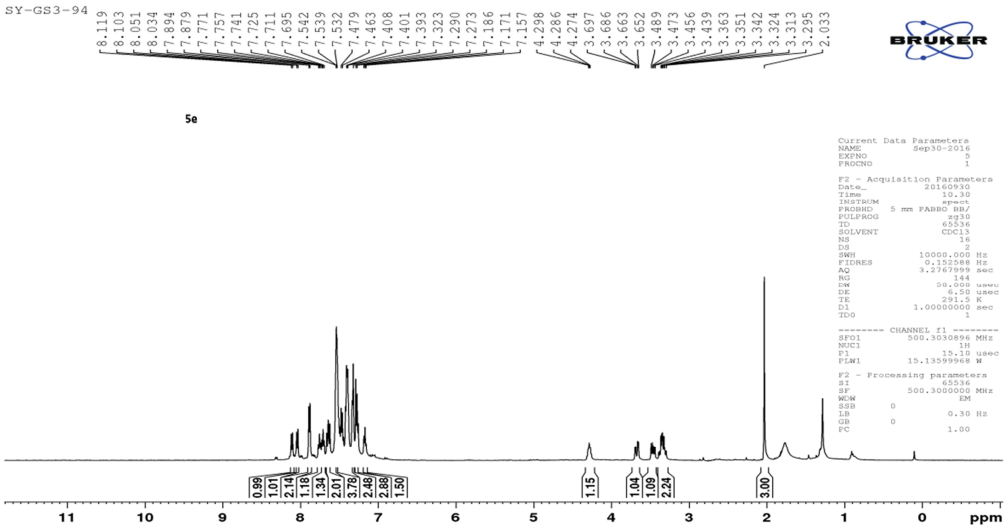
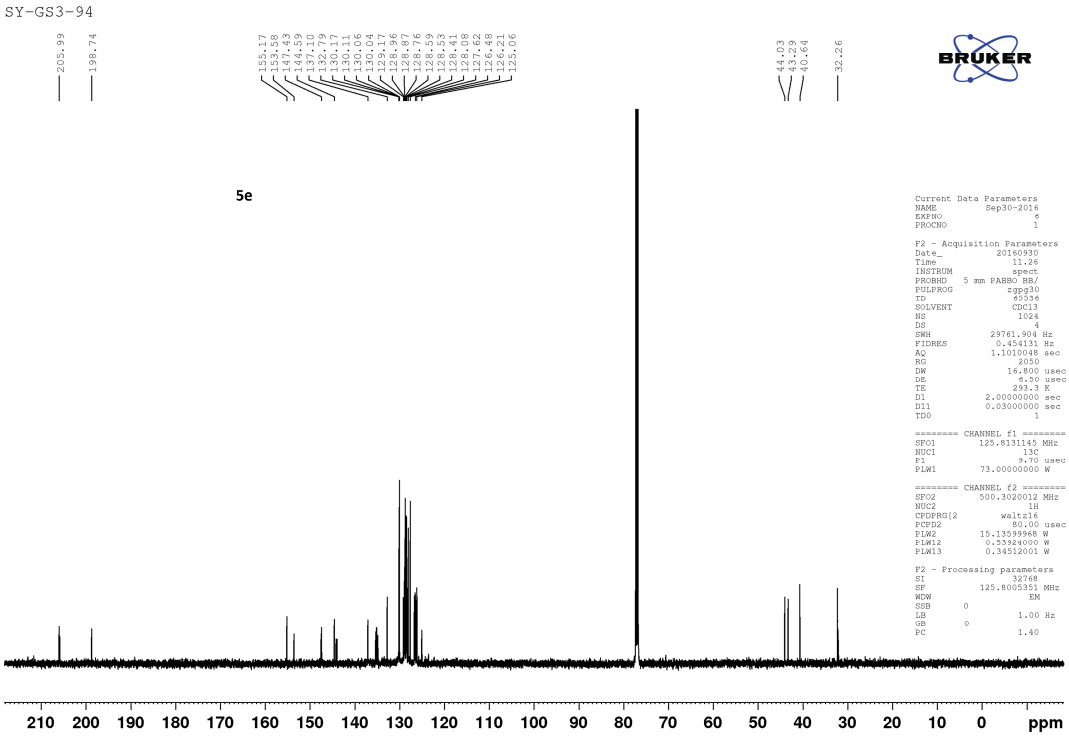

SY-GS3-94
5e
ppm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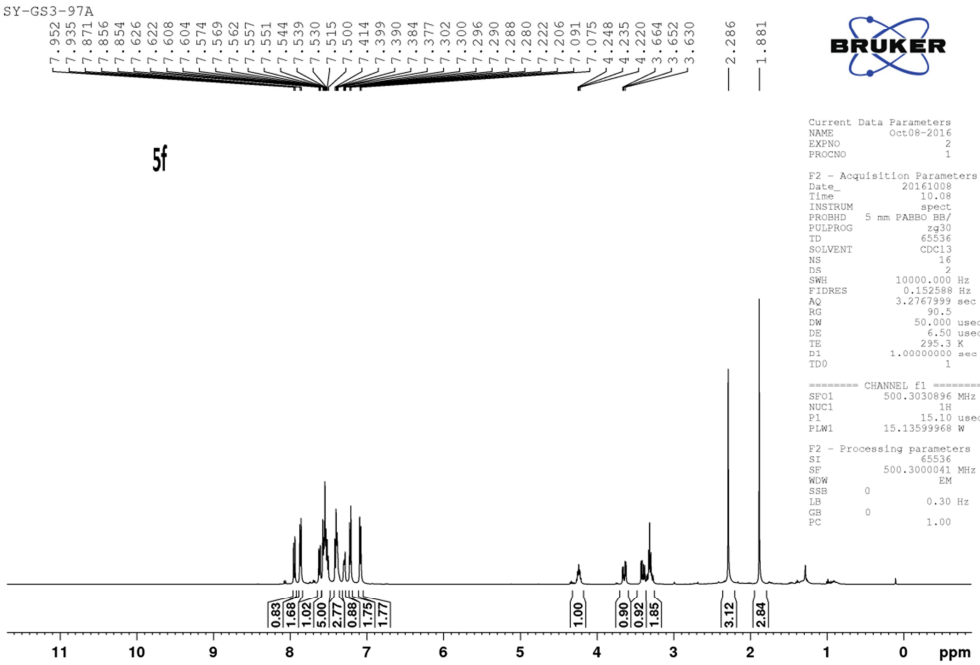
5e
ppm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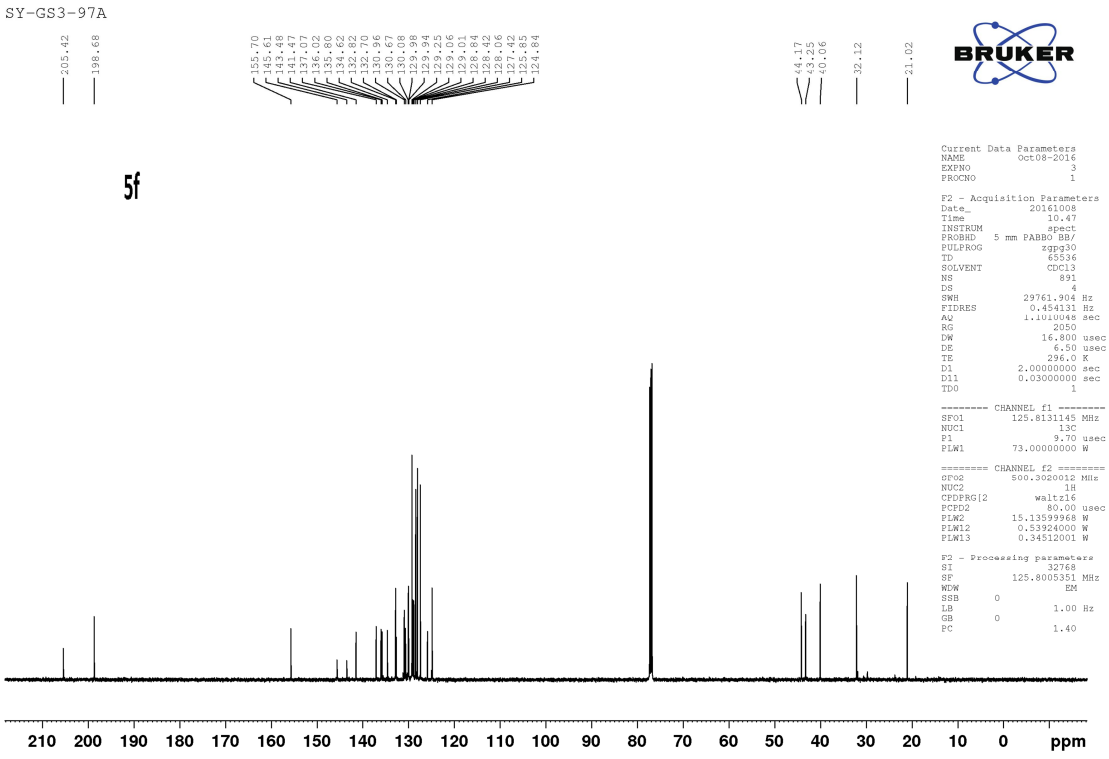
7.871
7.856
7.854
7.626
7.622
7.608
7.604
7.574
7.569
7.562
7.557
7.551
7.544
7.539
7.530
7.515
7.500
7.414
7.399
7.390
7.384
7.377
7.302
7.300
7.296
7.290
7.288
7.280
7.222
7.206
7.091
7.075
4.248
4.235
4.220
3.664
3.652
3.630
2.286
1.881
BRUKER
5f
Current Data Parameters
NAME Oct08-2016
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161008
Time 10.08
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 90.5
DW 50.000 usec
DE 6.50 usec
TE 295.3 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 500.3000041 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
0.83
1.68
1.02
5.00
2.77
0.88
1.75
1.77
1.00
0.90
0.92
1.85
3.12
2.84
11
10
9
8
7
6
5
4
3
2
1
0
ppm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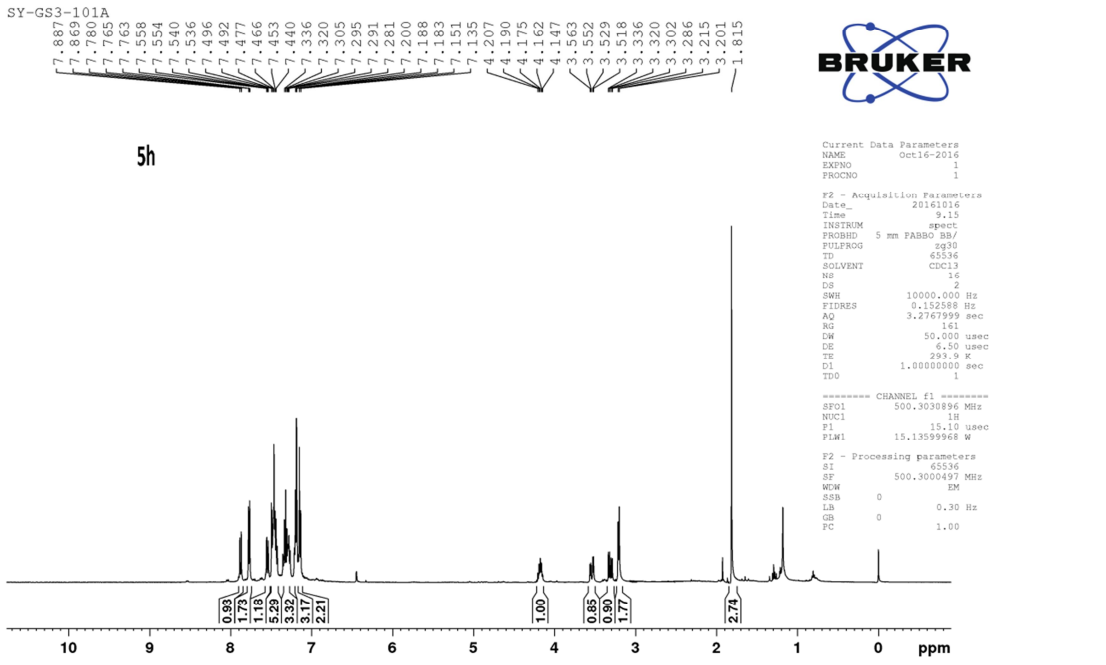
198.68
155.70
145.61
143.48
141.47
137.07
136.02
135.80
134.62
132.82
132.70
130.96
130.67
130.08
129.98
129.94
129.25
129.06
129.01
128.84
128.42
128.06
127.42
125.85
124.84
44.17
43.25
40.06
32.12
21.02
BRUKER
5f
Current Data Parameters
NAME Oct08-2016
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161008
Time 10.47
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 891
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 296.0 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 125.8005351 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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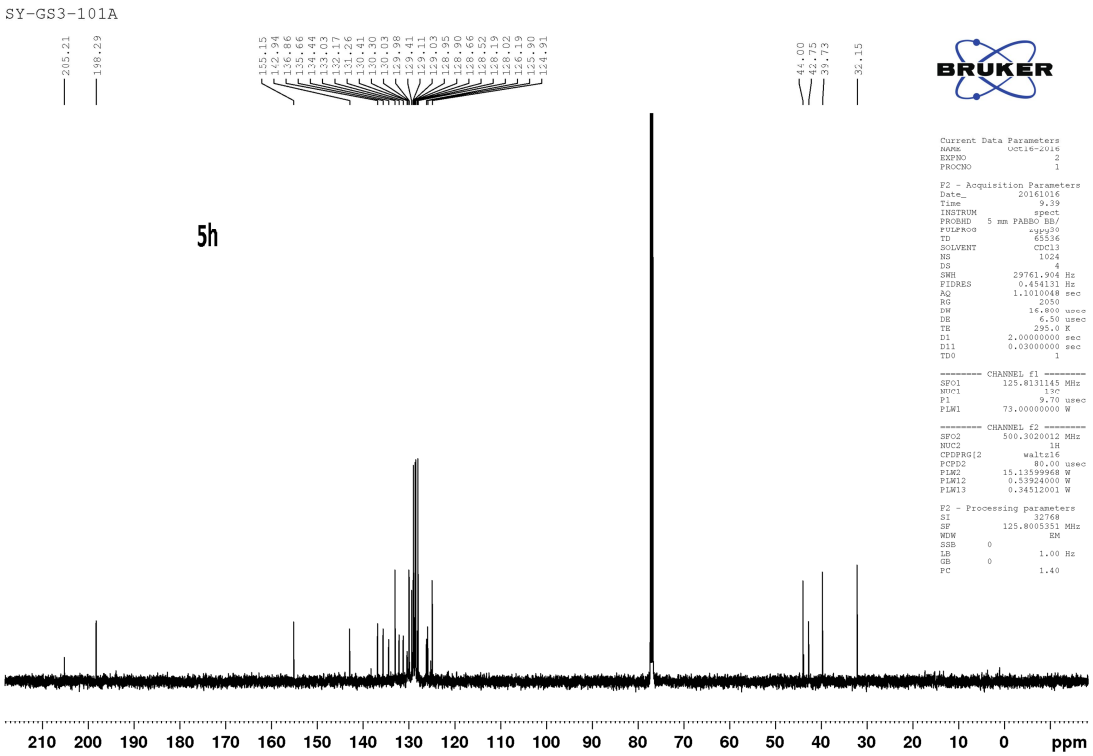
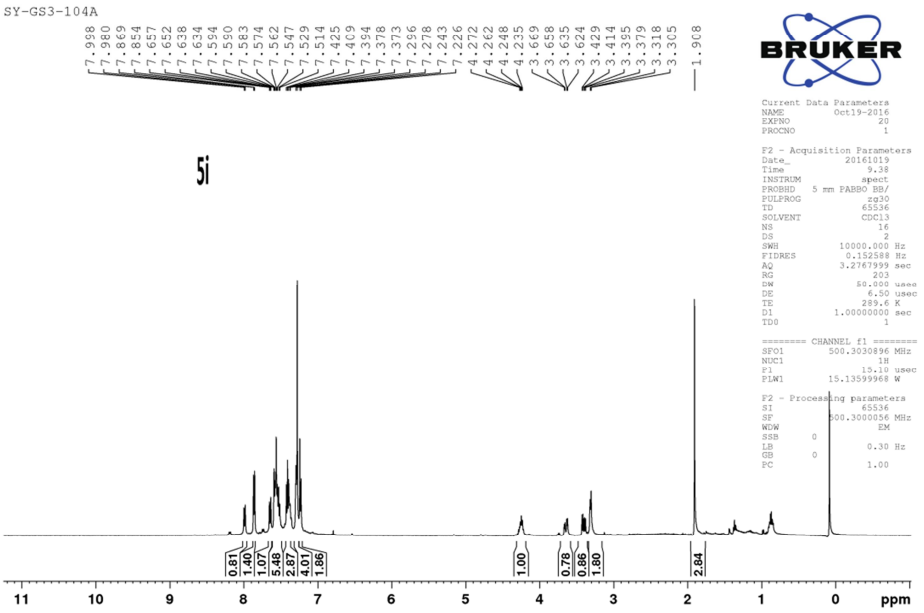
7.869
7.780
7.765
7.763
7.558
7.554
7.540
7.536
7.496
7.492
7.477
7.466
7.453
7.440
7.336
7.320
7.305
7.295
7.291
7.281
7.200
7.188
7.183
7.151
7.135
4.207
4.190
4.175
4.162
4.147
3.563
3.552
3.529
3.518
3.336
3.320
3.302
3.286
3.215
3.201
1.815
BRUKER
5h
Current Data Parameters
NAME Oct16-2016
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161016
Time 9.15
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 161
DW 50.000 usec
DE 6.50 usec
TE 293.9 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 500.3000497 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
0.93
1.75
1.18
5.29
3.32
3.17
2.21
1.00
0.85
0.90
1.77
2.74
10
9
8
7
6
5
4
3
2
1
0
ppm


SY-GS3-101A
205.21
198.29
155.15
142.94
136.86
135.66
134.44
133.03
132.17
131.26
130.41
130.30
130.03
129.98
129.41
129.11
129.03
128.95
128.90
128.66
128.52
128.19
128.02
126.19
125.90
124.91
44.00
42.75
39.73
30.15
BRUKER
5h
Current Data Parameters
NAME Oct16-2016
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161016
Time 9.39
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG cpyg30
TD 65536
SOLVENT CDCl3
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 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 125.8005351 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


SY-GS3-104A
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7.980
7.869
7.854
7.657
7.652
7.638
7.634
7.594
7.590
7.583
7.574
7.562
7.547
7.529
7.514
7.425
7.409
7.394
7.378
7.373
7.296
7.278
7.243
7.226
4.272
4.262
4.248
4.235
3.669
3.658
3.635
3.624
3.429
3.414
3.395
3.379
3.318
3.305
1.908
5i
BRUKER
Current Data Parameters
NAME Oct19-2016
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161019
Time 9.38
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 203
DW 50.000 usec
DE 6.50 usec
TE 289.6 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 500.3000056 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
0.81
1.40
1.07
5.48
2.87
4.01
1.86
1.00
0.78
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1.80
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10
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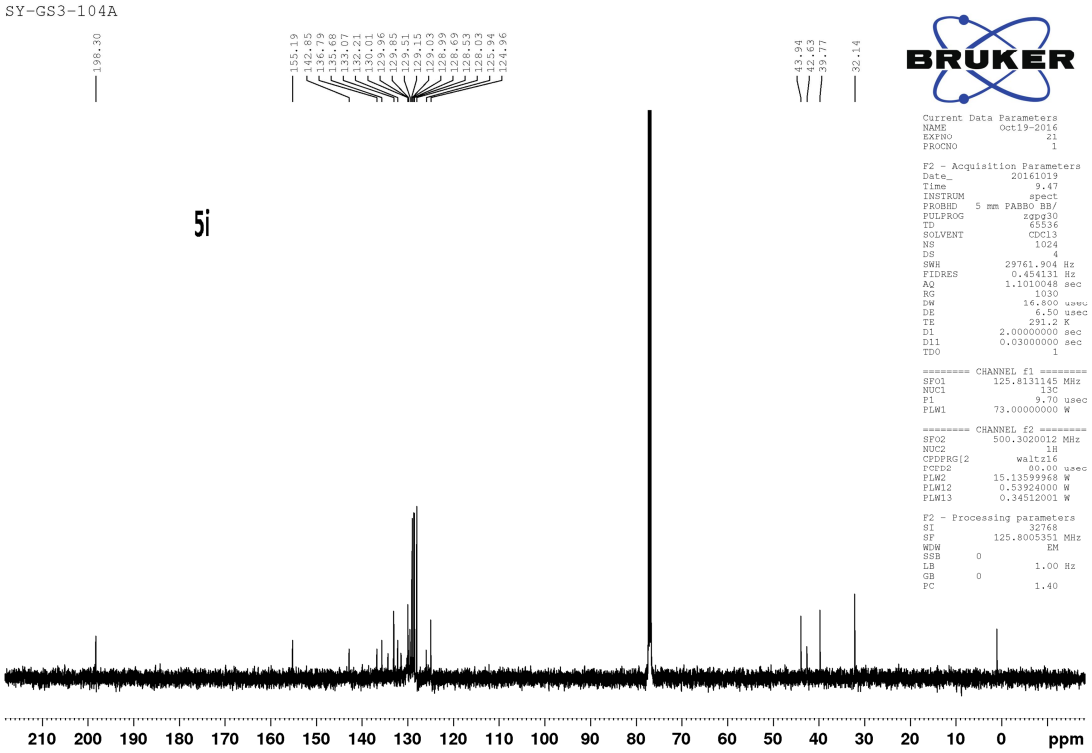
8
7
6
5
4
3
2
1
0
ppm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155.19
142.85
136.79
135.68
133.07
132.21
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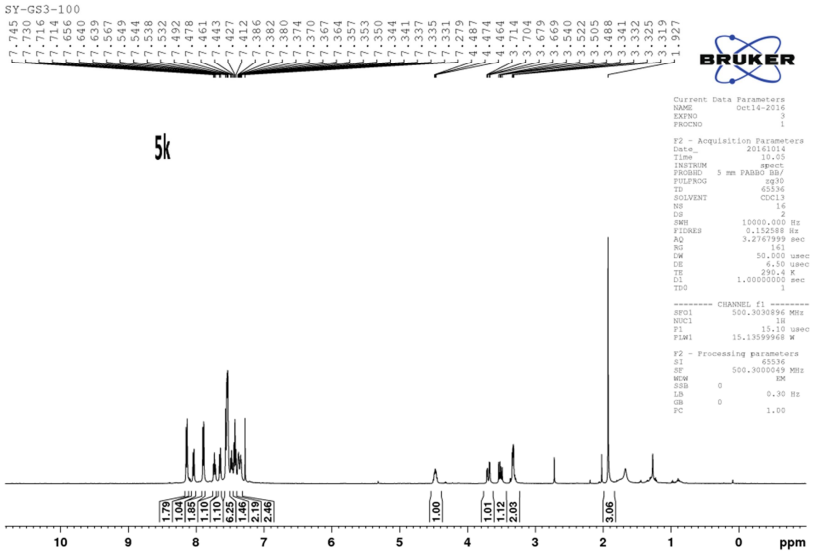
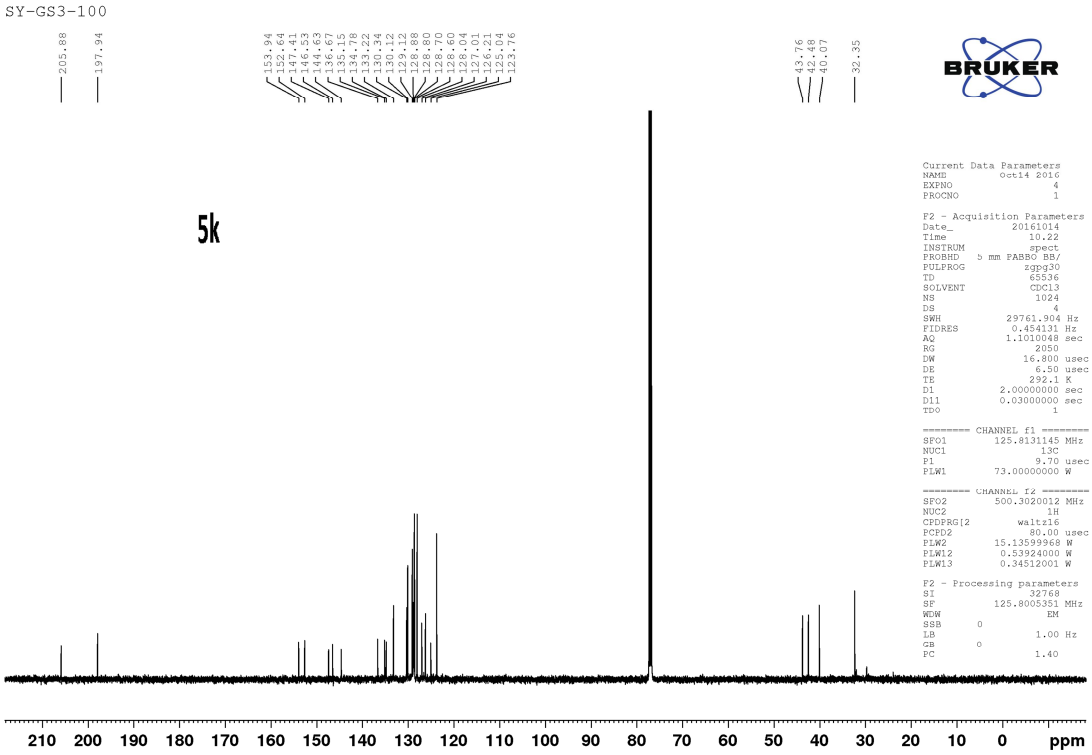
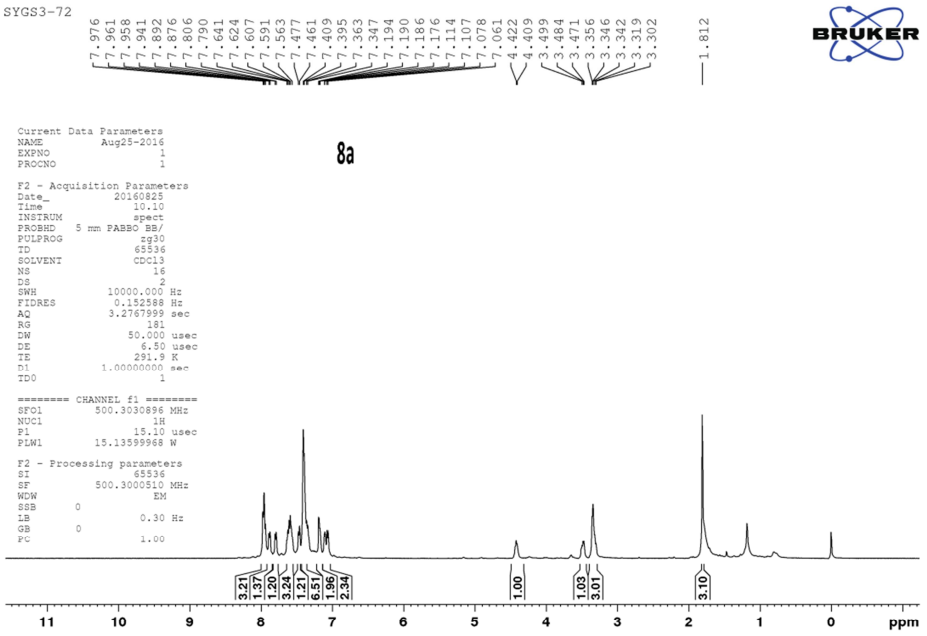
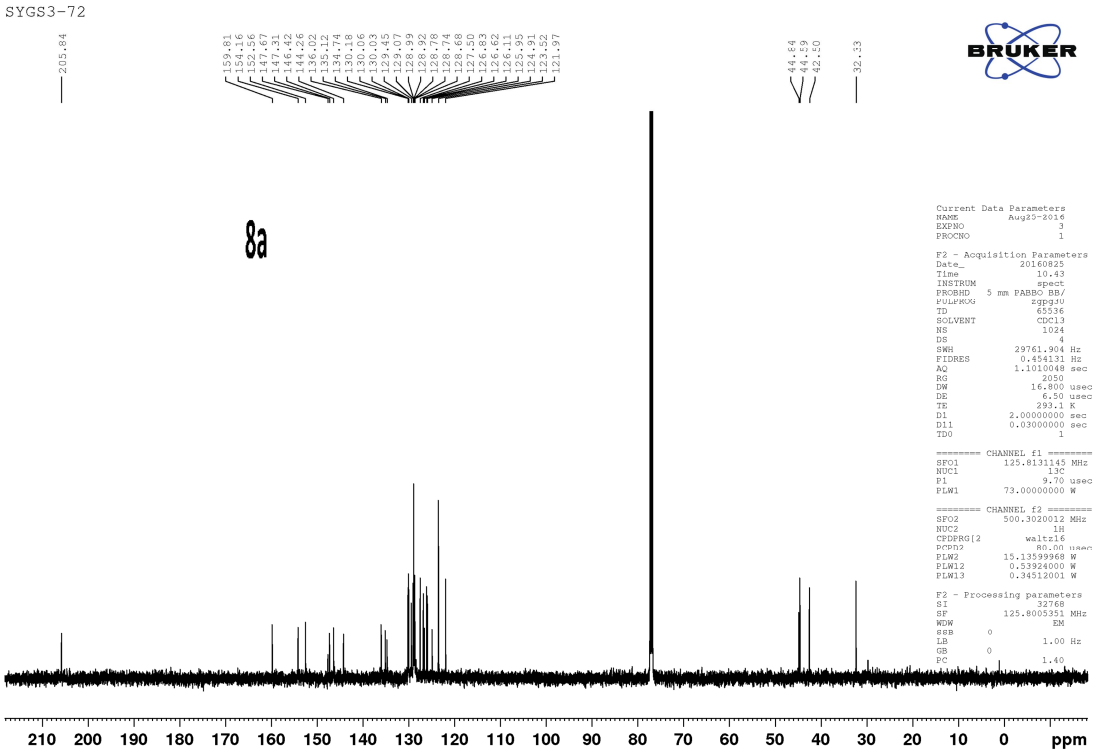
129.85
129.51
129.15
129.03
128.99
128.69
128.53
128.03
125.94
124.96
43.94
42.63
39.77
32.14
5i
BRUKER
Current Data Parameters
NAME Oct19-2016
EXPNO 21
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161019
Time 9.47
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 1030
DW 16.800 usec
DE 6.50 usec
TE 291.2 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
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

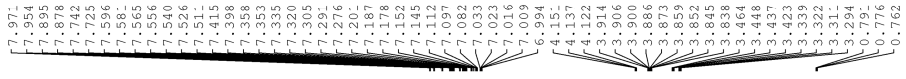

SY-GS3-100
5k
BRUKER
ppm


SY-GS3-100
5k
BRUKER
ppm


SYGS3-72
7.976
7.961
7.958
7.941
7.892
7.876
7.806
7.790
7.641
7.624
7.607
7.591
7.563
7.477
7.461
7.409
7.395
7.363
7.347
7.194
7.190
7.186
7.176
7.114
7.107
7.078
7.061
4.422
4.409
3.499
3.484
3.471
3.356
3.346
3.342
3.319
3.302
1.812
BRUKER
8a
Current Data Parameters
NAME Aug25-2016
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160825
Time 10.10
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 181
DW 50.000 usec
DE 6.50 usec
TE 291.9 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 500.3000510 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
3.21
1.37
1.20
3.24
1.21
6.51
1.96
2.34
1.00
1.03
3.01
3.10
11
10
9
8
7
6
5
4
3
2
1
0
ppm

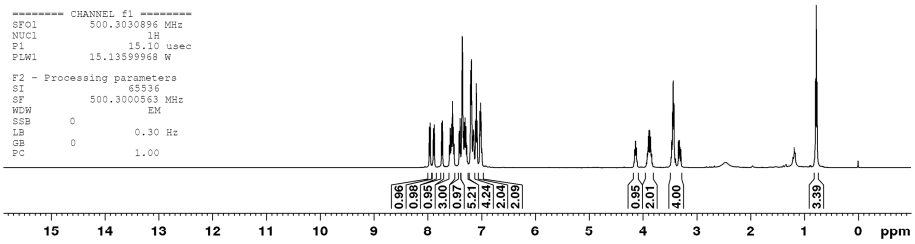

SYGS3-72
205.84
159.81
154.16
152.56
147.67
147.31
146.42
144.26
136.02
135.12
134.74
130.18
130.06
130.03
129.45
128.97
128.89
128.92
128.78
128.74
128.68
127.50
126.83
126.62
126.11
125.95
124.91
123.52
121.97
44.64
44.59
42.10
32.33
BRUKER
8a
Current Data Parameters
NAME Aug25-2016
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160825
Time 10.43
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg2U
TD 65536
SOLVENT CDCl3
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 293.1 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 125.8005351 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

SY-GS3-58

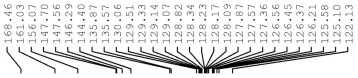


8b

```
Current Data Parameters
NAME          Jul22-2016
EXPNO                 50
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160722
Time              17.48
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                90.5
DW              50.000 usec
DE               6.50 usec
TE              290.5 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            500.3000563 MHz
WDW                 EM
SSB       0
LB              0.30 Hz
GB        0
PC              1.00
```

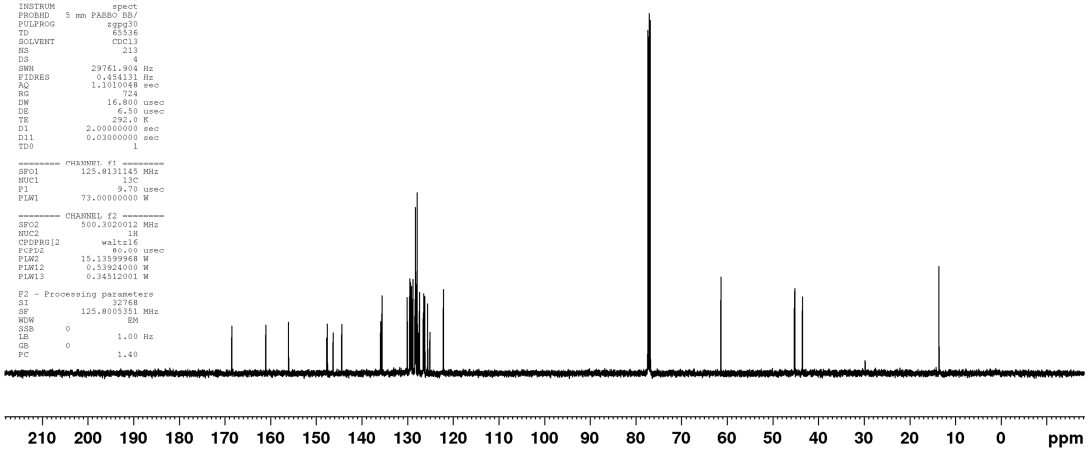


SY-GS3-58

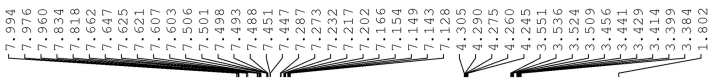


8b

```
Current Data Parameters
NAME          Jul22-2016
EXPNO                 51
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160722
Time              17.59
INSTRUM           spect
PROBHD  5 mm PABBO BB/
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                 213
DS                   4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ             1.10100048 sec
RG                 724
DW              16.800 usec
DE               6.50 usec
TE              292.0 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            125.8005351 MHz
WDW                 EM
SSB       0
LB              1.00 Hz
GB        0
PC              1.40
```



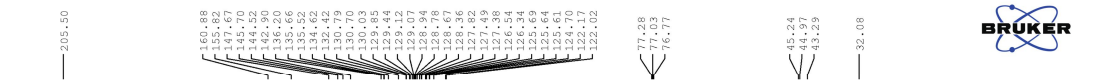
SY-GS3-58A-1



8c

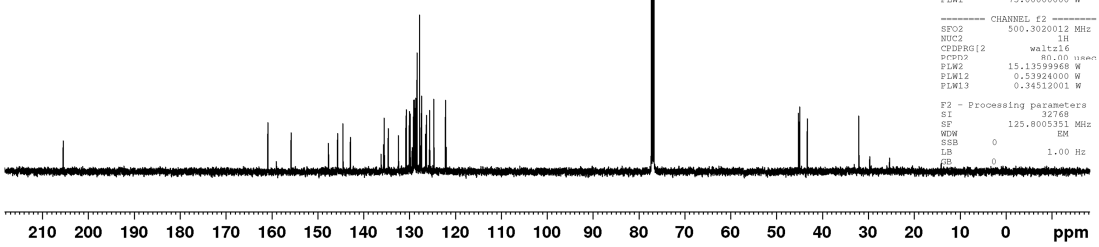
```
Current Data Parameters
NAME         Jul22-2016
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20160722
Time                9.29
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                  181
DW                50.000 usec
DE                 6.50 usec
TE                298.1 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
```


SY-GS3-58A-1

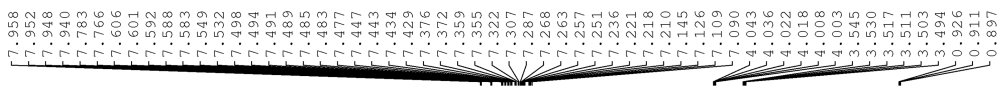


8c

```
Current Data Parameters
NAME         Jul22-2016
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_           20160722
Time                9.47
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  772
DS                    4
SWH           29761.904 Hz
FIDRES            0.454131 Hz
AQ             1.1010048 sec
RG                  912
DW                16.800 usec
DE                 6.50 usec
TE                299.5 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           125.8005351 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
```

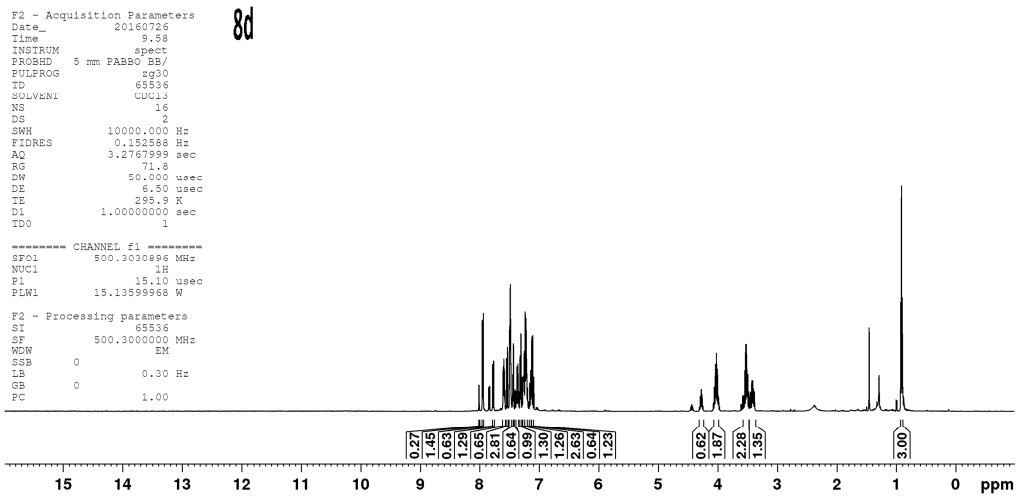


SY-GS3-60

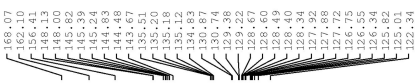



```
Current Data Parameters
NAME          Jul26-2016
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160726
Time               9.58
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                 71.8
DW                50.000 usec
DE                 6.50 usec
TE                295.9 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            500.3000000 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

8d

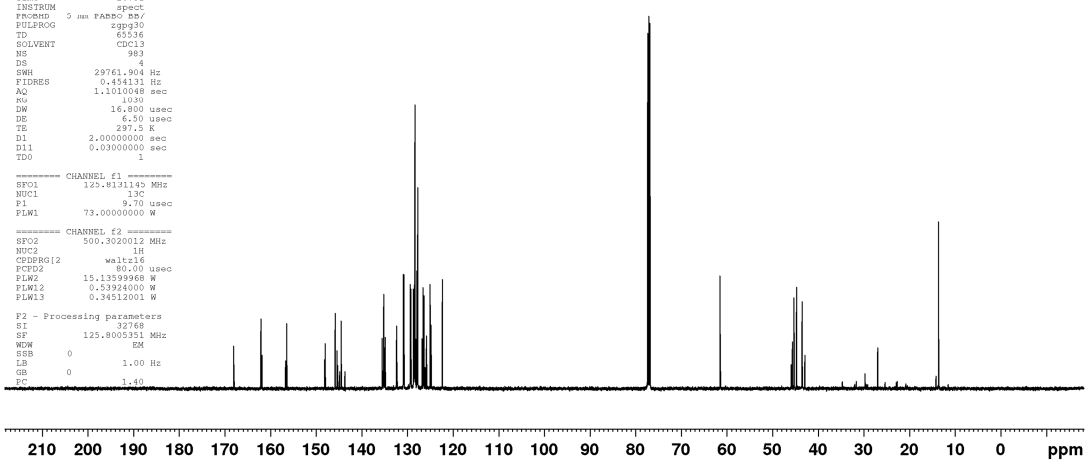


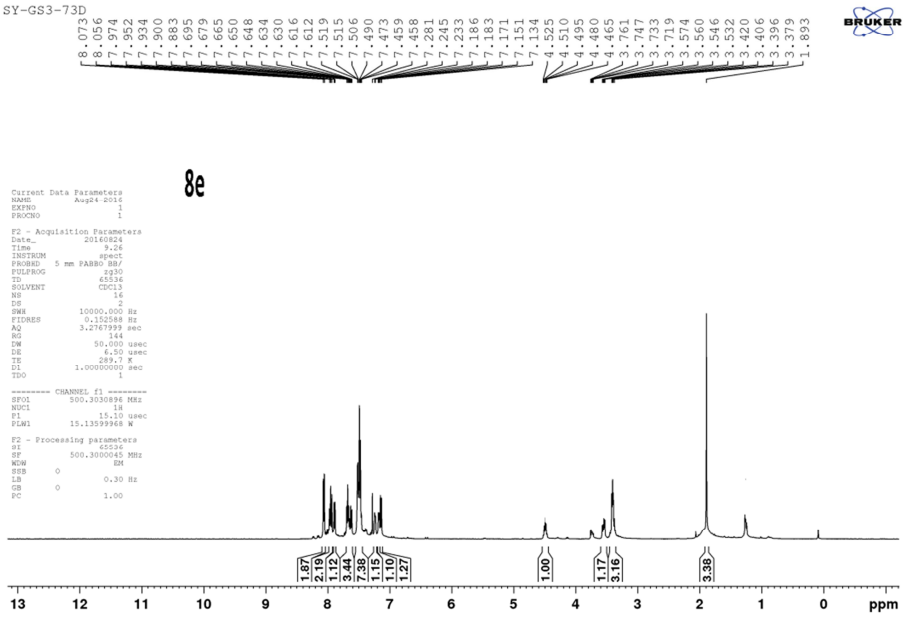
SY-GS3-60

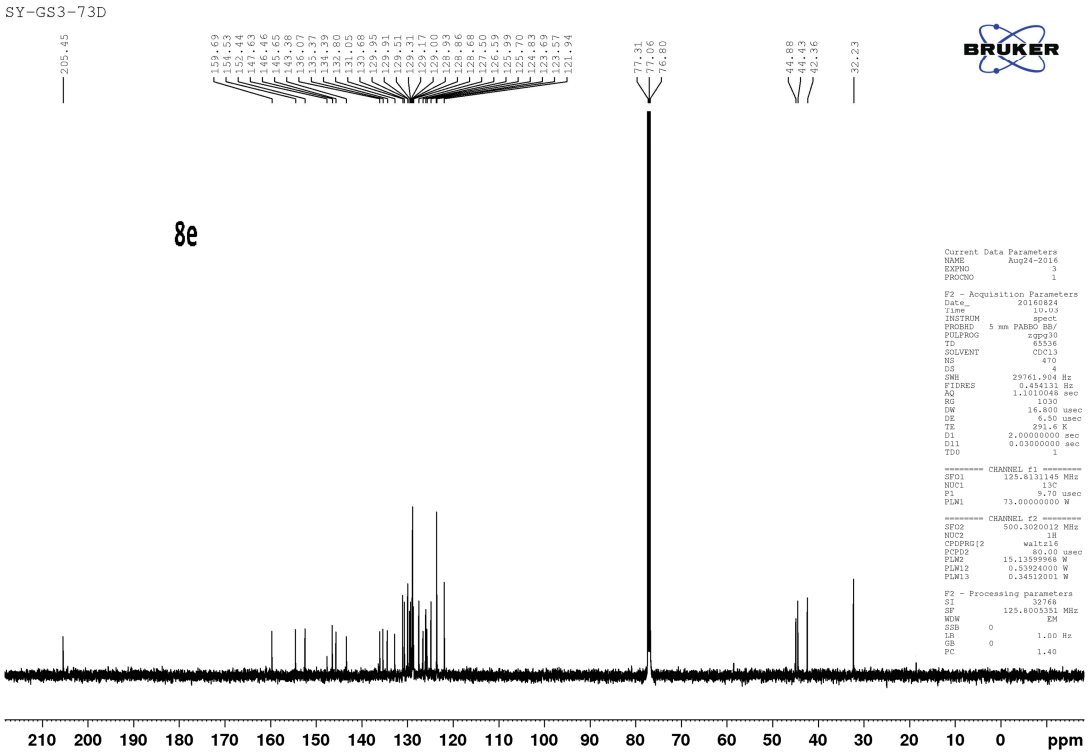


8d

```
Current Data Parameters
NAME          Jul26-2016
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20160726
Time              10.52
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  983
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                297.5 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            125.8005351 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



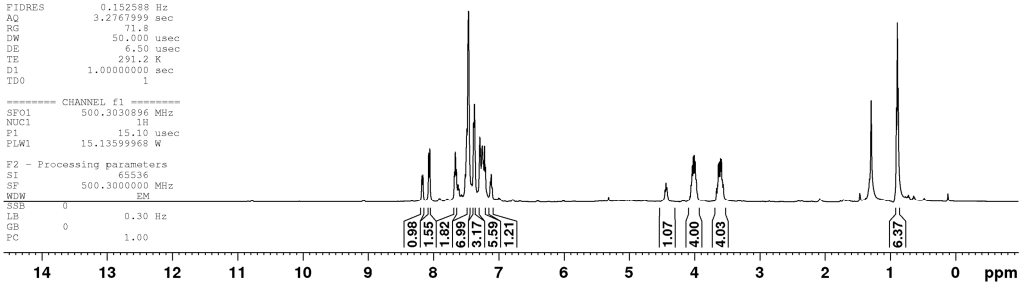

SY-GS3-73D
8.073
8.056
7.974
7.952
7.934
7.900
7.883
7.695
7.679
7.665
7.650
7.648
7.634
7.630
7.616
7.612
7.519
7.515
7.506
7.490
7.473
7.459
7.458
7.281
7.245
7.233
7.186
7.183
7.171
7.151
7.134
4.525
4.510
4.495
4.480
4.465
3.761
3.747
3.733
3.719
3.574
3.560
3.546
3.532
3.420
3.406
3.396
3.379
1.893
BRUKER
8e
Current Data Parameters
NAME Aug24-2016
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160824
Time 9.26
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 289.7 K
D1 1.00000000 sec
TDO 1
======== CHANNEL f1 ========
SFO1 500.3030896 MHz
NUC1 1H
P1 15.10 usec
PLW1 15.13599968 W
F2 - Processing parameters
SI 65536
SF 500.3000045 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.87
2.19
1.12
3.44
7.38
1.15
1.10
1.27
1.00
1.17
3.16
3.38
13
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm


SY-GS3-73D
205.45
159.69
154.53
152.44
147.63
146.46
145.65
143.38
136.07
134.77
134.39
132.80
131.05
130.68
129.95
129.91
129.31
129.21
129.17
129.00
128.93
128.86
128.68
127.50
126.59
126.39
124.70
124.83
123.69
123.57
121.94
77.31
77.06
76.80
44.88
44.43
42.36
32.23
BRUKER
8e
Current Data Parameters
NAME Aug24-2016
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160824
Time 10.03
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 470
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 291.6 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
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

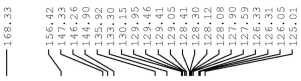
SY-GS3-61

8f

```
Current Data Parameters
NAME                  Jul26-2016
EXPNO                         50
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20160726
Time                       18.38
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                         71.8
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
```

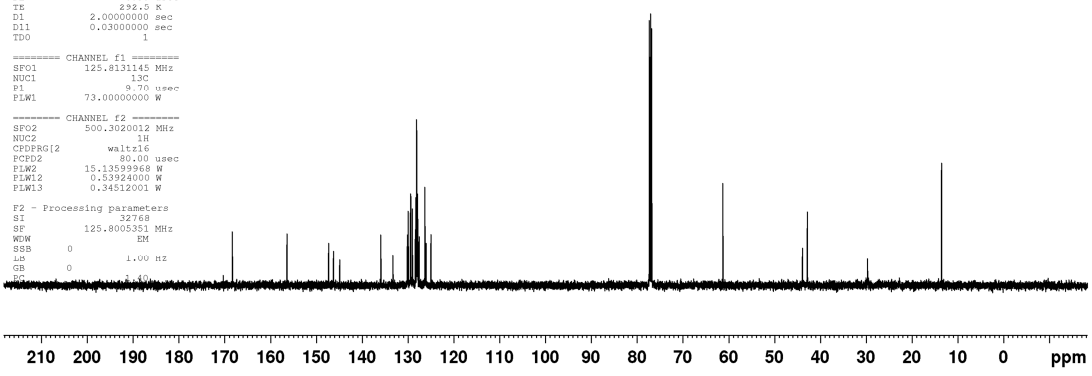


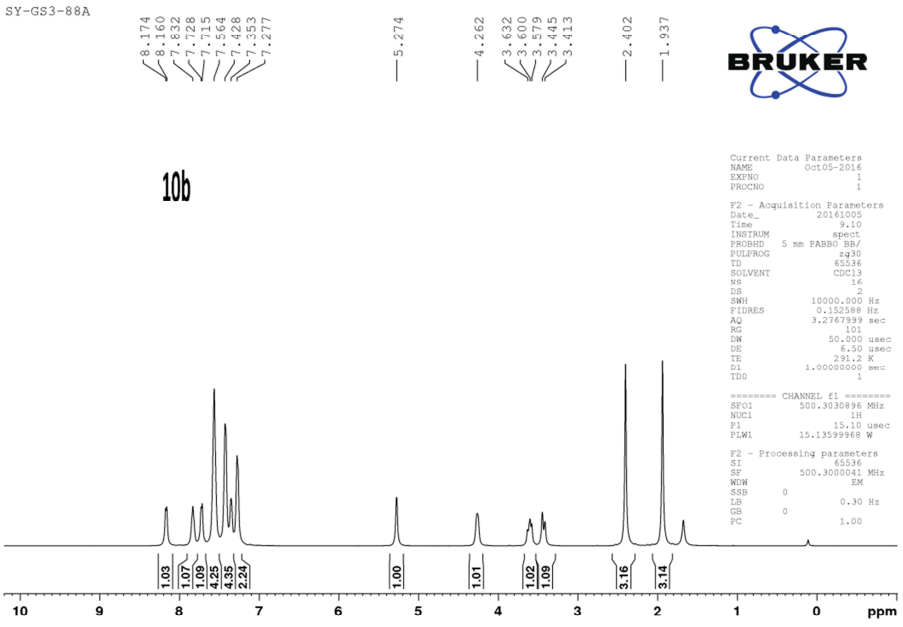
SY-GS3-61

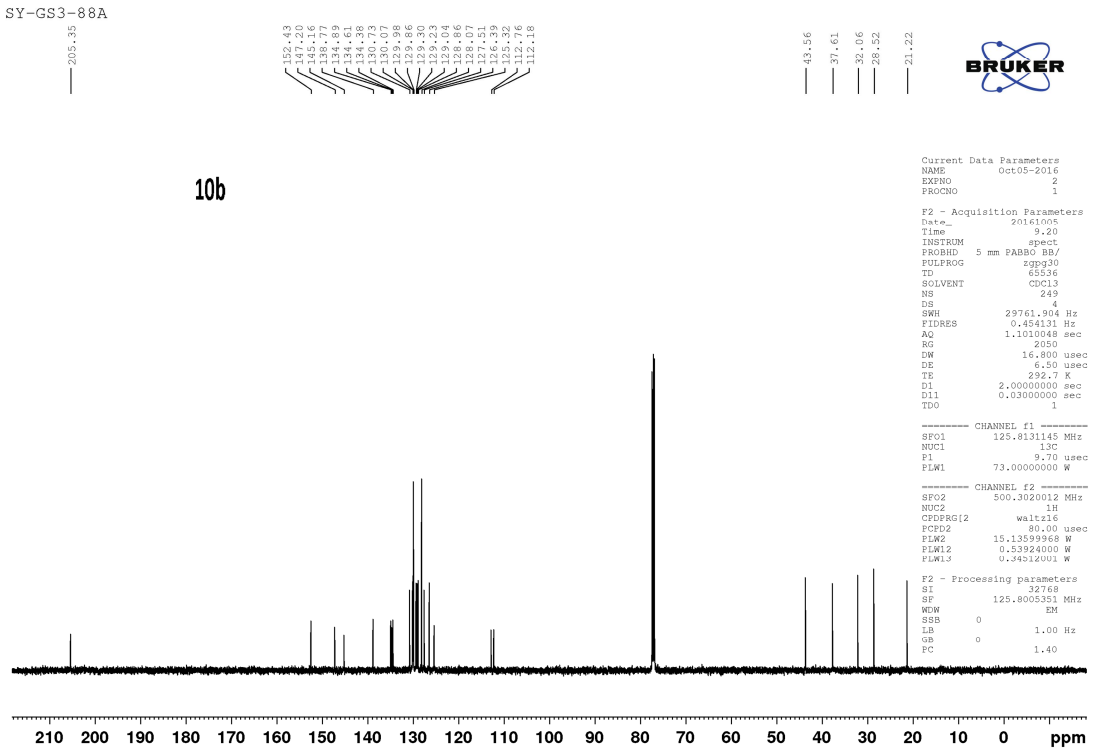


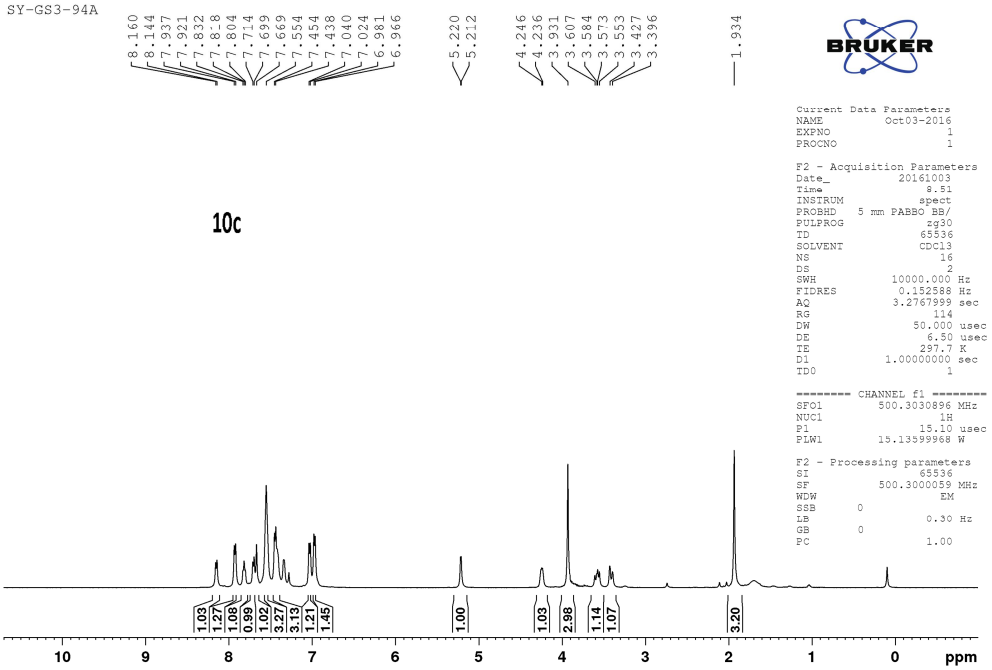
8f

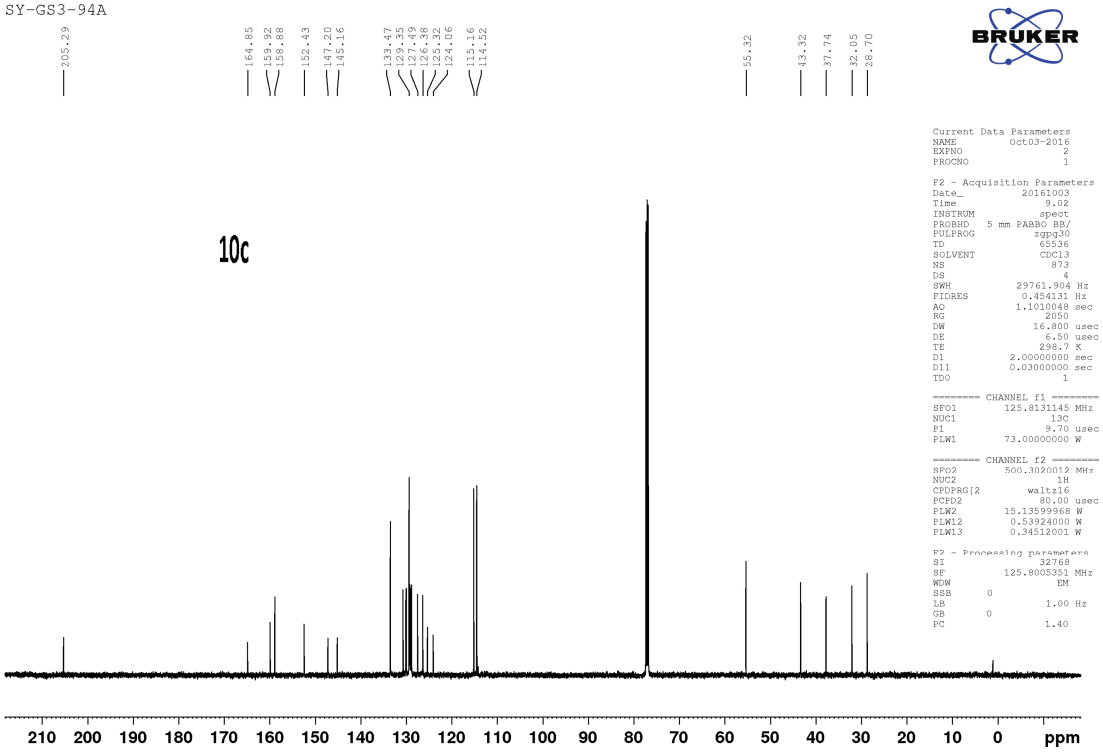
```
Current Data Parameters
NAME                  Jul26-2016
EXPNO                         51
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20160726
Time                       18.43
INSTRUM                    spect
PROBHD     5 mm PABBO BB/
PULPROG                  zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                           228
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                        292.5 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                  125.8005351 MHz
WDW                          EM
SSB                0
LB                        1.00 Hz
GB                 0
PC                        1.40
```

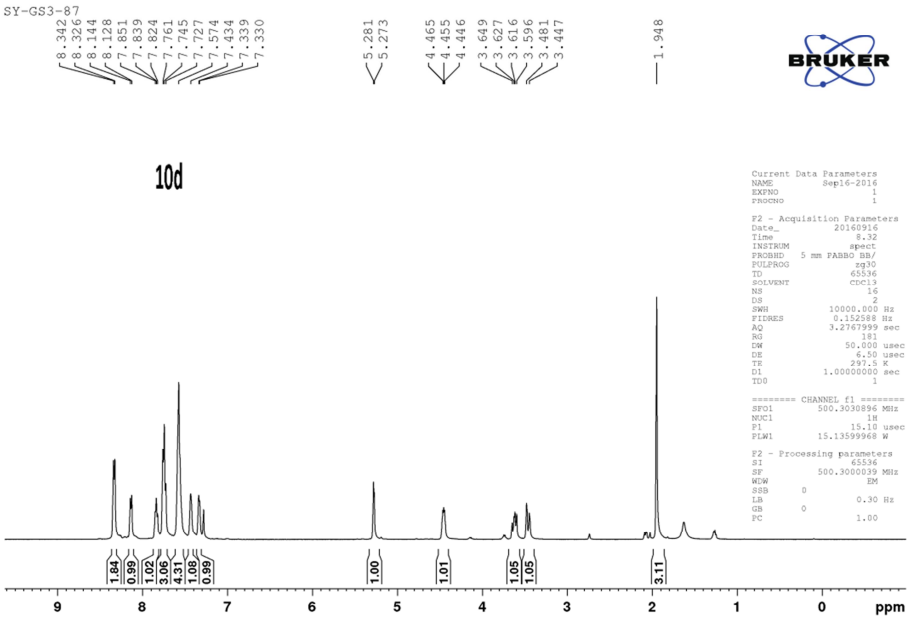


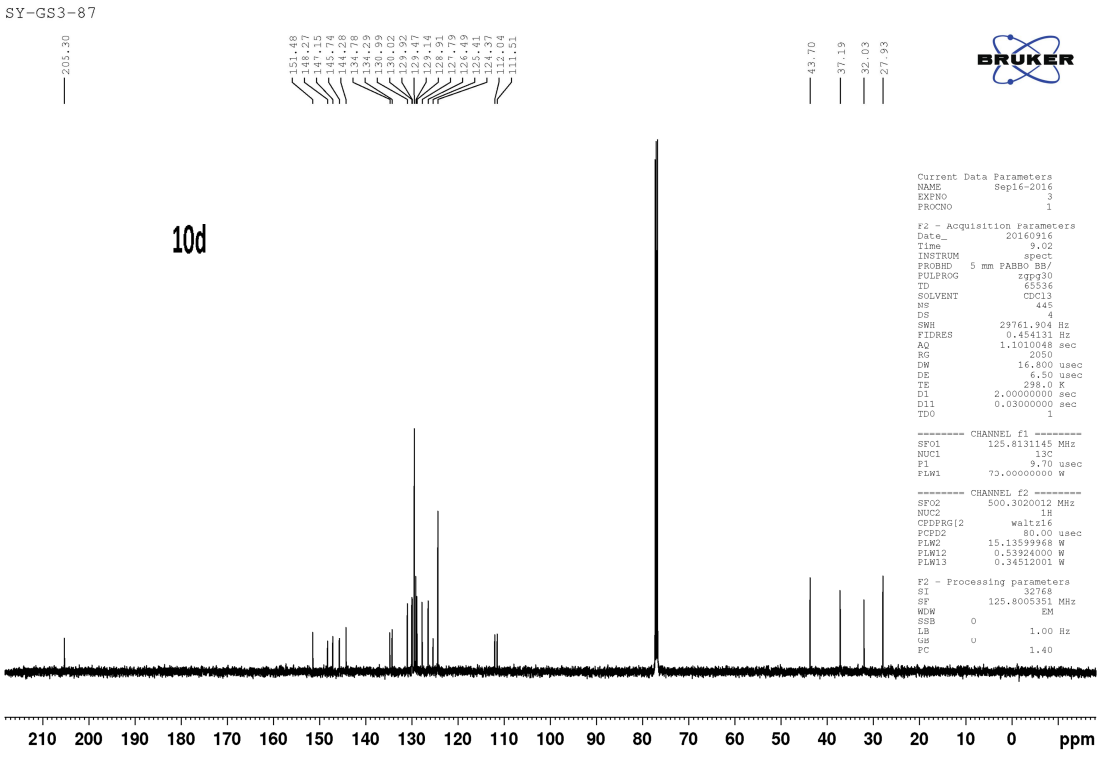

SY-GS3-88A
8.174
8.160
7.832
7.728
7.715
7.564
7.428
7.353
7.277
5.274
4.262
3.632
3.600
3.579
3.445
3.413
2.402
1.937
BRUKER
10b
Current Data Parameters
NAME Oct05-2016
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161005
Time 9.10
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 101
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 500.3000041 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.03
1.07
1.09
4.25
4.35
2.24
1.00
1.01
1.02
1.09
3.16
3.14
10 9 8 7 6 5 4 3 2 1 0 ppm


SY-GS3-88A
205.35
152.43
147.20
141.16
138.77
134.89
134.61
134.38
130.73
130.07
129.88
129.86
129.30
129.23
129.04
128.86
128.07
127.31
127.19
125.32
112.76
112.18
43.56
37.61
32.06
28.52
21.22
BRUKER
10b
Current Data Parameters
NAME Oct05-2016
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161005
Time 9.20
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 249
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 292.7 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
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

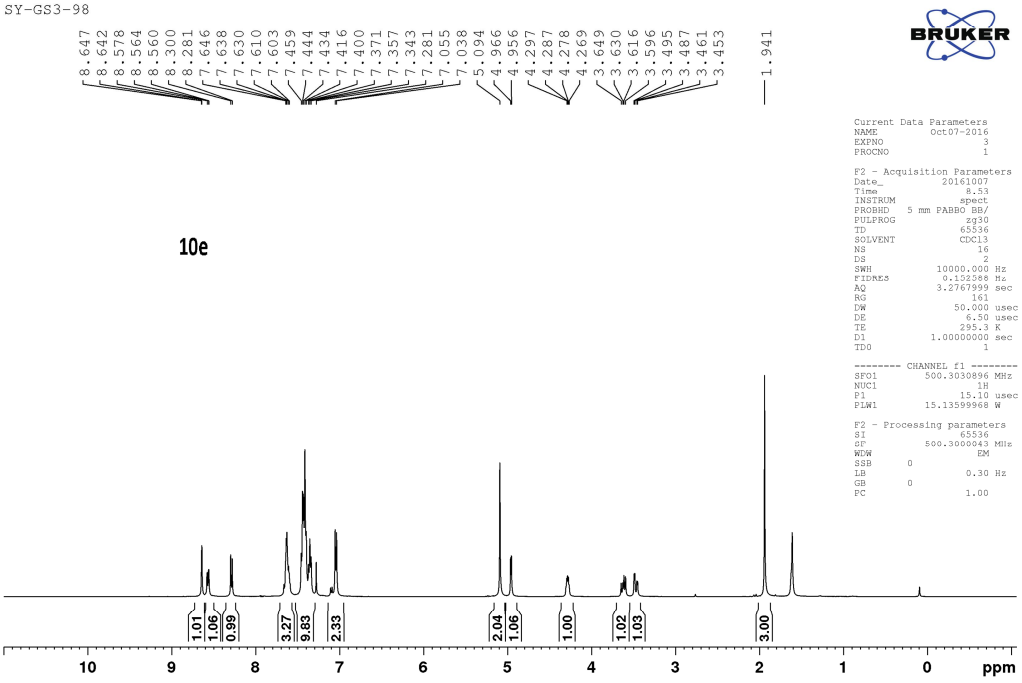

SY-GS3-94A
8.160
8.144
7.937
7.921
7.832
7.818
7.804
7.714
7.699
7.669
7.554
7.454
7.438
7.040
7.024
6.981
6.966
5.220
5.212
4.246
4.236
3.931
3.607
3.584
3.573
3.553
3.427
3.396
1.934
BRUKER
10c
Current Data Parameters
NAME Oct03-2016
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161003
Time 8.51
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 114
DW 50.000 usec
DE 6.50 usec
TE 297.7 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 500.3000059 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.03
1.27
1.08
0.99
1.02
3.27
3.13
1.21
1.45
1.00
1.03
2.98
1.14
1.07
3.20
10
9
8
7
6
5
4
3
2
1
0
ppm


SY-GS3-94A
205.29
164.85
159.92
158.88
152.43
147.20
145.16
133.47
129.35
127.49
126.38
125.32
124.06
115.16
114.52
55.32
43.32
37.74
32.05
28.70
BRUKER
10c
Current Data Parameters
NAME Oct03-2016
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161003
Time 9.02
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 873
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 298.7 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
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

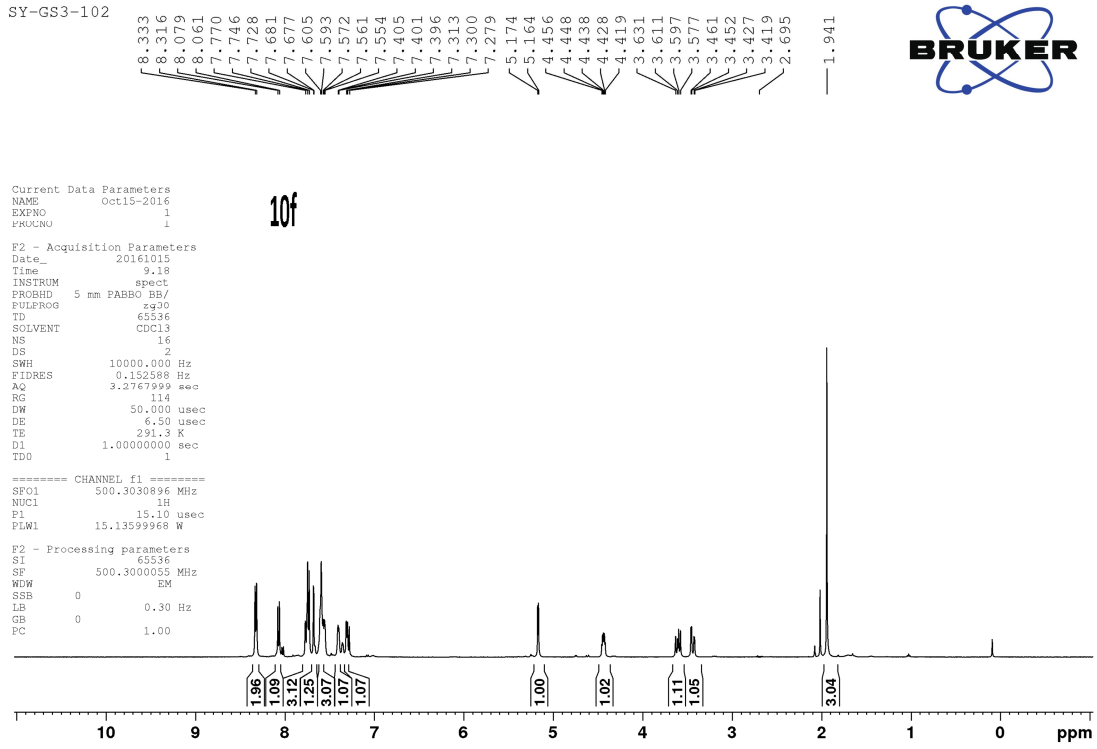

SY-GS3-87
8.342
8.326
8.144
8.128
7.851
7.839
7.824
7.761
7.745
7.727
7.574
7.434
7.339
7.330
5.281
5.273
4.465
4.455
4.446
3.649
3.627
3.616
3.596
3.481
3.447
1.948
BRUKER
10d
Current Data Parameters
NAME Sep16-2016
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160916
Time 8.32
INSTRUM spect
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 181
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 500.3000039 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.84
0.99
1.02
3.06
4.31
1.08
0.99
1.00
1.01
1.05
1.05
3.11
9
8
7
6
5
4
3
2
1
0
ppm


SY-GS3-87
205.30
151.48
148.27
147.15
145.74
144.28
134.29
130.99
130.02
129.92
129.47
129.14
128.91
128.79
126.49
125.41
124.37
112.04
111.51
43.70
37.19
32.03
27.93
BRUKER
10d
Current Data Parameters
NAME Sep16-2016
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160916
Time 9.02
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 445
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 298.0 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 125.8005351 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

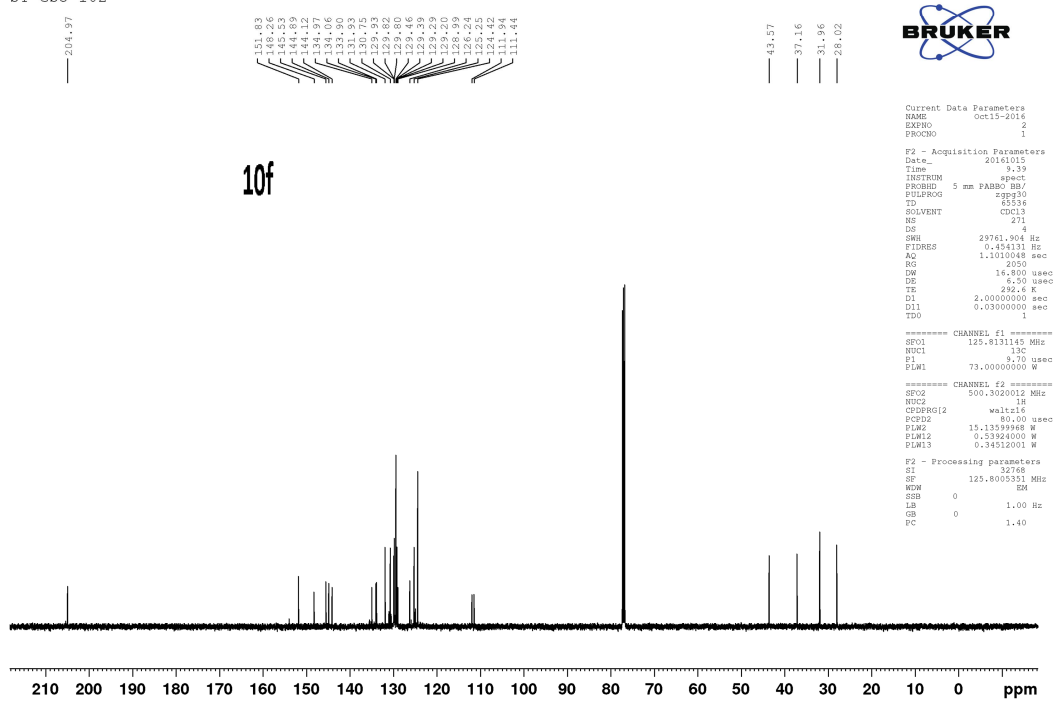
SY-GS3-98



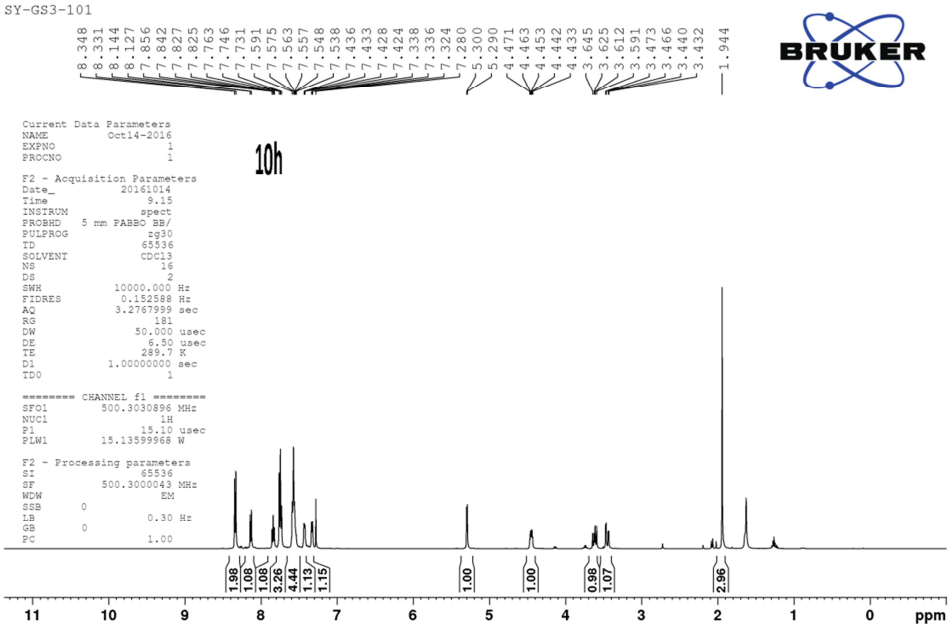
SY-GS3-102



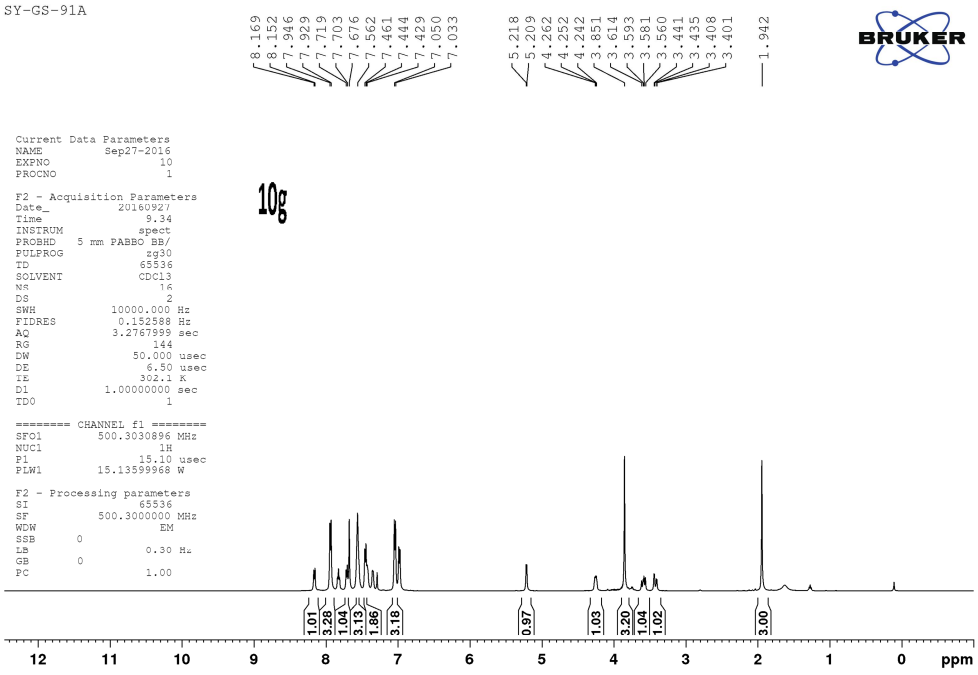
SY-GS3-102



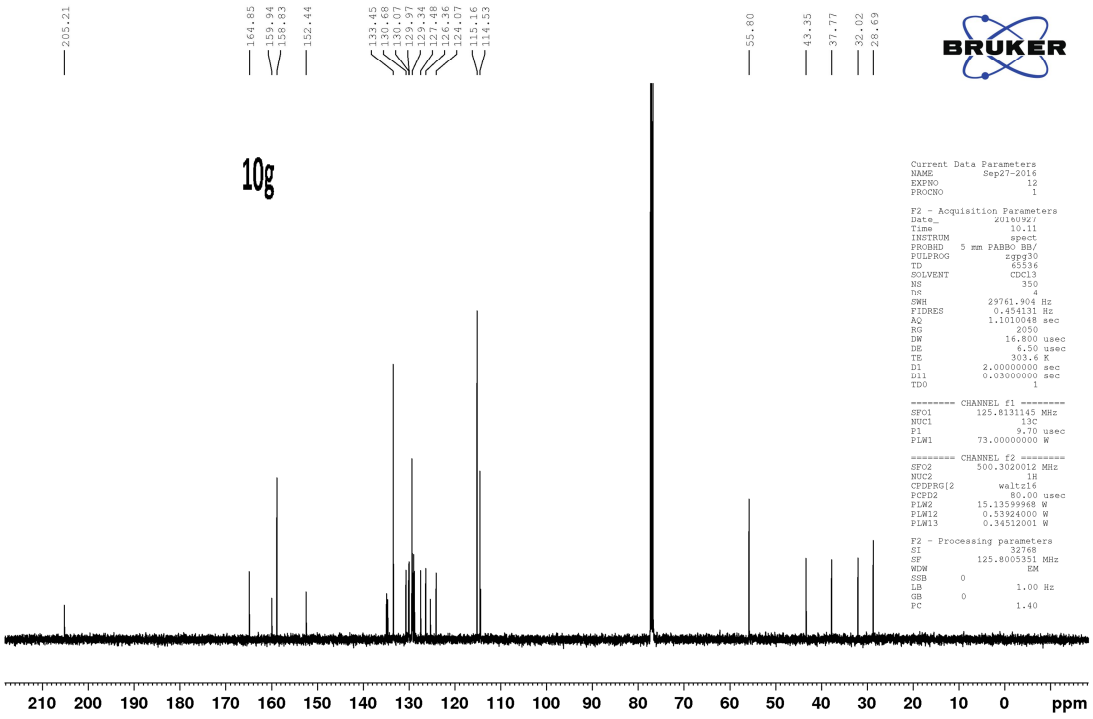
SY-GS3-101

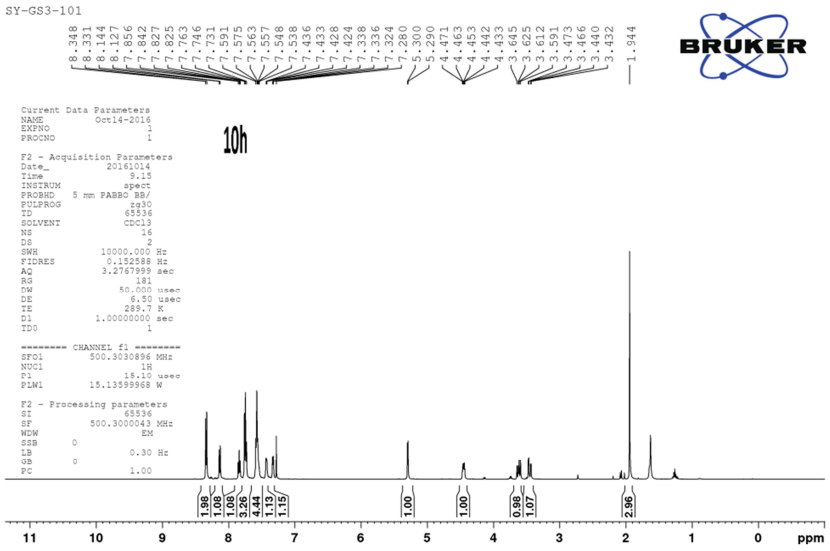


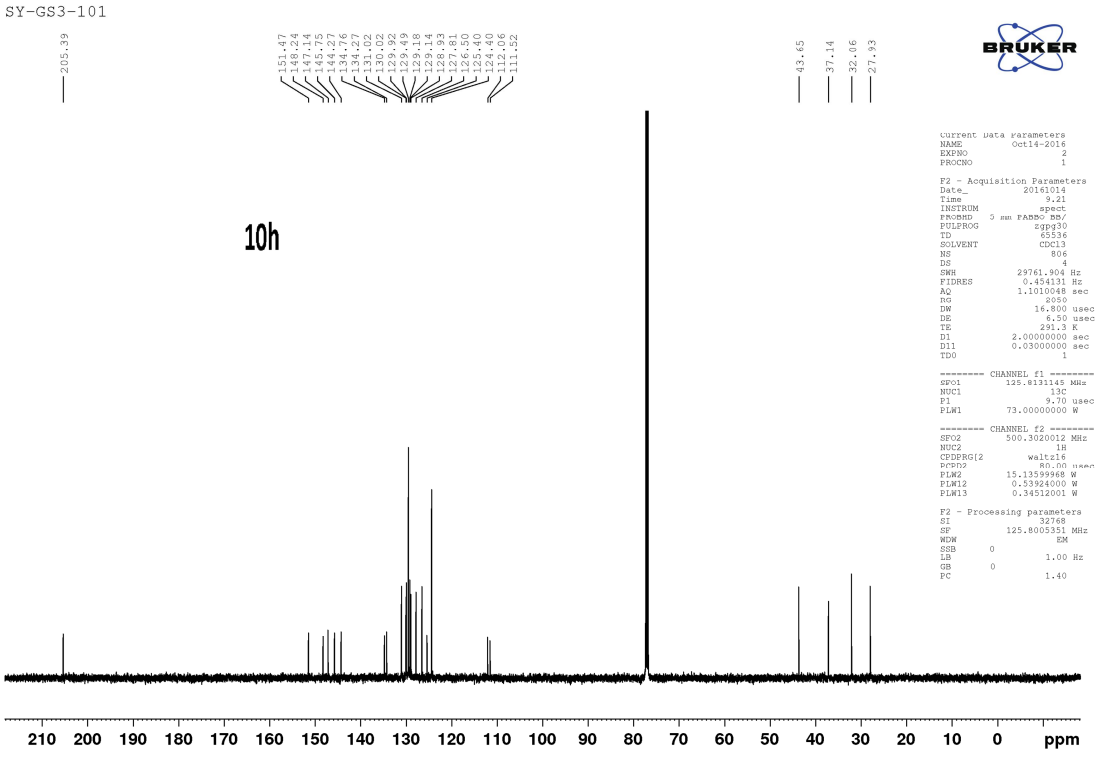
SY-GS-91A

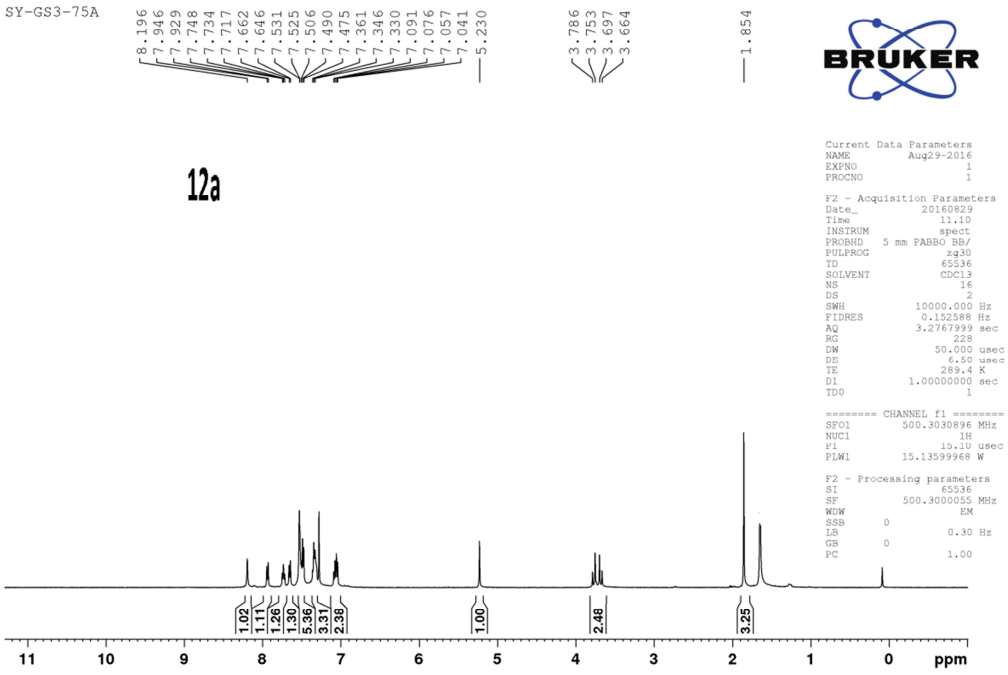


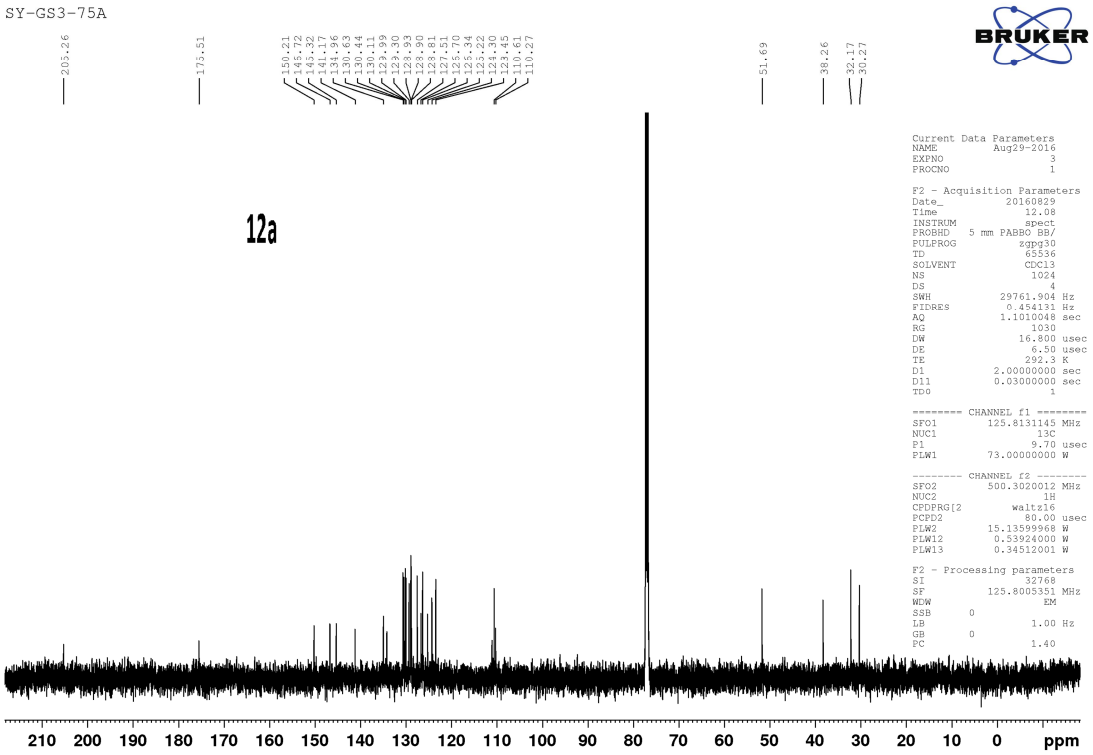
SY-GS-91A

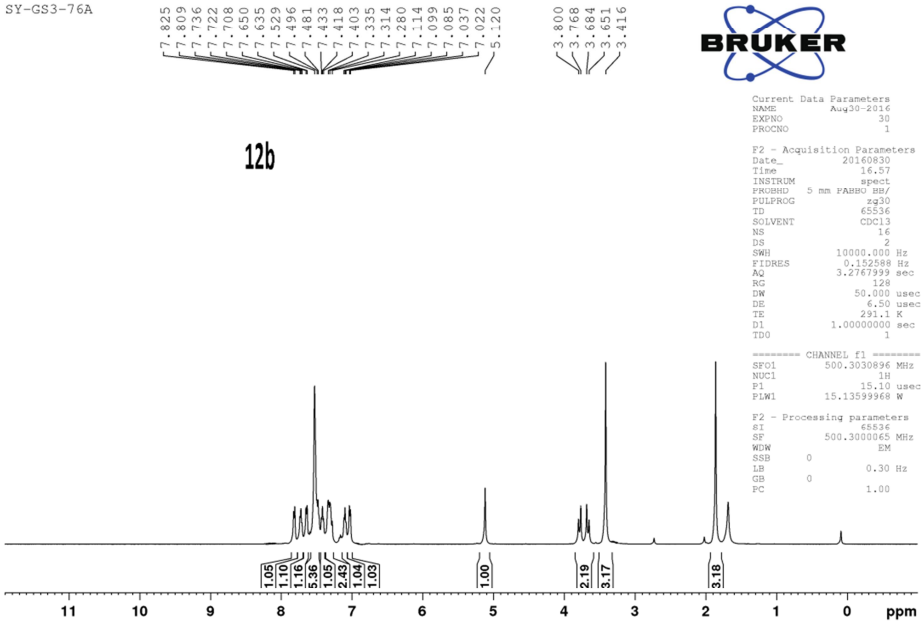


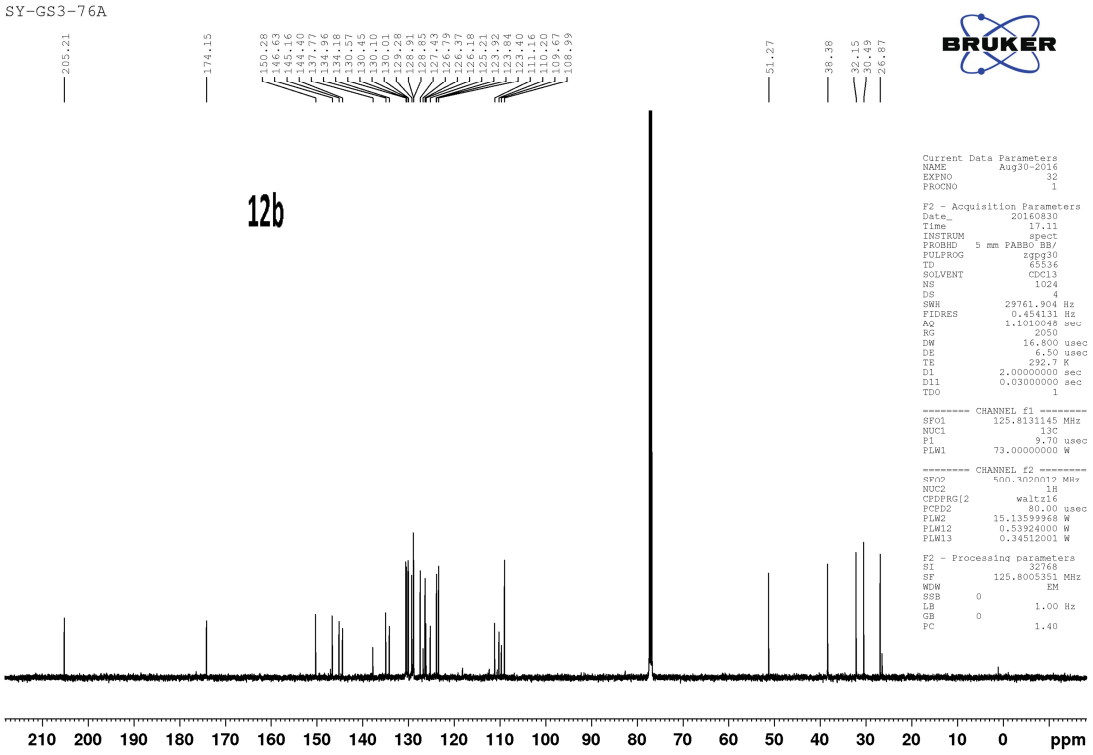

SY-GS3-101
10h
Current Data Parameters
NAME Oct14-2016
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161014
Time 9.15
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 181
DW 50.000 usec
DE 6.50 usec
TE 289.7 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 500.3000043 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm


SY-GS3-101
10h
Current Data Parameters
NAME Oct14-2016
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161014
Time 9.21
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 806
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 291.3 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 125.8005351 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
ppm

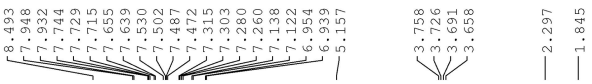

SY-GS3-75A
8.196
7.946
7.929
7.748
7.734
7.717
7.662
7.646
7.531
7.525
7.506
7.490
7.475
7.361
7.346
7.330
7.091
7.076
7.057
7.041
5.230
3.786
3.753
3.697
3.664
1.854
BRUKER
12a
Current Data Parameters
NAME Aug29-2016
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160829
Time 11.10
INSTRUM spect
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 228
DW 50.000 usec
DE 6.50 usec
TE 289.4 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 500.3000055 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.02
1.11
1.26
1.30
5.36
3.31
2.38
1.00
2.48
3.25
11
10
9
8
7
6
5
4
3
2
1
0
ppm


SY-GS3-75A
205.26
175.51
150.21
145.72
145.32
141.17
131.96
130.63
130.44
130.11
129.09
124.30
123.93
123.90
123.81
127.51
125.70
125.34
125.22
124.30
123.45
110.61
110.27
51.69
38.26
32.17
30.27
BRUKER
12a
Current Data Parameters
NAME Aug29-2016
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160829
Time 12.08
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
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 292.3 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 125.8005351 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

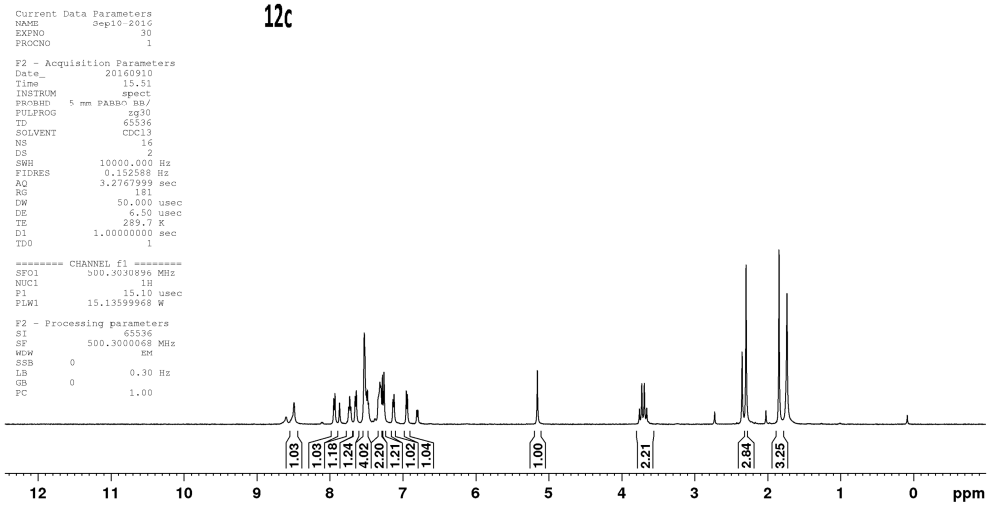

SY-GS3-76A
7.825
7.809
7.736
7.722
7.708
7.650
7.635
7.529
7.496
7.481
7.433
7.418
7.403
7.335
7.314
7.280
7.114
7.099
7.085
7.037
7.022
5.120
3.800
3.768
3.684
3.651
3.416
BRUKER
12b
Current Data Parameters
NAME Aug30-2016
EXPNO 30
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160830
Time 16.57
INSTRUM 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 291.1 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 500.3000065 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.05
1.10
1.16
5.36
1.05
2.43
1.04
1.03
1.00
2.19
3.17
3.18
11
10
9
8
7
6
5
4
3
2
1
0
ppm


SY-GS3-76A
205.21
174.15
150.28
146.63
145.16
144.40
137.77
134.96
134.18
130.57
130.45
130.10
130.01
129.28
128.91
128.85
127.43
126.79
126.37
126.18
125.21
123.82
123.64
123.40
111.16
110.20
109.67
108.99
51.27
38.38
32.15
30.49
26.87
BRUKER
12b
Current Data Parameters
NAME Aug30-2016
EXPNO 32
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160830
Time 17.11
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
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 292.7 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 500.3020013 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
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

SY-GS3-83-A



12c



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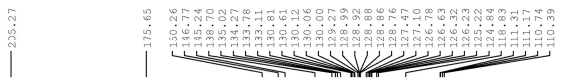

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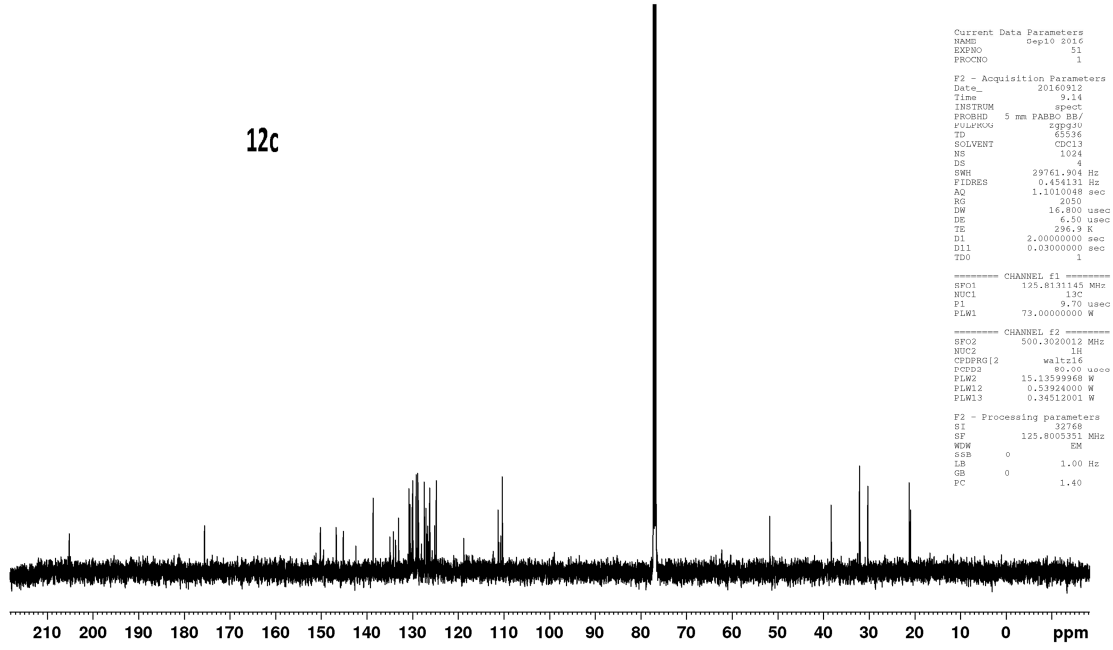

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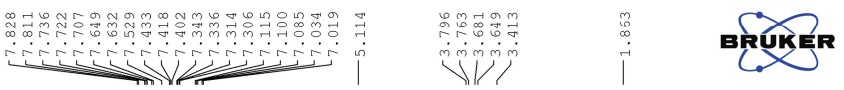

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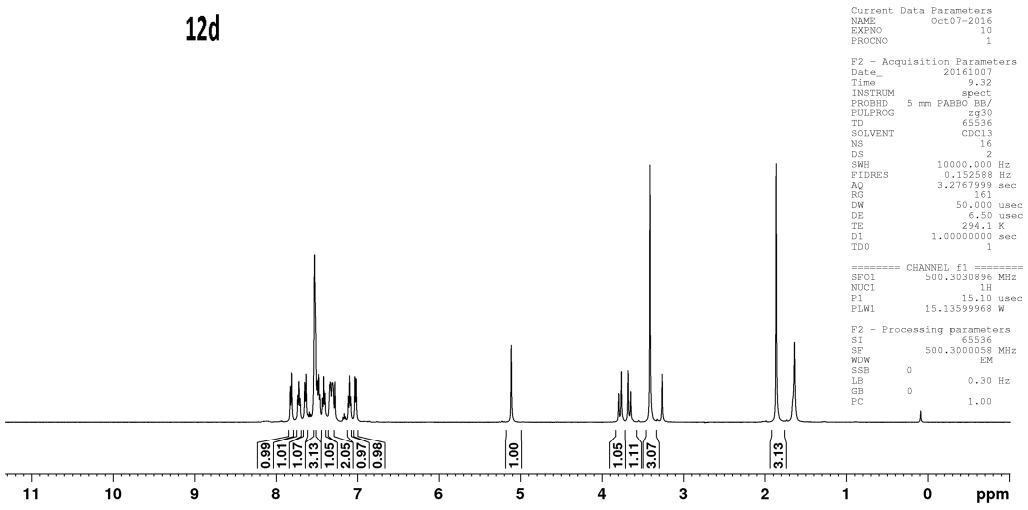

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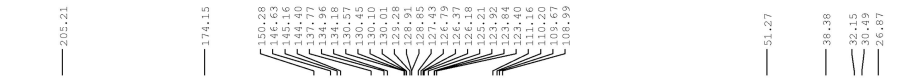

12d