

Anti-Adipogenic Activity of Secondary Metabolites Isolated from *Smilax sieboldii* Miq. on 3T3-L1 Adipocytes

Supplementary Material

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1. General Experimental Procedure

Optical rotations were measured using a JASCO P-2000 polarimeter (Tokyo, Japan). UV and FT-IR spectra were measured on a JASCO UV-550 and JASCO Model FT/IR-4100 spectrometer (Tokyo, Japan), respectively. Nuclear magnetic resonance (NMR) spectra were recorded on a Bruker Ascend III 700 spectrometer (Bruker BioSpin GmbH, Rheinstetten, Germany) in acetone-*d*₆, CDCl₃, and CD₃OD at room temperature. Chemical shifts are in ppm (δ), relative to tetramethylsilane as an internal standard, and coupling constants are in hertz; ¹H, ¹³C, DEPT, COSY, HSQC, and HMBC by using the standard pulse sequences. Electrospray ionization mass (ESI-MS) spectra were acquired on an Agilent 6130 series quadrupole LC/MS System (Agilent Technologies, CA, USA). HRESIMS spectra were recorded by a Triple TOF 5600+ mass spectrometer (AB SCIEX, MA, USA). Open column chromatography was performed using Diaion HP-20 adsorbent resin (Mitsubishi Chemical Corp, Tokyo, Japan). Medium-pressure liquid chromatography (MPLC) was conducted using a CombiFlash Rf flash chromatography system (Teledyne ISCO Inc., NE, USA), and the separations were performed on a RediSep[®] Rf C₁₈ column with a flow rate of 40 mL/min. Preparative high-performance liquid chromatography (HPLC) was performed on a Thermo Scientific Dionex Ultimate 3000 UHPLC system (Thermo Fisher Scientific Inc., MA, USA) equipped with an HPG-3200BX biocompatible binary semipreparative pump and a rapid separations PDA detector (Ultimate DAD-3000) controlled by Chromeleon 7.2 software. The separations were carried out on a Kromasil 100-5-C18 column (5 μm, 21.2 × 250 mm, Nouryon Chemicals Finance B.V., Amsterdam, Netherlands). Thin layer chromatography (TLC) was performed using DC-Fertigfolien ALUGRAM[®] SIL G/UV₂₅₄₊₃₆₆ (0.2 mm, Macherey-Nagel GmbH & Co. KG, Düren, Germany) plates, and spots were visualized by a 10% vanillin–sulfuric acid reagent. All chemicals and solvents were of analytical grade and were used without further purification.

Table S1. *In vitro* cytotoxicity of compounds 1–19.

Compounds	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19
IC ₅₀ (μM)	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC

IC₅₀ values of tested compounds were measured. 3T3-L1 preadipocytes (40% confluence) were treated with different concentrations (25, 50, and 100 μM) of tested compounds for 48 h. Statistical analysis was performed by Student's *t*-test. NC, no cytotoxicity.

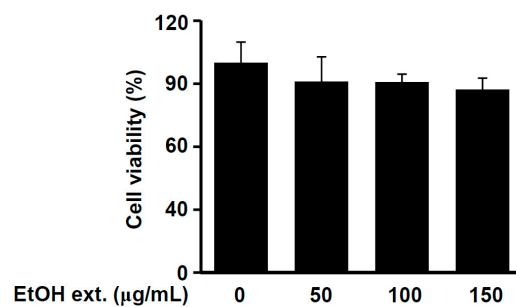


Figure S1. Effect of crude extract of *S. sieboldii* whole plant on cell viability by using MTT

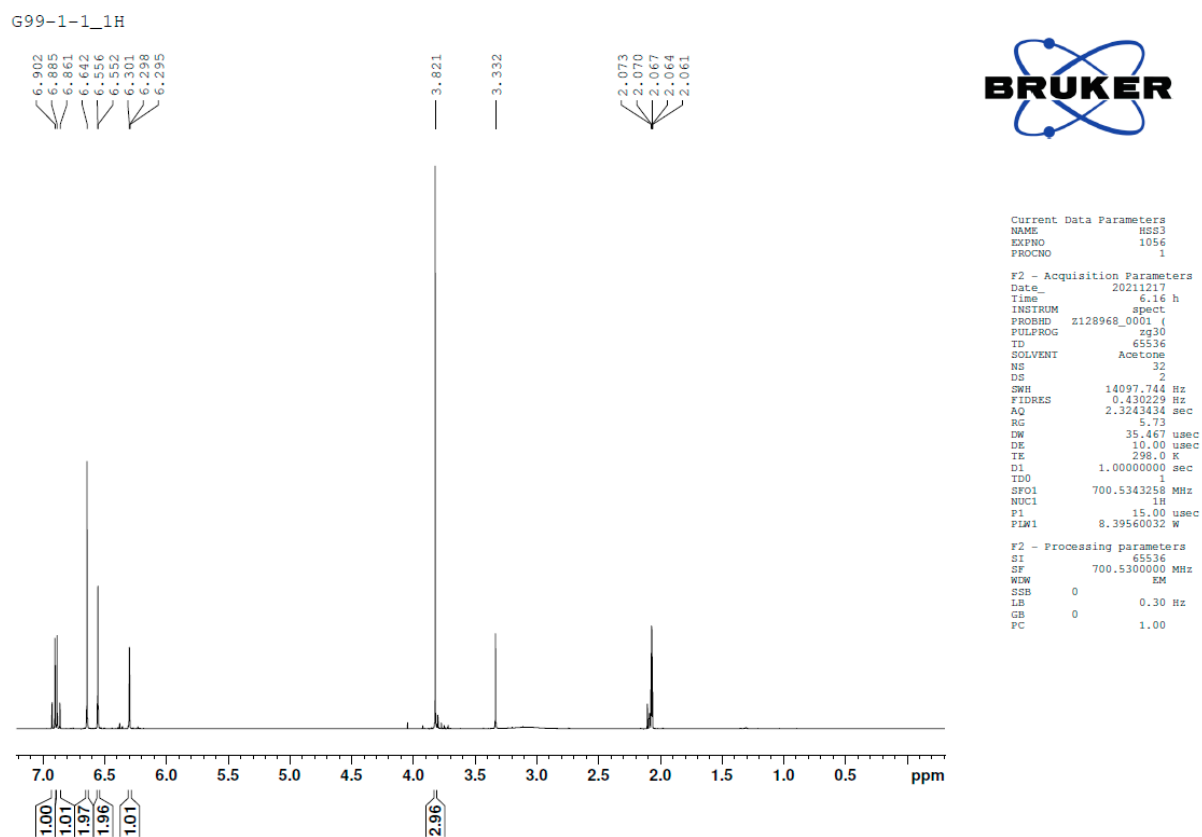
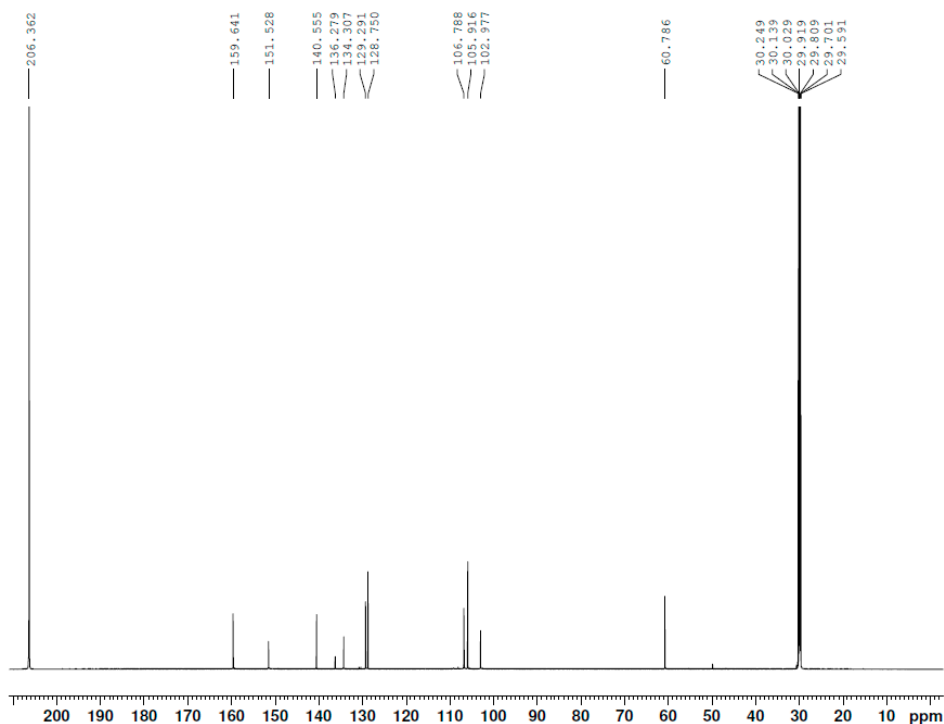


Figure S2. ^1H -NMR spectrum (acetone- d_6 , 700 MHz) of compound **1**

G99-1-1_13C



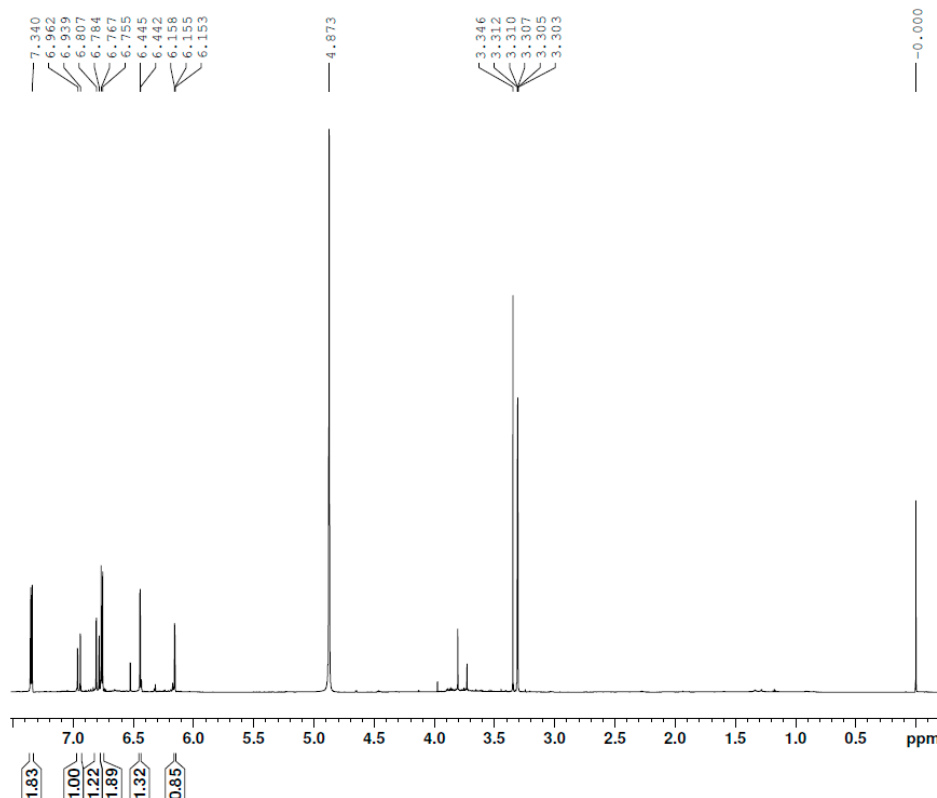
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FIDRES 1.304047 Hz
AQ 0.7668437 sec
RG 2050
DW 11.733 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 176.1660231 MHz
NUC1 13C
P1 12.00 usec
PLW1 32.96100006 W
SFO2 700.5328021 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 65.00 usec
PLW2 8.39560032 W
PLW12 0.44710001 W
PLW13 0.22513001 W

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WDW EM
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GB 0
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Figure S3. ^{13}C -NMR spectrum (acetone- d_6 , 175 MHz) of compound **1**

G99-3_1H



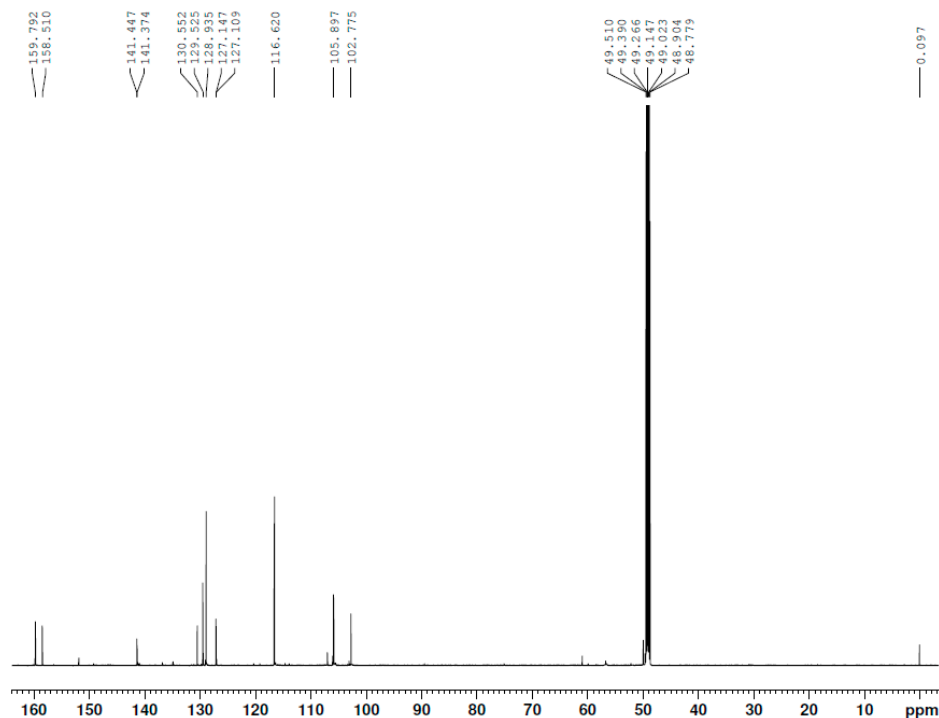
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FIDRES 0.430229 Hz
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RG 7.13
DW 35.467 usec
DE 10.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
SFO1 700.5343258 MHz
NUC1 1H
P1 15.00 usec
PLW1 8.39560032 W

F2 - Processing parameters
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WDW EM
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Figure S4. ^1H -NMR spectrum (acetone- d_6 , 700 MHz) of compound **2**

G99-3_13C



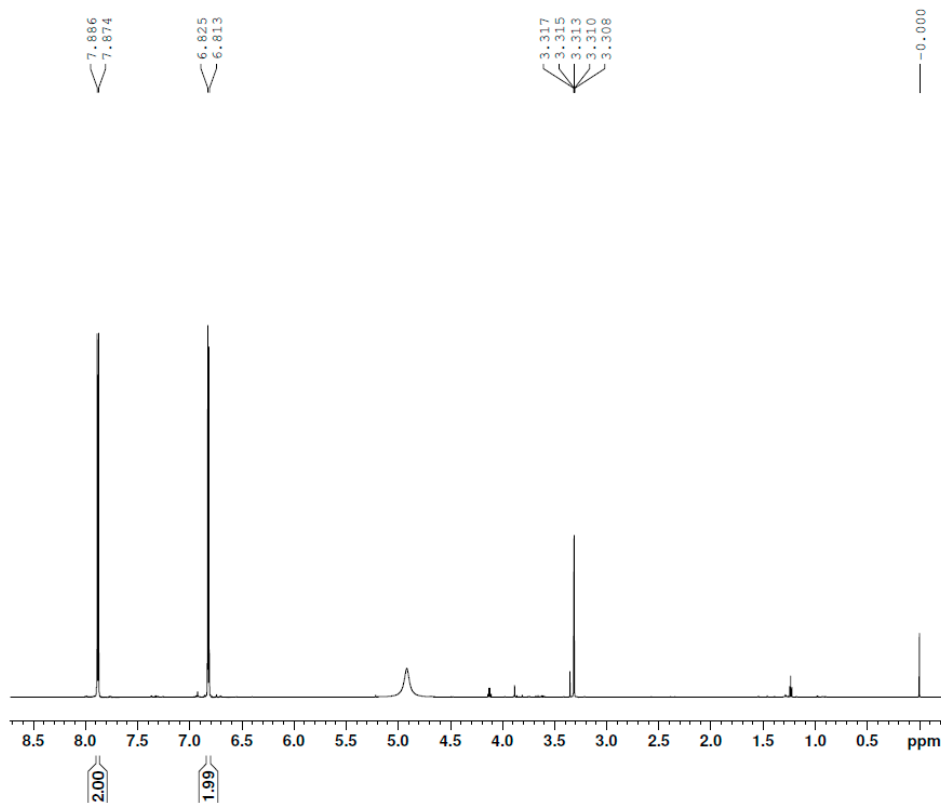
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SOLVENT MeOD
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FIDRES 1.304047 Hz
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RG 2050
DW 11.733 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0
SFO1 176.1660234 MHz
NUC1 13C
P1 12.00 usec
PLW1 32.86100006 W
SFO2 700.5328021 MHz
NUC2 1H
CPCPD2 waltz16
PCPD2 65.00 usec
PLW2 8.39560032 W
PLW12 0.44710001 W
PLW13 0.22513001 W

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GB 0
PC 1.40

Figure S5. ^{13}C -NMR spectrum (acetone- d_6 , 175 MHz) of compound **2**

G99-7_1H



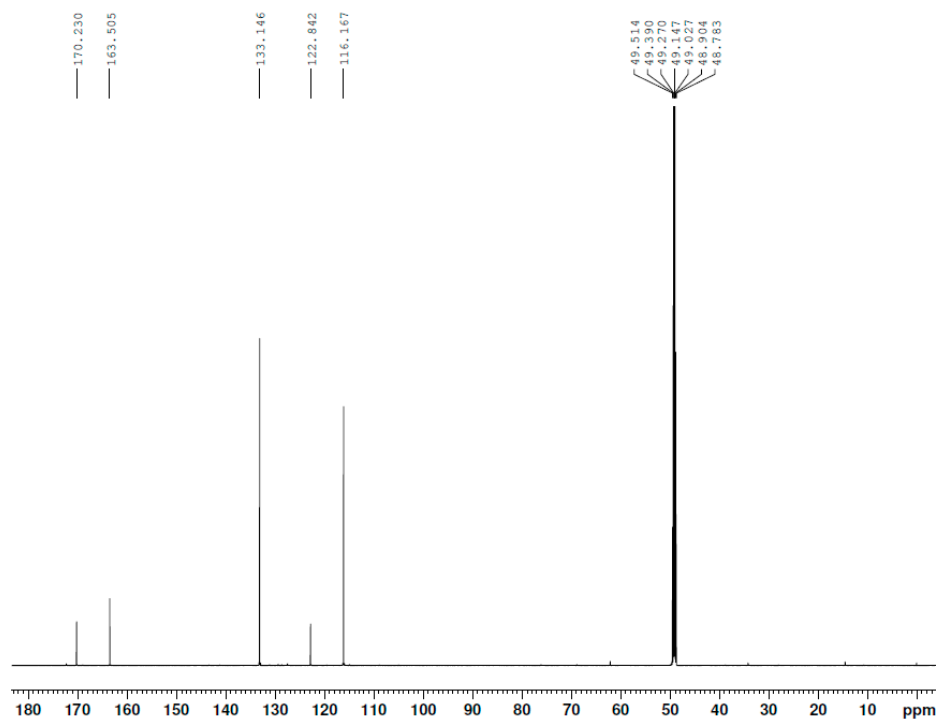
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FIDRES 0.430229 Hz
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RG 3.5
DW 35.467 usec
DE 10.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0
SFO1 700.5343258 MHz
NUC1 1H
P1 15.00 usec
PLW1 8.39560032 W

F2 - Processing parameters
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SF 700.5300115 MHz
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LB 0.30 Hz
GB 0
PC 1.00

Figure S6. ^1H -NMR spectrum (CD_3OD , 700 MHz) of compound **3**

G99-7_13C



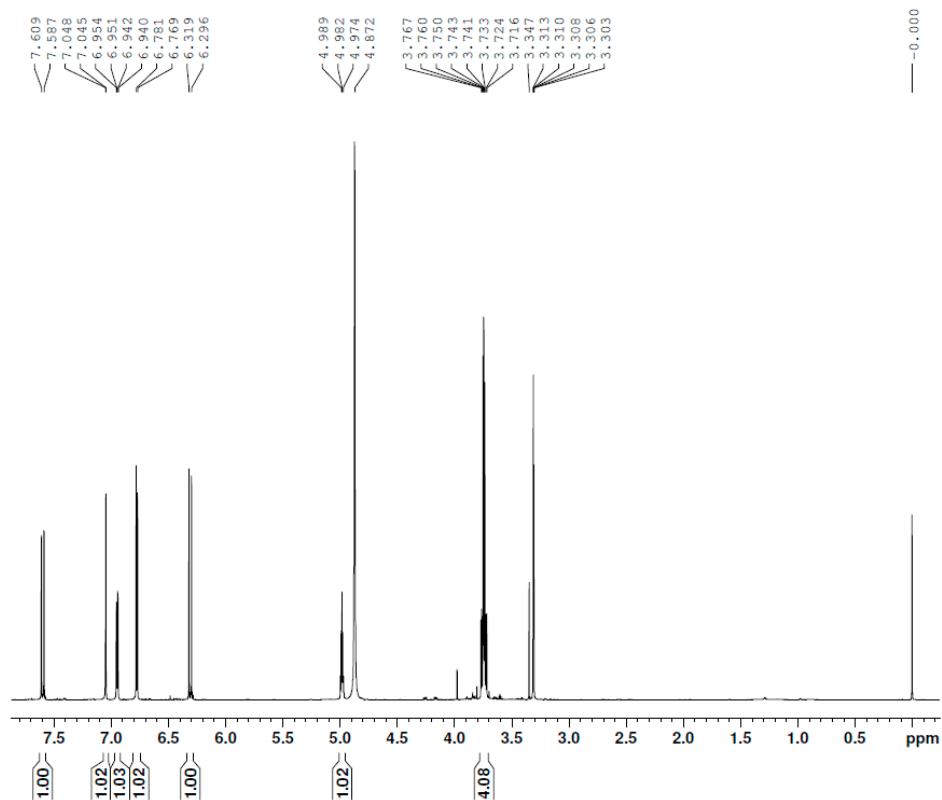
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SOLVENT MeOD
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FIDRES 1.304047 Hz
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RG 1440
DW 11.733 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 176.1660234 MHz
NUC1 13C
P1 12.00 usec
PLW1 32.86100006 W
SFO2 700.5328021 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 65.00 usec
PLW2 8.39560032 W
PLW12 0.44710001 W
PLW13 0.22513001 W

F2 - Processing parameters
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SF 176.1481379 MHz
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GB 0
PC 1.40

Figure S7. ^{13}C -NMR spectrum (CD_3OD , 175 MHz) of compound **3**

G99-8_1H



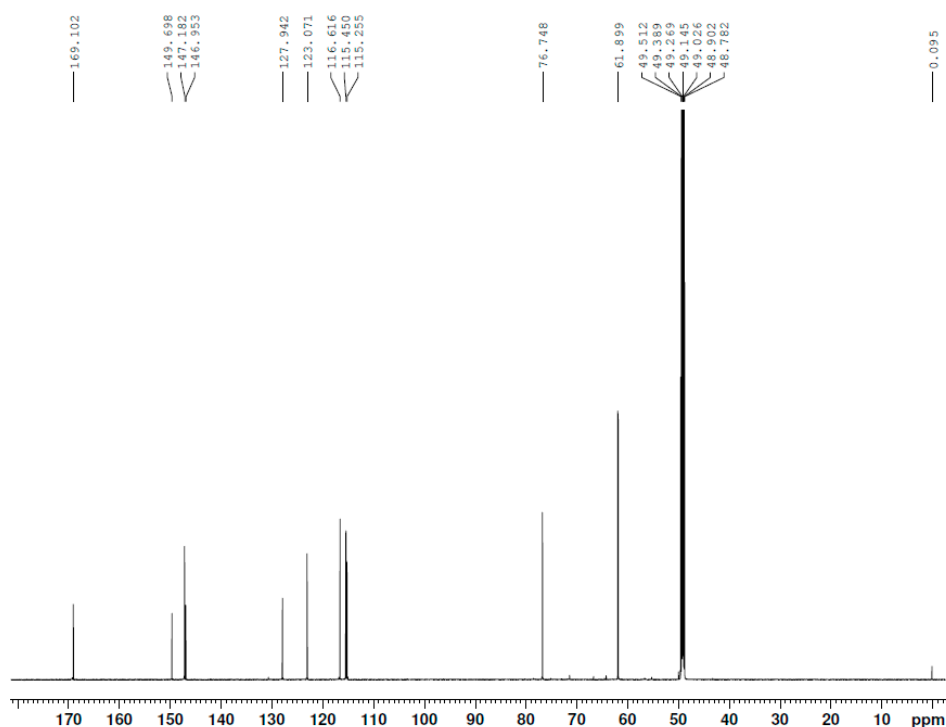
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TD 65536
SOLVENT MeOD
NS 32
DS 2
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FIDRES 0.430229 Hz
AQ 2.3243434 sec
RG 6.45
DW 35.467 usec
DE 10.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
SFO1 700.5343258 MHz
NUC1 1H
P1 15.00 usec
PLW1 8.39560032 W

F2 - Processing parameters
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SF 700.5300149 MHz
WDW EM
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GB 0
PC 1.00

Figure S8. ^1H -NMR spectrum (CD_3OD , 700 MHz) of compound **4**

G99-8_13C



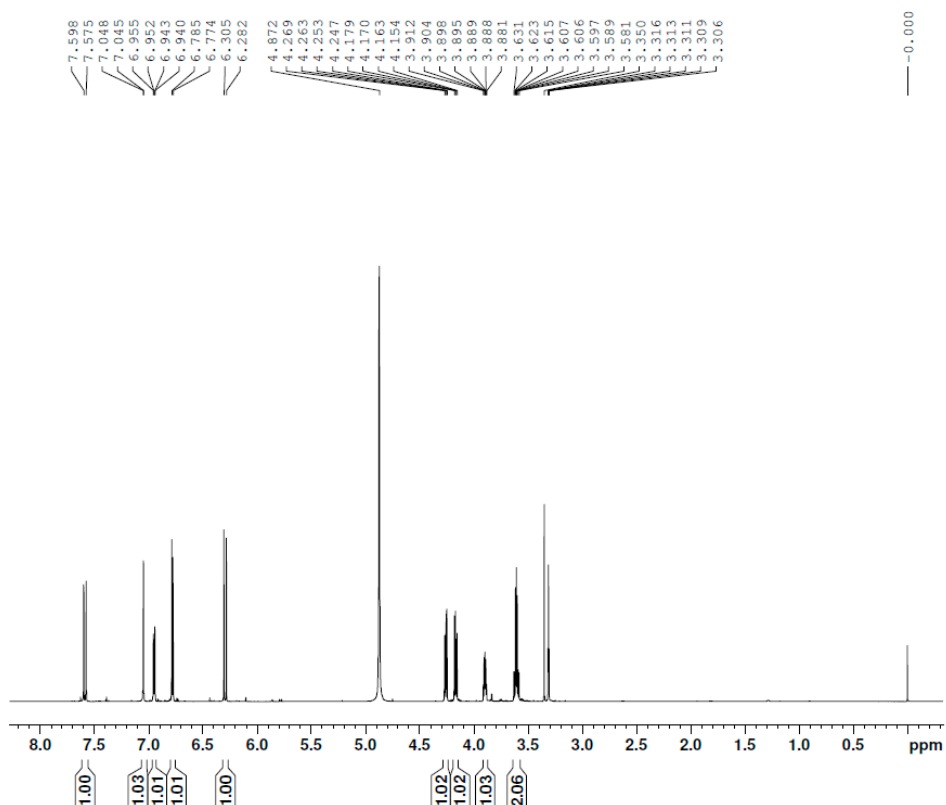
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SOLVENT MeOD
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FIDRES 1.304047 Hz
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RG 2050
DW 11.733 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 176.1660234 MHz
NUC1 13C
P1 12.00 usec
PLW1 32.86100006 W
SFO2 700.5328021 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 65.00 usec
PLW2 8.39560032 W
PLW12 0.44710001 W
PLW13 0.22513001 W

F2 - Processing parameters
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PC 1.40

Figure S9. ^{13}C -NMR spectrum (CD_3OD , 175 MHz) of compound 4

G99-15_1H



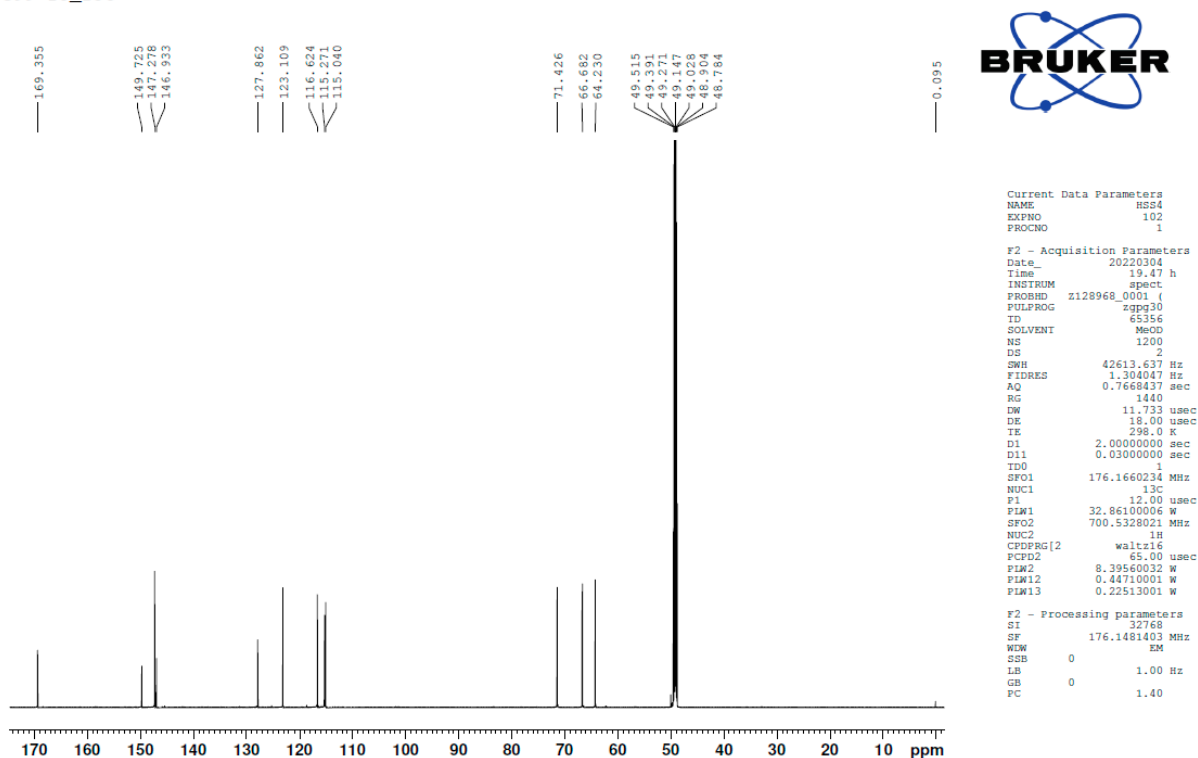
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SOLVENT MeOD
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FIDRES 0.430229 Hz
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RG 5.03
DW 35.467 usec
DE 10.00 usec
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PLW1 8.39560032 W

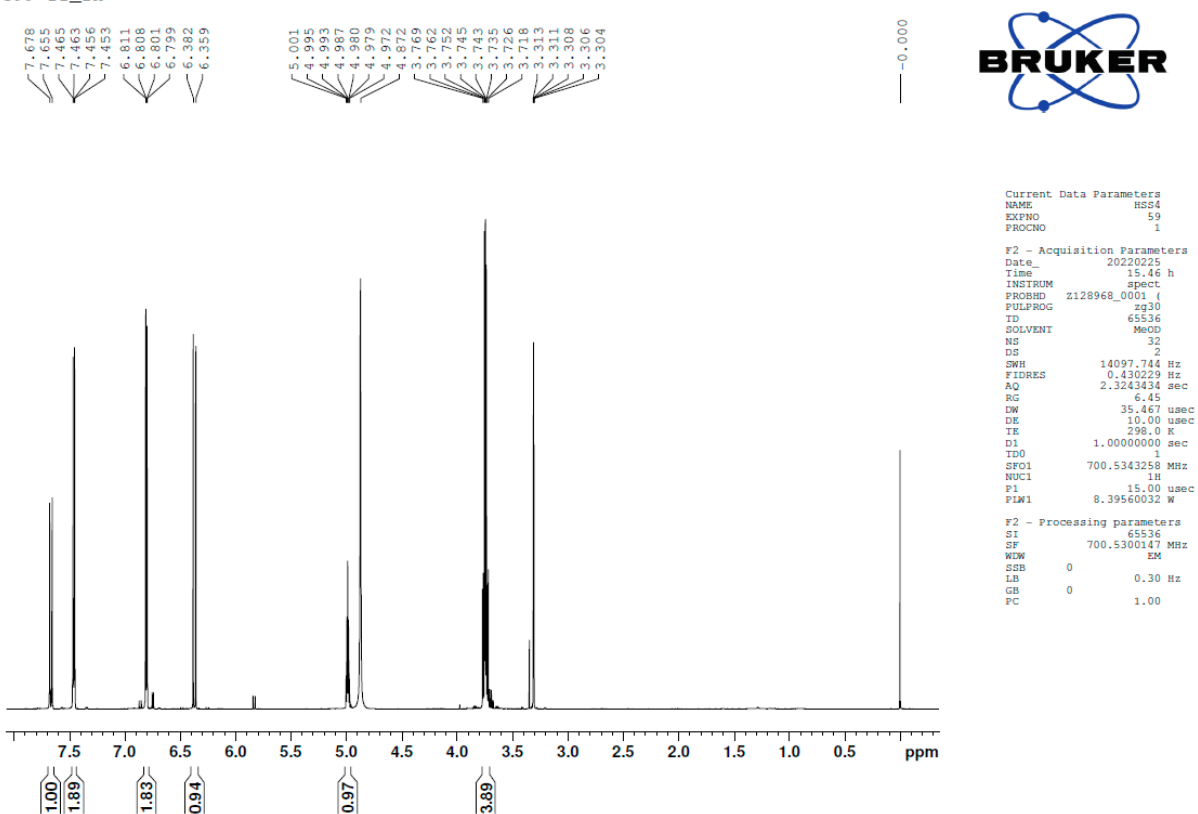
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Figure S10. ^1H -NMR spectrum (CD_3OD , 700 MHz) of compound 5

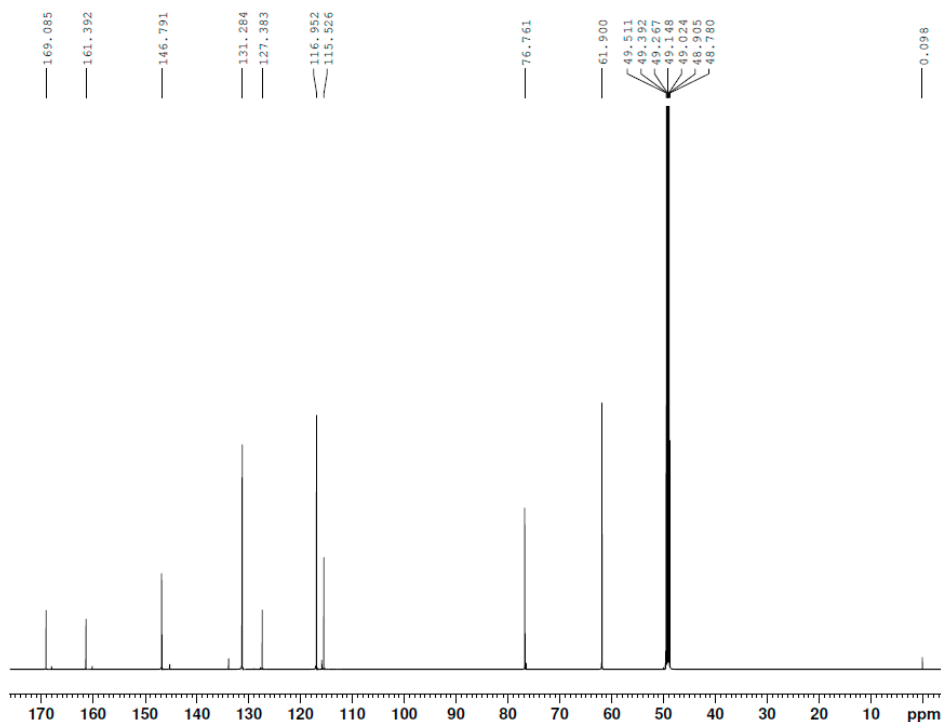
G99-15_13C

Figure S11. ^{13}C -NMR spectrum (CD_3OD , 175 MHz) of compound 5

G99-11_1H

Figure S12. ^1H -NMR spectrum (CD_3OD , 700 MHz) of compound 6

G99-11_13C



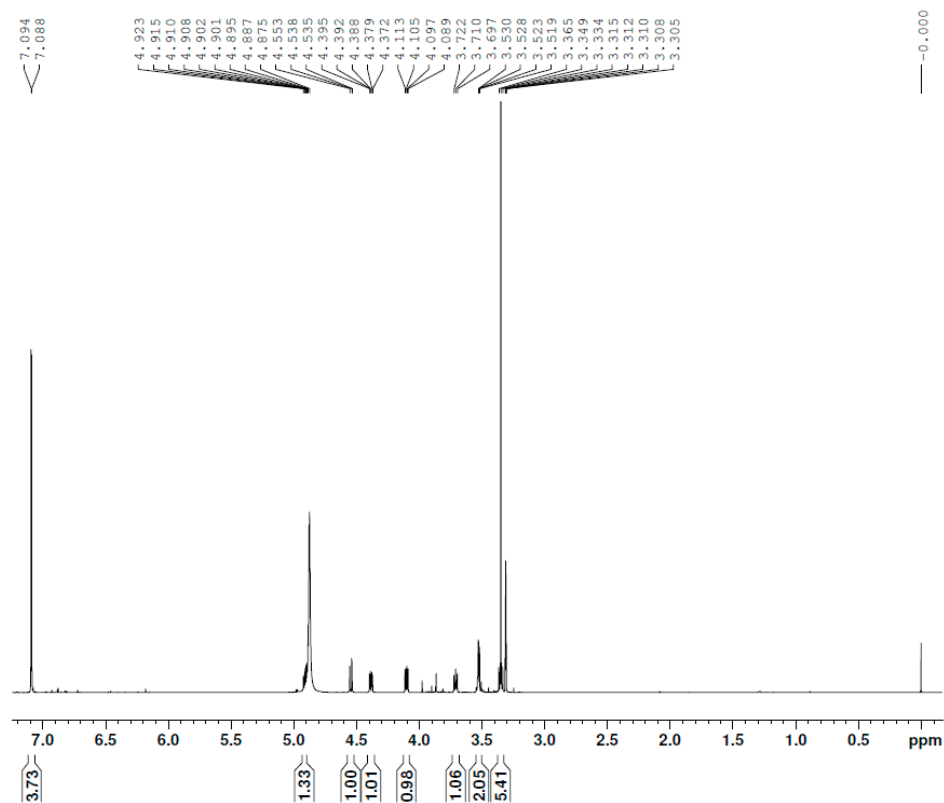
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FIDRES 1.304047 Hz
AQ 0.7668437 sec
RG 2050
DW 11.733 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 176.1660234 MHz
NUC1 13C
P1 12.00 usec
PLW1 32.86100006 W
SFO2 700.5328021 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 65.00 usec
PLW2 8.39560032 W
PLW12 0.44710001 W
PLW13 0.22513001 W

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Figure S13. ^{13}C -NMR spectrum (CD_3OD , 175 MHz) of compound 6

G99-12_1H



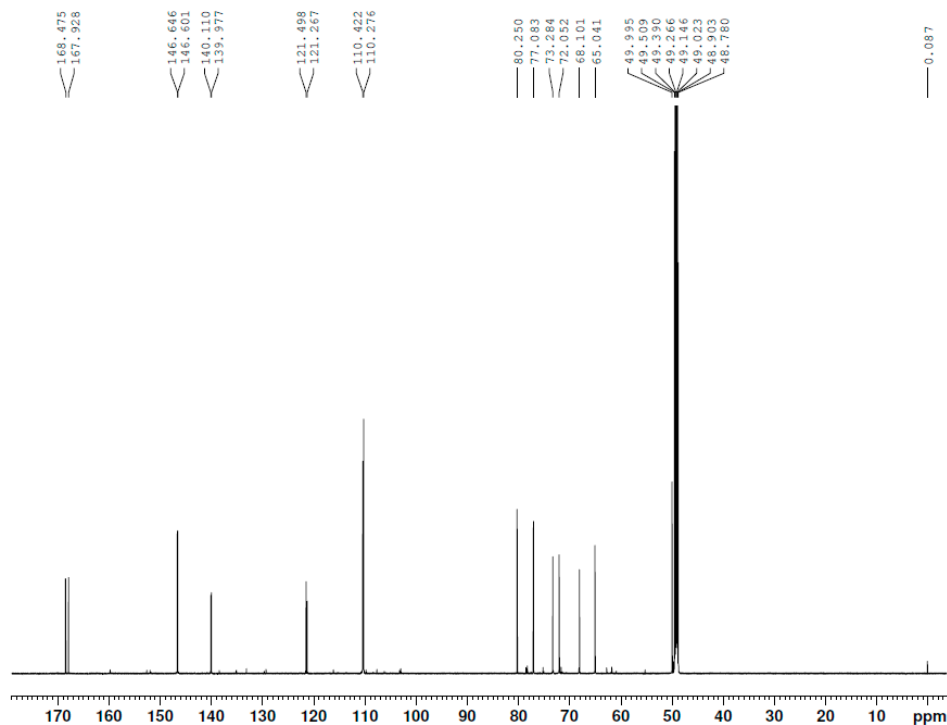
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RG 5.73
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DE 10.00 usec
TE 298.0 K
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TD0 1
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NUC1 1H
P1 15.00 usec
PLW1 8.39560032 W

F2 - Processing parameters
SI 65536
SF 700.5300134 MHz
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Figure S14. ^1H -NMR spectrum (CD_3OD , 700 MHz) of compound 7

G99-12_13C



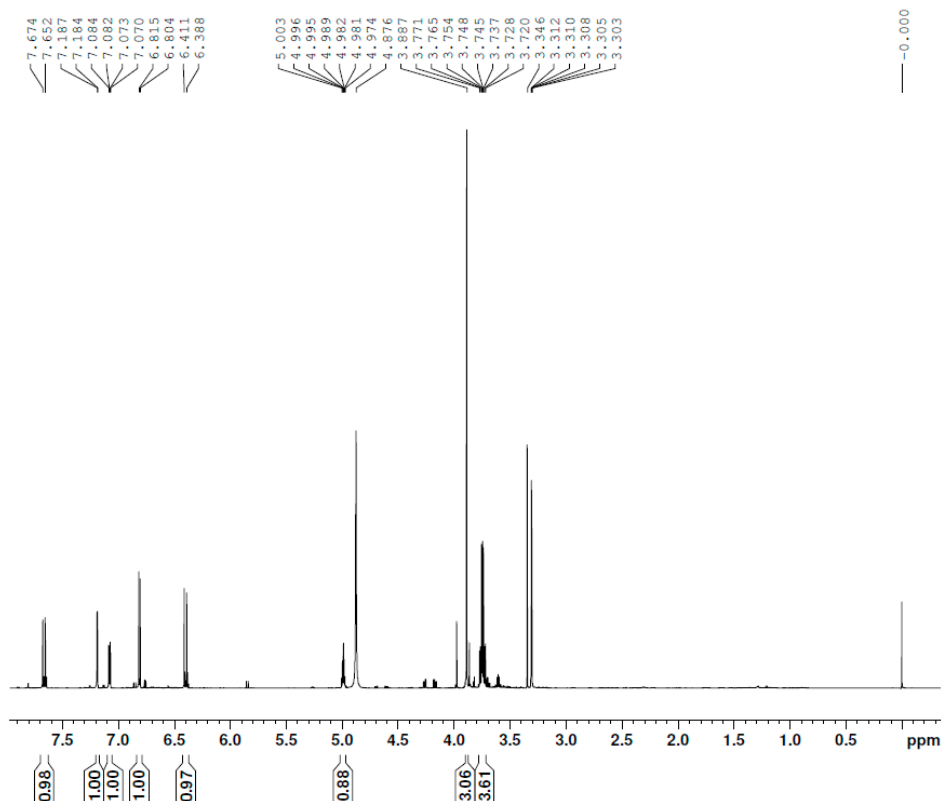
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RG 2050
DW 11.733 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 176.1660234 MHz
NUC1 13C
P1 12.00 usec
PLW1 32.86100006 W
SFO2 700.5328021 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 65.00 usec
PLW2 8.39560032 W
PLW12 0.44710001 W
PLW13 0.22513001 W

F2 - Processing parameters
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LB 1.00 Hz
GB 0
PC 1.40

Figure S15. ^{13}C -NMR spectrum (CD_3OD , 175 MHz) of compound **7**

G99-13_1H



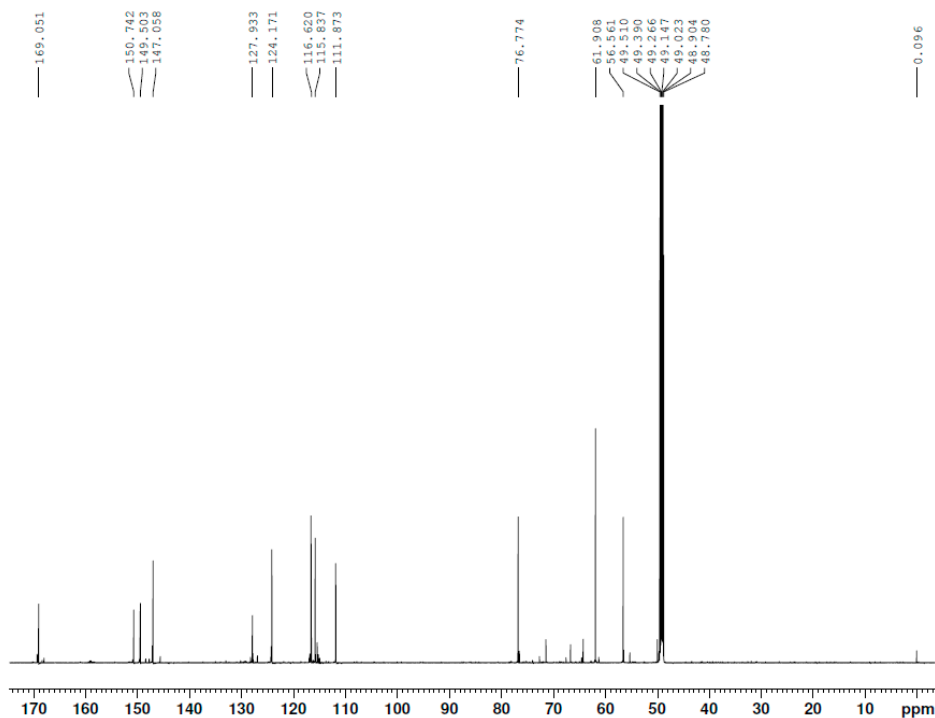
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RG 6.45
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DE 10.00 usec
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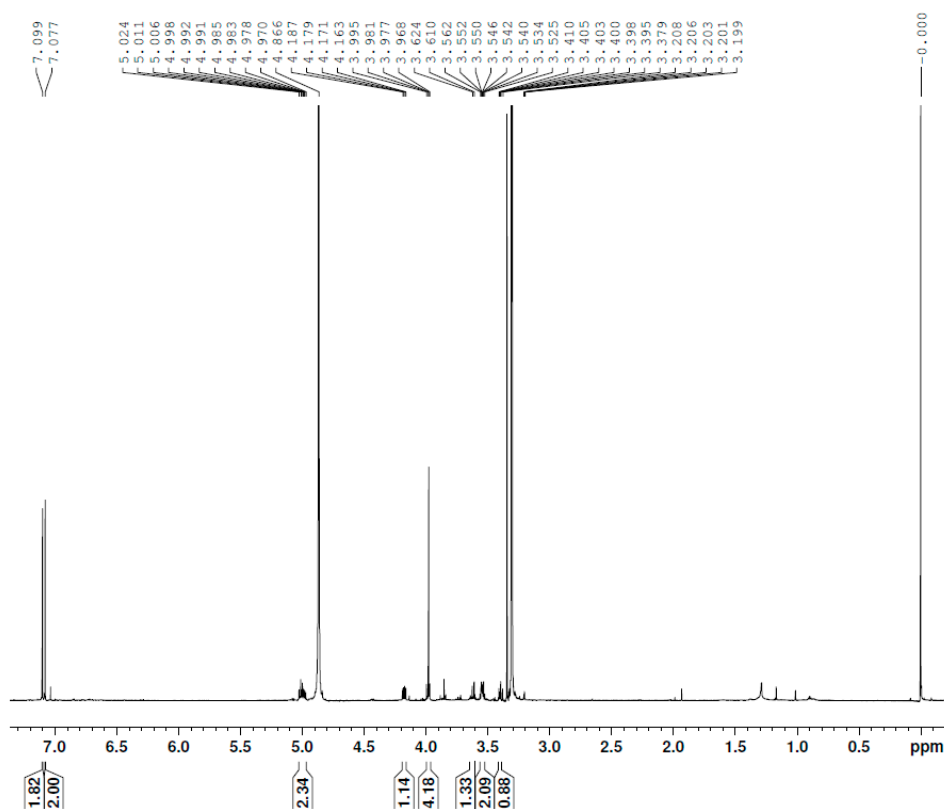
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Figure S16. ^1H -NMR spectrum (CD_3OD , 700 MHz) of compound **8**

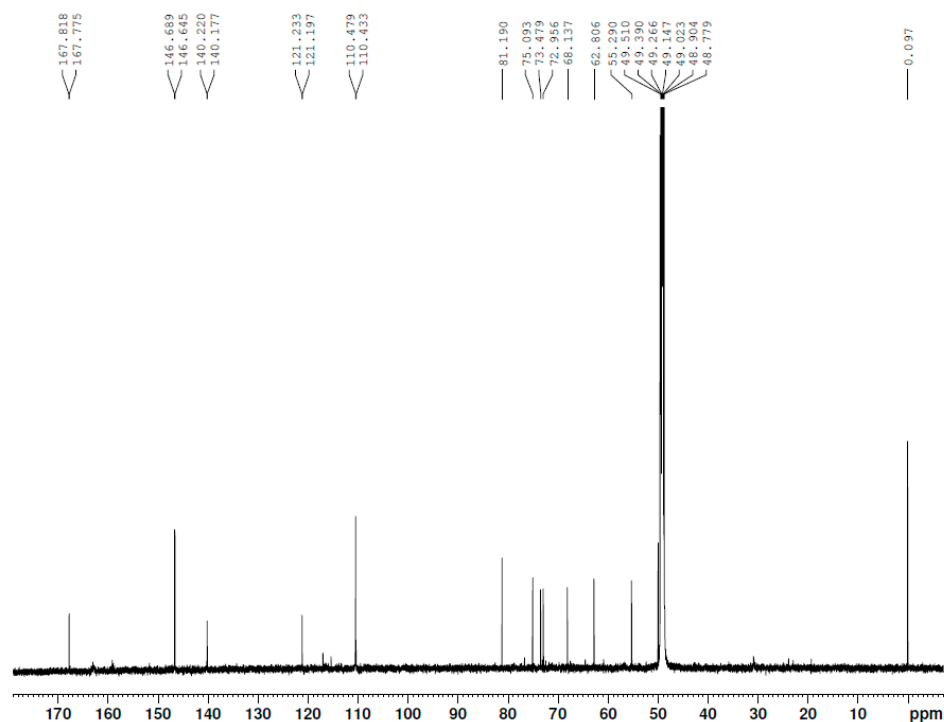
G99-13_13C

Figure S17. ^{13}C -NMR spectrum (CD_3OD , 175 MHz) of compound 8

G99-14_1H

Figure S18. ^1H -NMR spectrum (CD_3OD , 700 MHz) of compound 9

G99-14_13C



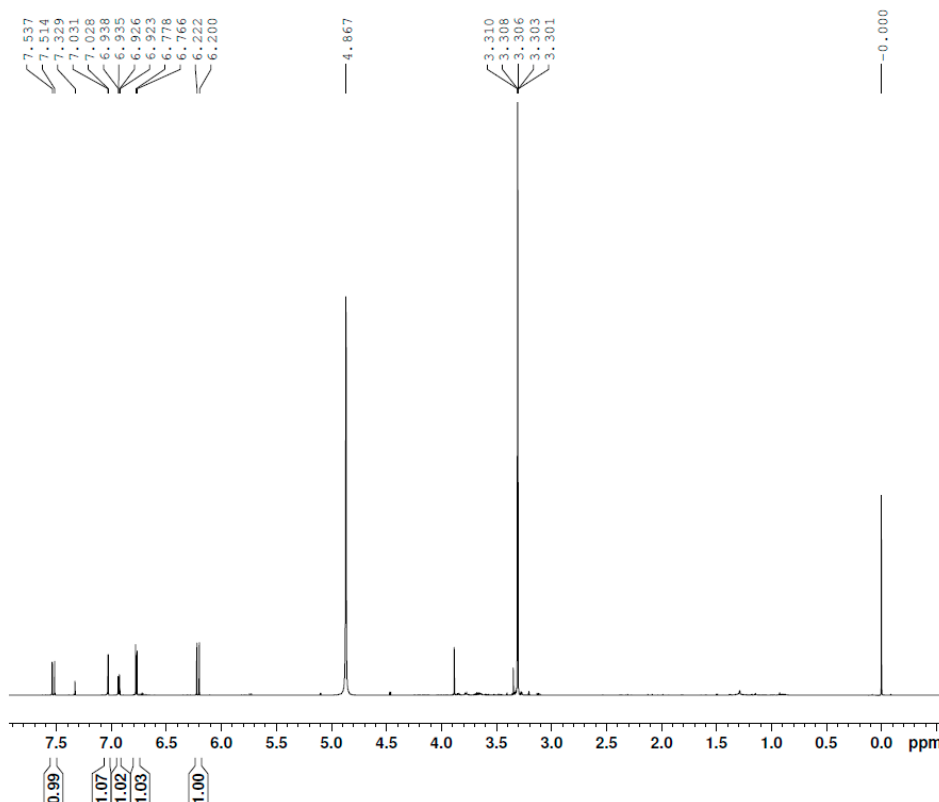
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FIDRES 1.304047 Hz
AQ 0.7668437 sec
RG 2050
DW 11.733 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0
SFO1 176.1660234 MHz
NUC1 13C
P1 12.00 usec
PLW1 32.86100006 W
SFO2 700.5328021 MHz
NUC2 1H
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SF 176.1481348 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Figure S19. ^{13}C -NMR spectrum (CD_3OD , 175 MHz) of compound **9**

G99-16_1H



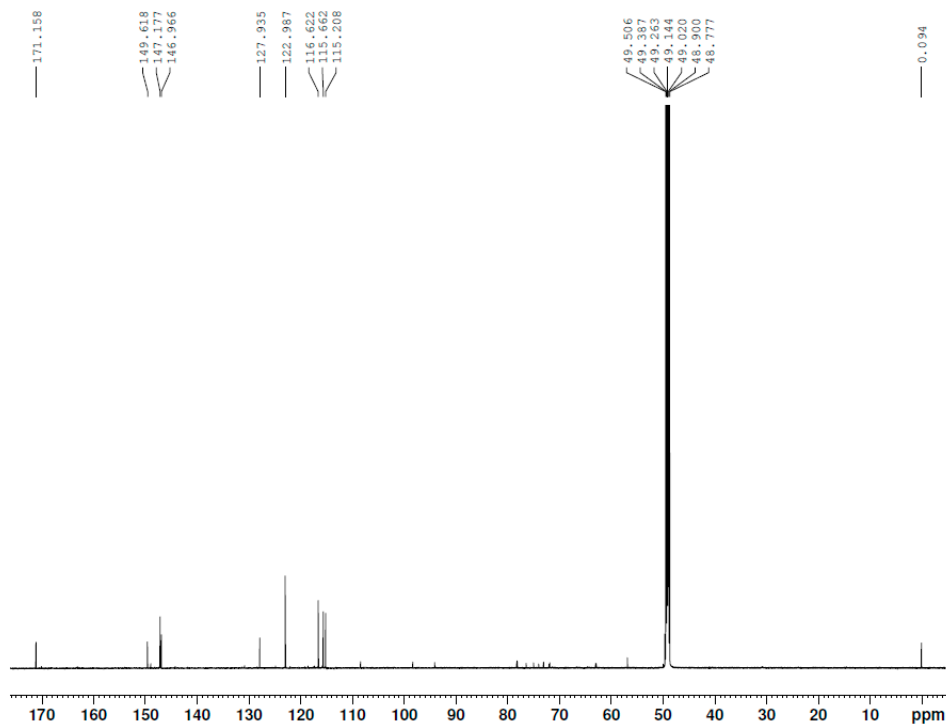
Current Data Parameters
NAME HSS4
EXPNO 108
PROCNO 1

F2 - Acquisition Parameters
Date_ 20220305
Time 1.10 h
INSTRUM spect
PROBHD z128968_0001 (
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 32
DS 2
SWH 14097.744 Hz
FIDRES 0.430229 Hz
AQ 2.3243434 sec
RG 6.45
DW 35.467 usec
DE 10.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0
SFO1 700.5343258 MHz
NUC1 1H
P1 15.00 usec
PLW1 8.39560032 W

F2 - Processing parameters
SI 65536
SF 700.5300165 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Figure S20. ^1H -NMR spectrum (CD_3OD , 700 MHz) of compound **10**

G99-16_13C



```

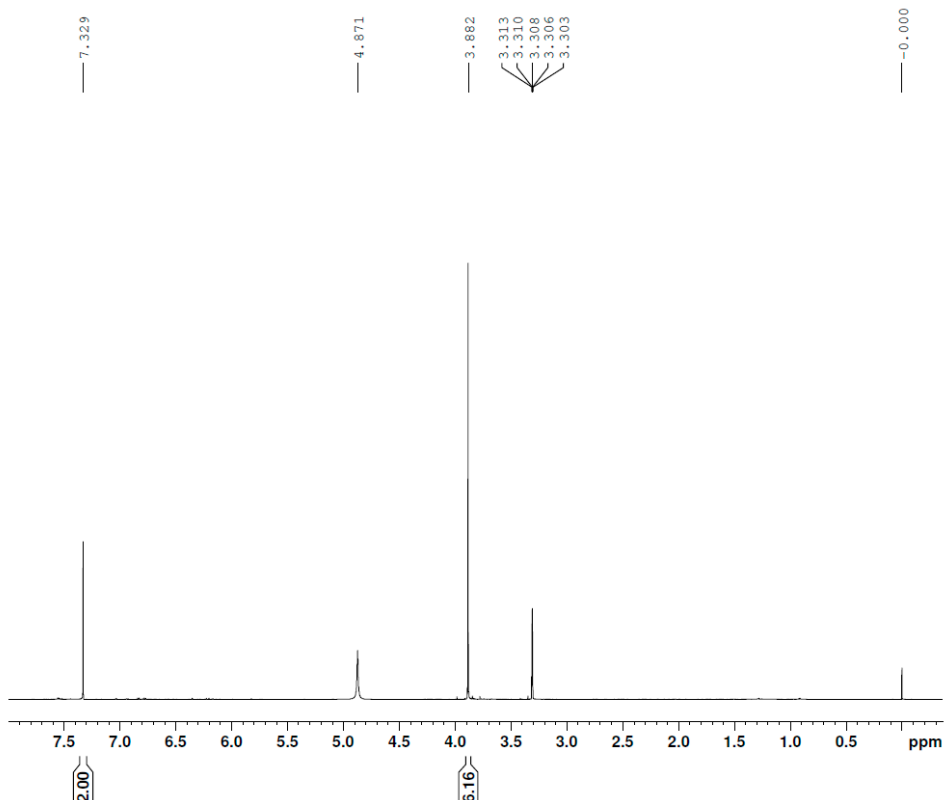
Current Data Parameters
NAME      HSS4
EXPNO     109
PROCNO    1

F2 - Acquisition Parameters
Date_     20220305
Time      7.29 h
INSTRUM   spect
PROBHD    Z128968_0001 (
PULPROG   zgpg30
TD         65536
SOLVENT   MeOD
NS         8000
DS         2
SWH        42613.637 Hz
FIDRES     1.304047 Hz
AQ         0.7668437 sec
RG         1440
RW         11.733 usec
DE         18.00 usec
TE         298.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
SF01       176.1660234 MHz
NUC1       13C
P1         12.00 usec
PLW1       32.86100006 W
SF02       700.5328021 MHz
NUC2       1H
CPDPRG2    waltz16
PCPD2      65.00 usec
PLW2       8.39560032 W
PLW12      0.44710001 W
PLW13      0.22513001 W

F2 - Processing parameters
SI         32768
SF         176.1481354 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```

Figure S21. ^{13}C -NMR spectrum (CD_3OD , 175 MHz) of compound **10**

G99-17_1H



```

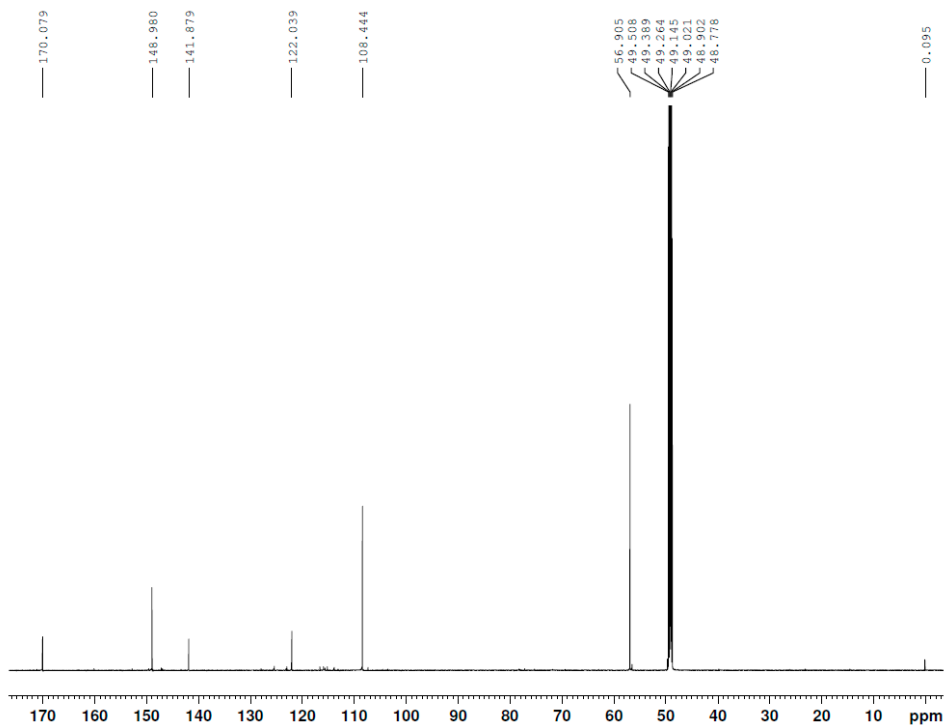
Current Data Parameters
NAME      HSS4
EXPNO     115
PROCNO    1

F2 - Acquisition Parameters
Date_     20220306
Time      2.50 h
INSTRUM   spect
PROBHD    Z128968_0001 (
PULPROG   zg30
TD         65536
SOLVENT   MeOD
NS         32
DS         2
SWH        14097.744 Hz
FIDRES     0.430229 Hz
AQ         2.3243434 sec
RG         6.45
RW         35.467 usec
DE         10.00 usec
TE         298.0 K
D1         1.00000000 sec
TD0        1
SF01       700.5343258 MHz
NUC1       1H
P1         15.00 usec
PLW1       8.39560032 W

F2 - Processing parameters
SI         65536
SF         700.5300147 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
```

Figure S22. ^1H -NMR spectrum (CD_3OD , 700 MHz) of compound **11**

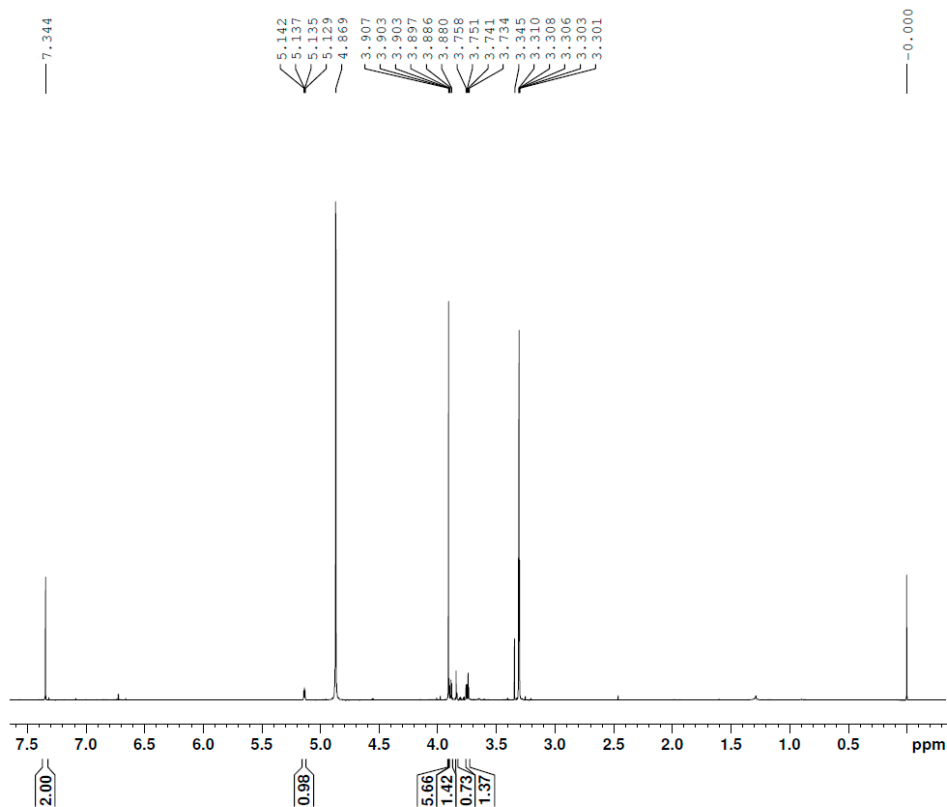
G99-17_13C



Current Data Parameters
NAME HSS4
EXPNO 116
PROCNO 1
F2 - Acquisition Parameters
Date_ 20220306
Time 4.26 h
INSTRUM spect
PROBHD Z128968_0001 (
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 2000
DS 2
SWH 42613.637 Hz
FIDRES 1.304047 Hz
AQ 0.7668437 sec
RG 12050
DW 11.733 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TDO 1
SFO1 176.1660234 MHz
NUC1 13C
P1 12.00 usec
PLM1 32.8610006 W
SFO2 700.5328021 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 65.00 usec
PLW2 8.39560032 W
PLW12 0.44710001 W
PLW13 0.22513001 W
F2 - Processing parameters
SI 32768
SF 176.1481364 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Figure S23. ^{13}C -NMR spectrum (CD_3OD , 175 MHz) of compound **11**

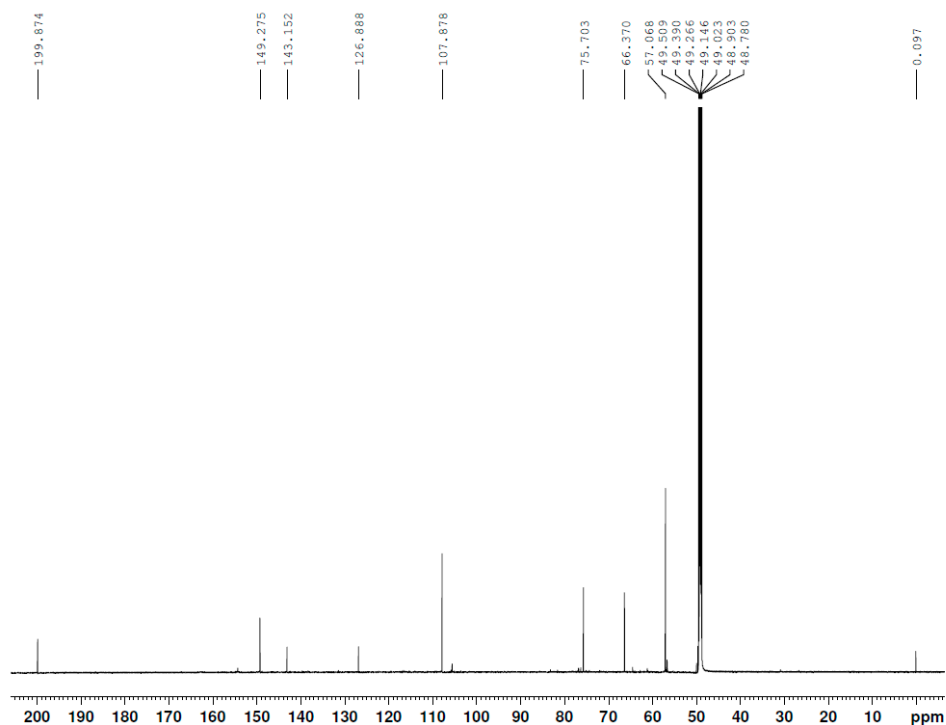
G99-18_1H



Current Data Parameters
NAME HSS4
EXPNO 122
PROCNO 1
F2 - Acquisition Parameters
Date_ 20220306
Time 10.29 h
INSTRUM spect
PROBHD Z128968_0001 (
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 32
DS 2
SWH 14097.744 Hz
FIDRES 0.430229 Hz
AQ 2.3243434 sec
RG 6.45
DW 35.467 usec
DE 10.00 usec
TE 298.0 K
D1 1.00000000 sec
TDO 1
SFO1 700.5343258 MHz
NUC1 1H
P1 15.00 usec
PLW1 8.39560032 W
F2 - Processing parameters
SI 65536
SF 700.5300164 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Figure S24. ^1H -NMR spectrum (CD_3OD , 700 MHz) of compound **12**

G99-18_13C



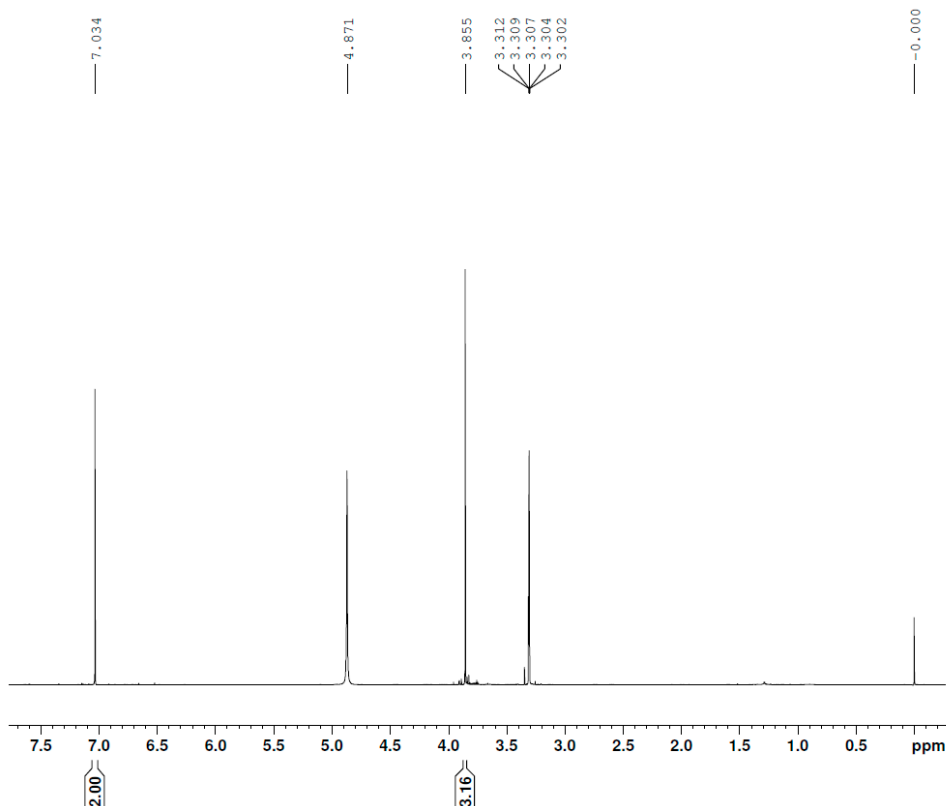
Current Data Parameters
NAME H554
EXPNO 123
PROCNO 1

F2 - Acquisition Parameters
Date_ 20220306
Time 16.48 h
INSTRUM spect
PROBHD Z128968_0001 (
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 8000
DS 2
SWH 42613.637 Hz
FIDRES 1.304047 Hz
AQ 0.7668437 sec
RG 1440
DW 11.733 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 176.1660234 MHz
NUC1 13C
P1 12.00 usec
PLW1 32.86100006 W
SFO2 700.5328021 MHz
NUC2 1H
CPDPRG2 waitt16
PCPD2 65.00 usec
PLW2 8.39560032 W
PLW12 0.44710001 W
PLW13 0.22513001 W

F2 - Processing Parameters
SI 32768
SF 176.1481350 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Figure S25. ^{13}C -NMR spectrum (CD_3OD , 175 MHz) of compound **12**

G99-19_1H



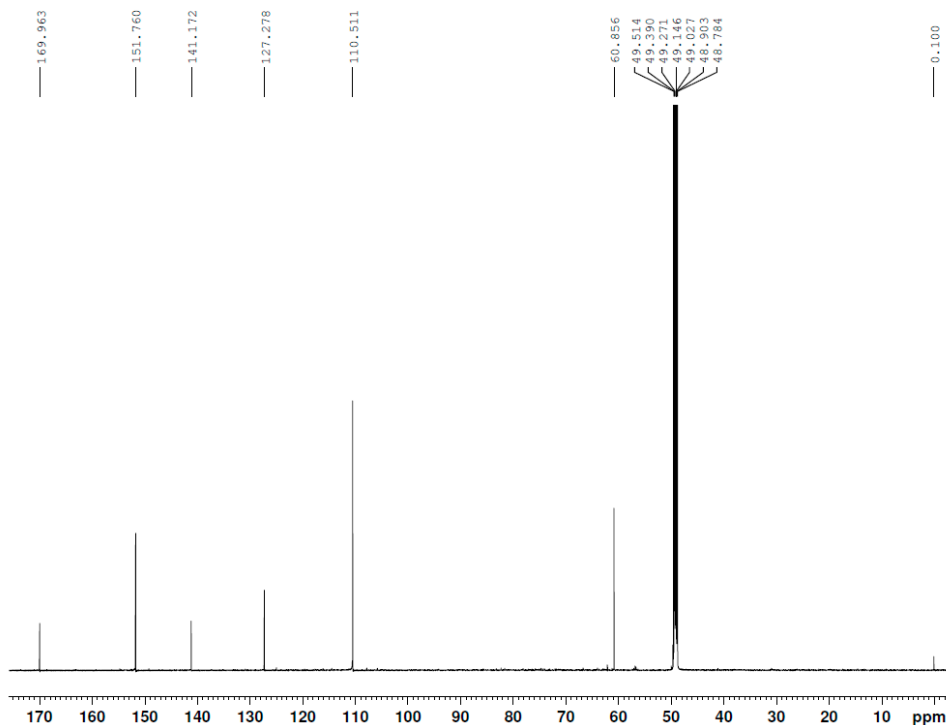
Current Data Parameters
NAME H554
EXPNO 129
PROCNO 1

F2 - Acquisition Parameters
Date_ 20220307
Time 12.11 h
INSTRUM spect
PROBHD Z128968_0001 (
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 32
DS 2
SWH 14097.744 Hz
FIDRES 0.430229 Hz
AQ 2.3243434 sec
RG 6.45
DW 35.467 usec
DE 10.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
SFO1 700.5343258 MHz
NUC1 1H
P1 15.00 usec
PLW1 8.39560032 W

F2 - Processing Parameters
SI 65536
SF 700.5300156 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Figure S26. ^1H -NMR spectrum (CD_3OD , 700 MHz) of compound **13**

G99-19_13C



```

Current Data Parameters
NAME      HSS4
EXPNO     130
PROCNO    1

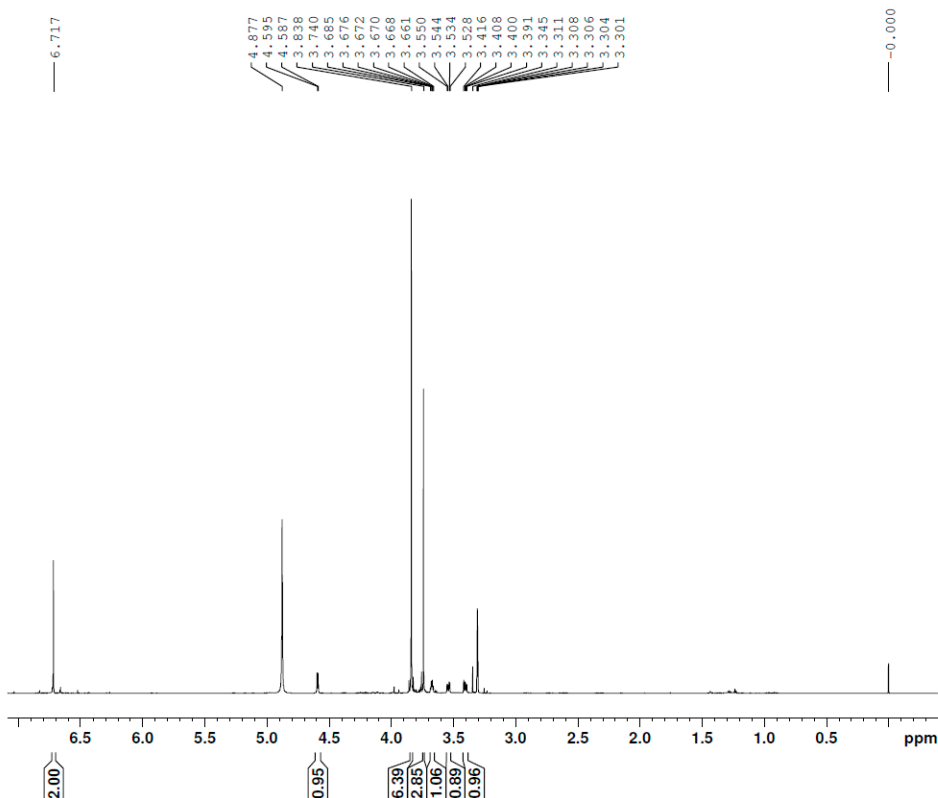
F2 - Acquisition Parameters
Date_     20220307
Time      15.21 h
INSTRUM   spect
PROBHD    Z128968_0001 (
PULPROG   zgpg30
TD         65536
SOLVENT    MeOD
NS         4000
DS         2
SWH        42613.637 Hz
FIDRES     1.304047 Hz
AQ         0.7668437 sec
RG         2050
DW         11.733 usec
DE         18.00 usec
TE         298.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
SF01       176.1660234 MHz
NUC1       13C
P1         12.00 usec
PLW1       32.96100006 W
SF02       700.5328021 MHz
NUC2       1H
CPDPRG2    waltz16
PCPD2      65.00 usec
PLW2       8.39560032 W
PLW12      0.44710001 W
PLW13      0.22513001 W

F2 - Processing parameters
SI         32768
SF         176.1481351 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40

```

Figure S27. ^{13}C -NMR spectrum (CD_3OD , 175 MHz) of compound **13**

G99-20_1H



```

Current Data Parameters
NAME      HSS4
EXPNO     136
PROCNO    1

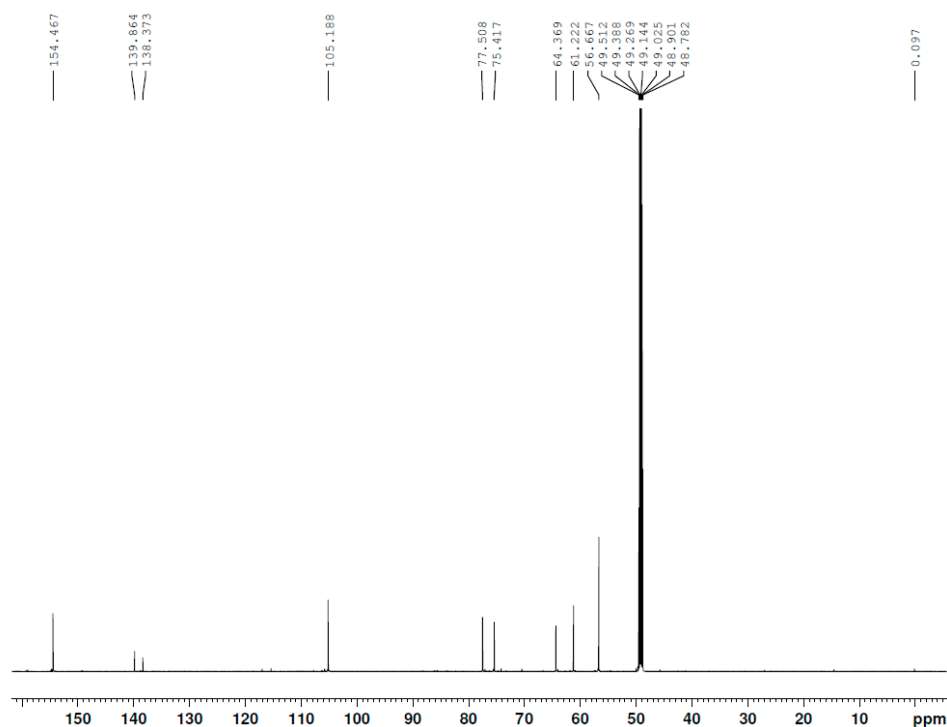
F2 - Acquisition Parameters
Date_     20220308
Time      3.12 h
INSTRUM   spect
PROBHD    Z128968_0001 (
PULPROG   zg30
TD         65536
SOLVENT    MeOD
NS         32
DS         2
SWH        14097.744 Hz
FIDRES     0.430229 Hz
AQ         2.3243434 sec
RG         6.45
DW         35.467 usec
DE         10.00 usec
TE         298.0 K
D1         1.00000000 sec
D11        1
SF01       700.5343258 MHz
NUC1       1H
P1         15.00 usec
PLW1       8.39560032 W

F2 - Processing parameters
SI         65536
SF         700.5300162 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00

```

Figure S28. ^1H -NMR spectrum (CD_3OD , 700 MHz) of compound **14**

G99-20_13C



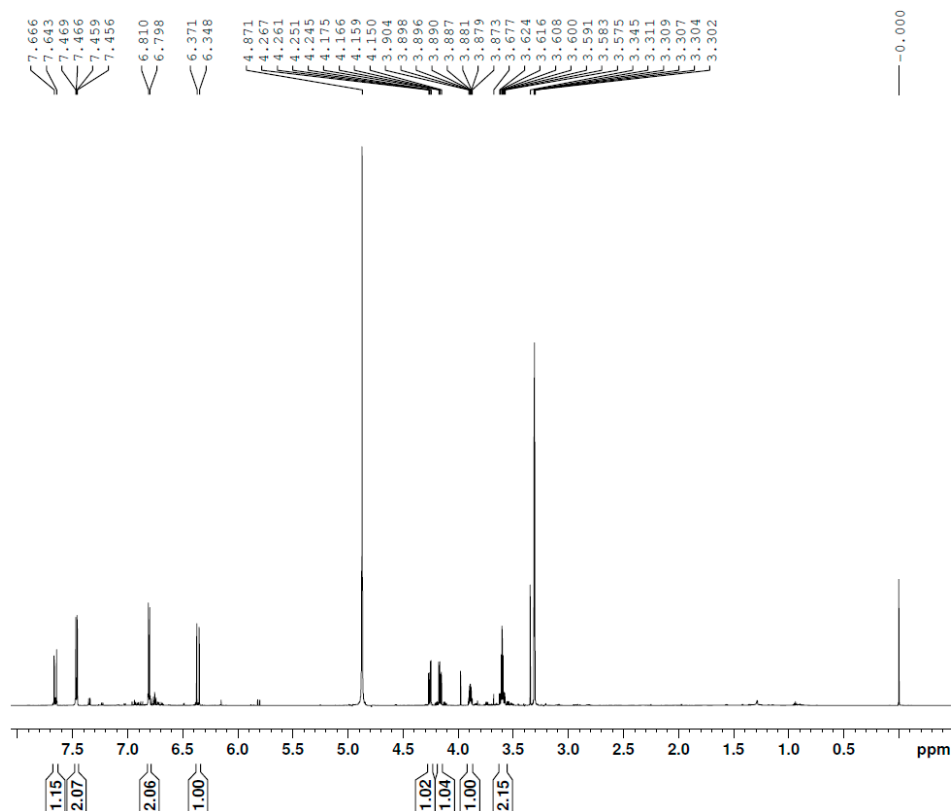
Current Data Parameters
NAME HSS4
EXPNO 137
PROCNO 1

F2 - Acquisition Parameters
Date_ 20220308
Time 4.29 h
INSTRUM spect
PROBHD Z128968_0001 ()
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 1600
DS 2
SWH 42613.637 Hz
FIDRES 1.304047 Hz
AQ 0.7668437 sec
RG 2050
DW 11.733 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SF01 176.1660234 MHz
NUC1 13C
P1 12.00 usec
PLW1 32.86100006 W
SF02 700.5328021 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 65.00 usec
PLW2 8.39560032 W
PLW12 0.44710001 W
PLW13 0.22513001 W

F2 - Processing parameters
SI 32768
SF 176.1481368 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Figure S29. ^{13}C -NMR spectrum (CD_3OD , 175 MHz) of compound **14**

G99-22_1H



Current Data Parameters
NAME HSS4
EXPNO 150
PROCNO 1

F2 - Acquisition Parameters
Date_ 20220308
Time 17.56 h
INSTRUM spect
PROBHD Z128968_0001 ()
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 32
DS 2
SWH 14097.744 Hz
FIDRES 0.430229 Hz
AQ 2.3243434 sec
RG 6.45
DW 35.467 usec
DE 10.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
SF01 700.5343258 MHz
NUC1 1H
P1 15.00 usec
PLW1 8.39560032 W

F2 - Processing parameters
SI 65536
SF 700.5300158 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Figure S30. ^1H -NMR spectrum (CD_3OD , 700 MHz) of compound **15**

G99-22_13C

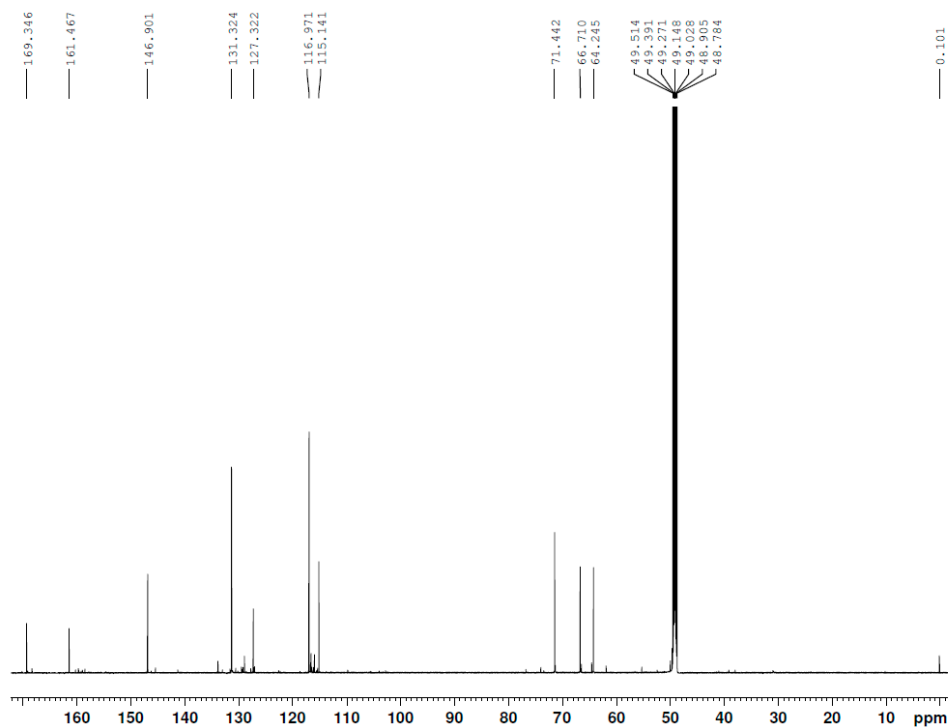


Figure S31. ^{13}C -NMR spectrum (CD_3OD , 175 MHz) of compound **15**

G99-10_1H

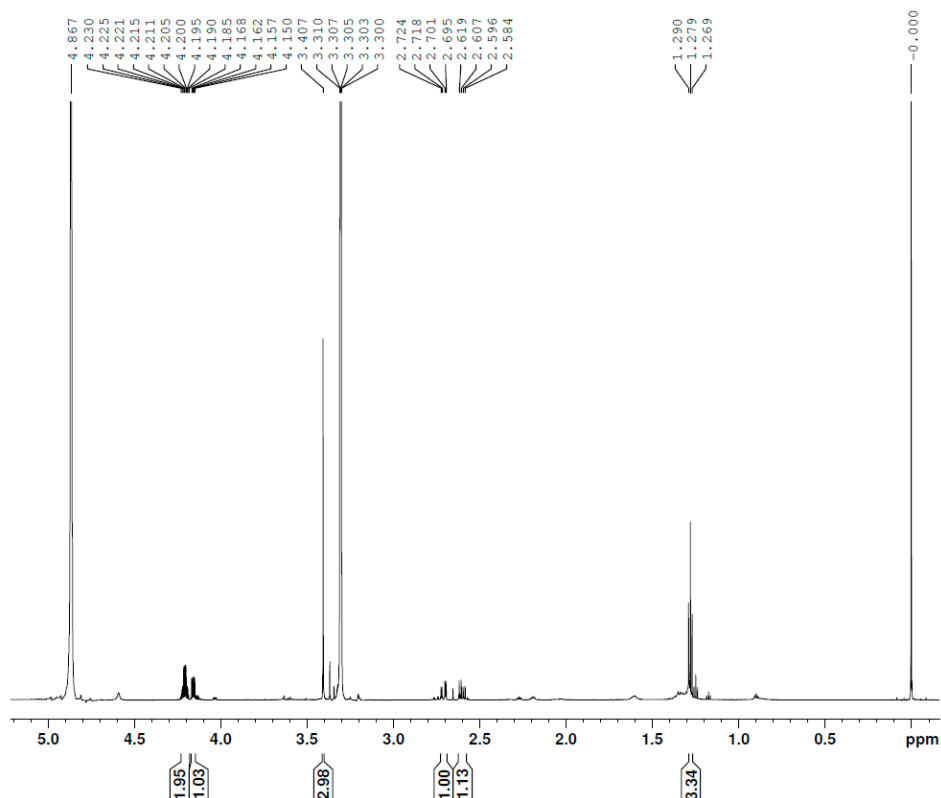
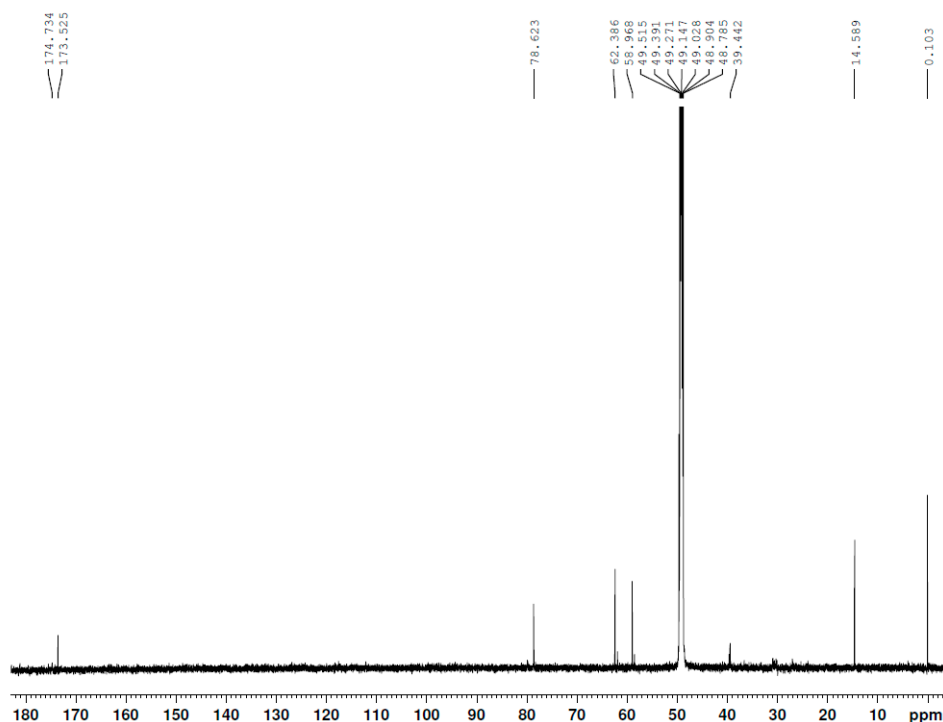


Figure S32. ^1H -NMR spectrum (CD_3OD , 700 MHz) of compound **16**

G99-10_13C



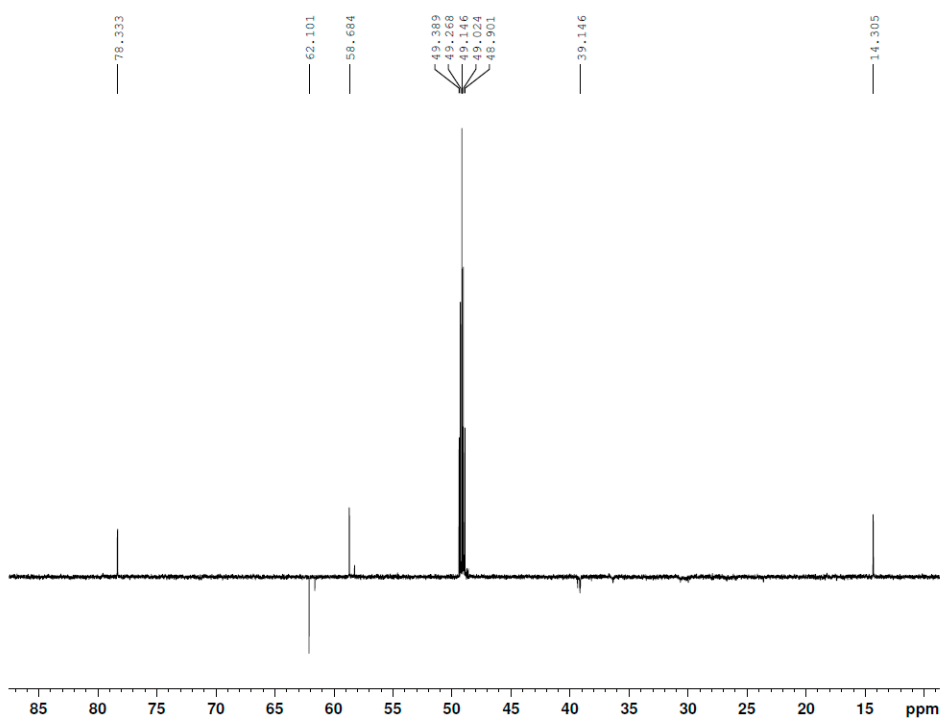
Current Data Parameters
NAME HSS4
EXPNO 294
PROCNO 1

F2 - Acquisition Parameters
Date_ 20220714
Time 23.39 h
INSTRUM spect
PROBHD z128968_0001 ()
PULPROG zgpg30
TD 65356
SOLVENT MeOD
NS 6000
DS 2
SWH 42613.637 Hz
FIDRES 1.304047 Hz
AQ 0.7668437 sec
RG 1440
DW 11.733 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 176.1660234 MHz
NUC1 13C
P1 12.00 usec
PLW1 32.86100006 W
SFO2 700.5328021 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 65.00 usec
PLW2 8.39560032 W
PLW12 0.44710001 W
PLW13 0.22513001 W

F2 - Processing parameters
SI 32768
SF 176.1481337 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Figure S33. ^{13}C -NMR spectrum (CD_3OD , 175 MHz) of compound 16

G99-10_DEPT135



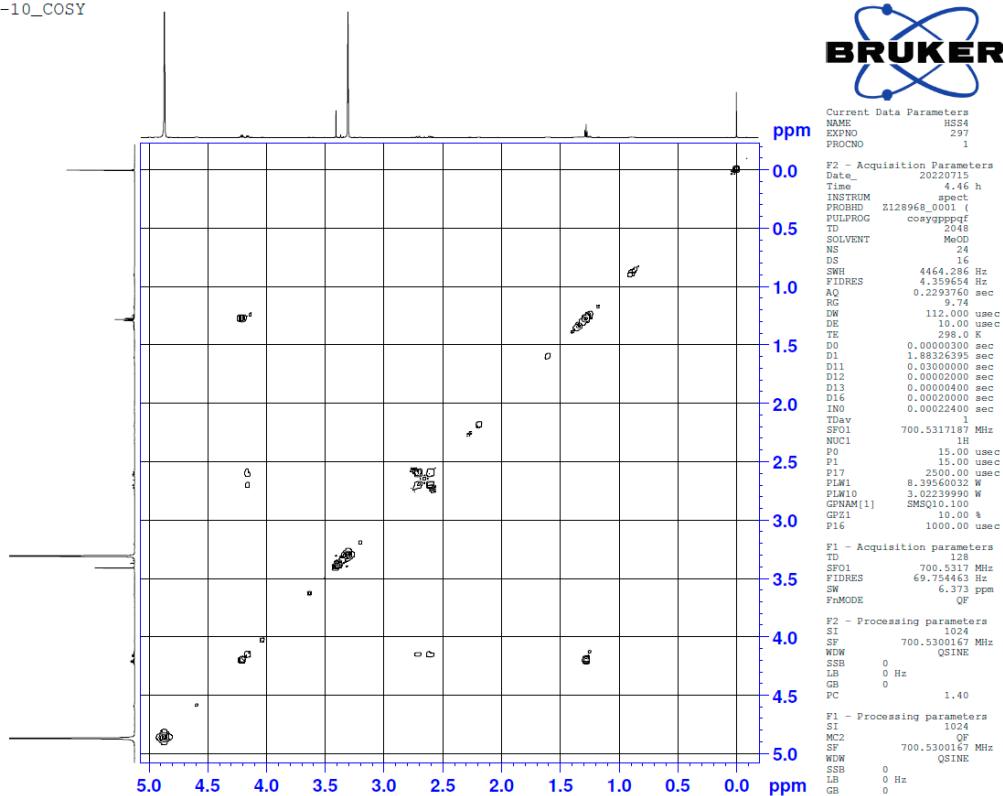
Current Data Parameters
NAME HSS4
EXPNO 296
PROCNO 1

F2 - Acquisition Parameters
Date_ 20220715
Time 4.44 h
INSTRUM spect
PROBHD z128968_0001 ()
PULPROG deptsp135
TD 65356
SOLVENT MeOD
NS 3000
DS 0
SWH 28409.092 Hz
FIDRES 0.866977 Hz
AQ 1.1534336 sec
RG 2050
DW 17.600 usec
DE 18.00 usec
TE 298.0 K
CNST2 145.0000000
D1 2.00000000 sec
D2 0.00344820 sec
D12 0.00002000 sec
TD0 1
SFO1 176.1625005 MHz
NUC1 13C
P1 12.00 usec
P13 2000.00 usec
PLW0 0 W
PLW1 32.86100006 W
SPNAM[5] Crp80comp.4
SFOAL5 0.500
SFOF5 0 Hz
SPW5 9.64000034 W
SFO2 700.5328021 MHz
NUC2 1H
CPDPRG2 waltz16
P3 15.00 usec
P4 30.00 usec
PCPD2 65.00 usec
PLW2 8.39560032 W
PLW12 0.44710001 W

F2 - Processing parameters
SI 32768
SF 176.1481342 MHz
WDW EM
GB 0

Figure S34. DEPT135 spectrum of compound 16

G99-10_COSY

Figure S35. ^1H - ^1H COSY spectrum of compound 16

G99-10_HSQC

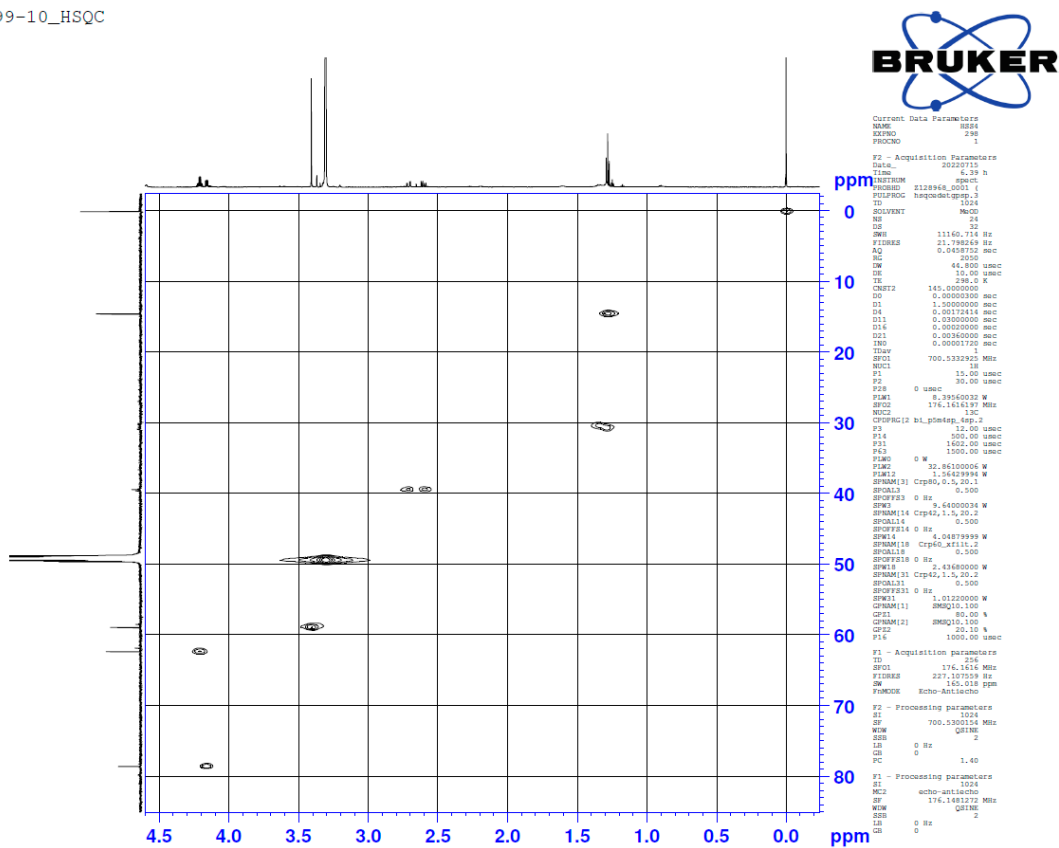


Figure S36. HSQC spectrum of compound 16

G99-10_HMBC

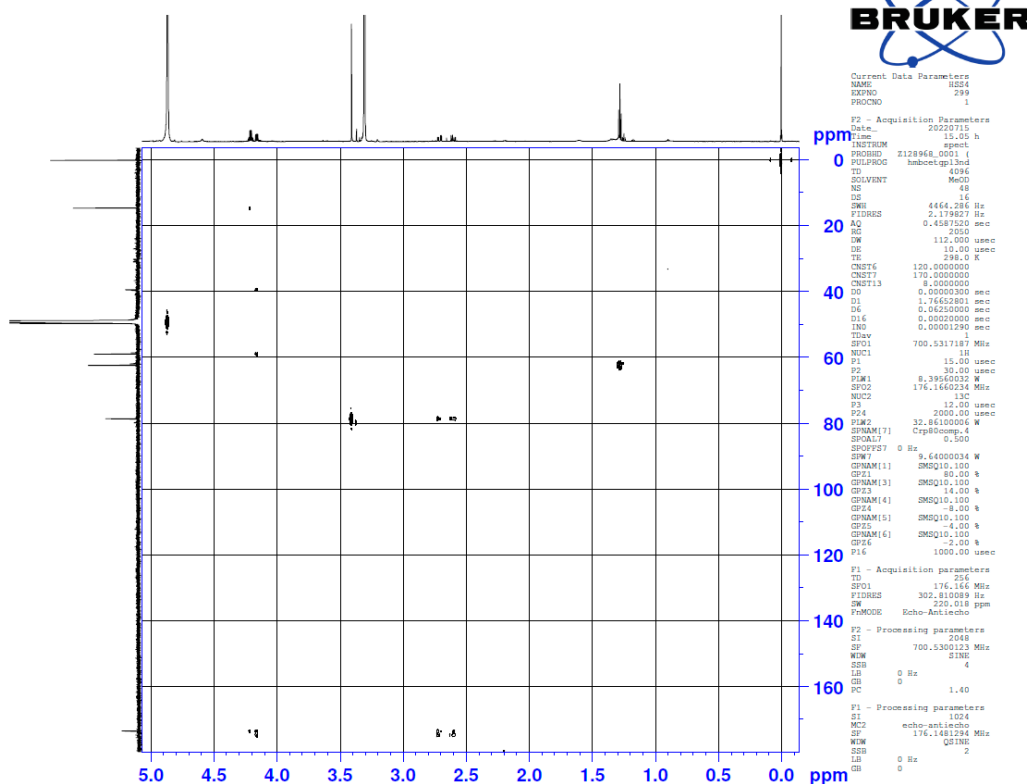


Figure S37. HMBC spectrum of compound 16

G99-10_HMBC

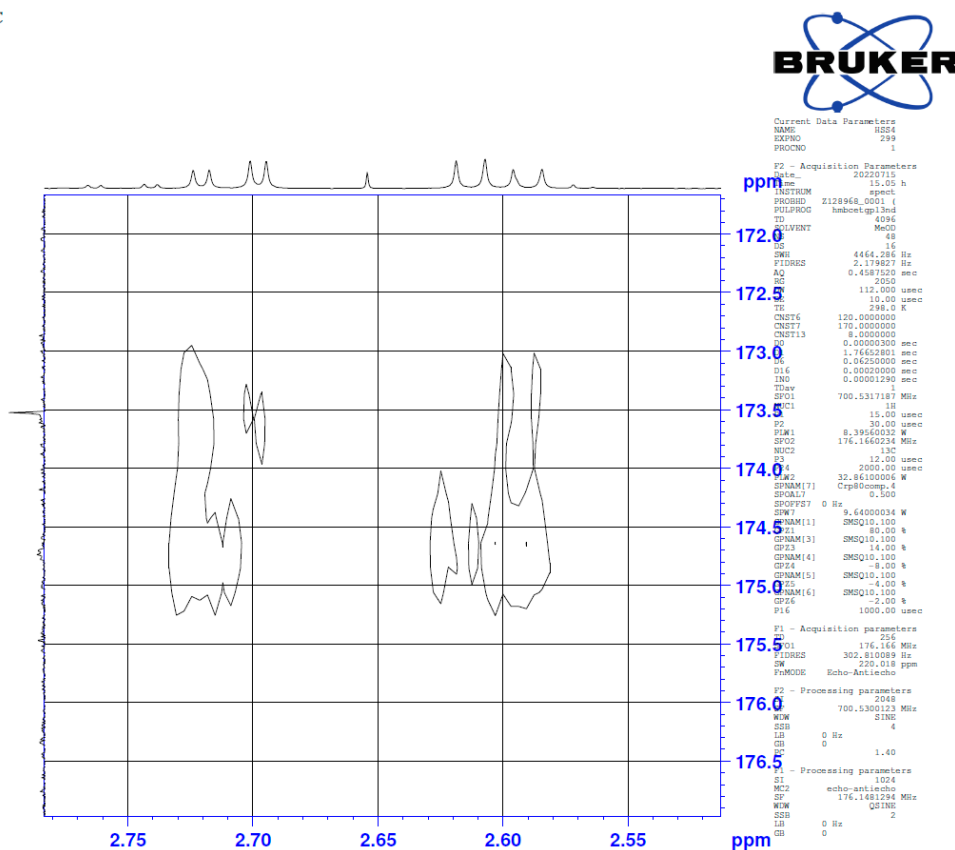


Figure S38. Expansion of HMBC spectrum of compound 16

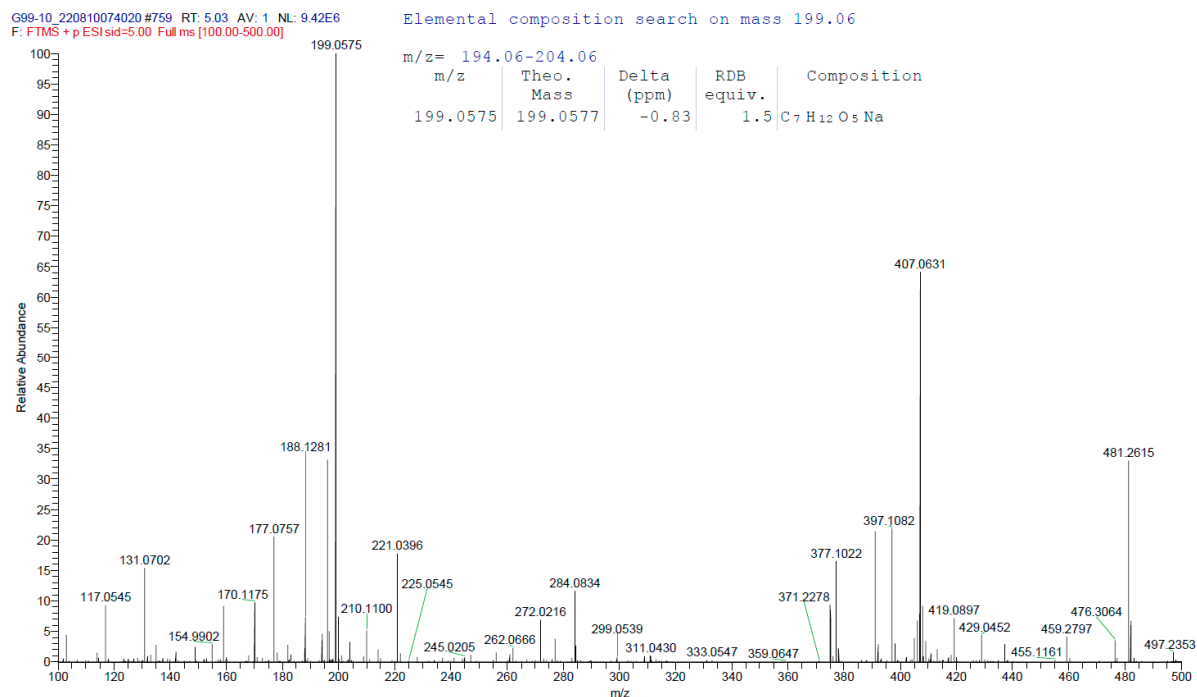


Figure S39. HRESIMS spectrum of compound 16

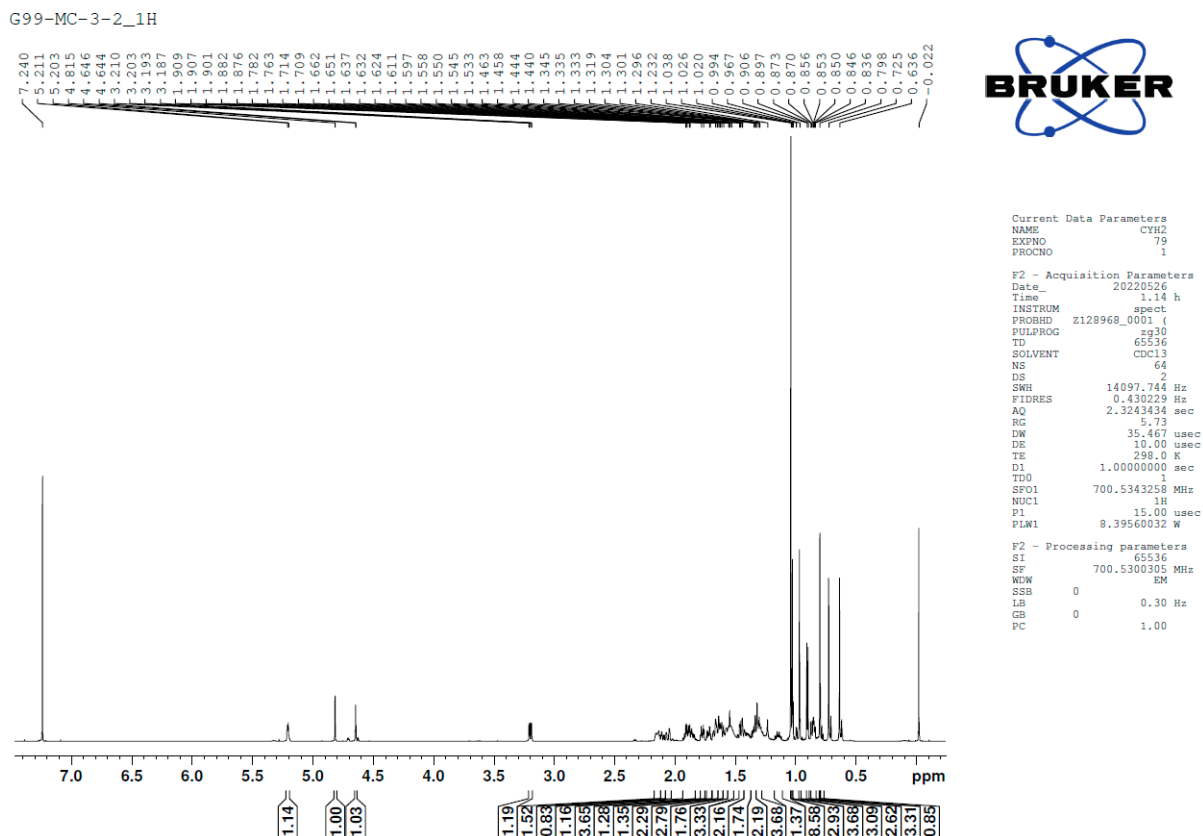
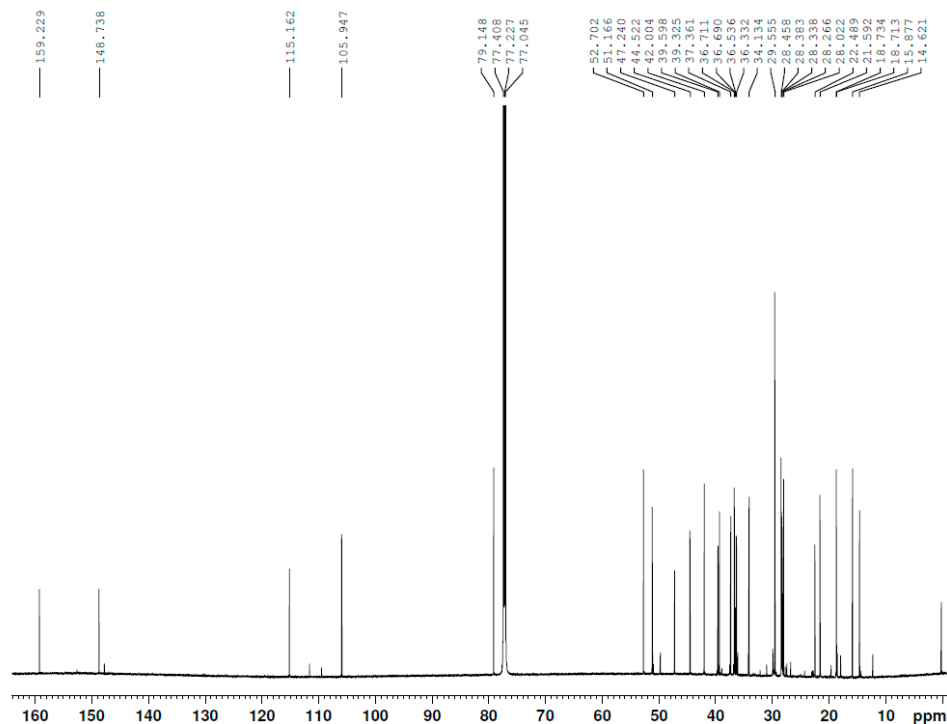


Figure S40. ¹H-NMR spectrum (CDCl₃, 700 MHz) of compound 17

G99-MC-3-2_13C



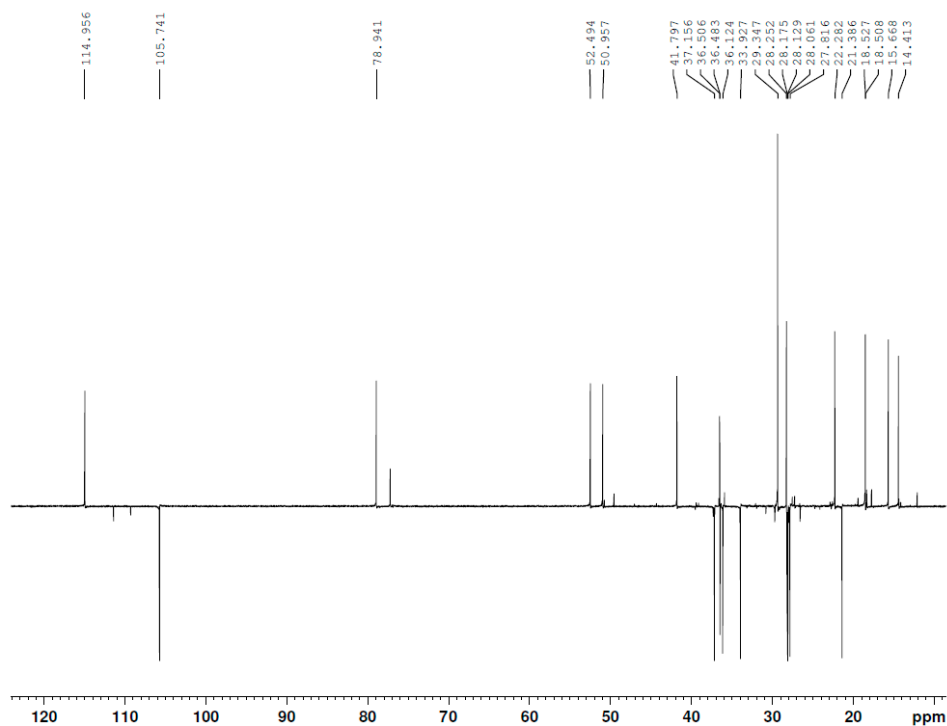
Current Data Parameters
NAME CYH2
EXPNO 80
PROCNO 1

F2 - Acquisition Parameters
Date_ 20220526
Time 6.00 h
INSTRUM spect
PROBHD Z128968_0001 ()
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 6000
DS 2
SWH 42613.637 Hz
FIDRES 1.300465 Hz
AQ 0.7689557 sec
RG 2050
DE 11.733 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 176.1660234 MHz
NUC1 13C
P1 12.00 usec
PLW1 32.86100006 W
SFO2 700.5328021 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 65.00 usec
PLW2 8.39560032 W
PLW12 0.44710001 W
PLW13 0.22513001 W

F2 - Processing parameters
SI 32768
SF 176.1483722 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Figure S41. ^{13}C -NMR spectrum (CDCl_3 , 175 MHz) of compound **17**

G99-MC-3-2_DEPT135



Current Data Parameters
NAME CYH2
EXPNO 84
PROCNO 1

F2 - Acquisition Parameters
Date_ 20220527
Time 14.36 h
INSTRUM spect
PROBHD Z128968_0001 ()
PULPROG deptsp135
TD 65536
SOLVENT CDCl3
NS 3634
DS 8
SWH 28409.092 Hz
FIDRES 0.866977 Hz
AQ 1.1534336 sec
RG 2050
DE 17.600 usec
TE 298.0 K
D1 145.0000000 sec
D11 2.00000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
TD0 1
SFO1 176.1625005 MHz
NUC1 13C
P1 12.00 usec
P13 2000.00 usec
PLW0 0 W
PLW1 32.86100006 W
SPNAM[5] Crp80comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SWH 9.64000034 W
SFO2 700.5328021 MHz
NUC2 1H
CPDPRG2 waltz16
P3 15.00 usec
P4 30.00 usec
PCPD2 65.00 usec
PLW2 8.39560032 W
PLW12 0.44710001 W

F2 - Processing parameters
SI 32768
SF 176.1484090 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Figure S42. DEPT135 spectrum of compound **17**

G99-MC-3-2_COSY

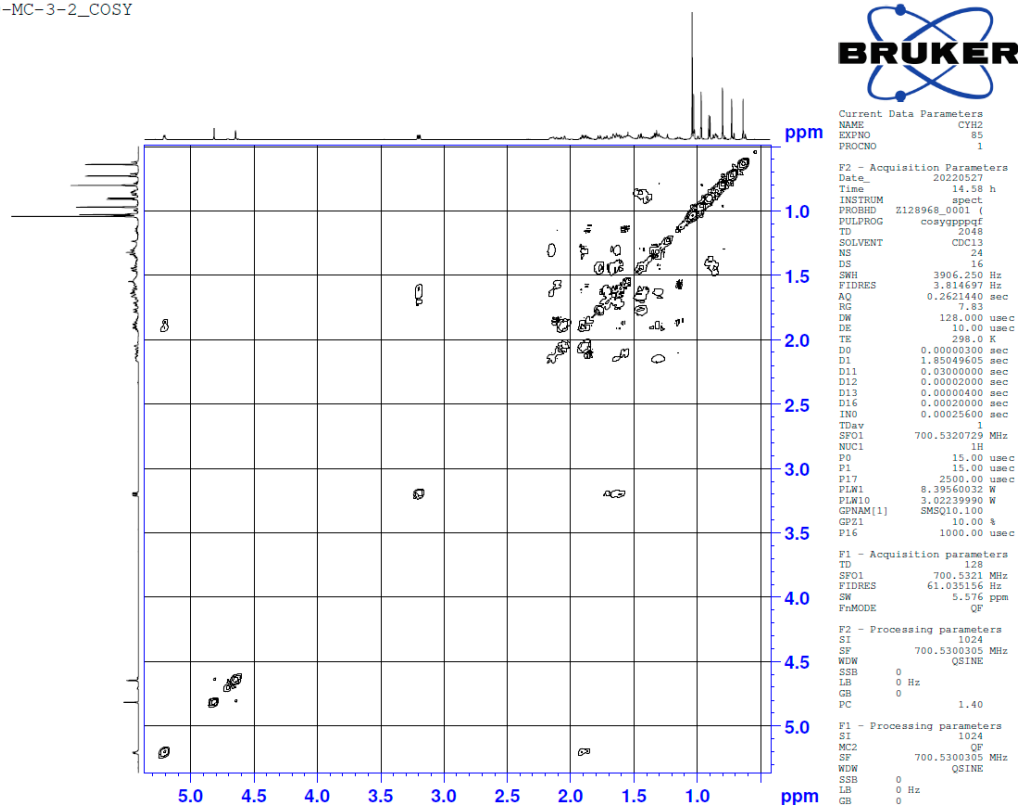


Figure S43. ^1H - ^1H COSY spectrum of compound **17**

G99-MC-3-2_HSQC

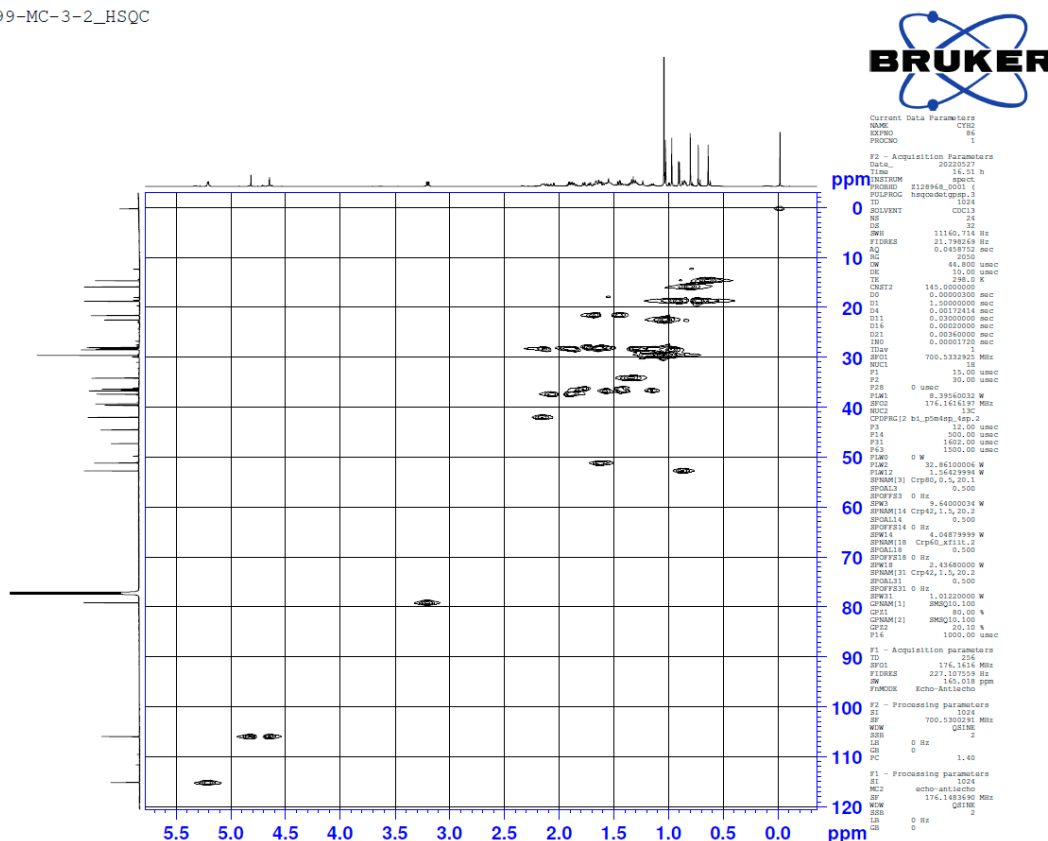


Figure S44. HSQC spectrum of compound **17**

G99-MC-3-2_HMBC

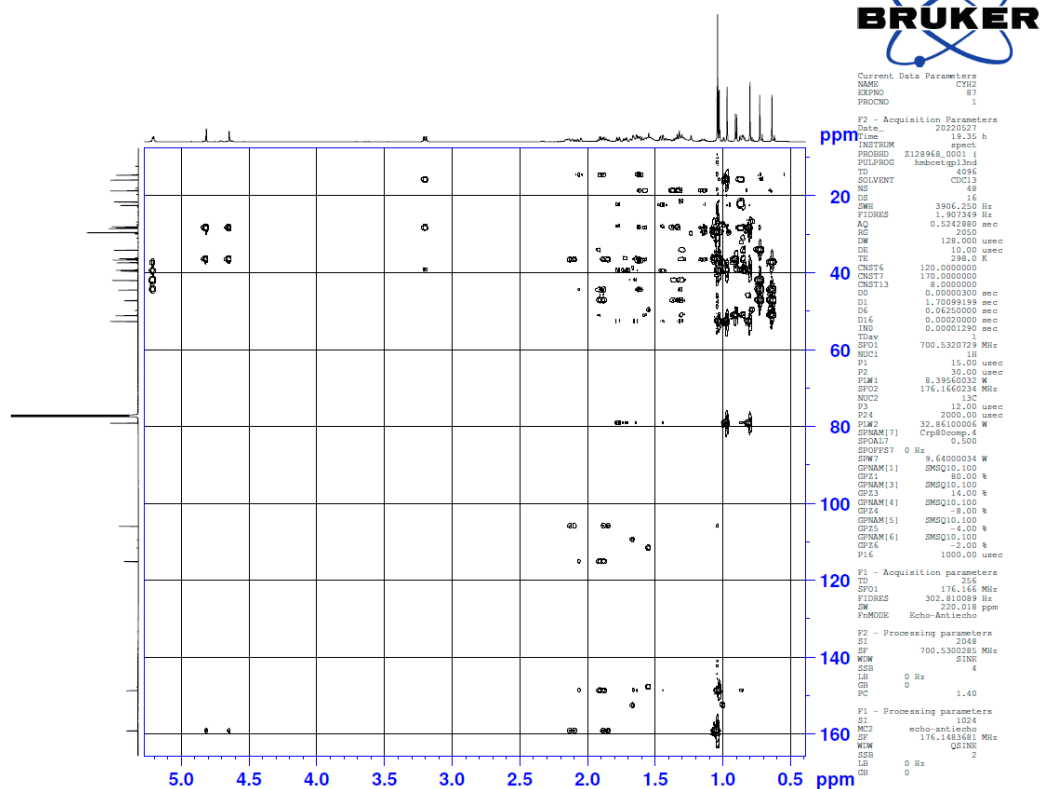


Figure S45. HMBC spectrum of compound 17

G99-MC-3-2_ROESY

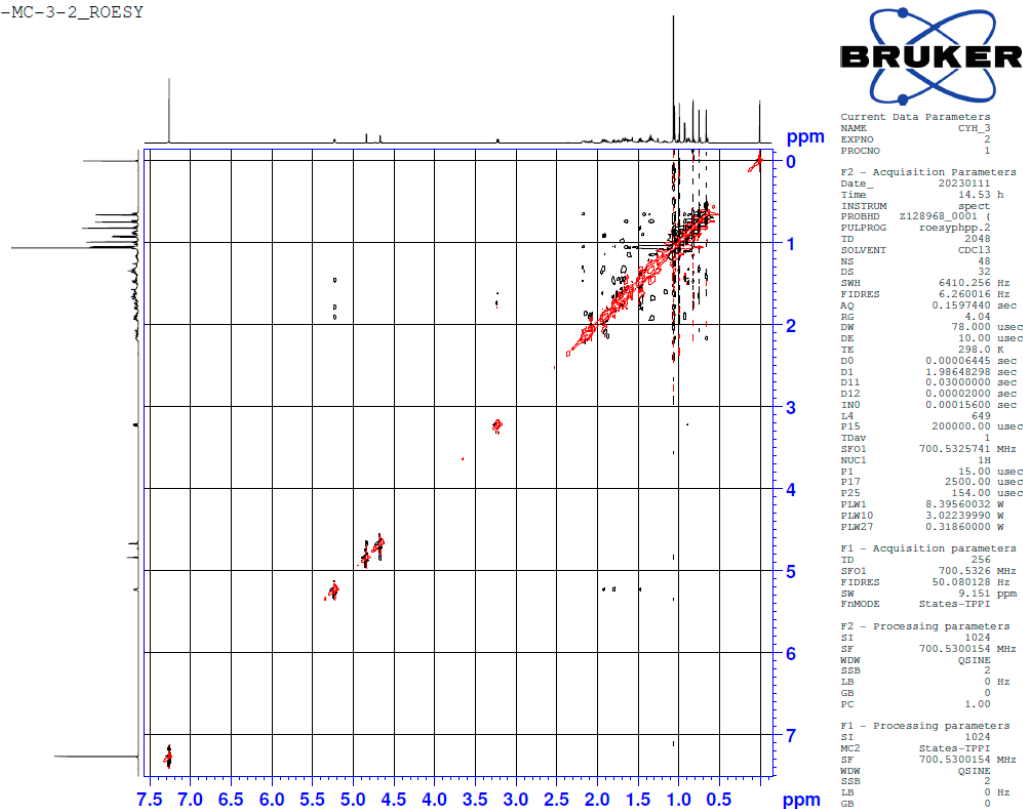


Figure S46. ROESY spectrum of compound 17

Light Source Na
 Monitor wavelength 589 nm
 D.I.T. 5 sec
 No. of cycle 5
 Cycle interval 0 sec
 Temp. Monitor Cell
 Temp. Corr. Factor None
 Aperture(S) 8.0mm
 Aperture(L) Auto
 Mode Specific O.R.
 Path Length 50 mm
 Concentration 0.114286 w/v%
 Water content of sample 0 %
 Factor 1

		No.	Sample No.	Mode	Calc. Data	Meas. Data	Monitor(deg)	PMT Voltage[V]	Temperature(C)
1	*	1	G99-MC-3P-1	Specific O.R.	44.2049	0.0253	0.0233	305	25.17
2	*	2	G99-MC-3P-2	Specific O.R.	48.2299	0.0276	0.0256	303	25.17
3	*	3	G99-MC-3P-3	Specific O.R.	50.6799	0.0290	0.0270	305	25.18
4	*	4	G99-MC-3P-4	Specific O.R.	39.8299	0.0228	0.0208	305	25.18
5	*	5	G99-MC-3P-5	Specific O.R.	49.1049	0.0281	0.0261	303	25.19
6	*	6	Avg.		46.4099				
7		7	S.D		4.3865				
8		8	C.V		9.4517				

Figure S47. Specific optical rotation of compound **17**

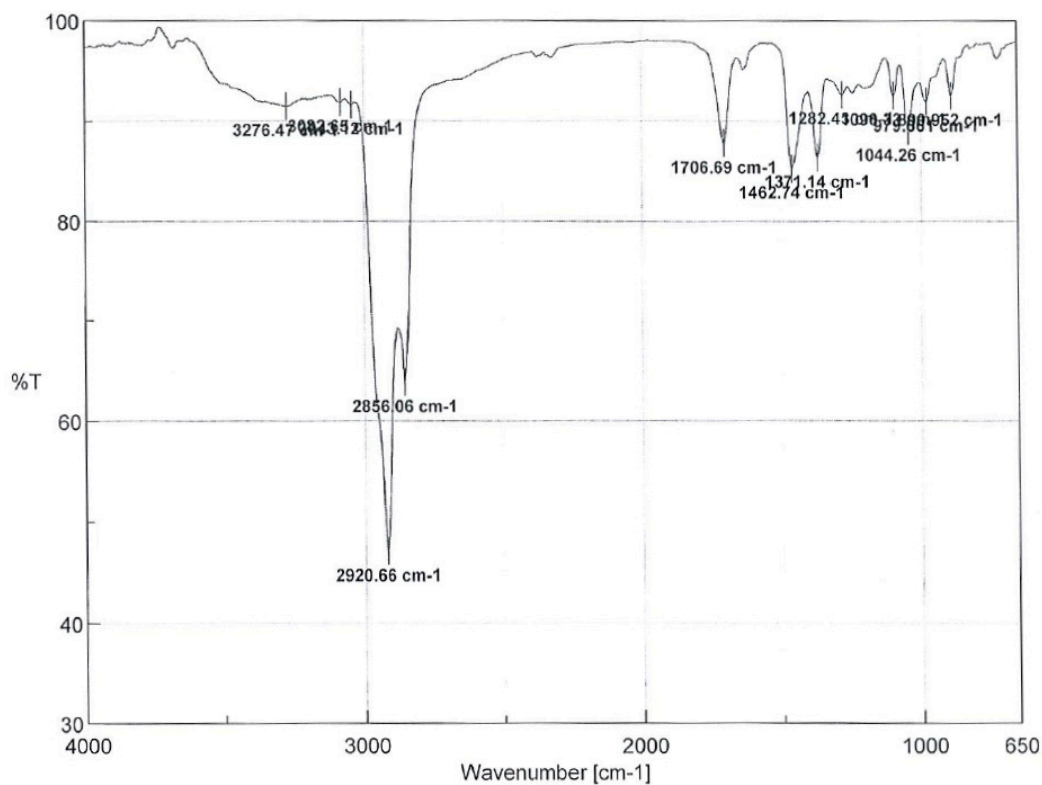


Figure S48. IR spectrum of compound **17**

g99-mc-3-2_230116104101#91-98 RT: 0.50-0.52 AV: 2 NL: 2.46E7
 F: FTMS + p APCI corona sid=5.00 Full ms [150.00-1500.00]

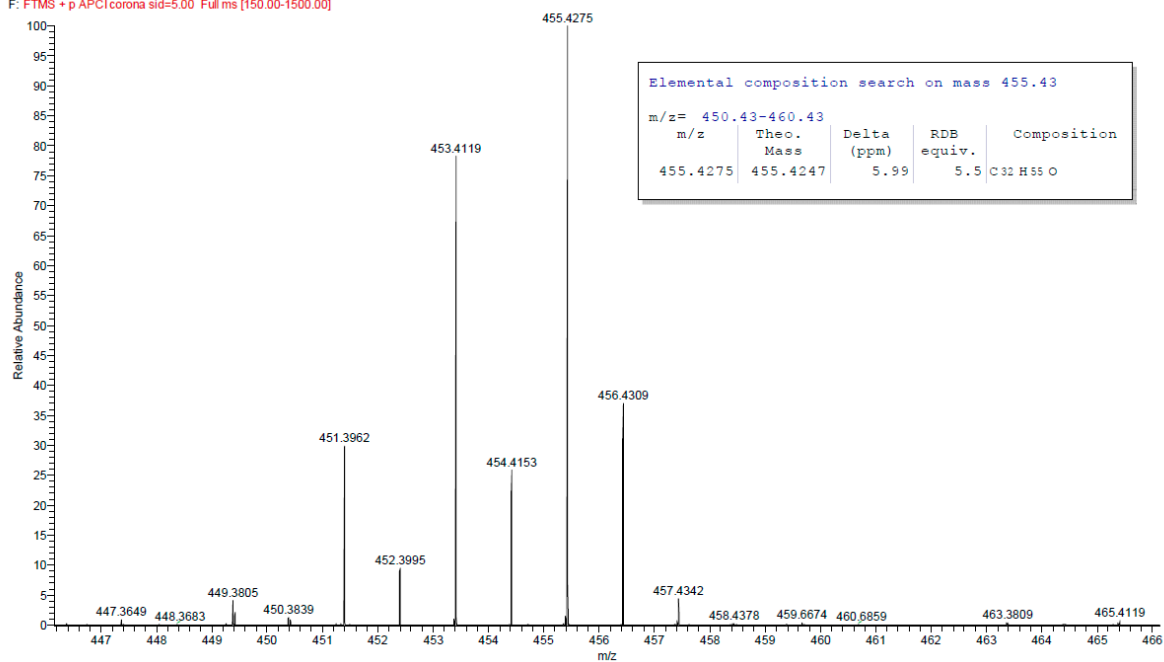
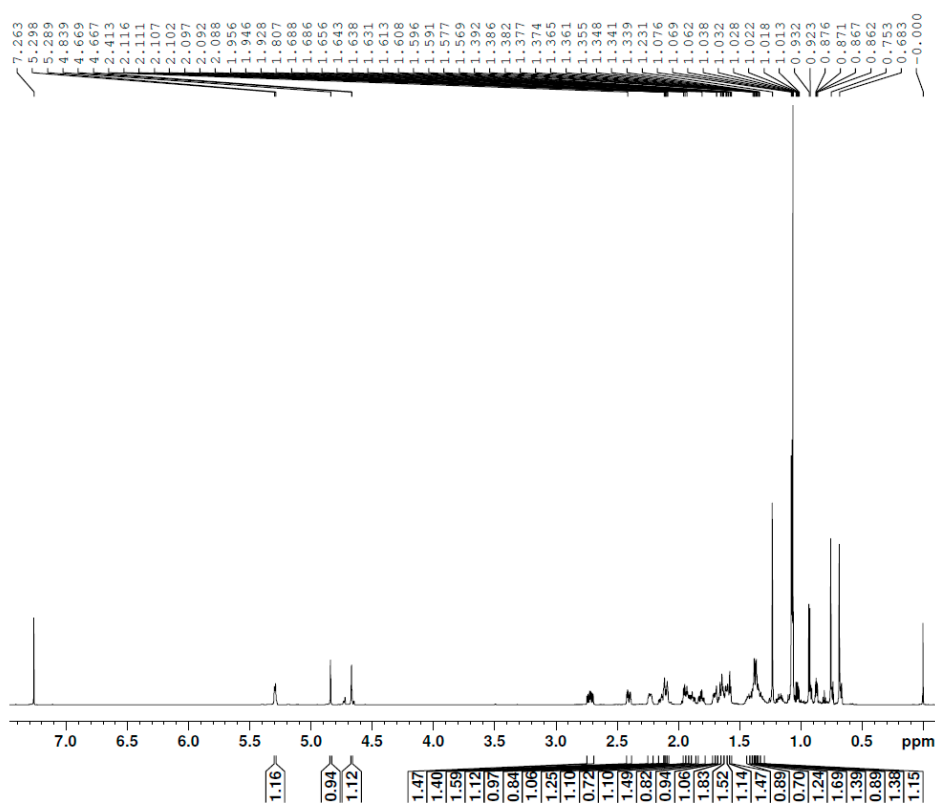


Figure S49. HRESIMS spectrum of compound **17**

G99-36F_1H



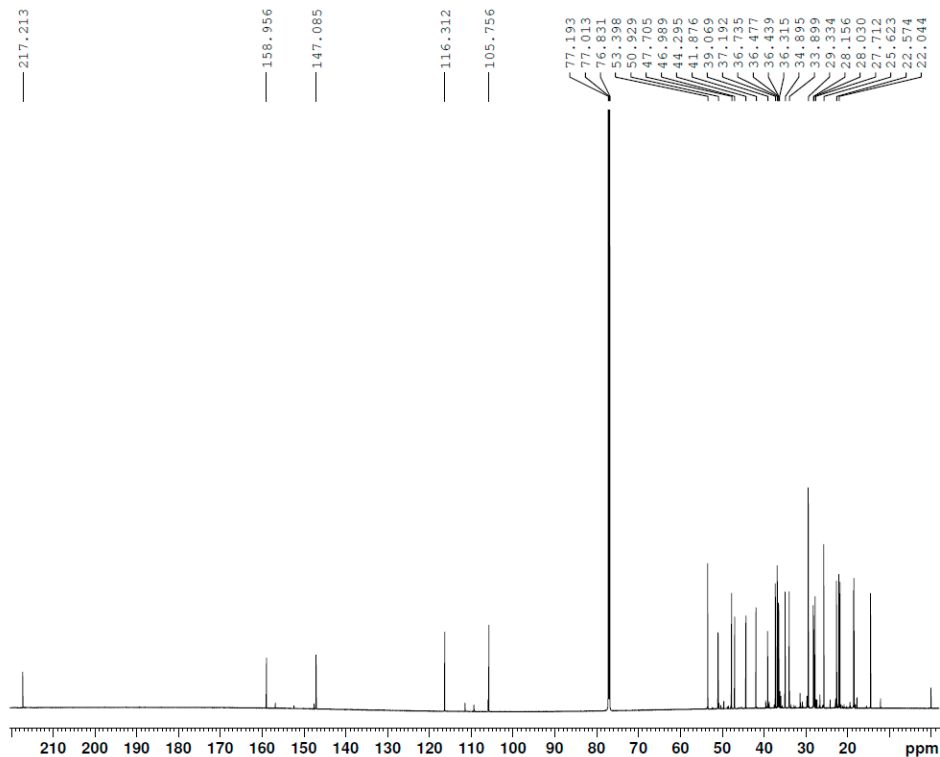
Current Data Parameters
 NAME H555
 EXPNO 74
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230315
 Time 17.21 h
 INSTRUM spect
 PROBHD z128968_0001 (
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 32
 DS 2
 SWH 14097.744 Hz
 FIDRES 0.430229 Hz
 AQ 2.3243434 sec
 RG 3.5
 DW 35.467 usec
 DE 10.00 usec
 TE 298.0 K
 D1 1.00000000 sec
 TD0
 SFO1 700.5343258 MHz
 NUC1 1H
 P1 15.00 usec
 PLW1 8.39560032 W

F2 - Processing parameters
 SI 65536
 SF 700.5300142 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Figure S50. ^1H -NMR spectrum (CDCl_3 , 700 MHz) of compound **18**

G99-36F_13C



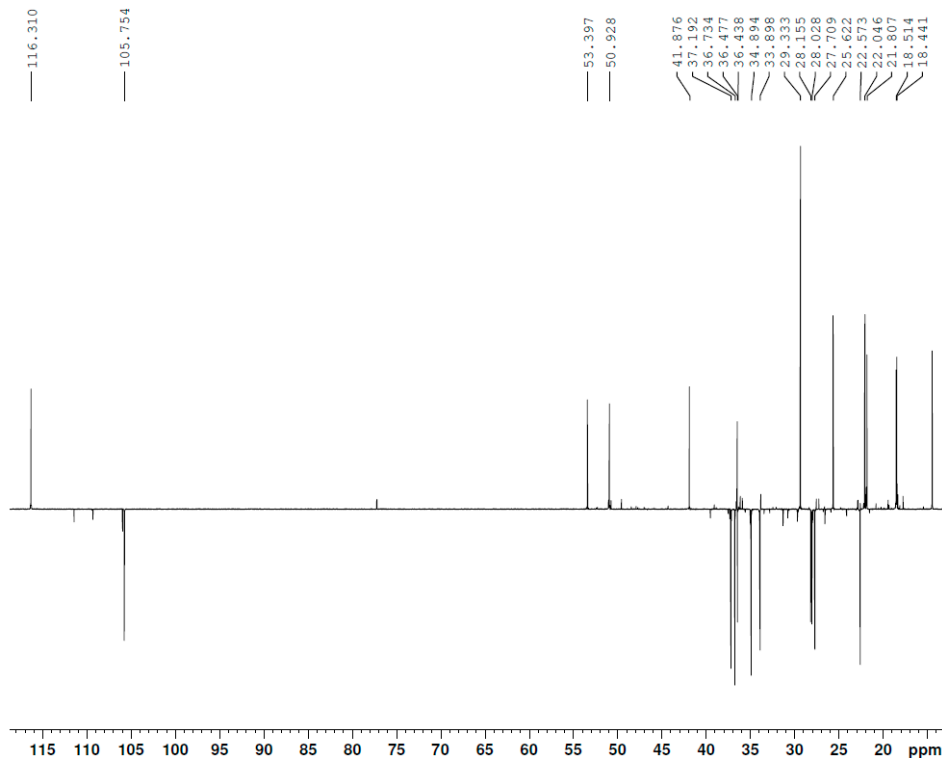
Current Data Parameters
 NAME H555
 EXPNO 75
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20230315
 Time 18.58 h
 INSTRUM spect
 PROBHD z128968_0001 (
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 2000
 DS 2
 SWH 42613.637 Hz
 FIDRES 1.300465 Hz
 AQ 0.7689557 sec
 RG 1440
 DW 11.733 usec
 DE 18.00 usec
 TE 298.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1
 SFO1 176.1660234 MHz
 NUC1 13C
 P1 12.00 usec
 PLW1 32.86100006 W
 SFO2 700.5328021 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 65.00 usec
 PLW2 8.39560032 W
 PLW12 0.44710001 W
 PLW13 0.22513001 W

F2 - Processing parameters
 SI 32768
 SF 176.1484110 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure S51. ^{13}C -NMR spectrum (CDCl_3 , 175 MHz) of compound **18**

G99-36F_DEPT135



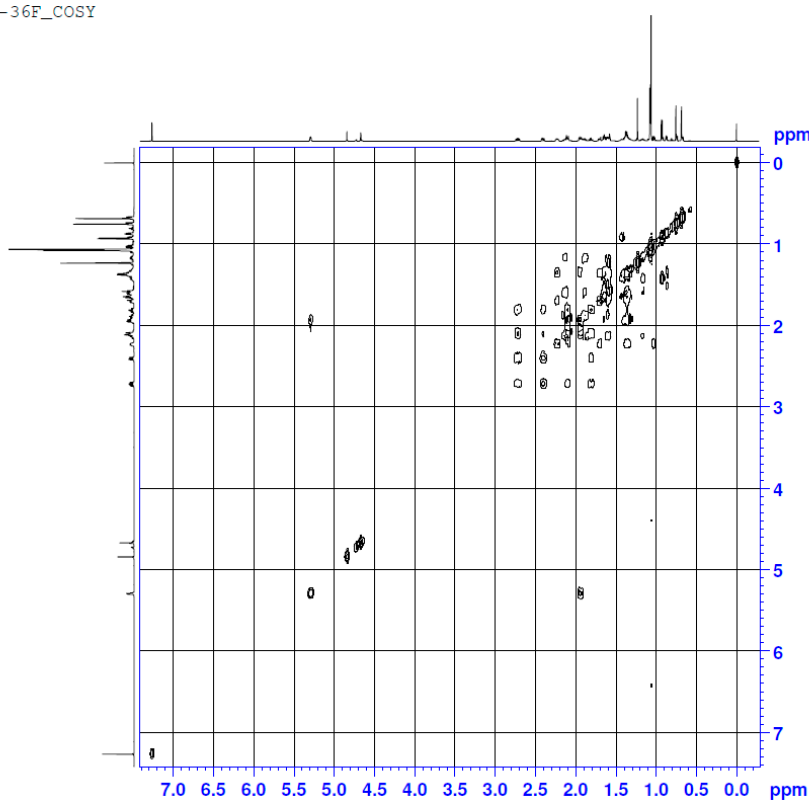
Current Data Parameters
NAME H555
EXPNO 77
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230315
Time 20.42 h
INSTRUM spect
PROBHD Z128968_0001 ()
PULPROG deptsp135
TD 65536
SOLVENT CDCl3
NS 1000
DS 8
SWH 28409.092 Hz
FIDRES 0.866977 Hz
AQ 1.1534336 sec
RG 2050
DW 17.600 usec
DE 18.00 usec
TE 298.0 K
CNST2 145.0000000
D1 2.00000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
TD0 1
SFO1 176.1625005 MHz
NUC1 13C
P1 12.00 usec
P13 2000.00 usec
PLW0 0 W
PLW1 32.86100006 W
SPNAM[5] Crp80comp.4
SFOALS 0.500
SFOF55 0 Hz
SPW5 9.64000034 W
SFO2 700.5328021 MHz
NUC2 1H
CPDPRG[2] waltz16
P3 15.00 usec
P4 30.00 usec
PCPD2 65.00 usec
PLW2 8.39560032 W
PLW12 0.44710001 W

F2 - Processing parameters
SI 32768
SF 176.1484113 MHz
WDW EM
SSR 0

Figure S52. DEPT135 spectrum of compound 18

G99-36F_COSY



Current Data Parameters
NAME H555
EXPNO 78
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230315
Time 20.44 h
INSTRUM spect
PROBHD Z128968_0001 ()
PULPROG cosygpgpgf
TD 2048
SOLVENT CDCl3
NS 8
DS 16
SWH 6097.561 Hz
FIDRES 5.954649 Hz
AQ 0.1679360 sec
RG 6.45
DW 82.000 usec
DE 10.00 usec
TE 298.0 K
D0 0.0000300 sec
D1 1.94470406 sec
D11 0.03000000 sec
D12 0.00005000 sec
D13 0.00000400 sec
D16 0.00020000 sec
IN0 0.00016400 sec
TDav 1
SFO1 700.5328001 MHz
NUC1 1H
P0 15.00 usec
P1 15.00 usec
F17 2500.00 usec
PLW1 8.39560032 W
PLW10 3.02239990 W
GFNAM[1] SMSQ10.100
GP21 10.00 %
F16 1000.00 usec

F1 - Acquisition parameters
TD 128
SFO1 700.5328 MHz
FIDRES 95.274391 Hz
SW 8.704 ppm
F2MODE QF

F2 - Processing parameters
SI 1024
SF 700.5300142 MHz
WDW QFTIME
SSB 0
LB 0 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 QF
SF 700.5300142 MHz
WDW QFTIME
SSB 0
LB 0 Hz
GB 0

Figure S53. ^1H - ^1H COSY spectrum of compound 18

G99-36F_HSQC

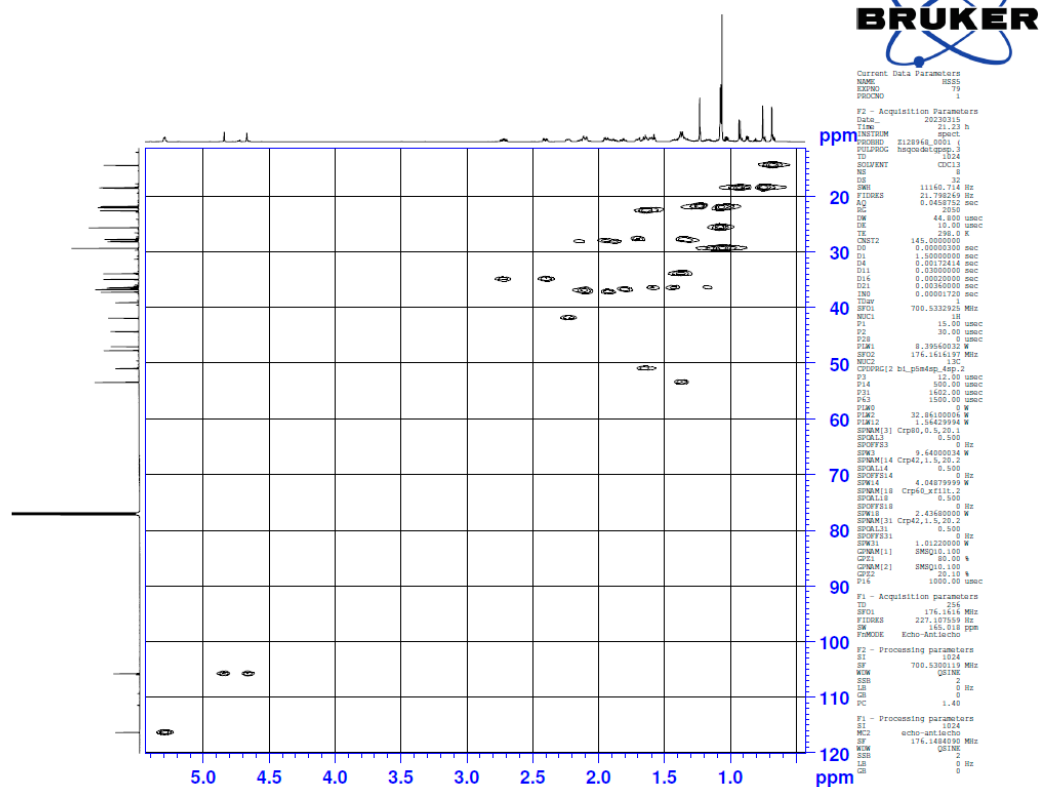


Figure S54. HSQC spectrum of compound **18**

G99-36F_HMBC

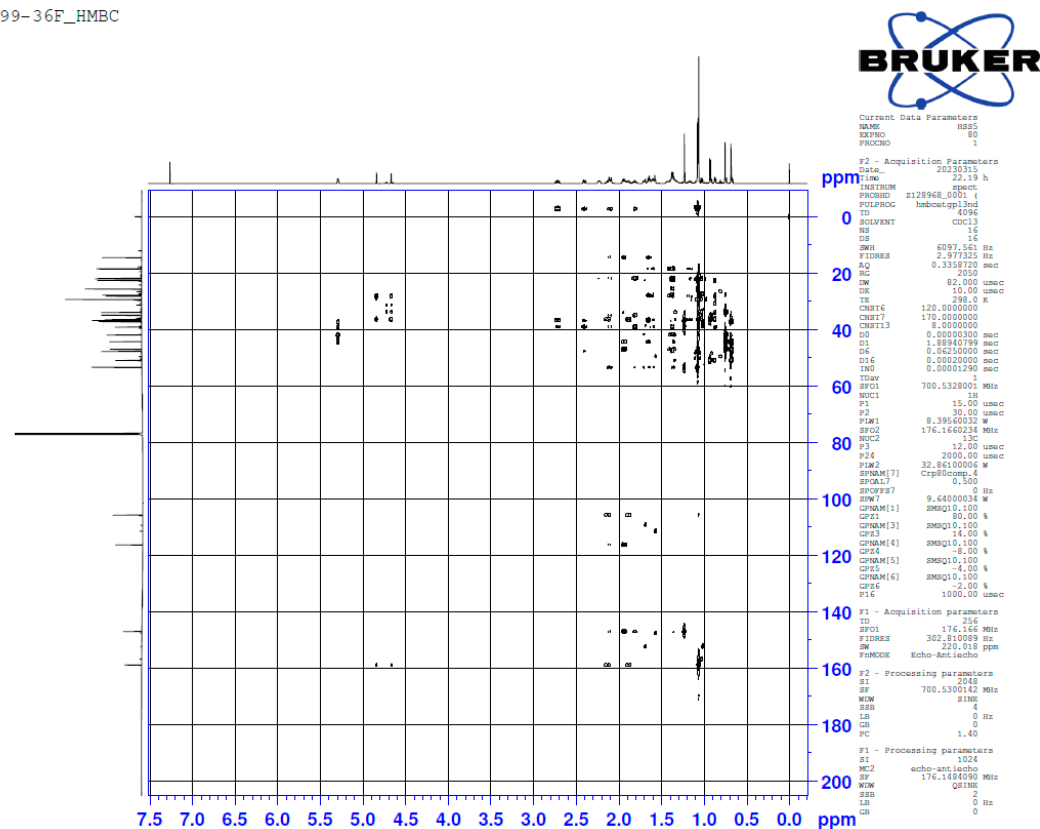


Figure S55. HMBC spectrum of compound **18**

G99-36F_ROESY

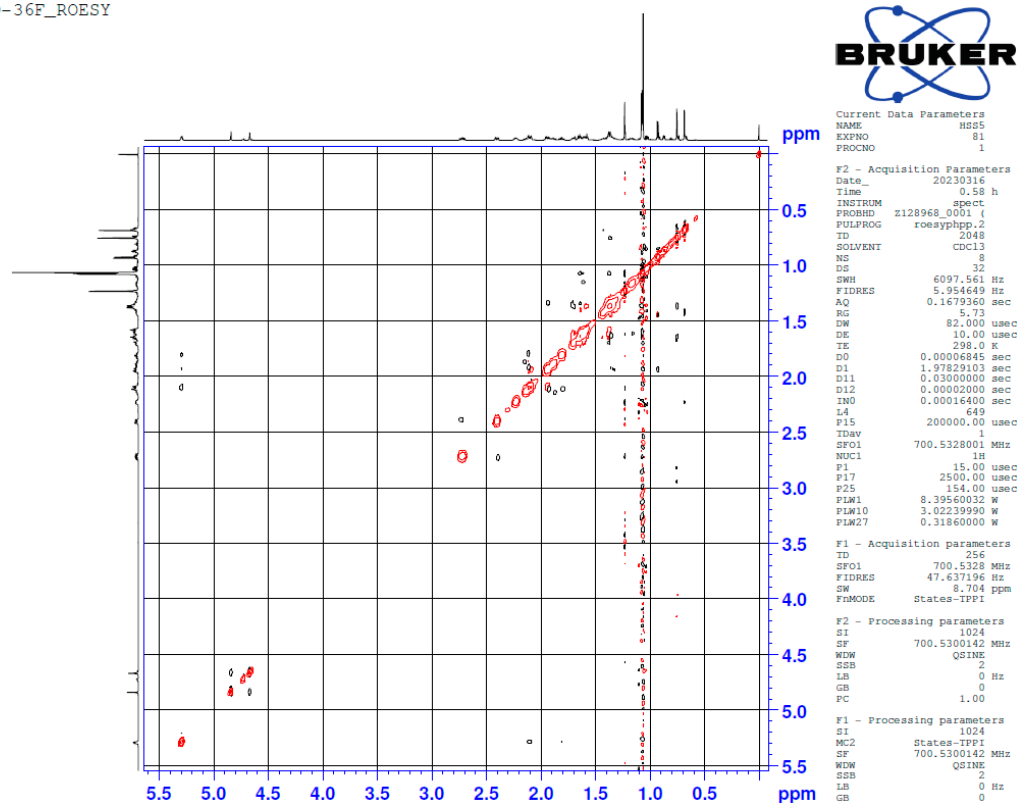


Figure S56. ROESY spectrum of compound 18

Light Source Na
 Monitor wavelength 589 nm
 D.I.T. 5 sec
 No. of cycle 5
 Cycle interval 0 sec
 Temp. Monitor Cell
 Temp. Corr. Factor None
 Aperture(S) 8.0mm
 Aperture(L) Auto
 Mode Specific O.R.
 Path Length 50 mm
 Concentration 0.357143 w/v%
 Water content of sample 0 %
 Factor 1

		No.	Sample No.	Mode	Calc. Data	Meas. Data	Monitor(deg)	PMT Voltage[V]	Temperature(C)
1	*	1	G99-36_1-1	Specific O.R.	28.9408	0.0517	0.0527	303	23.27
2	*	2	G99-36_1-2	Specific O.R.	26.7008	0.0477	0.0487	304	23.27
3	*	3	G99-36_1-3	Specific O.R.	24.5728	0.0439	0.0449	303	23.27
4	*	4	G99-36_1-4	Specific O.R.	25.0768	0.0448	0.0458	304	23.27
5	*	5	G99-36_1-5	Specific O.R.	26.8128	0.0479	0.0489	304	23.27
6	*	6	Avg.		26.4208				
7		7	S.D		1.7178				
8		8	C.V		6.5018				

Figure S57. Specific optical rotation of compound 18

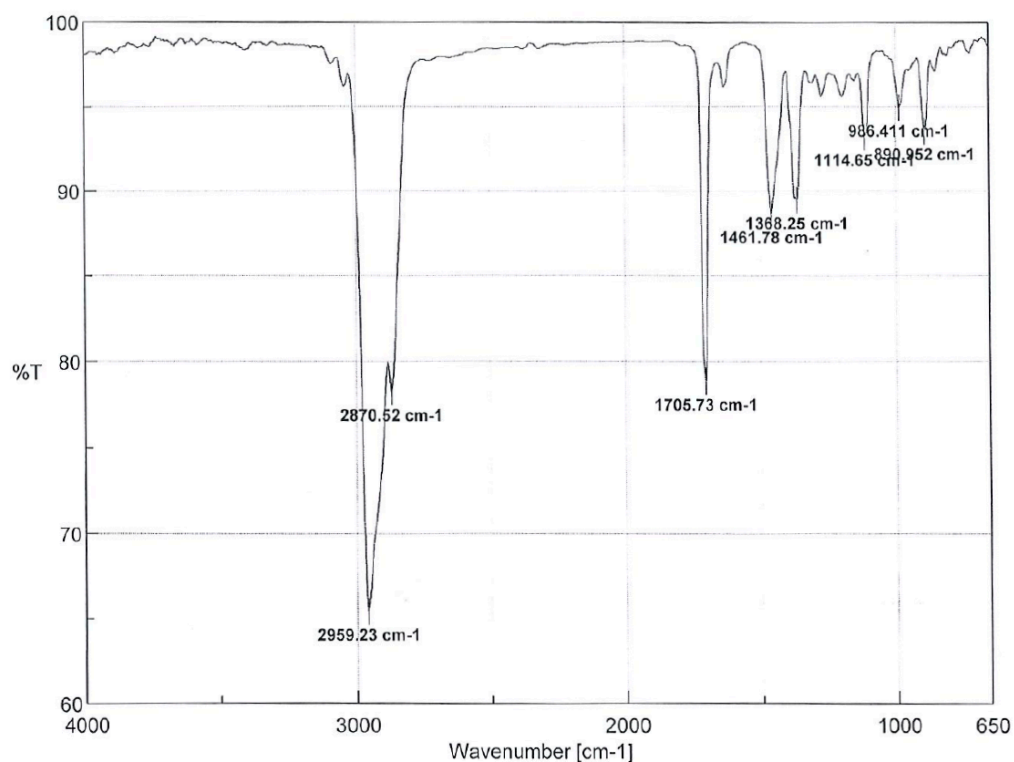


Figure S58. IR spectrum of compound 18

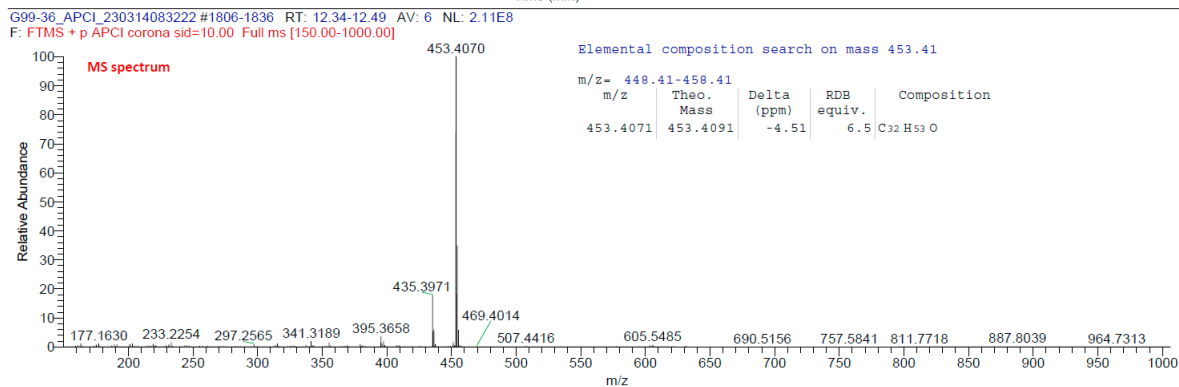
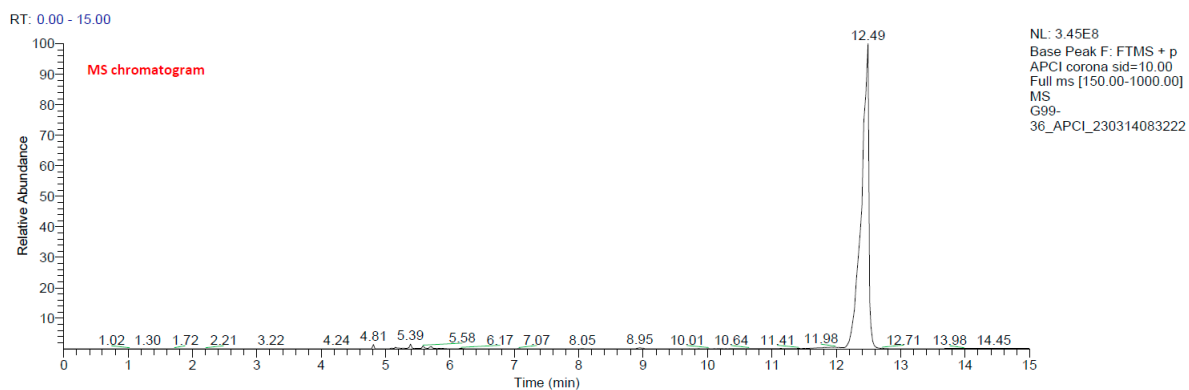
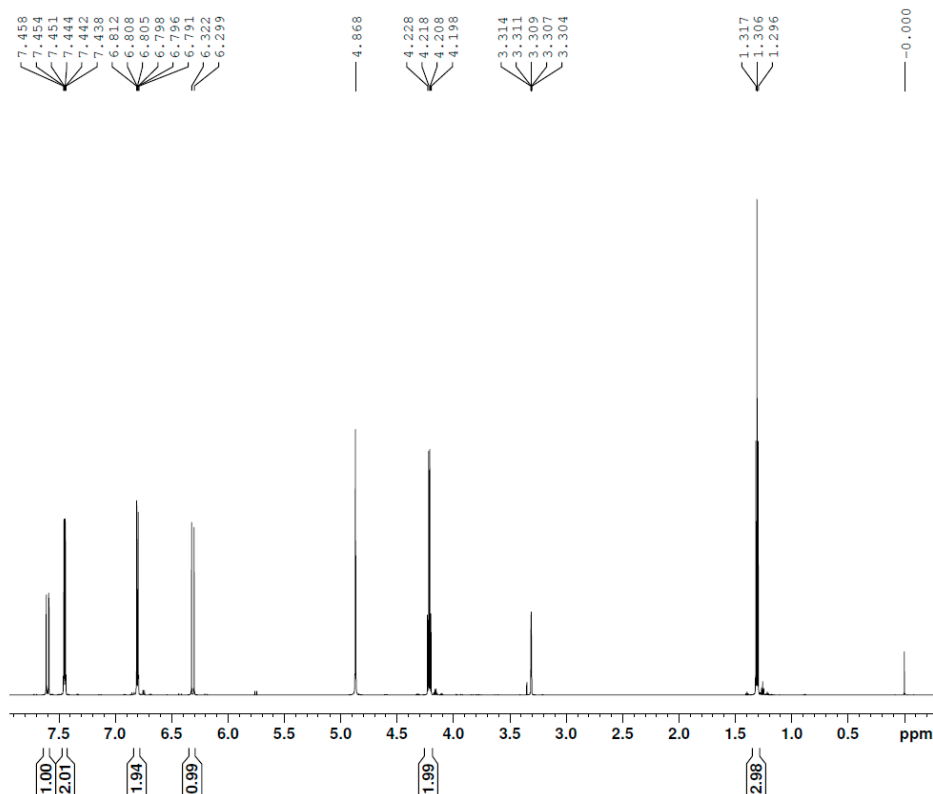


Figure S59. HRESIMS spectrum of compound 18

G99-MC-4-4-1_1H



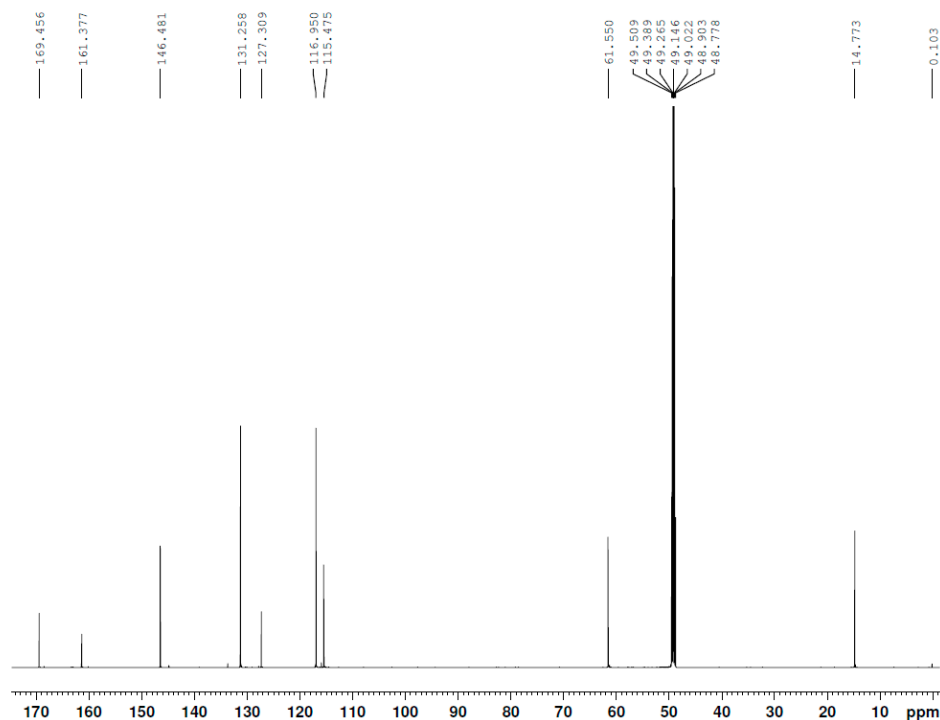
Current Data Parameters
NAME CYH2
EXPNO 39
PROCNO 1

F2 - Acquisition Parameters
Date_ 20220413
Time 8.33 h
INSTRUM spect
PROBHD Z128968_0001 ()
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 32
DS 2
SWH 14097.744 Hz
FIDRES 0.430229 Hz
AQ 2.3243434 sec
RG 6.45
DW 35.467 usec
DE 10.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
SFO1 700.5343258 MHz
NUC1 1H
P1 15.00 usec
PLW1 8.39560032 W

F2 - Processing parameters
SI 65536
SF 700.5300141 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Figure S60. ^1H -NMR spectrum (CD_3OD , 700 MHz) of compound **19**

G99-MC-4-4-1_13C



Current Data Parameters
NAME CYH2
EXPNO 40
PROCNO 1

F2 - Acquisition Parameters
Date_ 20220413
Time 10.09 h
INSTRUM spect
PROBHD Z128968_0001 ()
PULPROG zgpg30
TD 65356
SOLVENT MeOD
NS 2000
DS 2
SWH 42613.637 Hz
FIDRES 1.304047 Hz
AQ 0.7668437 sec
RG 1440
DW 11.713 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 176.1660234 MHz
NUC1 13C
P1 12.00 usec
PLW1 32.86100006 W
SFO2 700.5328021 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 65.00 usec
PLW2 8.39560032 W
PLW12 0.44710001 W
PLW13 0.22513001 W

F2 - Processing parameters
SI 32768
SF 176.1481376 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Figure S61. ^{13}C -NMR spectrum (CD_3OD , 175 MHz) of compound **19**