

Supporting Information

Microwave assisted unprotected Sonogashira reaction in water for the synthesis of multi-replaced aromatic acetylene compounds

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Supporting Information

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1. General remarks

All reagents and solvents were from commercial sources and were used without further purification. All reactions were monitored by thin-layer chromatography (TLC) on 0.25 mm silica gel plates with fluorescent indicator (GF₂₅₄) and visualized under UV light. With tetramethylsilane (TMS) as internal standard, the ¹H and ¹³C NMR spectra were recorded on Bruker AV-300 (300 and 75 MHz) apparatus at 25 °C. Samples were prepared as solutions in deuterated solvent. EI-MS was collected on shimadzu GCMS-2010 instruments. All target compounds were purified via silica gel (60 Å, 70-230 mesh) column chromatography. Abbreviations of solvents and chemicals are as followed: TEA: triethylamine, DIPA: diisopropanolamine, Pyr.: pyridine, DIPEA: N, N- diisopropylethylamine, CD: cyclodextrin, DMF: N, N- dimethylformamide.

2. General procedures

2.1 Synthesis

The reaction raw materials, tetrakis (4-bromophenyl) methane, TEA and catalyst were added to the microwave tube. After adding water, the microwave tube was closed and stirred well at room temperature. The reaction mixture was then subjected to microwave irradiation at specified temperature. After completion of the reaction, the mixture was cooled to room temperature, and the solvent was evaporated under reduced pressure and purified by silica gel column chromatography.

Tetrakis (4-((trimethylsilyl) ethynyl) phenyl) methane (1P)

1S (0.1 g, 1.5 mmol), trimethylsilylacetylene (0.23 mL, 15 mmol), TEA (0.22 mL, 15 mmol), PPh₃ (13 mg, 0.5 mmol), Pd (PPh₃)₂Cl₂ (17 mg, 0.24 mmol), CuI (6 mg, 0.32 mmol), 18-Crown-6 (21 mg, 0.75 mmol), solvent (2.0 mL) were added to the microwave tube and mixed. The temperature was raised to 115 °C at 270 watts for 15 minutes. After the reaction was completed, extraction was performed with DCM, and the yield was calculated by taking 1/50. And the crude product was purified by column chromatography to afford the titled compound. ¹H-NMR (300 MHz, CDCl₃): δ [ppm] = 7.34- 7.31(d, *J* = 9.0 Hz, 8H), 7.05- 7.02(d, *J* = 9.0 Hz, 8H), 0.23(s, 36H), ¹³C-NMR (300 MHz, CDCl₃): δ [ppm] = 146.10, 131.48, 130.82, 121.29, 104.71, 94.89, 64.83, 0.07, EI-MS found 704, 616, 531, 343, 73.

1,4-bis((trimethylsilyl)ethynyl)benzene (2P)

2S (0.3 g, 1.27 mmol), trimethylsilylacetylene (0.9 mL, 6.36 mmol), TEA (0.88 mL, 6.36 mmol), PPh₃ (110 mg, 0.42 mmol), Pd (PPh₃)₂Cl₂ (143 mg, 0.20 mmol), CuI (53 mg, 0.28 mmol), 18-Crown-6 (168 mg, 0.64 mmol), solvent (10.0 mL) were added to the microwave tube and mixed. The temperature was raised to 115 °C at 270 watts for 15 minutes. After the reaction was completed, extraction was performed with DCM, and the yield was calculated by taking 1/100. And the crude product was purified by column chromatography to afford the titled compound. ¹H- NMR(300MHz, (CD₃)₂CO): δ [ppm] = 7.45(s, 4H), 0.23(s, 18H), ¹³C-NMR(300MHz, CDCl₃): δ [ppm] = 131.88, 123.25, 104.68, 96.43, 0.05, EI-MS found 270, 255, 197, 120.

1,3,5-tris((trimethylsilyl)ethynyl)benzene (3P)

3S (0.1 g, 0.32 mmol), trimethylsilylacetylene (0.33 mL, 2.38 mmol), TEA (0.33 mL,

2.38 mmol), PPh₃ (27 mg, 2.38 mmol), Pd (PPh₃)₂Cl₂ (36 mg, 0.05 mmol), CuI (13 mg, 0.07 mmol), 18-Crown-6 (42 mg, 0.16 mmol), solvent (2.0 mL) were added to the microwave tube and mixed. The temperature was raised to 115 °C at 270 watts for 5 minutes. After the reaction was completed, extraction was performed with DCM, and the yield was calculated by taking 1/50. And the crude product was purified by column chromatography to afford the titled compound. ¹H- NMR(300MHz, (CD₃)₂CO): δ [ppm] = 7.51(s, 3H), 0.28(s, 27H), ¹³C- NMR(300MHz, CDCl₃): δ [ppm] = 135.08, 123.81, 103.30, 95.74, 0.02, EI-MS found 366, 351, 168, 73.

1,2,4,5-tetrakis(trimethylsilyl)ethynylbenzene (4P)

4S (0.3 g, 0.76 mmol), trimethylsilylacetylene (0.36 mL, 7.62 mmol), TEA (0.33 mL, 7.62 mmol), PPh₃ (66 mg, 0.25 mmol), Pd (PPh₃)₂Cl₂ (86 mg, 0.12 mmol), CuI (32 mg, 0.17 mmol), 18-Crown-6 (101 mg, 0.38 mmol), solvent (10.0 mL) were added to the microwave tube and mixed. The temperature was raised to 130 °C at 270 watts for 15 minutes. After the reaction was completed, extraction was performed with DCM, and the yield was calculated by taking 1/100. And the crude product was purified by column chromatography to afford the titled compound. ¹H- NMR(300MHz, (CD₃)₂CO): δ [ppm] = 7.55(s, 2H), 0.27(s, 36H), ¹³C- NMR(300MHz, CDCl₃): δ [ppm] = 136.11, 125.44, 102.10, 0.02, EI-MS found 462, 447, 359, 73.

4,4'-bis(trimethylsilyl)ethynyl-1,1'-biphenyl (5P)

5S (0.1 g, 0.32 mmol), trimethylsilylacetylene (0.23 mL, 1.60 mmol), TEA (0.22 mL, 1.60 mmol), PPh₃ (28 mg, 0.11 mmol), Pd (PPh₃)₂Cl₂ (36 mg, 0.05 mmol), CuI (14 mg, 0.07 mmol), 18-Crown-6 (44 mg, 0.16 mmol), solvent (2.0 mL) were added to the microwave tube and mixed. The temperature was raised to 130 °C at 270 watts for 30 minutes. After the reaction was completed, extraction was performed with DCM, and the yield was calculated by taking 1/50. And the crude product was purified by column chromatography to afford the titled compound. ¹H- NMR(300MHz, (CD₃)₂CO): δ [ppm] = 7.73- 7.70(d, *J* = 9.0 Hz, 4H), 7.57- 7.54(d, *J* = 9.0 Hz, 4H), 0.25 (s, 18H), ¹³C- NMR(300MHz, CDCl₃): δ [ppm] = 140.32, 132.60, 126.87, 122.54, 104.96, 95.34, 0.10, EI-MS found 346, 331, 158, 131, 73, 43.

((5'-(4-((trimethylsilyl)ethynyl)phenyl)-[1,1':3',1''-terphenyl]-4,4''-diyl)bis(ethyne-2,1-diyl))bis(trimethylsilane) (6P)

6S (0.1 g, 0.32 mmol), trimethylsilylacetylene (0.34 mL, 2.34 mmol), TEA (0.33 mL, 2.34 mmol), PPh₃ (28 mg, 0.11 mmol), Pd (PPh₃)₂Cl₂ (36 mg, 0.05 mmol), CuI (13 mg, 0.07 mmol), 18-Crown-6 (42 mg, 0.16 mmol), solvent (2.0 mL) were added to the microwave tube and mixed. The temperature was raised to 115 °C at 270 watts for 10 minutes. After the reaction was completed, extraction was performed with DCM, and the yield was calculated by taking 1/50. And the crude product was purified by column chromatography to afford the titled compound. ¹H- NMR(300MHz, (CD₃)₂CO): δ [ppm] = 7.99(s, 3H), 7.92- 7.89(d, *J* = 9.0 Hz, 6H), 7.62- 7.59(d, *J* = 9.0 Hz, 6H), 0.26(s, 27H), ¹³C- NMR(300MHz, CDCl₃): δ [ppm] = 141.81, 140.88, 132.63, 127.19, 125.28, 122.64, 104.94, 95.35, 0.11, EI-MS found 594, 579, 282, 183, 73.

Tris(4-((trimethylsilyl)ethynyl)phenyl)amine (7P)

7S (0.1 g, 0.21 mmol), trimethylsilylacetylene (0.22 mL, 1.56 mmol), TEA (0.22 mL, 1.56 mmol), PPh₃ (18 mg, 0.07 mmol), Pd (PPh₃)₂Cl₂ (24 mg, 0.03 mmol), CuI (9 mg, 0.05 mmol), 18-Crown-6 (27 mg, 0.11 mmol), solvent (2.0 mL) were added to the microwave tube

and mixed. The temperature was raised to 115 °C at 270 watts for 5 minutes. After the reaction was completed, extraction was performed with DCM, and the yield was calculated by taking 1/50. And the crude product was purified by column chromatography to afford the titled compound. ¹H- NMR(300MHz, (CD₃)₂CO): δ [ppm] = 7.42- 7.39(d, *J* = 9.0 Hz, 6H), 7.06- 7.03(d, *J* = 9.0 Hz, 6H), 0.22(s, 27H), ¹³C- NMR(300MHz, CDCl₃): δ [ppm] = 146.89, 133.29, 123.93, 117.94, 104.99, 94.07, 0.16, EI-MS found 533, 518, 252, 163, 73.

2,4,6-tris(4-((trimethylsilyl)ethynyl)phenyl)-1,3,5-triazine (8P**)**

8S (0.1 g, 0.18 mmol), trimethylsilylacetylene (0.19 mL, 1.37 mmol), TEA (0.19 mL, 1.37 mmol), PPh₃ (16 mg, 0.06 mmol), Pd (PPh₃)₂Cl₂ (21 mg, 0.03 mmol), CuI (8 mg, 0.04 mmol), 18-Crown-6 (24 mg, 0.10 mmol), solvent (2.0 mL) were added to the microwave tube and mixed. The temperature was raised to 115 °C at 270 watts for 15 minutes. After the reaction was completed, extraction was performed with DCM, and the yield was calculated by taking 1/50. And the crude product was purified by column chromatography to afford the titled compound. ¹H- NMR(300MHz, (CD₃)₂CO): δ [ppm] = 8.87- 8.84(d, *J* = 9.0 Hz, 6H), 7.79- 7.76(d, *J* = 9.0 Hz, 6H), 0.33(s, 27H), ¹³C- NMR(300MHz, CDCl₃): δ [ppm] = 171.07, 135.83, 132.31, 128.83, 127.49, 104.80, 97.60, 0.06, EI-MS found 597, 582, 284, 184, 73, 43.

2,2',7,7'-tetrakis((trimethylsilyl)ethynyl)-9,9'-spirobi[fluorene] (9P**)**

9S (0.1 g, 0.16 mmol), trimethylsilylacetylene (0.22 mL, 1.58 mmol), TEA (0.22 mL, 1.58 mmol), PPh₃ (14 mg, 0.05 mmol), Pd (PPh₃)₂Cl₂ (18 mg, 0.03 mmol), CuI (7 mg, 0.03 mmol), 18-Crown-6 (21 mg, 0.08 mmol), solvent (2.0 mL) were added to the microwave tube and mixed. The temperature was raised to 130 °C at 270 watts for 25 minutes. After the reaction was completed, extraction was performed with DCM, and the yield was calculated by taking 1/50. And the crude product was purified by column chromatography to afford the titled compound. ¹H- NMR(300MHz, (CD₃)₂CO): δ [ppm] = 8.08- 8.05(d, *J* = 9.0 Hz, 4H), 7.58-7.54(dd, *J* = 3.0 Hz, 6.0 Hz, 4H), 6.79(s, 4H), 0.13(s, 36H), ¹³C- NMR(300MHz, CDCl₃): δ [ppm] = 148.37, 141.71, 132.70, 128.16, 123.32, 120.77, 105.43, 95.64, 65.48, 0.37, EI-MS found 700, 597, 509, 73.

2.2 Relative percentage content changes of different substitution products

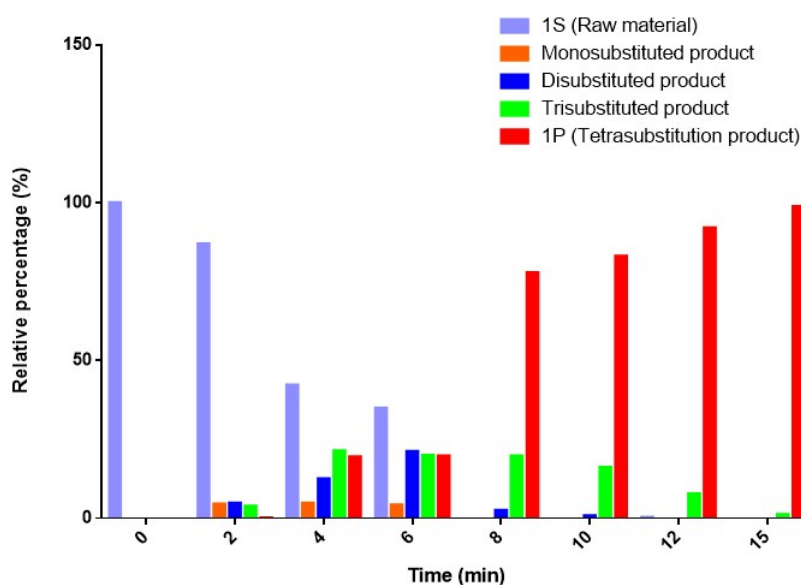


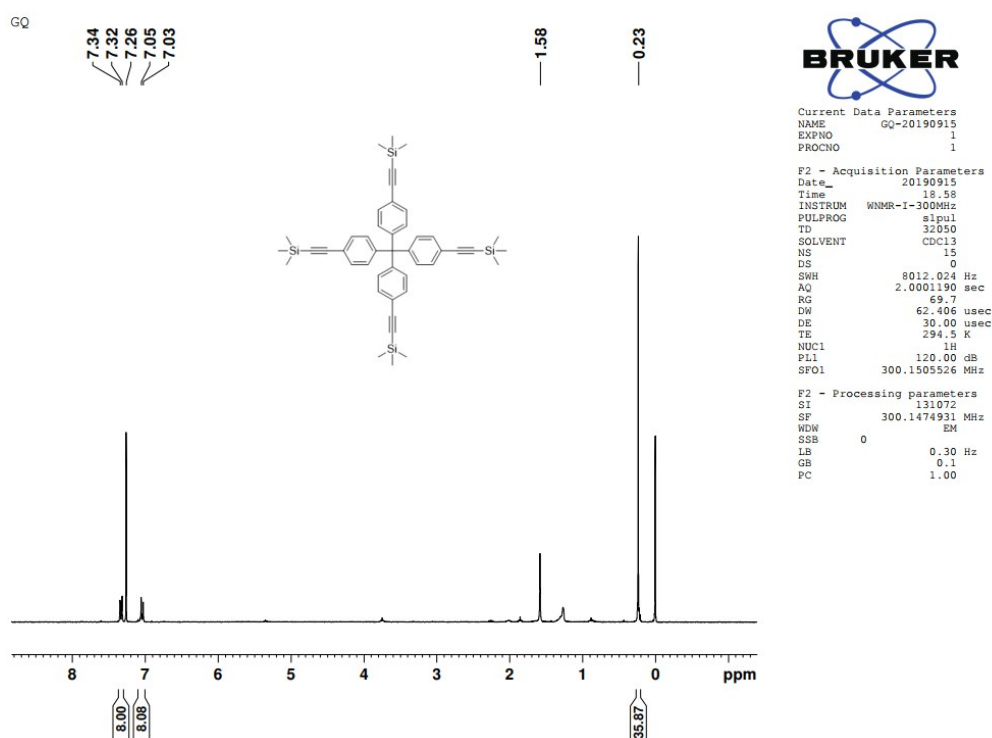
Fig. S1 Relative percentage content changes of different substitution products

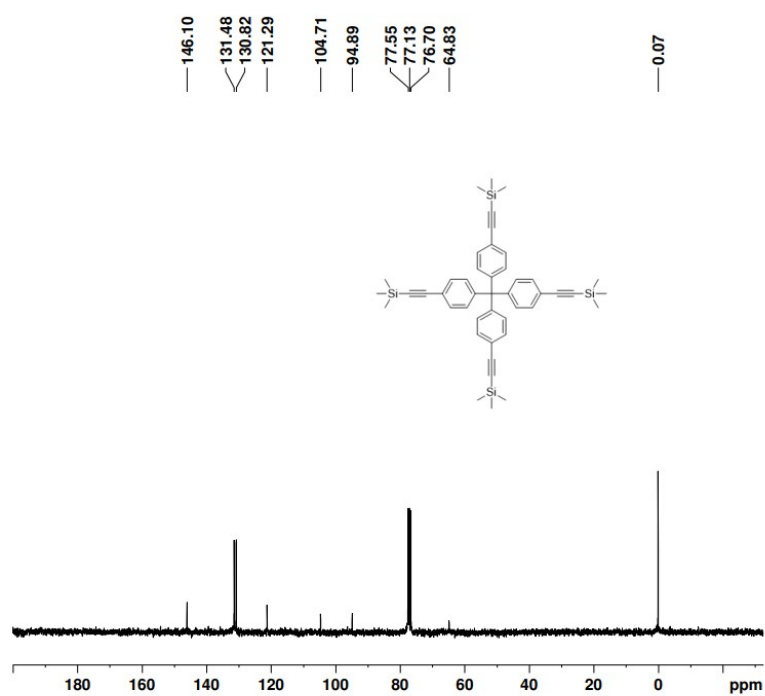
2.3 Yield calculation

The fumaric acid was dissolved in deuterated acetone, and ultrasonically mixed to prepare an internal standard solution of 1 mg/mL. The reaction mixture was taken 1/50 or 1/100, dissolved in 0.6 mL of the internal standard solution, and the yield was calculated by ^1H - NMR. The olefin hydrogen atom of fumaric acid was used as an internal standard to integrate hydrogen, and the yield was calculated by the relative integral area of the hydrogen atom in the silicon methyl group of products.

3. Spectra data

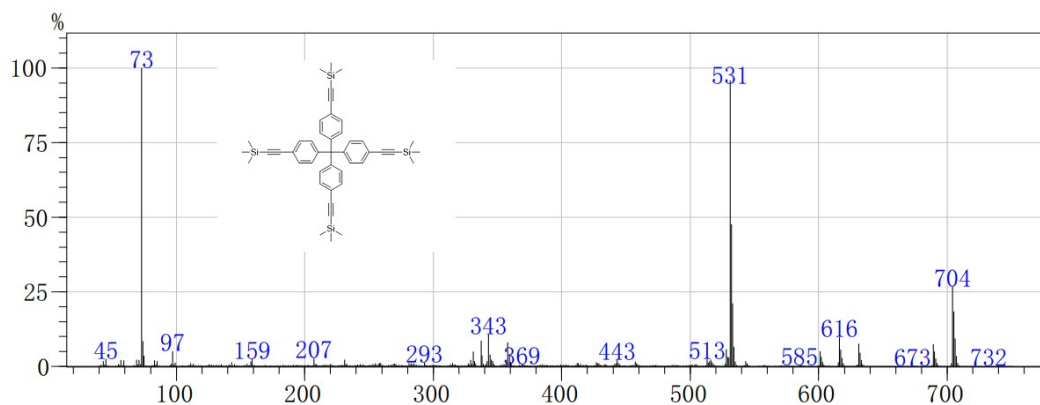
3.1 Spectra data of products



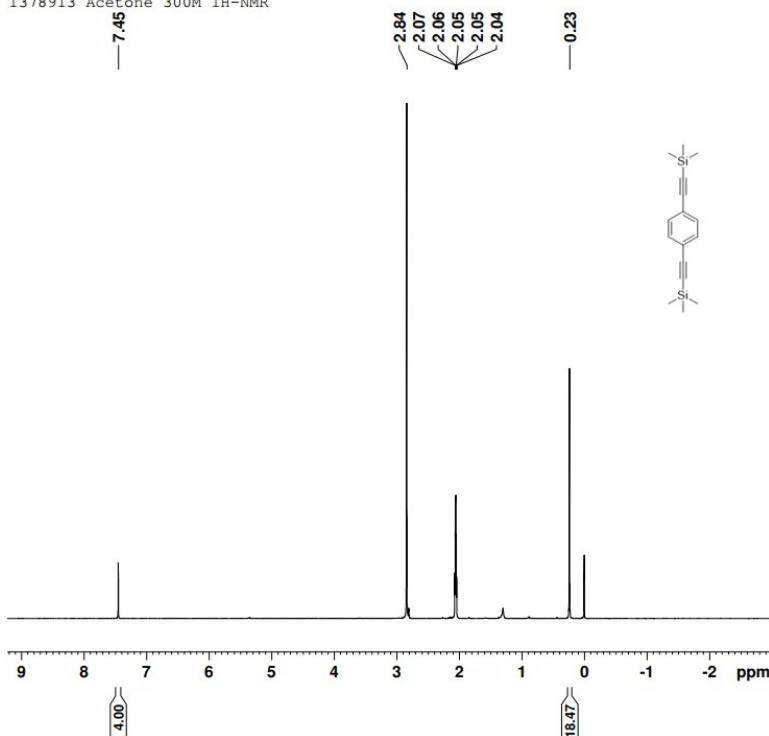


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PROCNO 1

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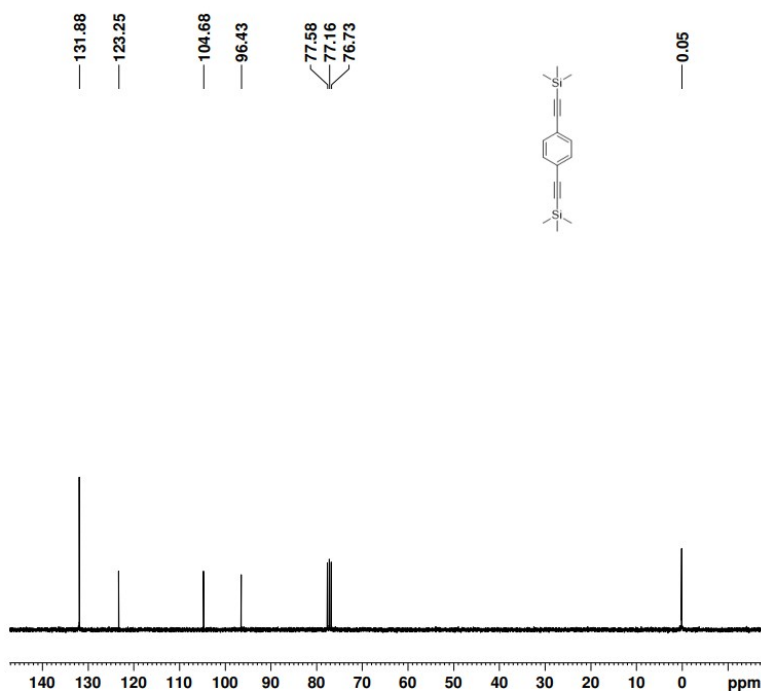
1378913 Acetone 300M 1H-NMR



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 SOLVENT Acetone
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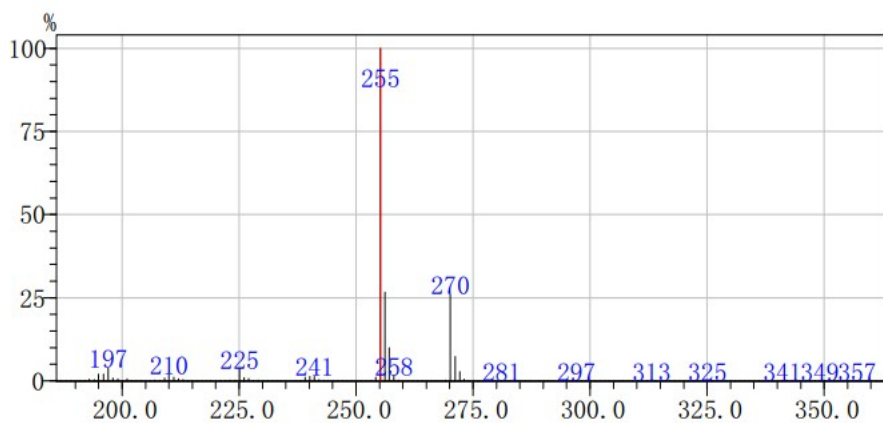
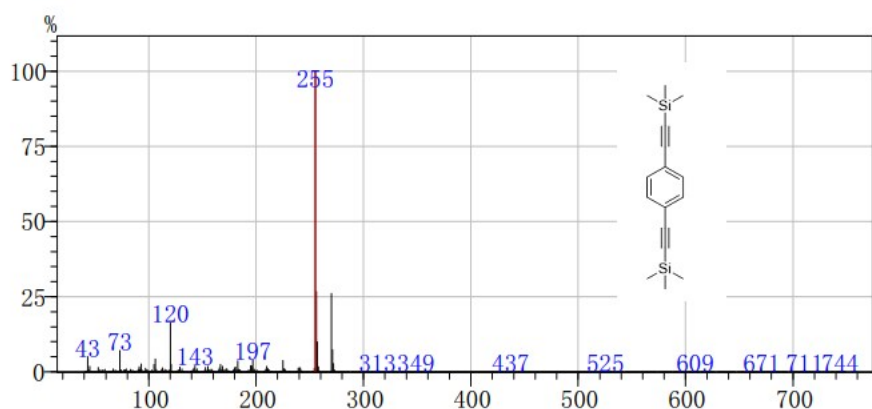
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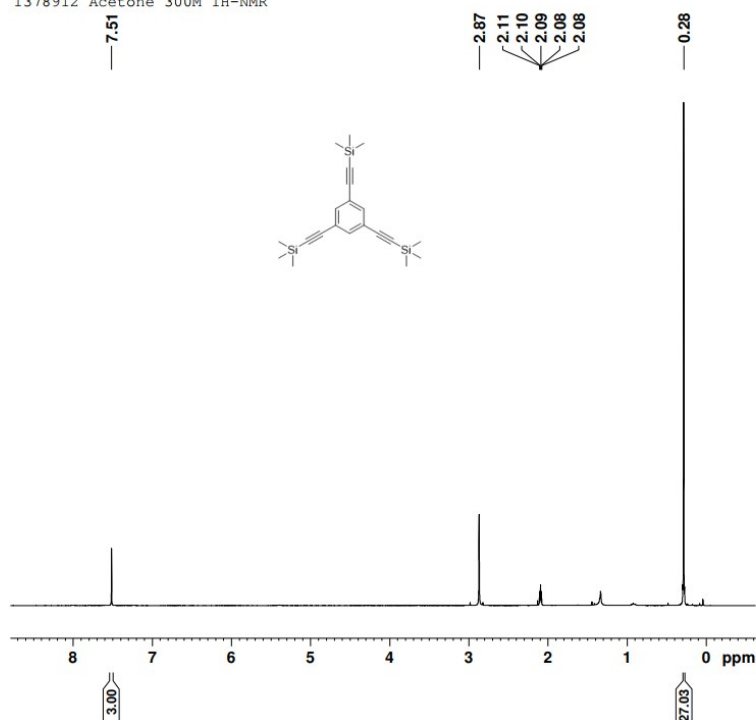
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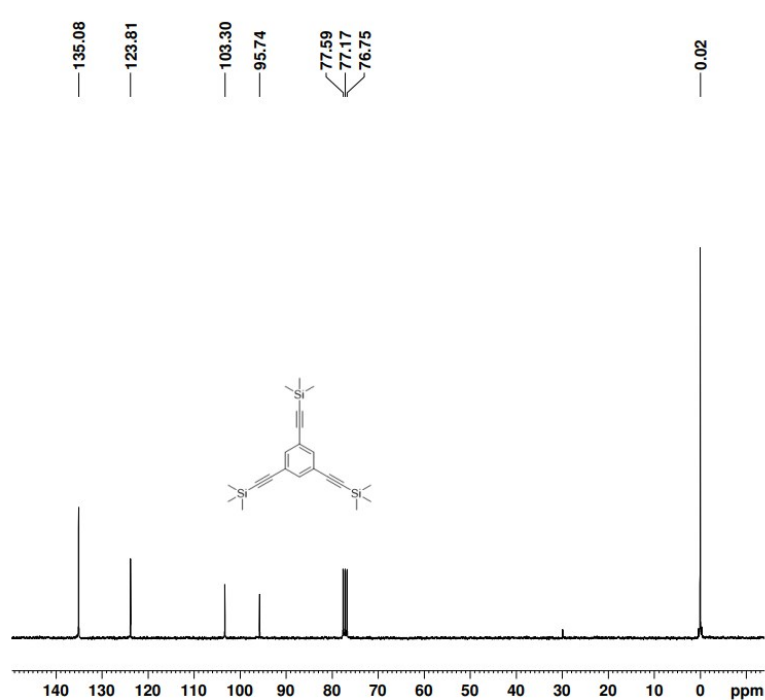
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1378912 Acetone 300M 1H-NMR



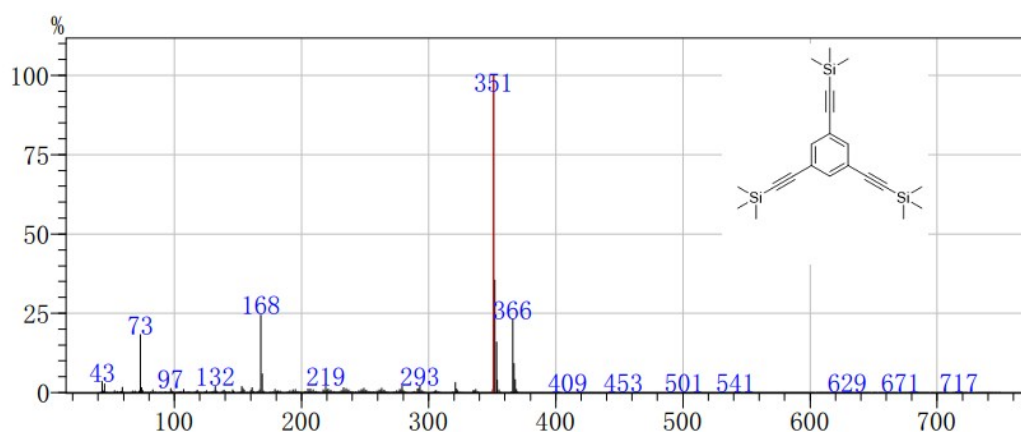
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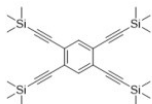
1378914 Acetone- d_6 300M ^1H -NMR

Chemical structure: *[Si](*)#C#Cc1ccc(cc1)C#C[Si](*)(*)#C

^1H -NMR spectrum (ppm):

- 7.55 (s, 2.00H)
- 2.83 (s, 2.07H)
- 2.07 (s, 2.06H)
- 2.05 (s, 2.05H)
- 2.04 (s, 2.04H)
- 0.27 (s, 36.23H)

Integration values: 2.00, 36.23



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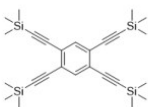
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Chemical structure: C[Si](C)(C)C#Cc1cc(C#C[Si](C)(C)C)c(C#C[Si](C)(C)C)c1C#C[Si](C)(C)C

¹³C NMR peaks (ppm): 136.11, 125.44, 102.10, 77.56, 77.14, 76.71, 0.02.

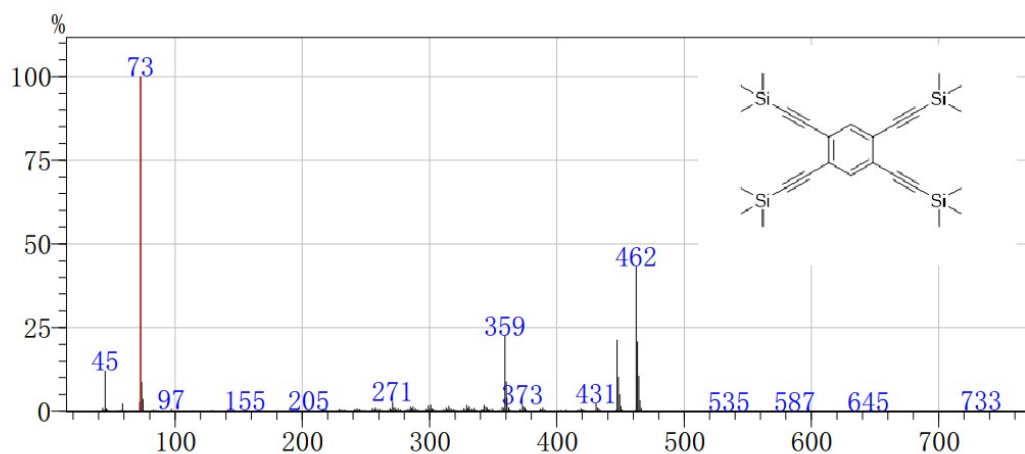


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1378911

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7.58
7.55

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2.07
2.06
2.05
2.05
2.04

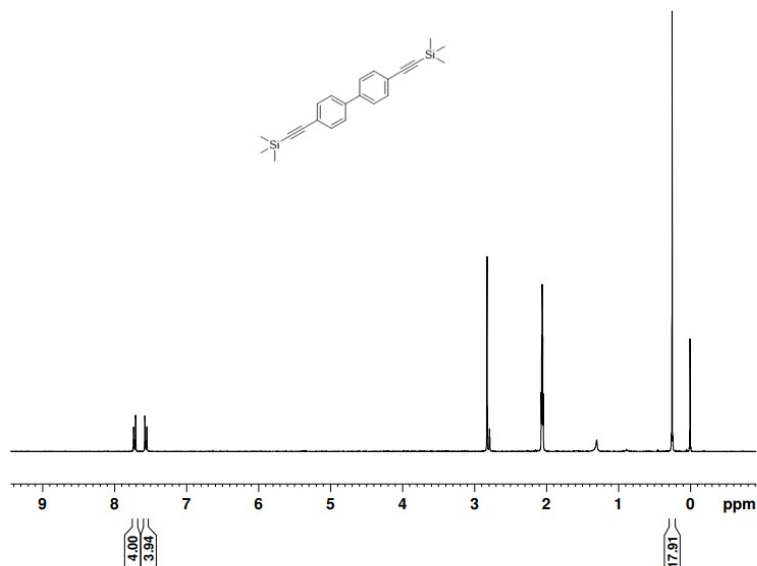
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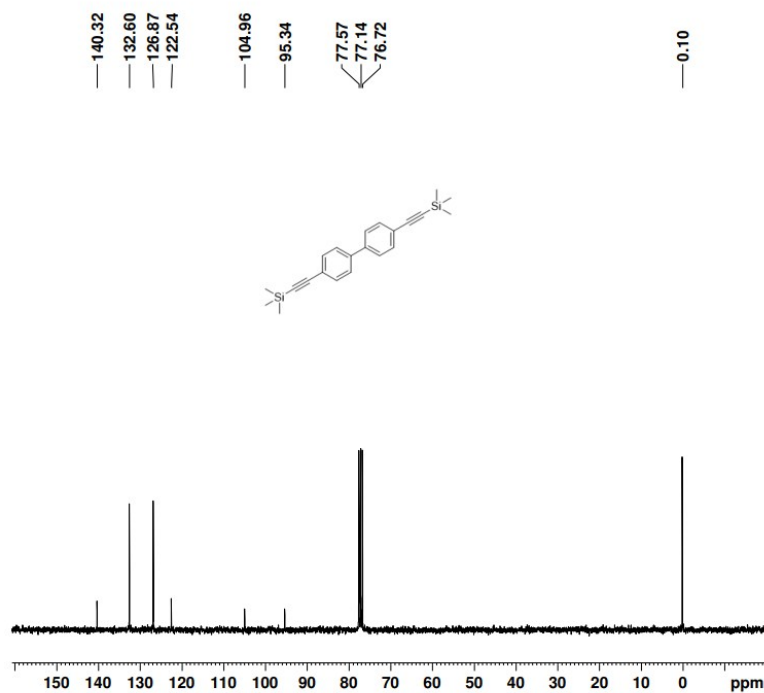


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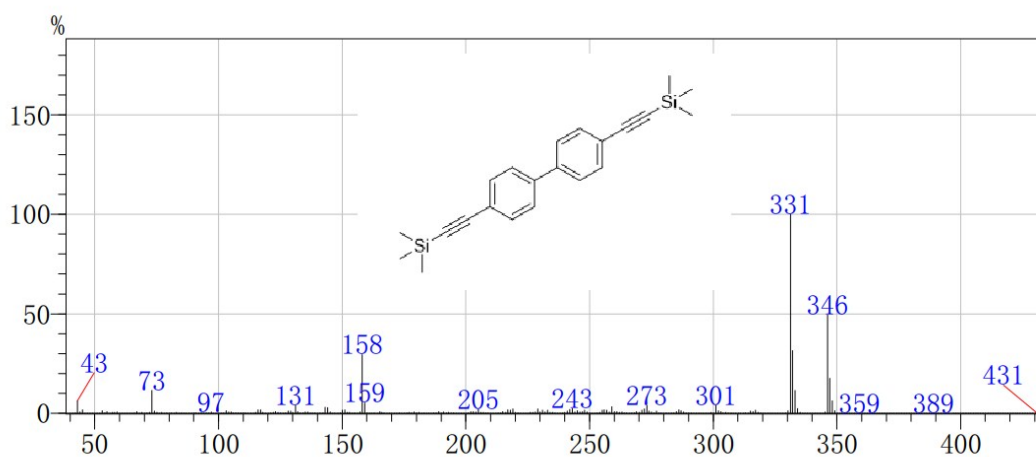




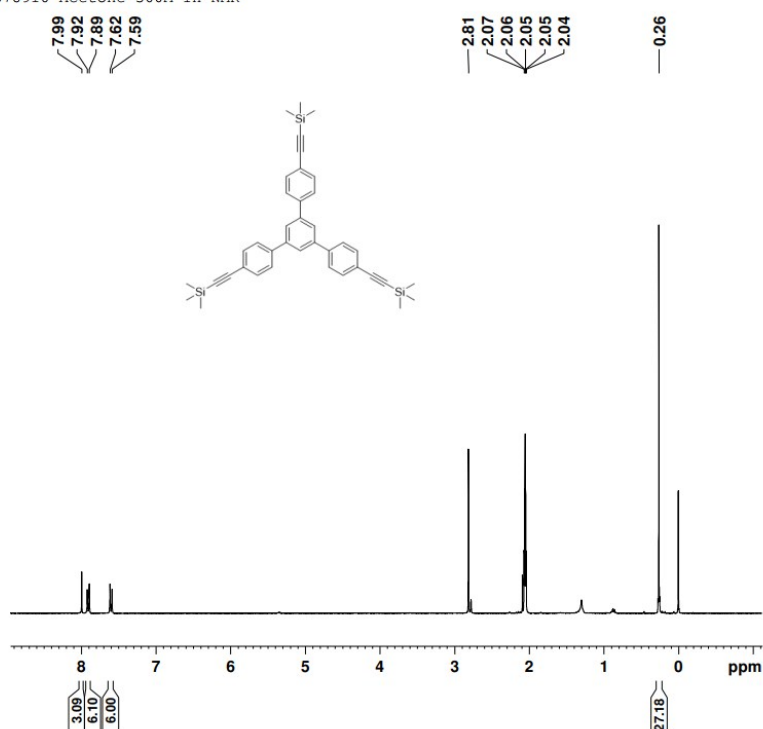
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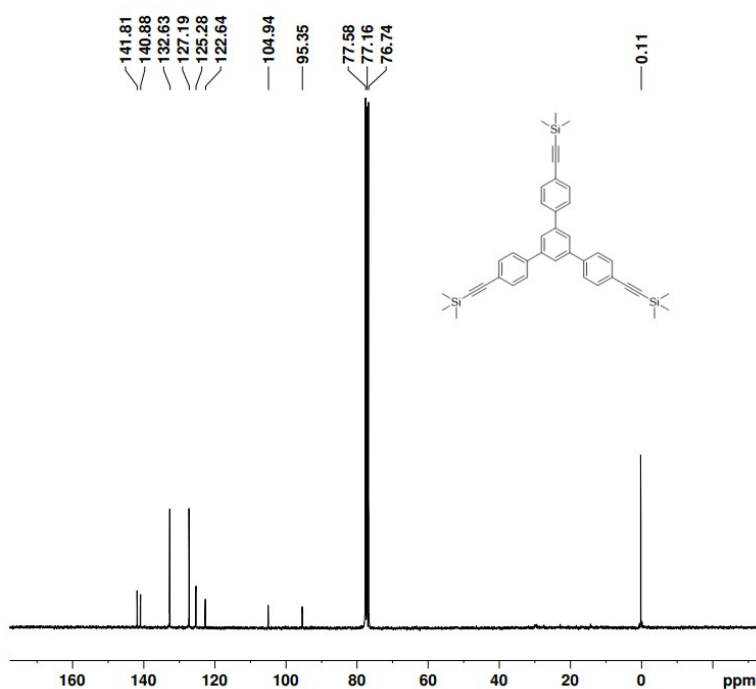
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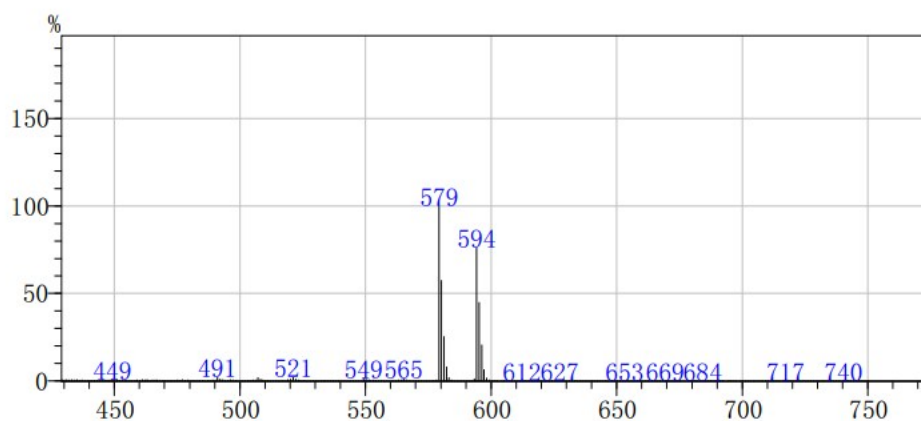
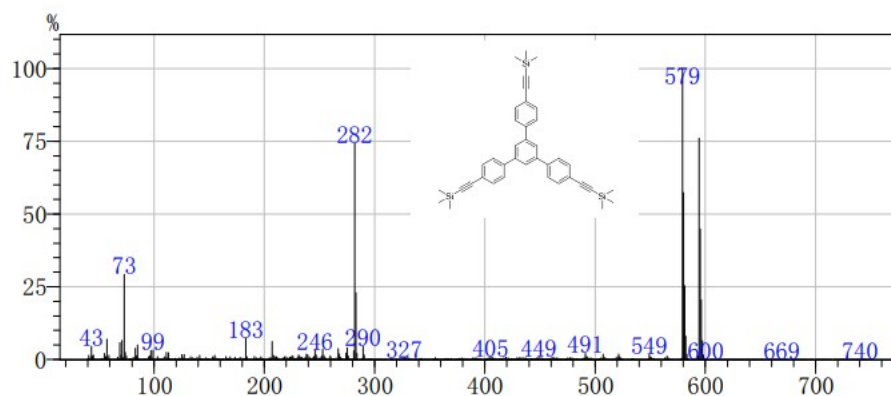
1378910 Acetone 300M 1H-NMR



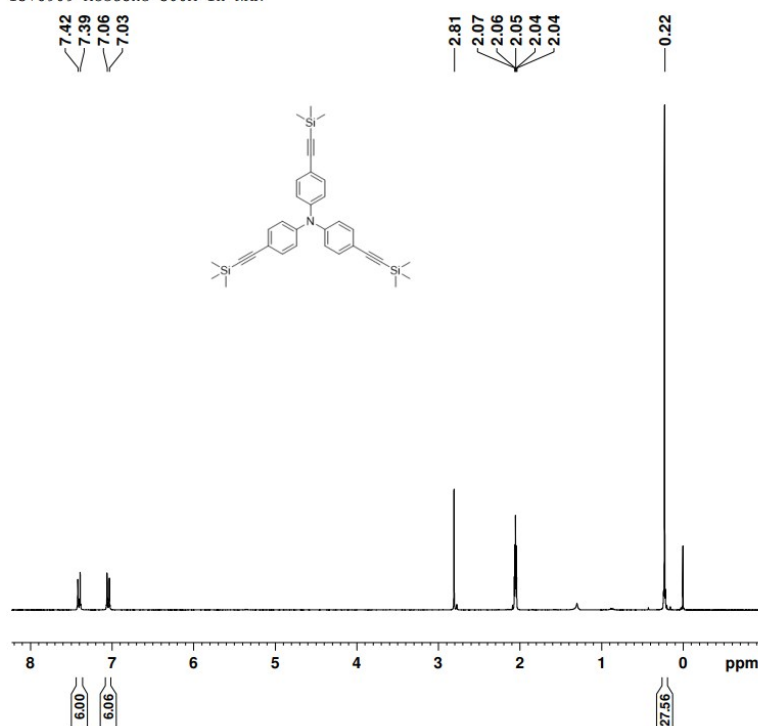
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Current Data Parameters
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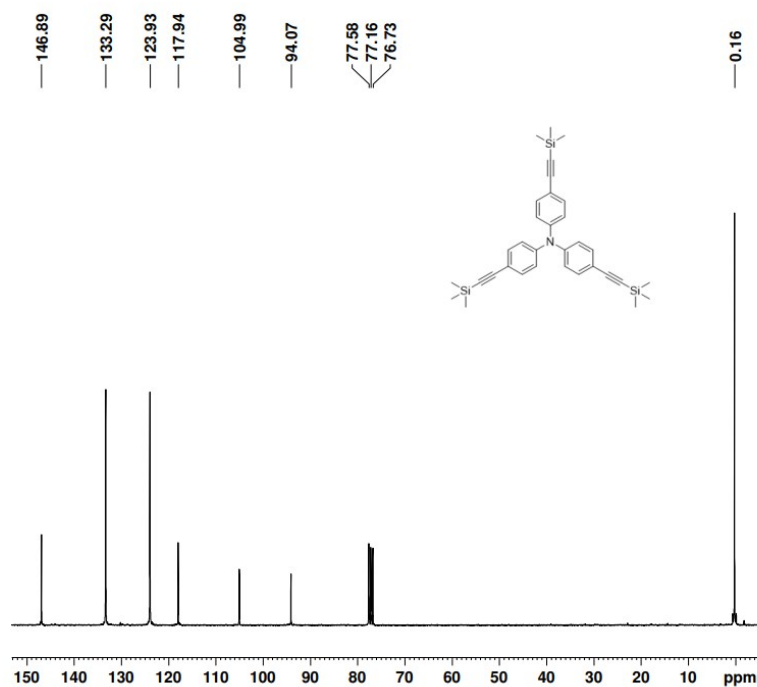
1378909 Acetone 300M 1H-NMR



Current Data Parameters
NAME 1378909-20190821
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190821
Time 18.29
INSTRUM WNMRI-300MHz
PULPROG zgpg30
TD 32050
SOLVENT Acetone
NS 15
DS 0
SWH 8012.024 Hz
AQ 2.0001190 sec
RG 63.6
DW 62.406 usec
DE 30.00 usec
TE 295.5 K
NUC1 1H
PL1 120.00 dB
SFO1 300.1505526 MHz

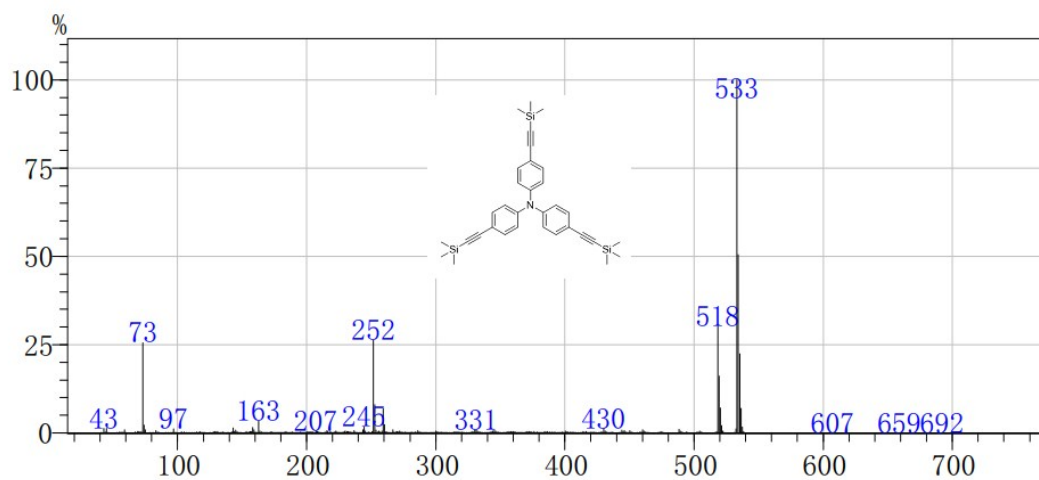
F2 - Processing parameters
SI 131072
SF 300.1490519 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0.1
PC 1.00

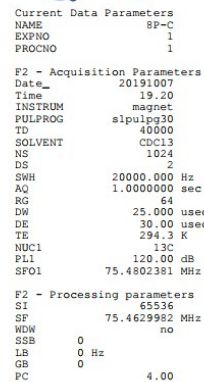
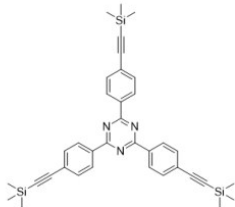
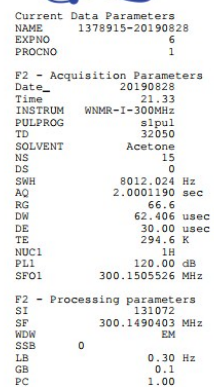
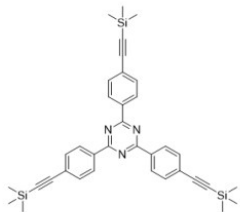


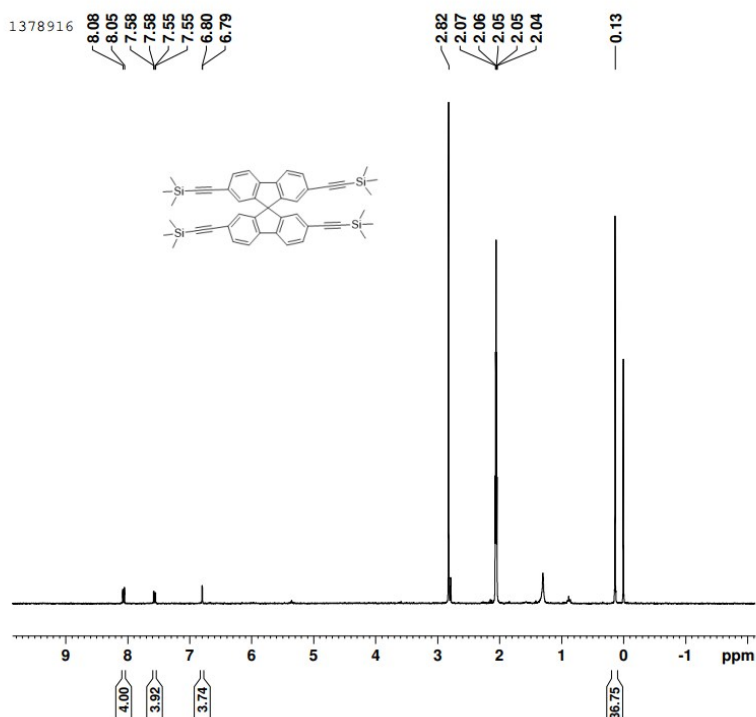
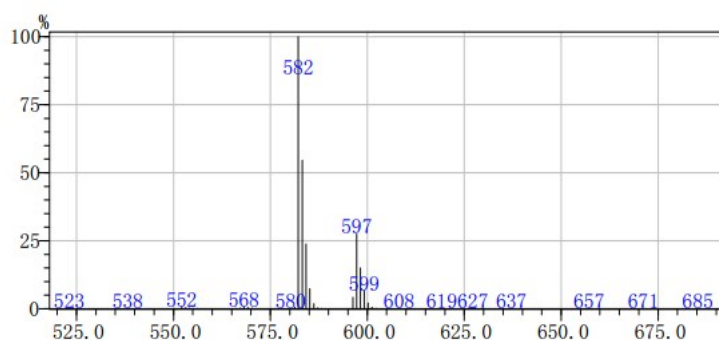
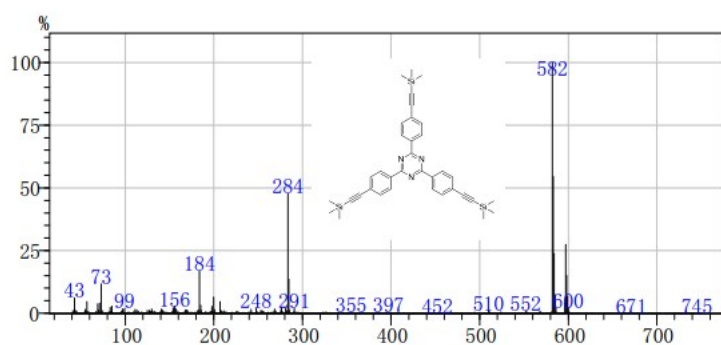
Current Data Parameters
NAME 7P-C
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20191007
Time 17.37
INSTRUM magnet
PULPROG spulpg30
TD 40000
SOLVENT CDCl3
NS 10000
DS 2
SWH 20000.000 Hz
AQ 1.0000000 sec
RG 64
DW 25.000 usec
DE 30.00 usec
TE 294.0 K
NUC1 13C
PL1 120.00 dB
SFO1 75.4802381 MHz

F2 - Processing parameters
SI 65536
SF 75.4726891 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 4.00



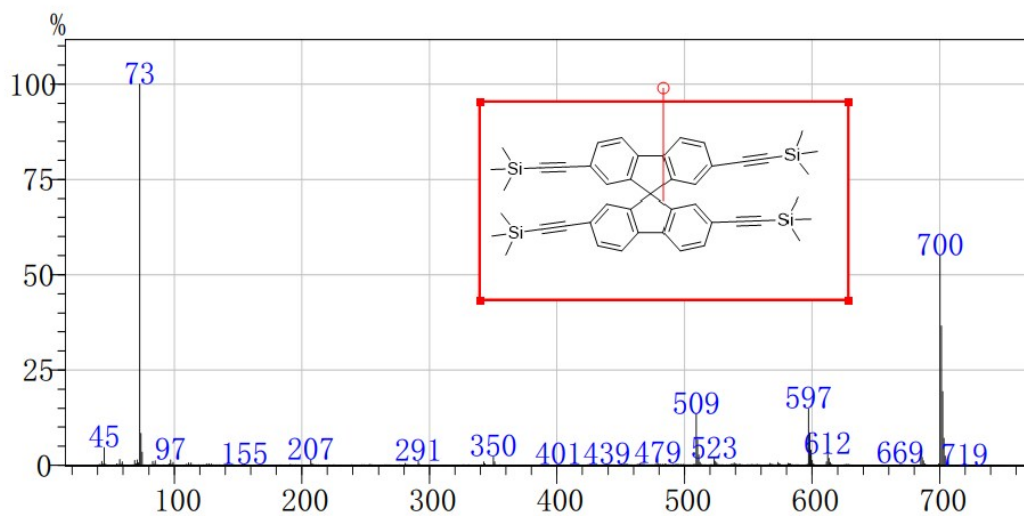
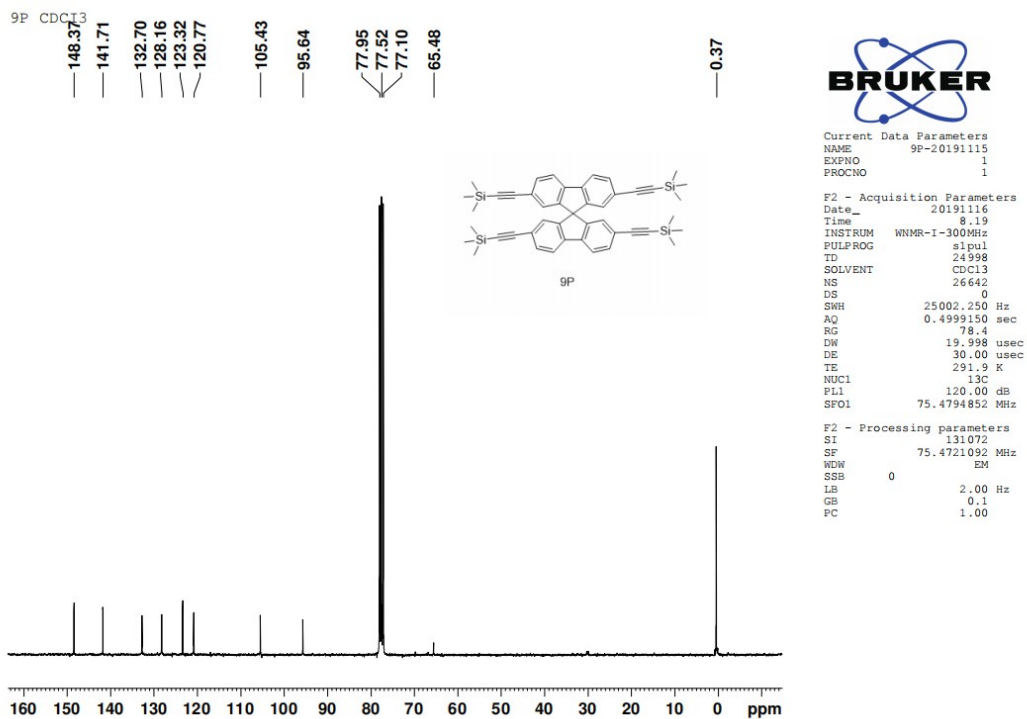




Current Data Parameters
 NAME 1378916-20190828
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20190828
 Time 21.14
 INSTRUM WNMN-1-300MHz
 PULPROG zgpg30
 TD 32050
 SOLVENT Acetone
 NS 15
 DS 0
 SWH 8012.024 Hz
 AQ 2.0001190 sec
 RG 35.2
 DW 62.406 usec
 DE 30.00 usec
 TE 294.6 K
 NUC1 ¹H
 PL1 120.00 dB
 SFO1 300.150526 MHz

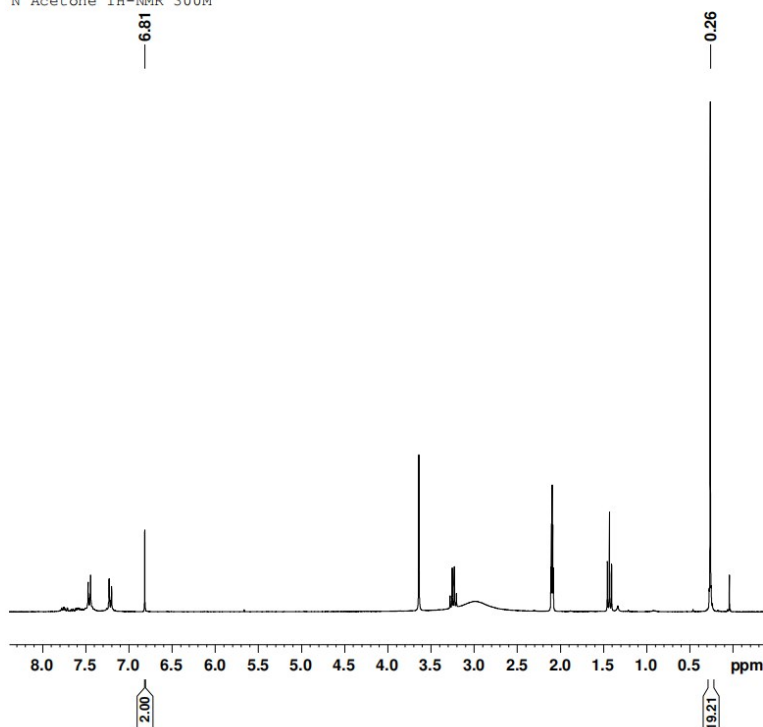
F2 - Processing parameters
 SI 131072
 SF 300.1490507 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0.1
 PC 1.00



3.2 Spectra data for calculating yield

Table 1, Entry 2

N Acetone 1H-NMR 300M

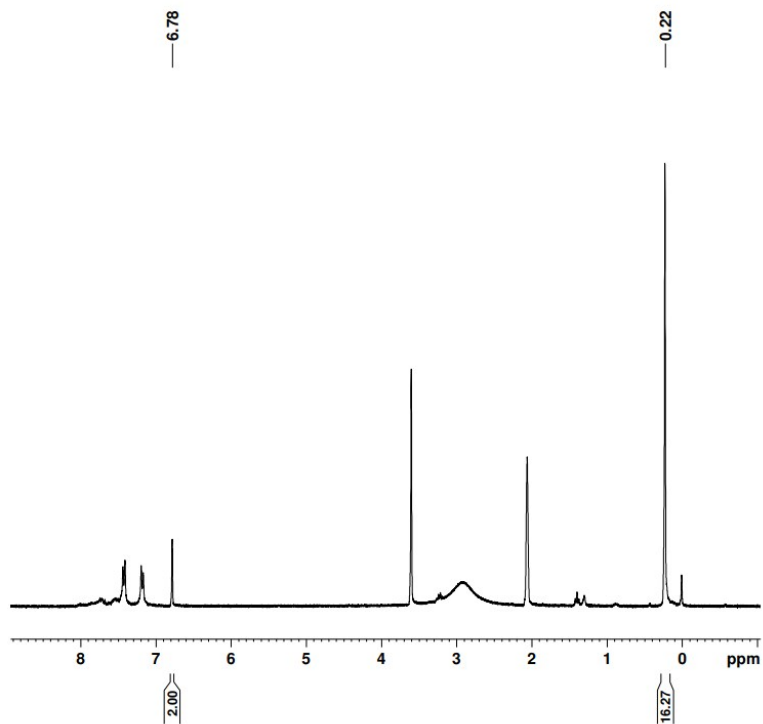


Current Data Parameters
NAME N-20190812
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190812
Time 19.35
INSTRUM WNMN-I-300MHz
PULPROG sipul
TD 32050
SOLVENT Acetone
NS 15
DS 0
SWH 8012.024 Hz
AQ 2.0001190 sec
RG 58.2
DN 62.406 usec
DE 30.00 usec
TE 295.4 K
NUC1 1H
PL1 120.00 dB
SFO1 300.1505526 MHz

F2 - Processing parameters
SI 131072
SF 300.1490403 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0.1
PC 1.00

Table 1, Entry 3

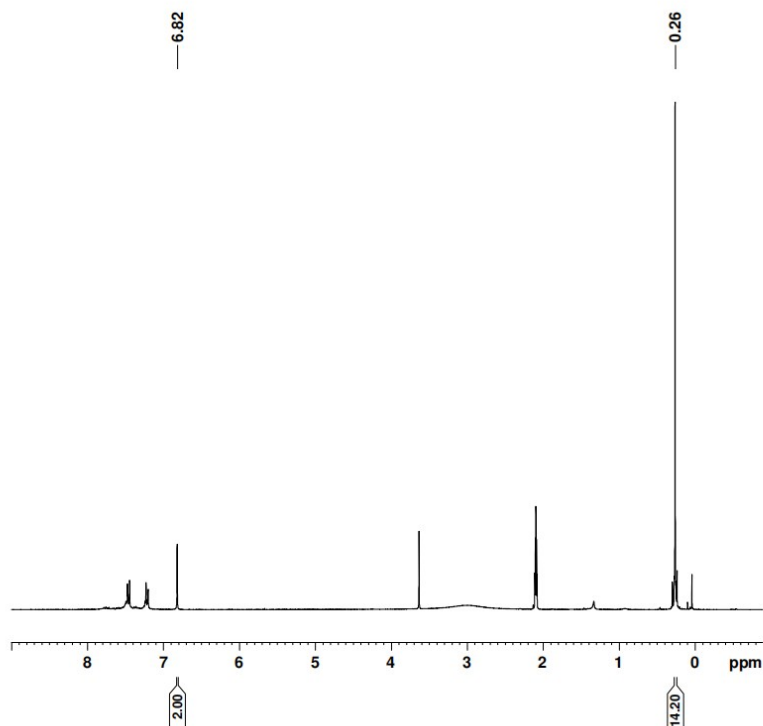


Current Data Parameters
NAME t3
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20191014
Time 18.16
INSTRUM magnet
PULPROG alpu130
TD 48978
SOLVENT Acetone
NS 16
DS 2
SWH 6122.000 Hz
AQ 4.0001636 sec
RG 64
DN 81.673 usec
DE 30.00 usec
TE 295.5 K
NUC1 1H
PL1 120.00 dB
SFO1 300.1514562 MHz

F2 - Processing parameters
SI 65536
SF 300.1496643 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 4.00

Table 1, Entry 4



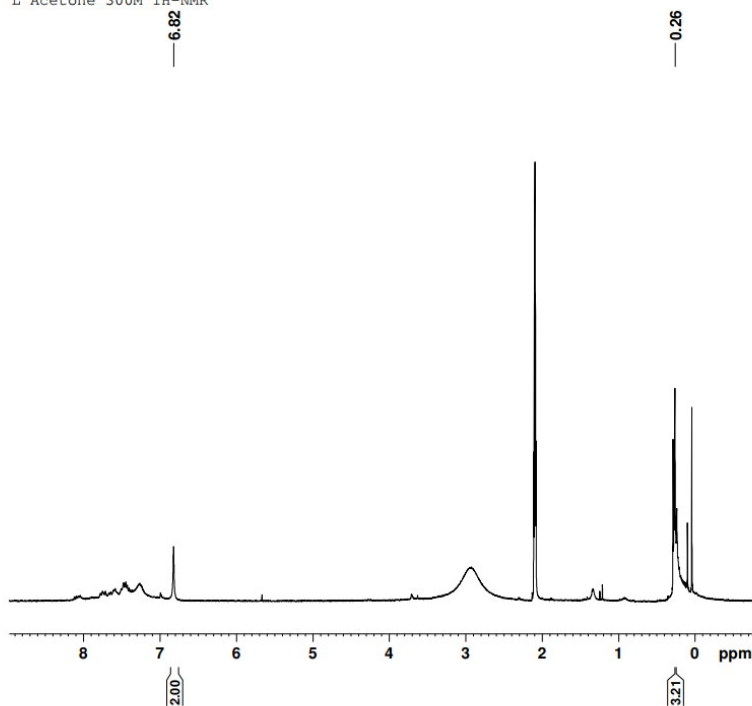
Current Data Parameters
NAME T10-20190626
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190626
Time 20.33
INSTRUM WNMRI-300MHz
PULPROG zgpg30
TD 32050
SOLVENT Acetone
NS 15
DS 0
SWH 8012.024 Hz
AQ 2.0001190 sec
RG 35.2
DW 62.406 usec
DE 30.00 usec
TE 294.3 K
NUC1 1H
PL1 120.00 dB
SFO1 300.150526 MHz

F2 - Processing parameters
SI 131072
SF 300.1490403 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0.1
PC 1.00

Table 1, Entry 5

L Acetone 300M 1H-NMR



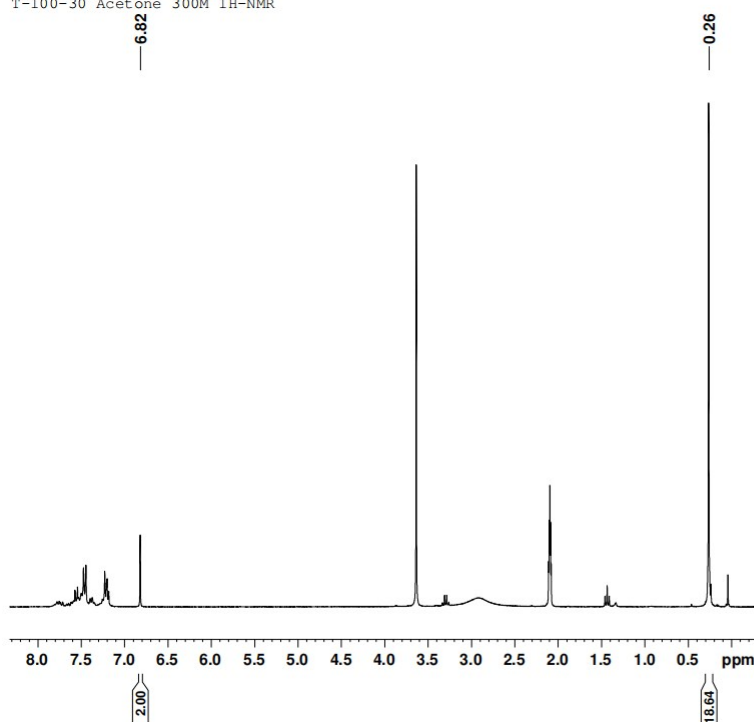
Current Data Parameters
NAME L-20190813
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190813
Time 8.18
INSTRUM WNMRI-300MHz
PULPROG zgpg30
TD 32050
SOLVENT Acetone
NS 15
DS 0
SWH 8012.024 Hz
AQ 2.0001190 sec
RG 35.2
DW 62.406 usec
DE 30.00 usec
TE 294.8 K
NUC1 1H
PL1 120.00 dB
SFO1 300.150526 MHz

F2 - Processing parameters
SI 131072
SF 300.1490403 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Table 1, Entry 6

T-100-30 Acetone 300M 1H-NMR

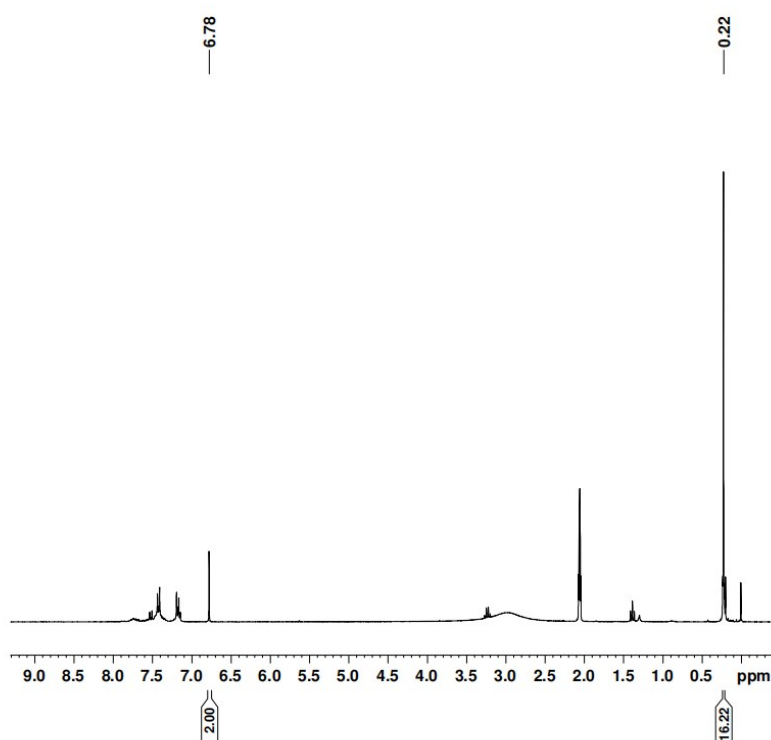


Current Data Parameters
NAME T-100-30-20191218
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20191218
Time 19.48
INSTRUM WNMRI-300MHz
PULPROG zgpg30
TD 32768
SOLVENT Acetone
NS 15
DS 0
SWH 8012.024 Hz
AQ 2.0001190 sec
RG 59.7
DW 62.406 usec
DE 30.00 usec
TE 294.0 K
NUC1 1H
FL1 120.00 dB
SFO1 300.1505526 MHz

F2 - Processing parameters
SI 131072
SF 300.1490403 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0.1
PC 1.00

Table 2, Entry 2



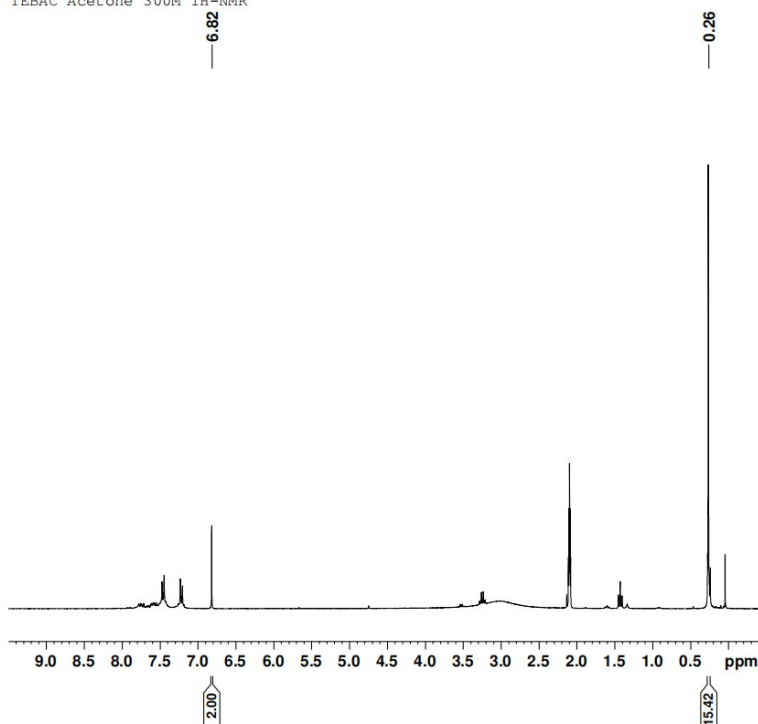
Current Data Parameters
NAME CD-20190816
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190816
Time 20.51
INSTRUM WNMRI-300MHz
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 15
DS 0
SWH 8012.024 Hz
AQ 2.0001190 sec
RG 60.5
DW 62.406 usec
DE 30.00 usec
TE 294.8 K
NUC1 1H
FL1 120.00 dB
SFO1 300.1505526 MHz

F2 - Processing parameters
SI 131072
SF 300.1490504 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0.1
PC 1.00

Table 2, Entry 3

TEBAC Acetone 300M 1H-NMR



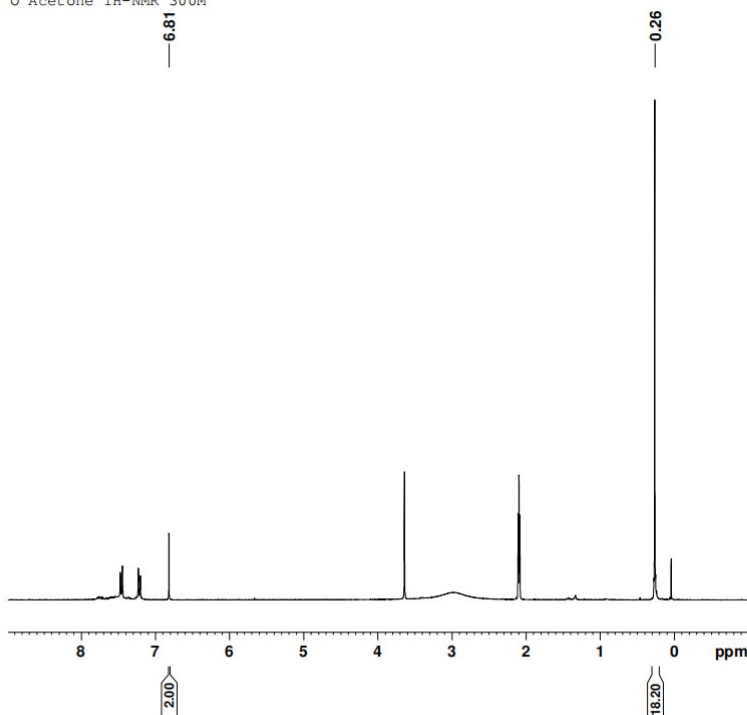
Current Data Parameters
NAME TEBAC-20190821
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190821
Time 19.11
INSTRUM WNMN-I-300MHz
PULPROG slpul
TD 32050
SOLVENT Acetone
NS 15
DS 0
SWH 8012.024 Hz
AQ 2.0001190 sec
RG 60.1
DW 62.406 usec
DE 30.00 usec
TE 296.6 K
NUC1 1H
PL1 120.00 dB
SFO1 300.1505526 MHz

F2 - Processing parameters
SI 131072
SF 300.1490403 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0.1
PC 1.00

Table 2, Entry 4

O Acetone 1H-NMR 300M



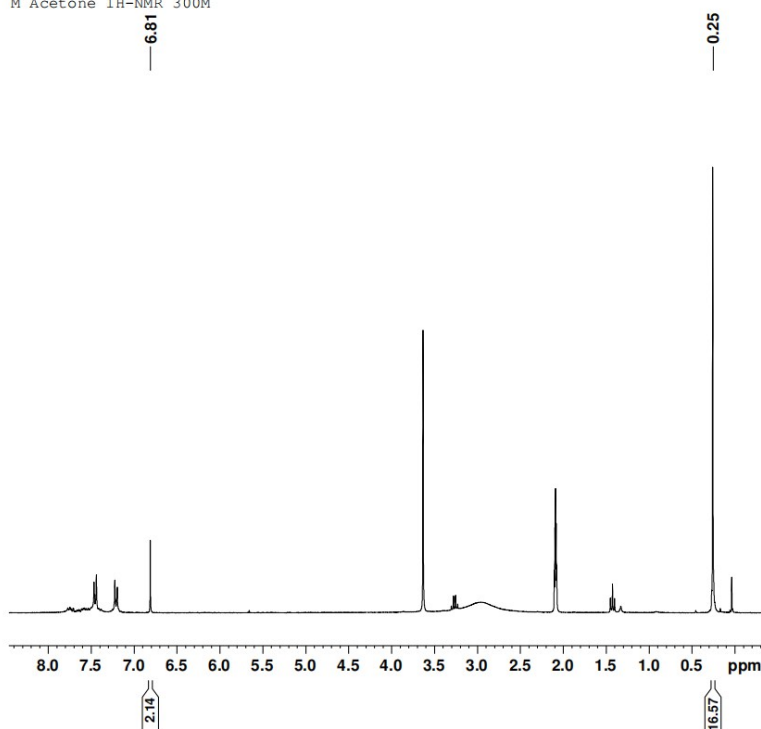
Current Data Parameters
NAME O-20190812
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190812
Time 19.55
INSTRUM WNMN-I-300MHz
PULPROG slpul
TD 32050
SOLVENT Acetone
NS 15
DS 0
SWH 8012.024 Hz
AQ 2.0001190 sec
RG 35.2
DW 62.406 usec
DE 30.00 usec
TE 295.4 K
NUC1 1H
PL1 120.00 dB
SFO1 300.1505526 MHz

F2 - Processing parameters
SI 131072
SF 300.1490403 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0.1
PC 1.00

Table 2, Entry 5

M Acetone 1H-NMR 300M



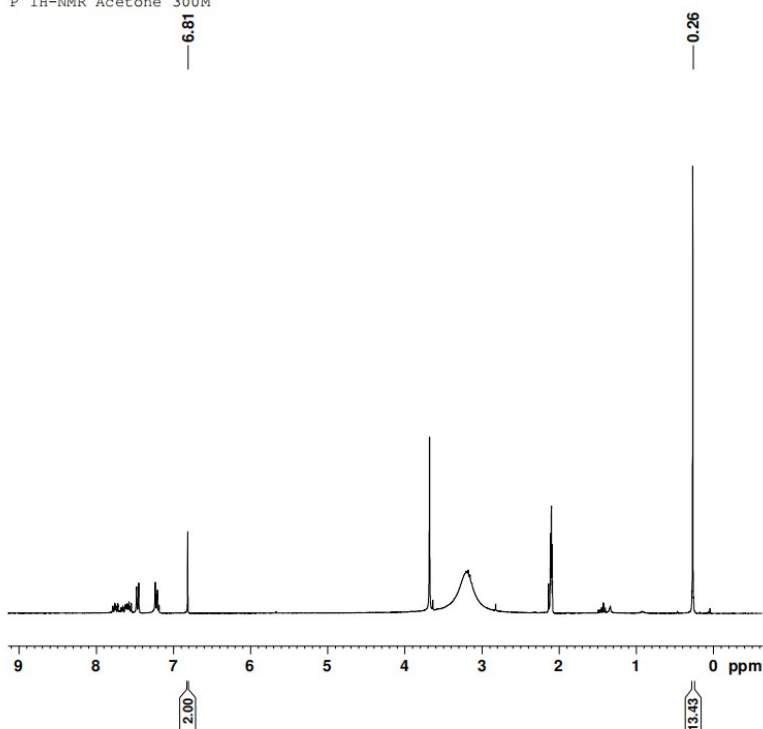
Current Data Parameters
NAME M-20190812
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190812
Time 19.42
INSTRUM WNMN-I-300MHz
PULPROG slpul
TD 32050
SOLVENT Acetone
NS 15
DS 0
SWH 8012.024 Hz
AQ 2.0001190 sec
RG 59
DW 62.406 usec
DE 30.00 usec
TE 295.4 K
NUC1 1H
PL1 120.00 dB
SFO1 300.150526 MHz

F2 - Processing parameters
SI 131072
SF 300.1490403 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0.1
PC 1.00

Table 2, Entry 6

P 1H-NMR Acetone 300M



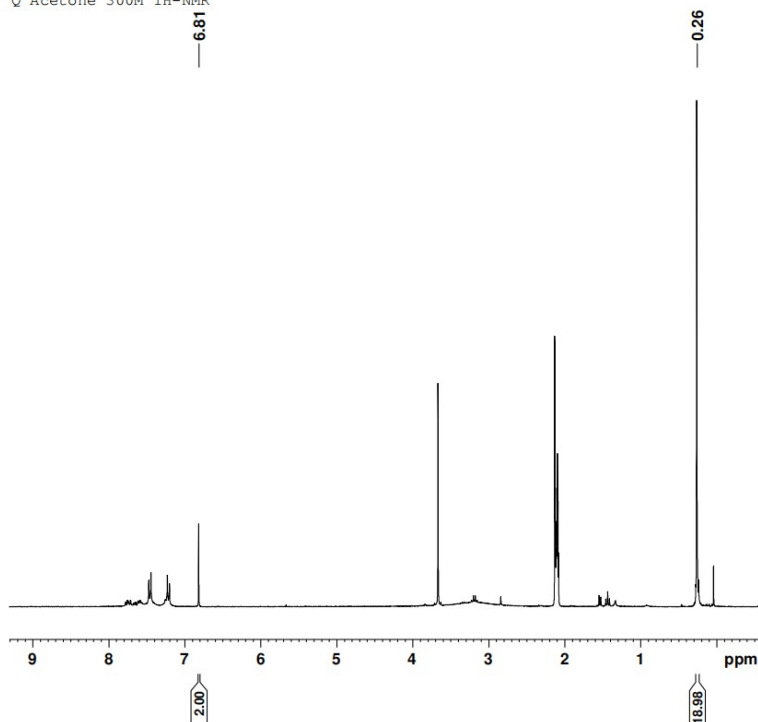
Current Data Parameters
NAME P-20190812
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190812
Time 19.49
INSTRUM WNMN-I-300MHz
PULPROG slpul
TD 32050
SOLVENT Acetone
NS 15
DS 0
SWH 8012.024 Hz
AQ 2.0001190 sec
RG 55
DW 62.406 usec
DE 30.00 usec
TE 295.4 K
NUC1 1H
PL1 120.00 dB
SFO1 300.150526 MHz

F2 - Processing parameters
SI 131072
SF 300.1490403 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0.1
PC 1.00

Table 2, Entry 8

Q Acetone 300M 1H-NMR



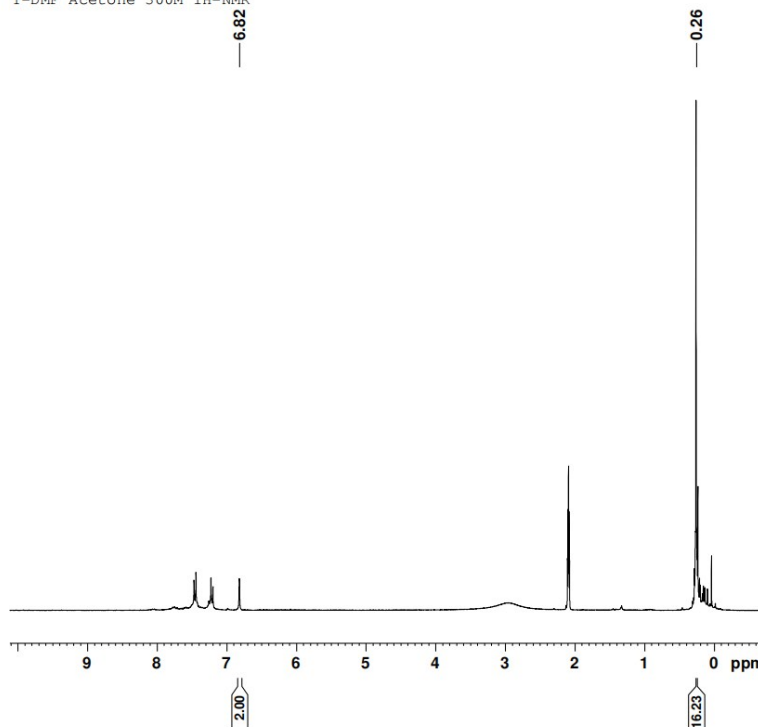
Current Data Parameters
NAME Q-20190813
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190813
Time 8.26
INSTRUM WNMRI-300MHz
PULPROG zgpg30
TD 32050
SOLVENT Acetone
NS 15
DS 0
SWH 8012.024 Hz
AQ 2.0001190 sec
RG 35.2
DW 62.406 usec
DE 30.00 usec
TE 294.9 K
NUC1 1H
PL1 120.00 dB
SFO1 300.150526 MHz

F2 - Processing parameters
SI 131072
SF 300.1490403 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0.1
PC 1.00

Table 2, Entry 10

T-DMF Acetone 300M 1H-NMR



Current Data Parameters
NAME T-DMF-20190712
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190712
Time 20.14
INSTRUM WNMRI-300MHz
PULPROG zgpg30
TD 32050
SOLVENT Acetone
NS 15
DS 0
SWH 8012.024 Hz
AQ 2.0001190 sec
RG 57.7
DW 62.406 usec
DE 30.00 usec
TE 294.4 K
NUC1 1H
PL1 120.00 dB
SFO1 300.150526 MHz

F2 - Processing parameters
SI 131072
SF 300.1490403 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0.1
PC 1.00

Table 2, Entry 11

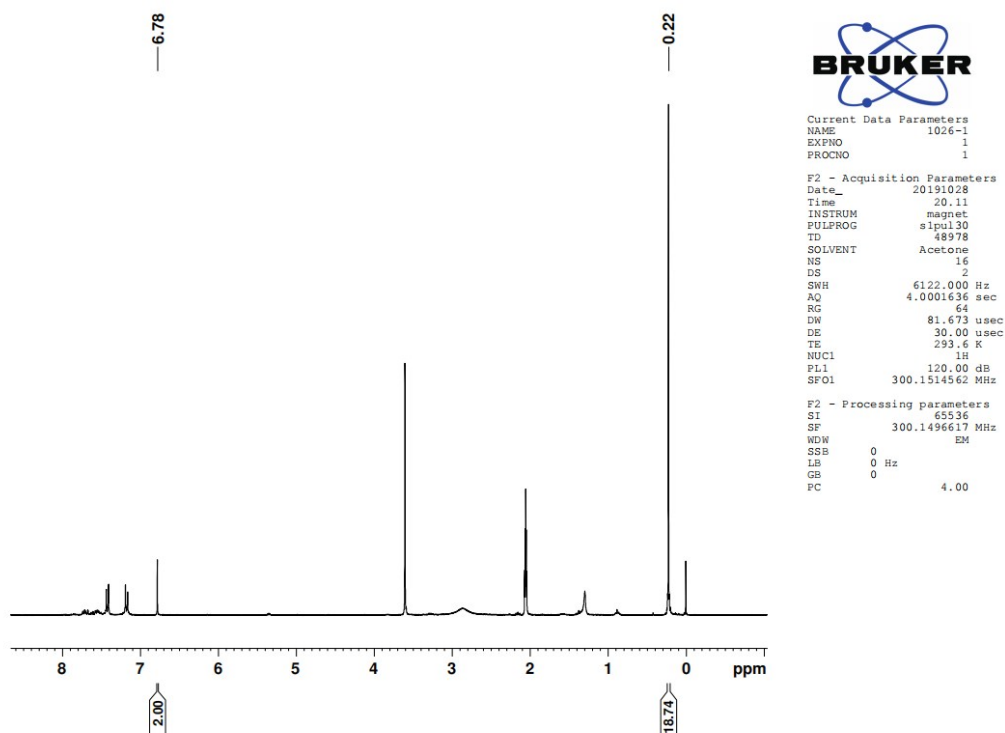


Table 2, Entry 12

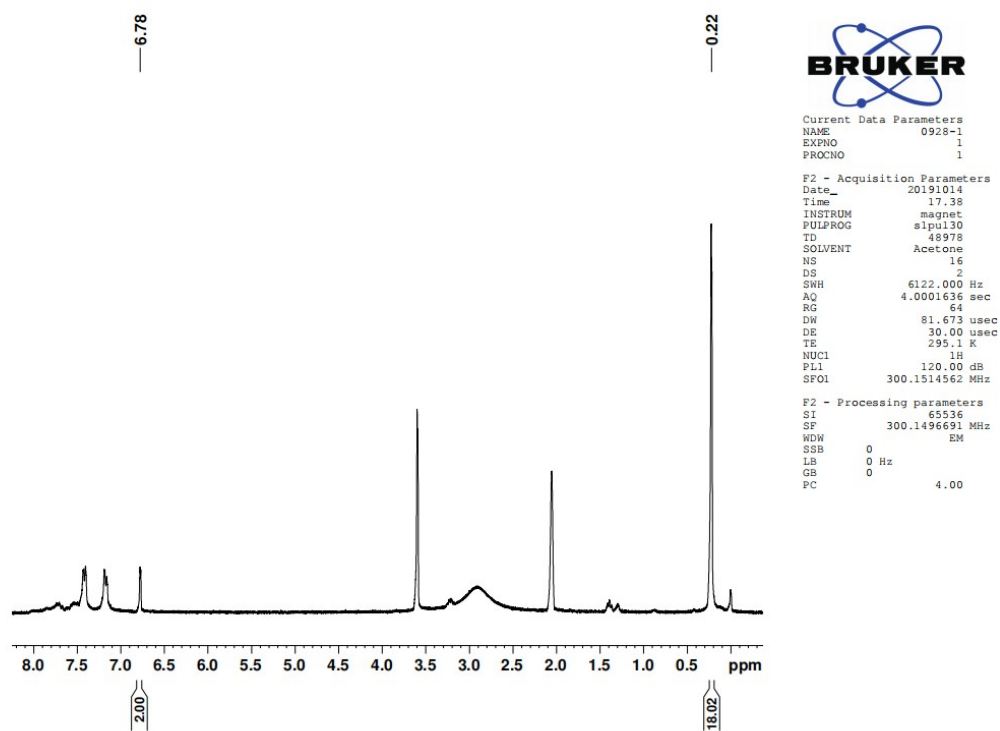
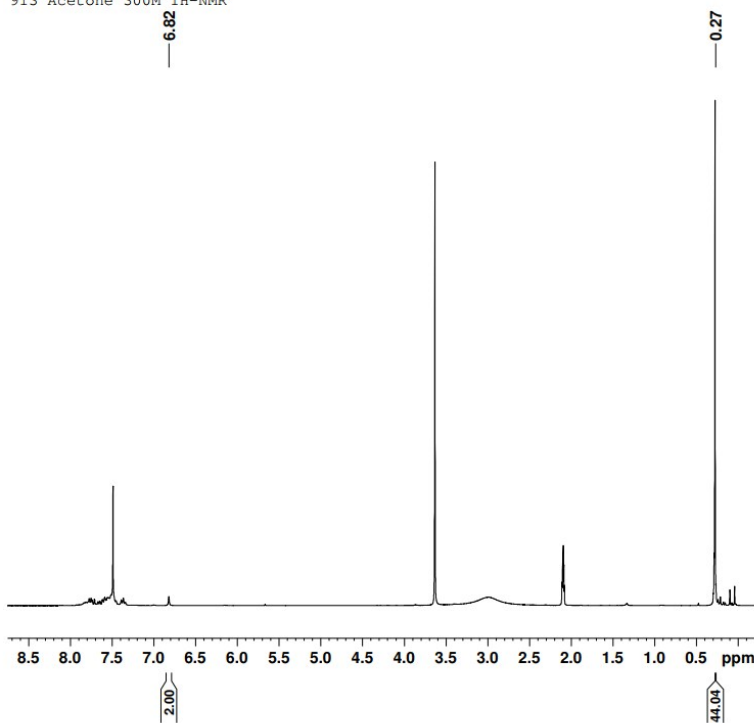


Table 3, Entry 1 (2P)

913 Acetone 300M 1H-NMR



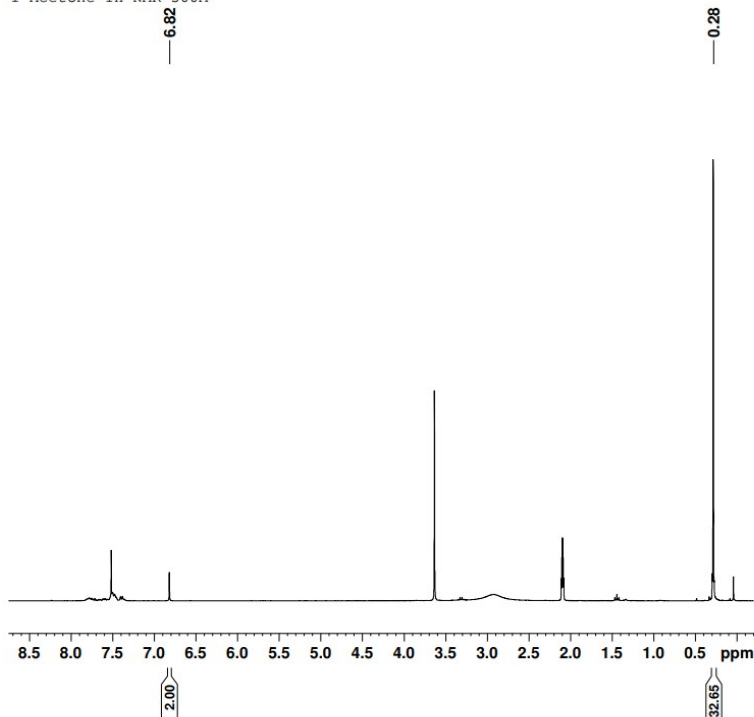
Current Data Parameters
 NAME 913-20190827
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20190827
 Time 18.42
 INSTRUM WNMN-1-300MHz
 PULPROG zgpg30
 TD 32050
 SOLVENT Acetone
 NS 15
 DS 0
 SWH 8012.024 Hz
 AQ 2.0001190 sec
 RG 55.2
 DW 62.406 usec
 DE 30.00 usec
 TE 294.6 K
 NUC1 1H
 PL1 120.00 dB
 SFO1 300.1505526 MHz

F2 - Processing parameters
 SI 131072
 SF 300.1490403 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0.1
 PC 1.00

Table 3, Entry 2 (3P)

1 Acetone 1H-NMR 300M



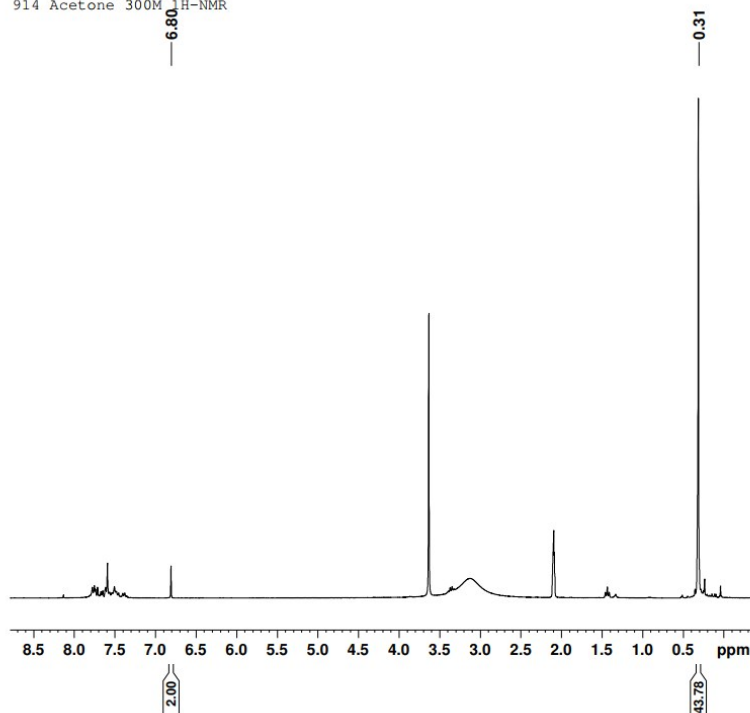
Current Data Parameters
 NAME 1-20190831
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20190831
 Time 18.31
 INSTRUM WNMN-1-300MHz
 PULPROG zgpg30
 TD 32050
 SOLVENT Acetone
 NS 15
 DS 0
 SWH 8012.024 Hz
 AQ 2.0001190 sec
 RG 58.4
 DW 62.406 usec
 DE 30.00 usec
 TE 294.5 K
 NUC1 1H
 PL1 120.00 dB
 SFO1 300.1505526 MHz

F2 - Processing parameters
 SI 131072
 SF 300.1490403 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0.1
 PC 1.00

Table 3, Entry 3 (4P)

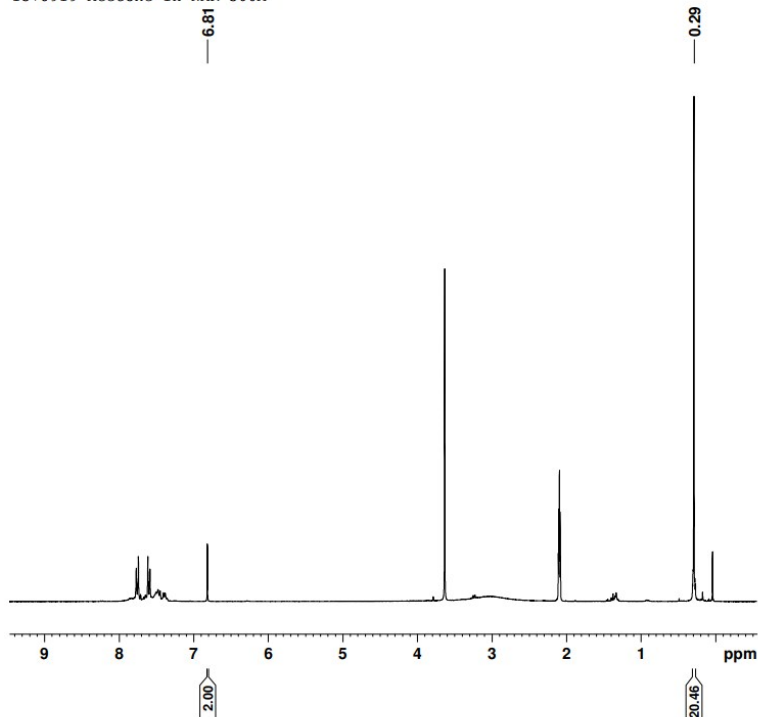
914 Acetone 300M ¹H-NMR



Current Data Parameters
 NAME 914-20190827
 EXPNO 1
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20190827
 Time 18.54
 INSTRUM WNMN-1-300MHz
 PULPROG zgpg30
 TD 32050
 SOLVENT Acetone
 NS 15
 DS 0
 SWH 8012.024 Hz
 AQ 2.0001190 sec
 RG 34.3
 DW 62.406 usec
 DE 30.00 usec
 TE 294.0 K
 NUC1 1H
 PL1 120.00 dB
 SFO1 300.1505526 MHz
 F2 - Processing parameters
 SI 131072
 SF 300.1490403 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0.1
 PC 1.00

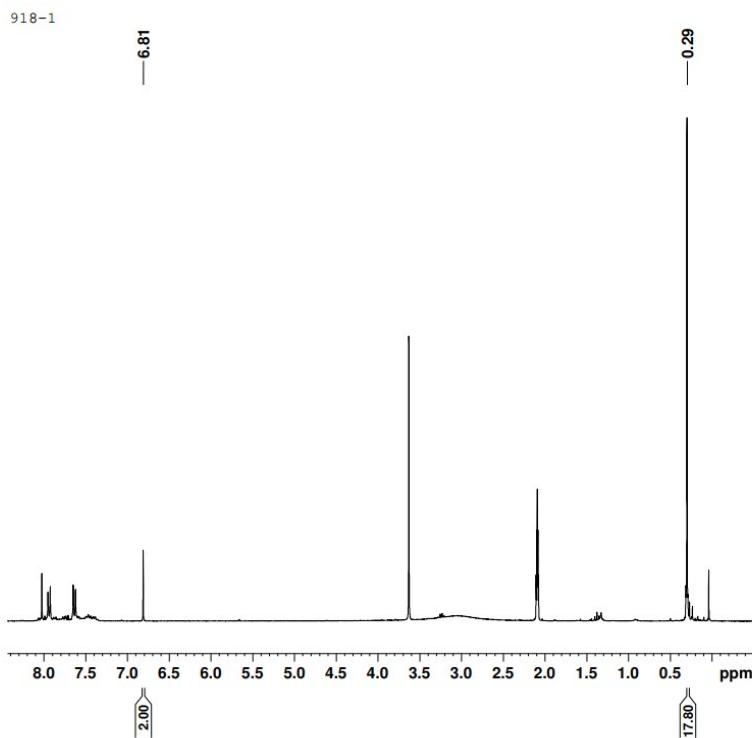
Table 3, Entry 4 (5P)

1378919 Acetone 1H-NMR 300M



Current Data Parameters
 NAME 1378919-20190831
 EXPNO 1
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20190831
 Time 18.37
 INSTRUM WNMN-1-300MHz
 PULPROG zgpg30
 TD 32050
 SOLVENT Acetone
 NS 15
 DS 0
 SWH 8012.024 Hz
 AQ 2.0001190 sec
 RG 34.3
 DW 62.406 usec
 DE 30.00 usec
 TE 294.0 K
 NUC1 1H
 PL1 120.00 dB
 SFO1 300.1505526 MHz
 F2 - Processing parameters
 SI 131072
 SF 300.1490403 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0.1
 PC 1.00

Table 3, Entry 5 (6P)

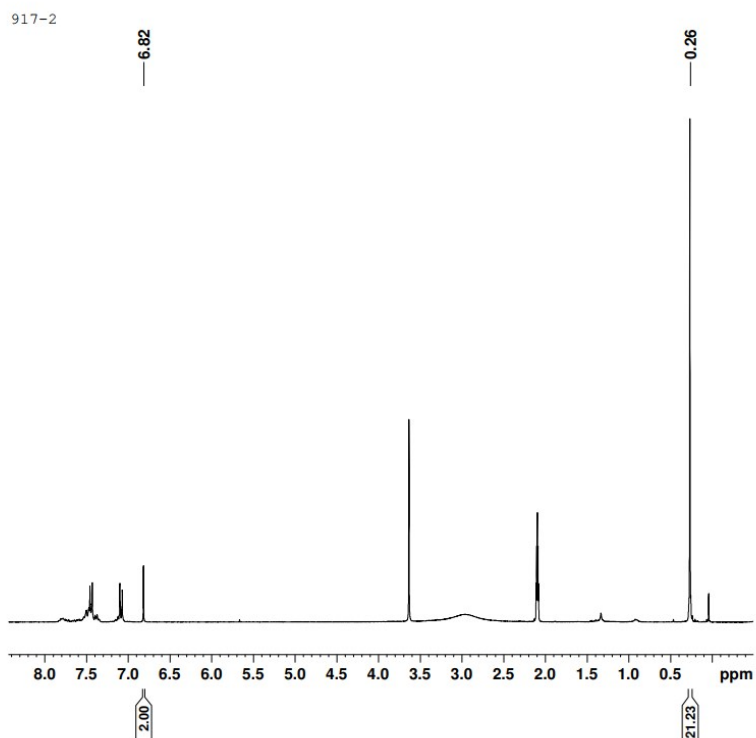


Current Data Parameters
 NAME 918-1-20190828
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20190828
 Time 21.40
 INSTRUM WNMN-I-300MHz
 PULPROG sgpr1
 TD 32050
 SOLVENT Acetone
 NS 15
 DS 0
 SWH 8012.024 Hz
 AQ 2.0001190 sec
 RG 35.2
 DW 62.406 usec
 DE 30.00 usec
 TE 294.6 K
 NUC1 1H
 PL1 120.00 dB
 SFO1 300.1505526 MHz

F2 - Processing parameters
 SI 131072
 SF 300.1490403 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0.1
 PC 1.00

Table 3, Entry 6 (7P)



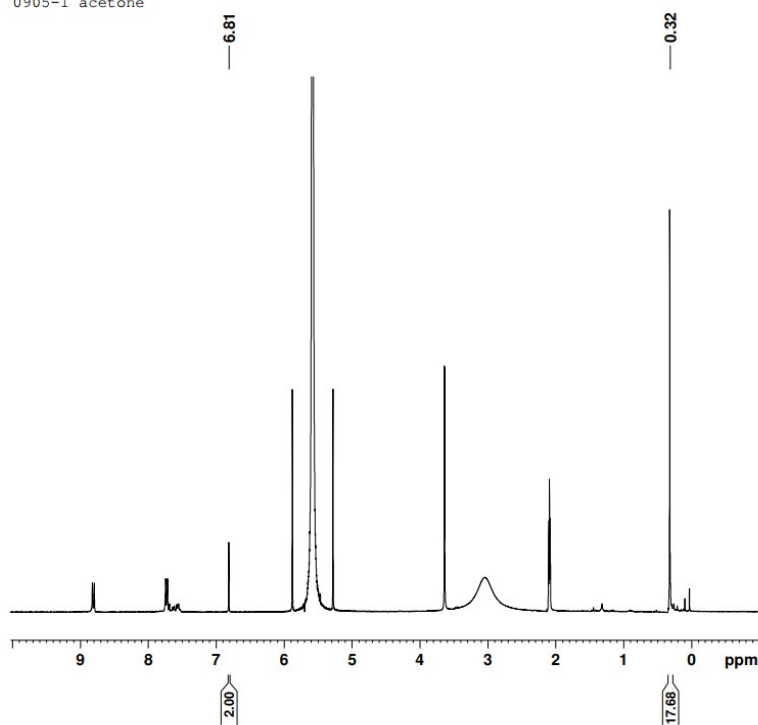
Current Data Parameters
 NAME 917-2-20190828
 EXPNO 4
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20190828
 Time 22.05
 INSTRUM WNMN-I-300MHz
 PULPROG sgpr1
 TD 32050
 SOLVENT Acetone
 NS 15
 DS 0
 SWH 8012.024 Hz
 AQ 2.0001190 sec
 RG 35.2
 DW 62.406 usec
 DE 30.00 usec
 TE 294.6 K
 NUC1 1H
 PL1 120.00 dB
 SFO1 300.1505526 MHz

F2 - Processing parameters
 SI 131072
 SF 300.1490403 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0.1
 PC 1.00

Table 3, Entry 7 (8P)

0905-1 acetone

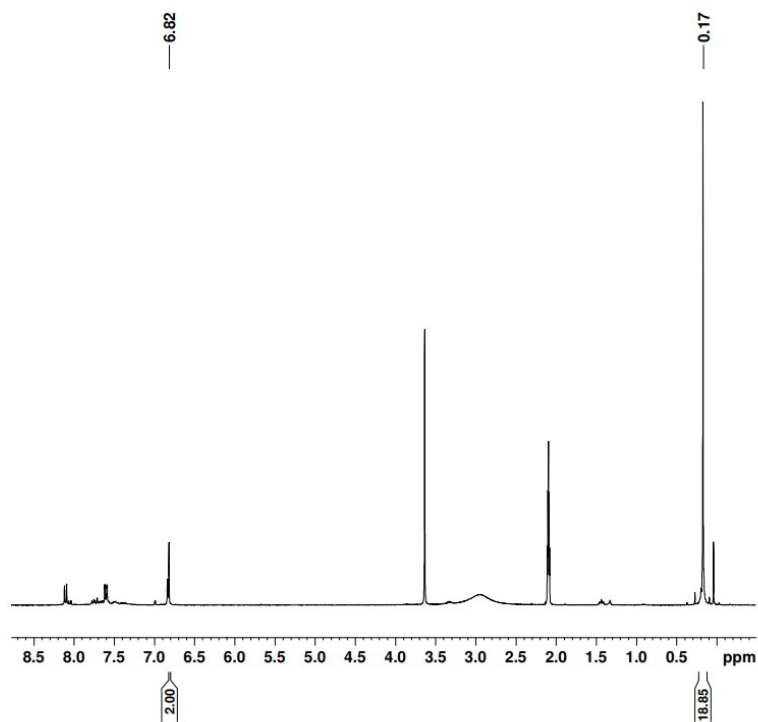


Current Data Parameters
 NAME 0905-1-20190920
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20190920
 Time 19.53
 INSTRUM WNMRI-300MHz
 PULPROG zgpg30
 TD 32768
 SOLVENT Acetone
 NS 15
 DS 0
 SWH 8012.024 Hz
 AQ 2.0001190 sec
 RG 36.8
 DW 62.406 usec
 DE 30.00 usec
 TE 295.4 K
 NUC1 1H
 PL1 120.00 dB
 SFO1 300.1505526 MHz

F2 - Processing parameters
 SI 131072
 SF 300.1490403 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0.1
 PC 1.00

Table 3, Entry 8 (9P)



Current Data Parameters
 NAME 0905-2-20190909
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20190909
 Time 18.50
 INSTRUM WNMRI-300MHz
 PULPROG zgpg30
 TD 32768
 SOLVENT Acetone
 NS 15
 DS 0
 SWH 8012.024 Hz
 AQ 2.0001190 sec
 RG 36.8
 DW 62.406 usec
 DE 30.00 usec
 TE 292.9 K
 NUC1 1H
 PL1 120.00 dB
 SFO1 300.1505526 MHz

F2 - Processing parameters
 SI 131072
 SF 300.1490403 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0.1
 PC 1.00