Supplementary Information

For

Nickel-Catalysed Cycloaddition Oligomerization of 1,6-Diynes to Medium-Size Cyclic Polyenes

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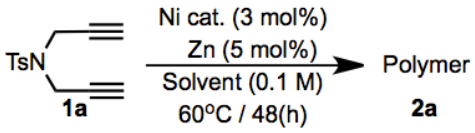
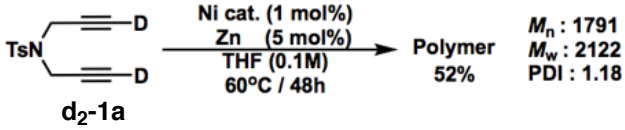
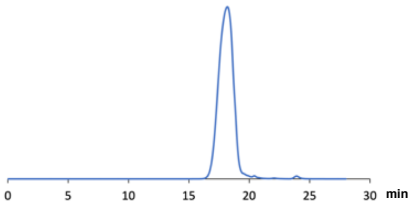
S-1. Solvent effects



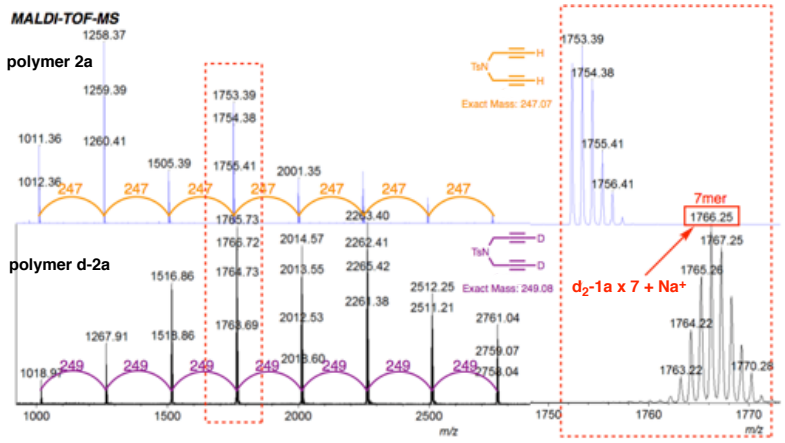
entry	Solvent	yield	Polymer[a] M_n	M_w	PDI	entry	Solvent	yield	Polymer[a] M_n	M_w	PDI
1	THF	70%.	1908	2339	1.23	4	DMA	49%	1735	2016	1.16
2	DMSO	38%	1837	2144	1.17	5	NMP	59%	1852	2230	1.20
3	Dioxane	64%	1616	1915	1.18	6	*t*-BuOH	No reaction			

S-2. Results for the reaction of deuterated diyne **d_2-1a**

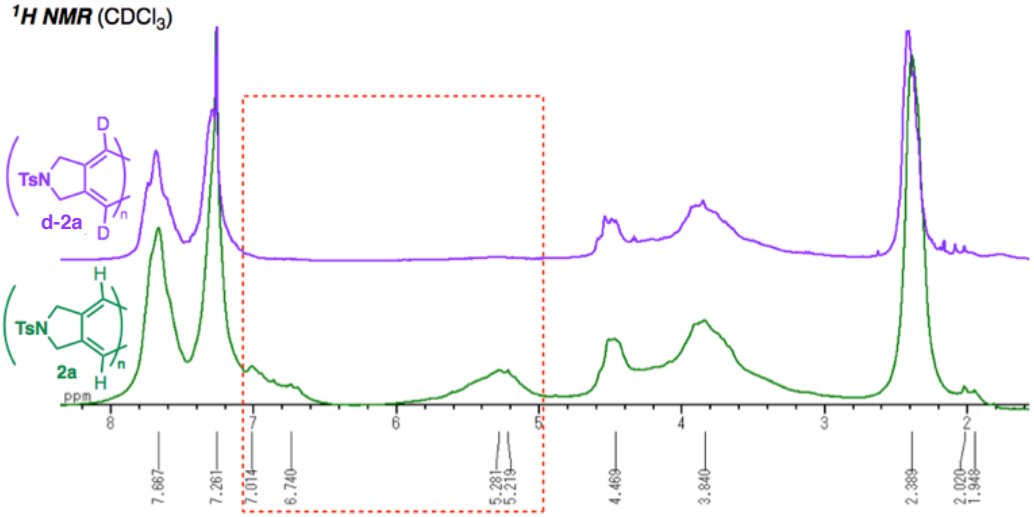




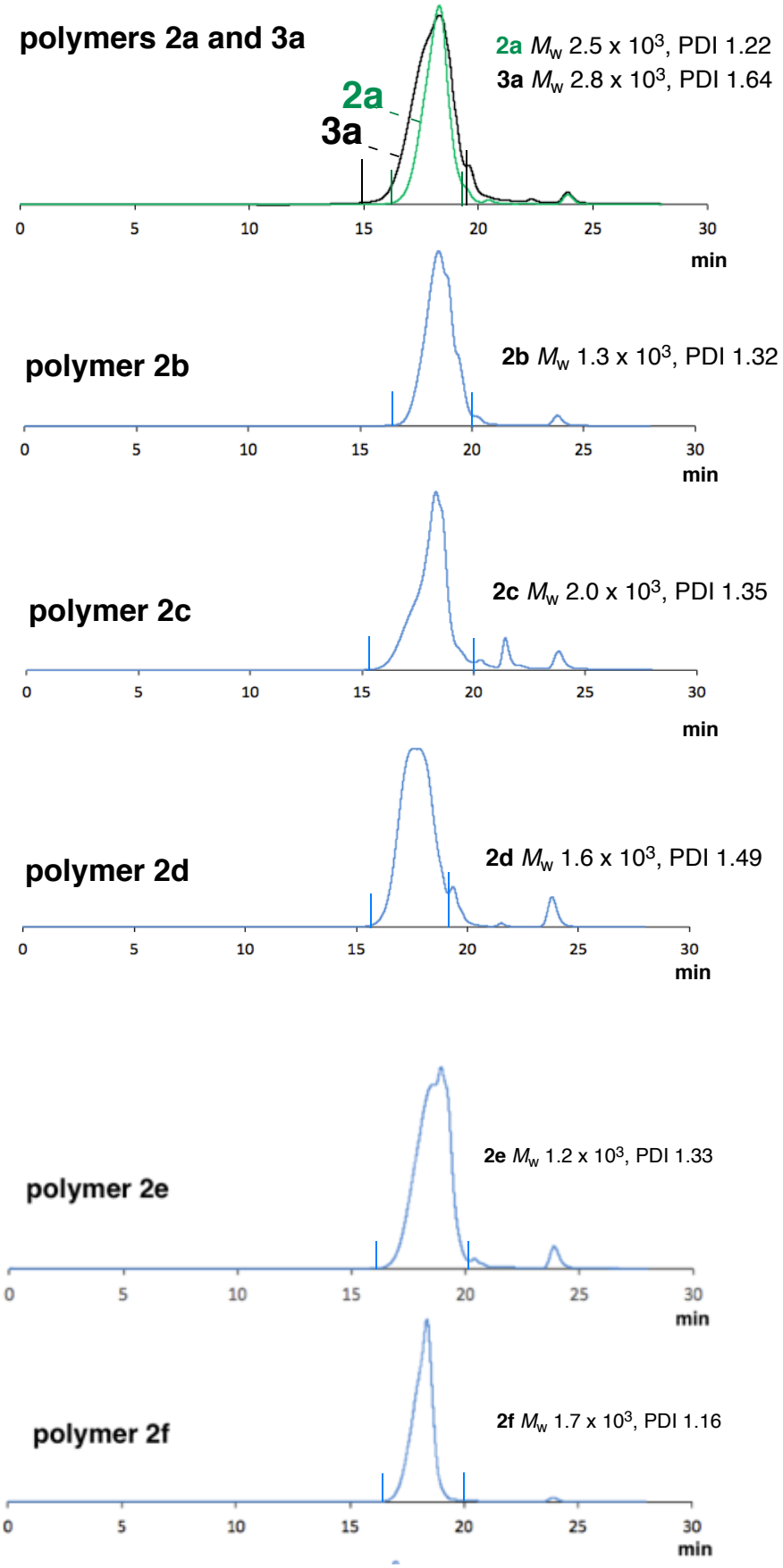
GPC profile



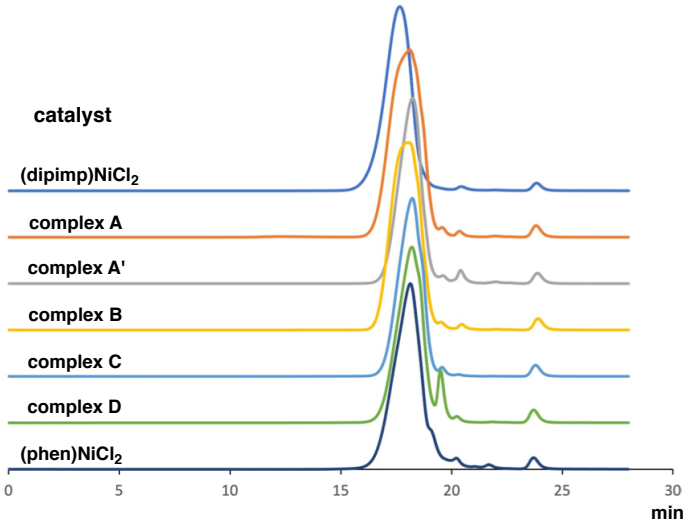
MALDI-TOF-MS spectra

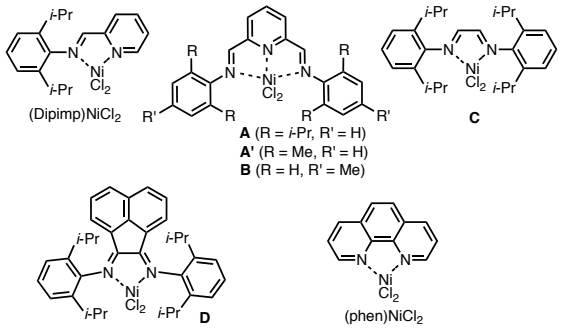


S-3 GPC profiles of crude polymers prepared by the reaction catalyzed by complex A



S-4 GPC profiles of crude polymers derived from diyne 1a with various catalysts





Experimental Procedures

General. NMR spectra were recorded in deuterated chloroform ($CDCl_3$) at 600, 500, and 400 MHz for ^1H and 150, 125, and 100 MHz for ^{13}C on JEOL JNM-ECA600, -ECA500, and –ECZ400 spectrometers, respectively. Chemical shifts are reported in parts per million (ppm, δ) relative to trimethylsilane (Me_4Si, δ 0.00) or residual $CHCl_3$ (δ 7.26 for ^1H NMR), and $CDCl_3$ (δ 77.0 for ^{13}C NMR). MALDI-TOF mass spectra were recorded using a Shimadzu Biotech Axima CFRplus with curved field reflection (CFR) in reflection ion mode with a laser (λ = 337 nm). IR spectra were recorded on a JASCO IR FT/IR 4100 spectrometer. UV–vis absorption spectra were recorded using Shimazdu UV-2450 spectrometers. Thermogravimetry/differential thermal analysis (TG/DTA) was carried out using Seiko Instruments Inc. EXSTAR6000 TG/DTA6200 under nitrogen (heating rate: 10 °C /min). High-resolution mass spectroscopy (HR–MS) was performed on a JEOL Accu TOF T-100 instrument equipped with electrospray ionization (ESI) unit. The molecular weights (M_n, M_w) and polydispersities (M_w/M_n) of the polymers were determined with a TOSOH HLC-8020 gel permeation chromatograph (GPC) unit [eluent: THF; calibration: polystyrene standards] using two TSK-gel columns (2 × Multipore H_{XL}-M). All reactions sensitive to oxygen and/or moisture were performed under an argon atmosphere. Dry solvents [THF, *N*,*N*-dimethylformamide (DMF), dichloromethane (CH_2Cl_2), toluene, *N*-methyl-2-pyrrolidone (NMP), and diethyl ether (ether)] were purchased from Kanto Chemicals. (Dipimp)$NiCl_2$,[1] complexes **A**,[2] **B**,[2] **C**,[3] **D**,[4] and (phen)$NiCl_2$[5] were prepared by the reported procedures. Diynes **1a**,[6] **1b**,[7] **1c**,[8] **1d**,[9] **1e**,[10] **1f**,[6] and **1a-d$_2$**[11] were prepared according to the literature.

4-(((*tert*-Butyldimethylsilyl)oxy)methyl)-*N*,*N*-di(prop-2-yn-1-yl)benzenesulfonamide (1a'). To a mixture of 4-(hydroxymethyl)-*N*,*N*-di(prop-2-yn-1-yl)benzenesulfonamide[12] (0.810 g, 3.08 mmol) and imidazole (0.462 g, 6.78 mmol) in DMF (15 mL) was added *t*-BuMe_2SiCl (0.556 g, 3.70 mmol) and the mixture was stirred for 12 h at room temperature. After the addition of water, the mixture was extracted with Et_2O. The combined organic layers were washed with brine, dried over $MgSO_4$, filtered, and concentrated. The residue was purified by recrystallization in hexane to provide **1a'** (1.09 g) in 94% yield. ^1H NMR (400 MHz, $CDCl_3$) δ 7.69 (d, *J* = 8.8 Hz, 2H, Ar), 7.35 (d, *J* = 8.8 Hz, 2H, Ar), 4.69 (s, 2H, OCH_2), 4.07 (d, *J* = 2.0 Hz, 4H, NCH_2), 2.03 (t, *J* = 2.0 Hz, 2H, CH), 0.84 (s, 9H, *t*Bu), -0.01 (s, 6H, $SiCH_3$); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.3, 136.4, 127.9, 126.1, 76.1, 64.2, 36.2, 25.9, 18.4, -5.3; IR (ATR) 3279, 2953, 2927, 2884, 2857, 2120, 1600, 1471, 1409, 1361, 1342, 1322, 1254, 1210, 1154, 1124, 1091, 1017, 955, 890, 834, 775 cm^{-1}. HR-MS (ESI^+) for $C_{19}H_{27}NO_3SSiNa$: Calcd. 400.1373, Found 400.1392.

General procedure for nickel-catalyzed cycloaddition cyclooligomerization of 1,6-diyne. Under argon atmosphere, to a stirred mixture of Zn powder (3.3 mg, 0.05 mmol) and diyne **1** (1.0 mmol) in THF (4 mL) was added a solution of complex **A** (6.0 mg, 0.01 mmol) in THF (6 mL) at 60 °C. After stirring for 48 h at this temperature, the mixture was allowed to cool to ambient temperature and 4 mL of Et_2O was added. The resulting mixture was passed through a pad

of Celite and the filtrate was concentrated under reduced pressure. The residue was chromatographed on silica gel to obtain cyclic polyene **2**.

Polymer **2a** (317 mg) was obtained in 78% yield from **1a** (247 mg, 1.00 mmol).

Polymer **2b** (108 mg) was obtained in 55% yield from **1b** (197 mg, 1.00 mmol).

Polymer **2c** (105 mg) was obtained in 41% yield from **1c** (255 mg, 1.00 mmol).

Polymer **2d** (143 mg) was obtained in 74% yield from **1d** (193 mg, 1.00 mmol).

Polymer **2e** (71 mg) was obtained in 37% yield from **1e** (191 mg, 1.00 mmol).

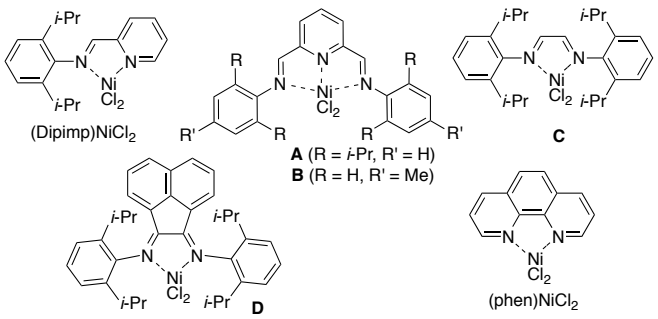
Polymer **2f** (175 mg) was obtained in 78% yield from **1f** (292 mg, 1.00 mmol).

Copolymerization of 1a and 1a'. Under argon atmosphere, to a stirred mixture of Zn powder (9.8 mg, 0.03 mmol) and diyne **1a** (649 mg 2.625 mmol) and **1a'** (93 mg, 0.375 mmol) in THF (12 mL) was added a solution of complex **A** (18.0 mg, 0.03 mmol) in THF (18 mL) at 60 °C. After stirring for 48 h at this temperature, the mixture was allowed to cool to ambient temperature and 15 mL of Et_2O was added. The resulting mixture was passed through a pad of Celite and the filtrate was concentrated under reduced pressure. The corresponding COT **4a** and benzene derivatives were removed from the crude residue by chromatography on silica gel to obtain a mixture of polyene **2** and **2a'-OTBS** (total 412 mg) in ~56% yield.

Desilylation of 2a'-OTBS and isolation of 2a'-OH. To a solution of **2a'-OTBS** (412 mg, approximately 1.67 mmol) in THF (5 mL) was added *n*-Bu_4NF (3.3 mL, 1.0 M in THF, 3.3 mmol) at room temperature. After being stirred for 12 h, water was added. The mixture was extracted with AcOEt (15 mL), dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The residue was chromatographed on silica gel (hexane/AcOEt) to give **2a** (105 mg) and **2a'-OH** (126 mg) in 14% yield and ~17% yield (based on **1a** and **1a'** (total 3.0 mmol)), respectively.

Catalysts

Nickel complexes, (dipimp)$NiCl_2$, complex **A**,[13] complex **B**,[14,15] complex **C**,[14,15] complex **D**,[16] and (phen)$NiCl_2$[17] were prepared according to the reported procedure.



References

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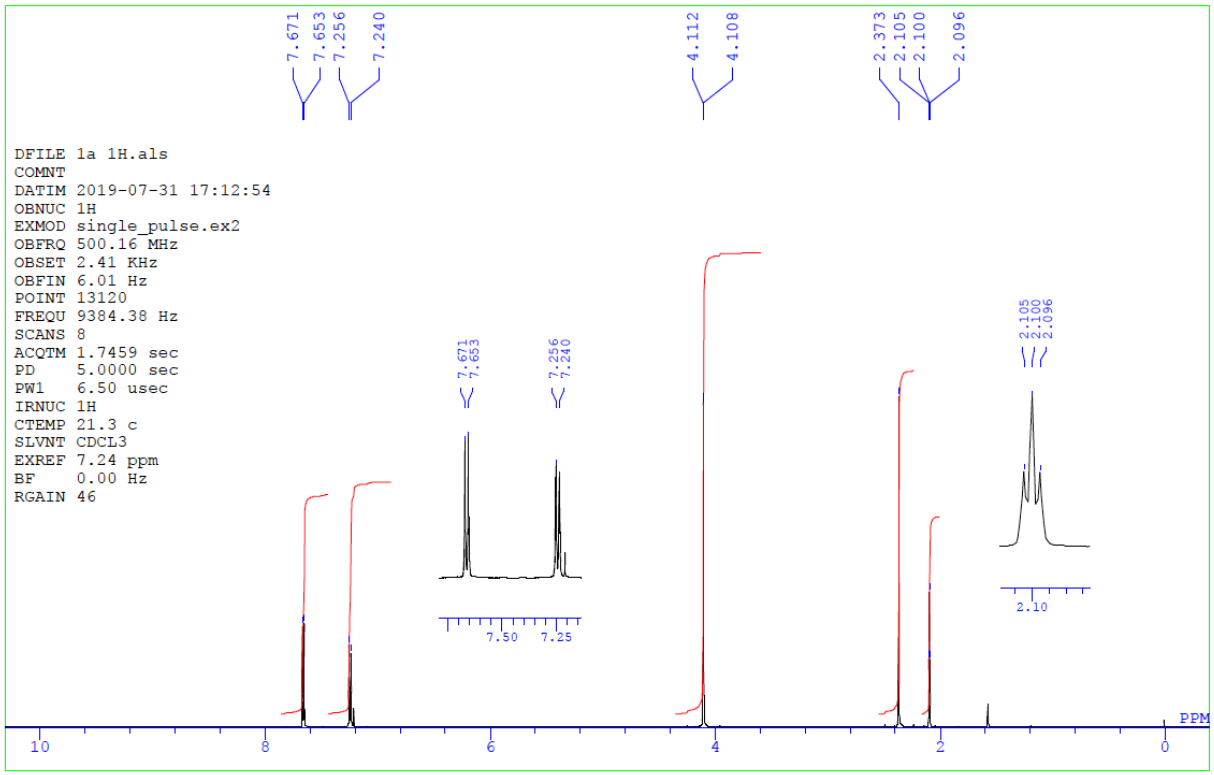
Spectroscopic Data

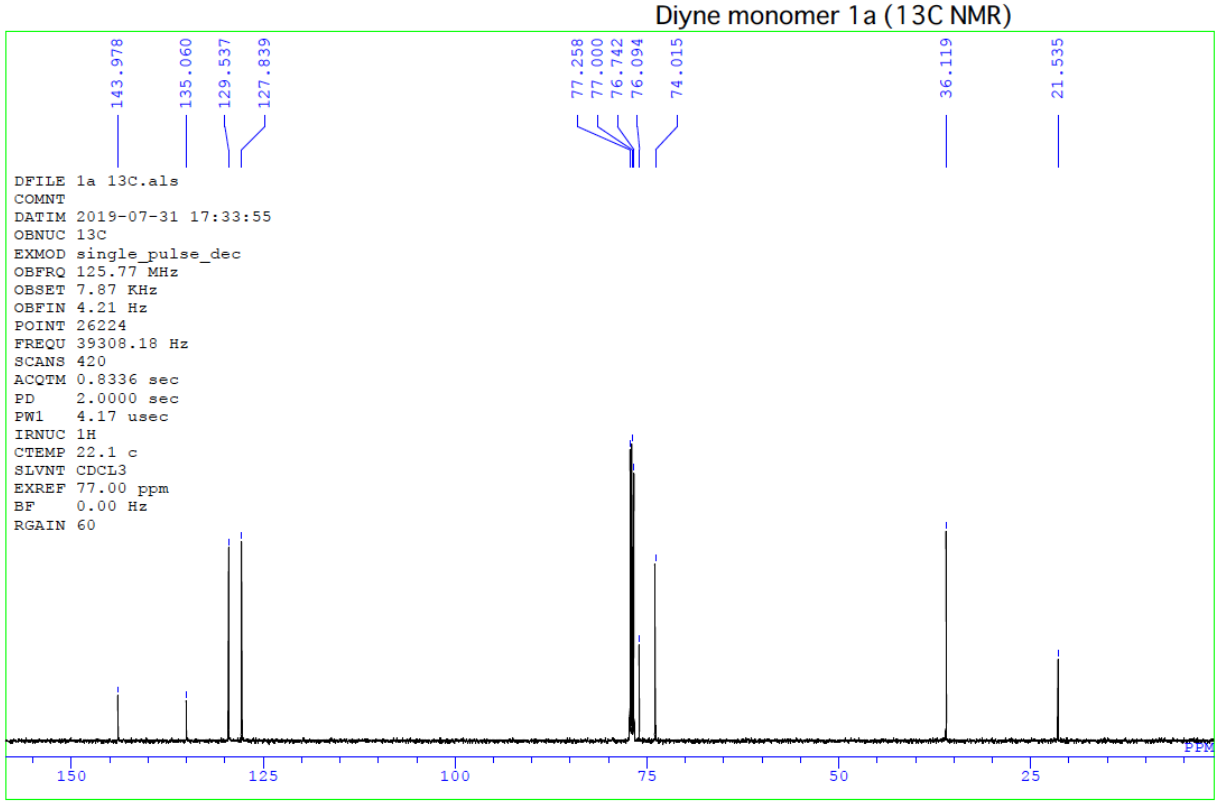
NMR Spectra

Diyne monomer 1a

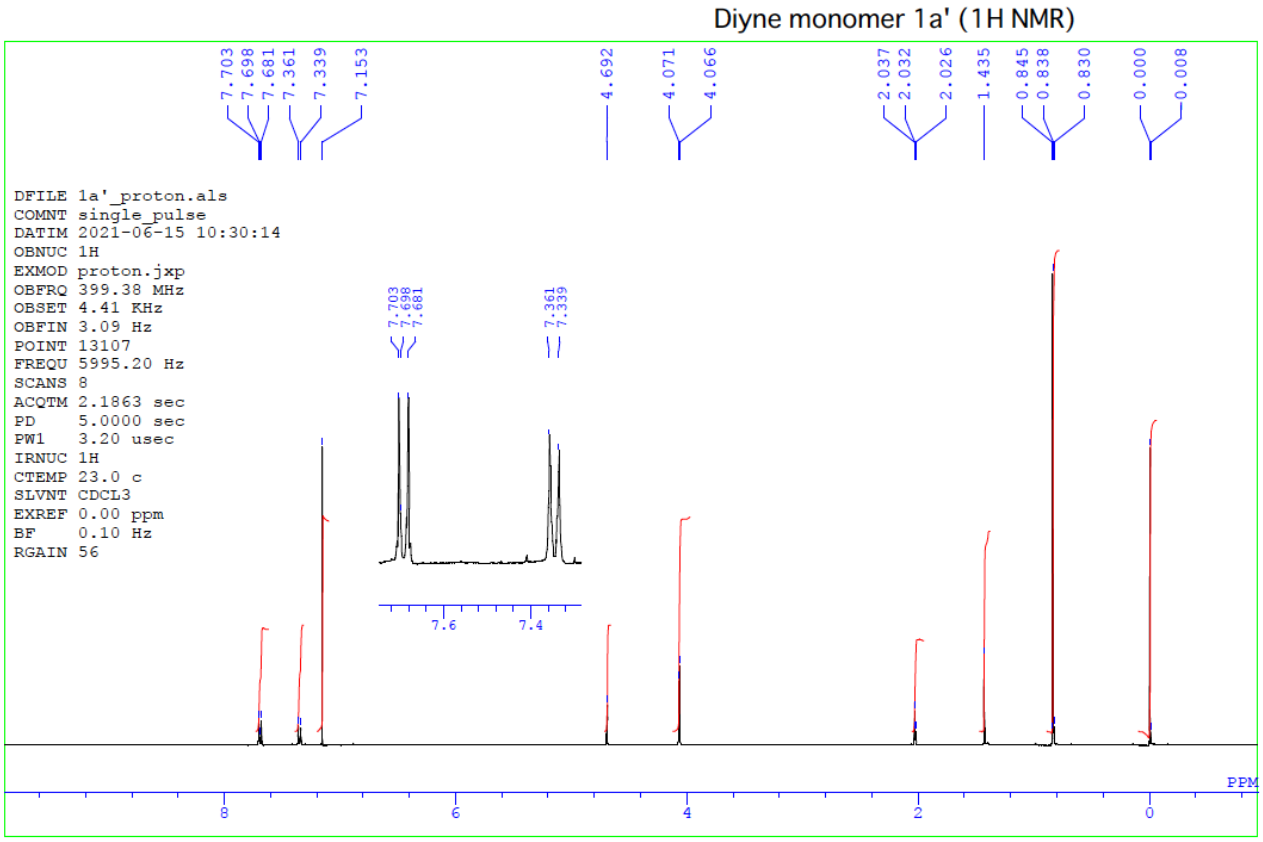
Diyne monomer 1a (1H NMR)

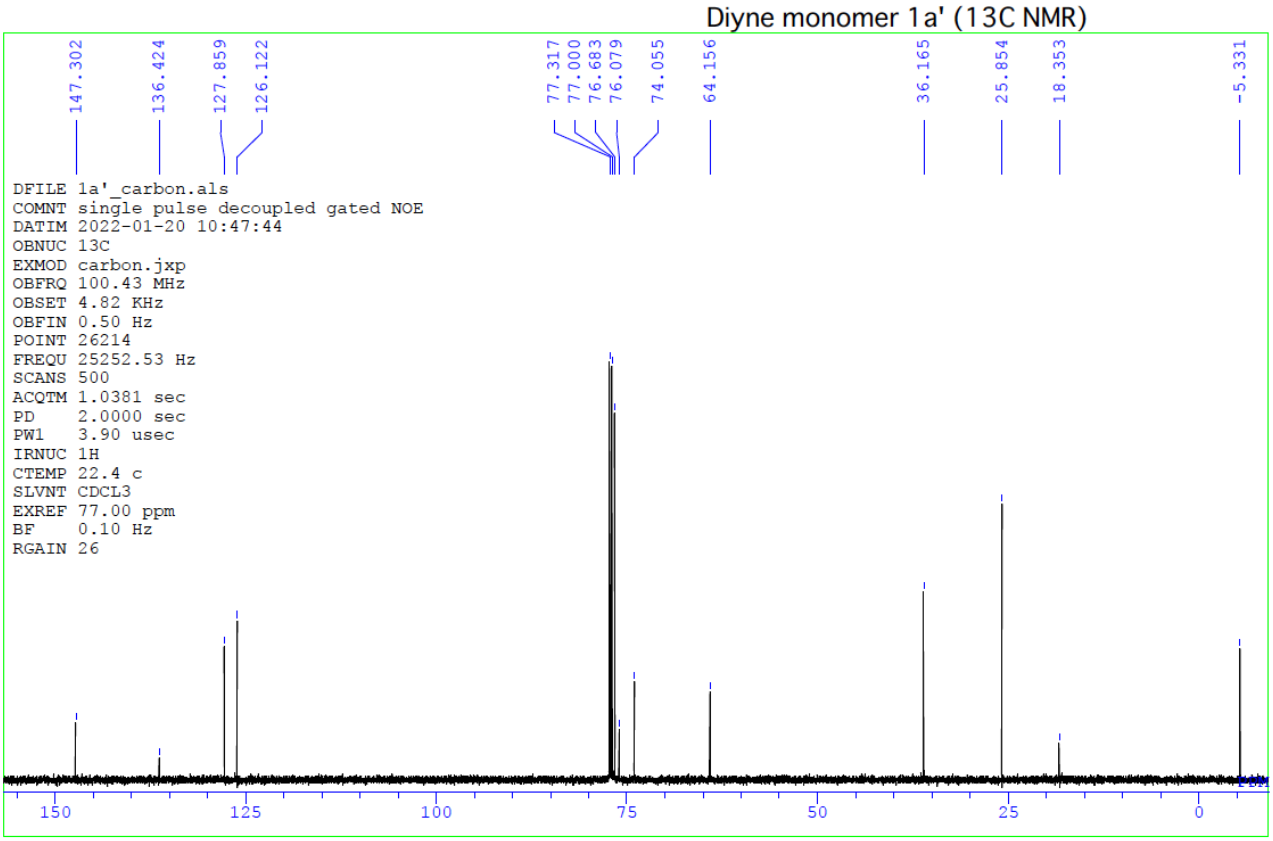


Diyne monomer 1a (13C NMR)

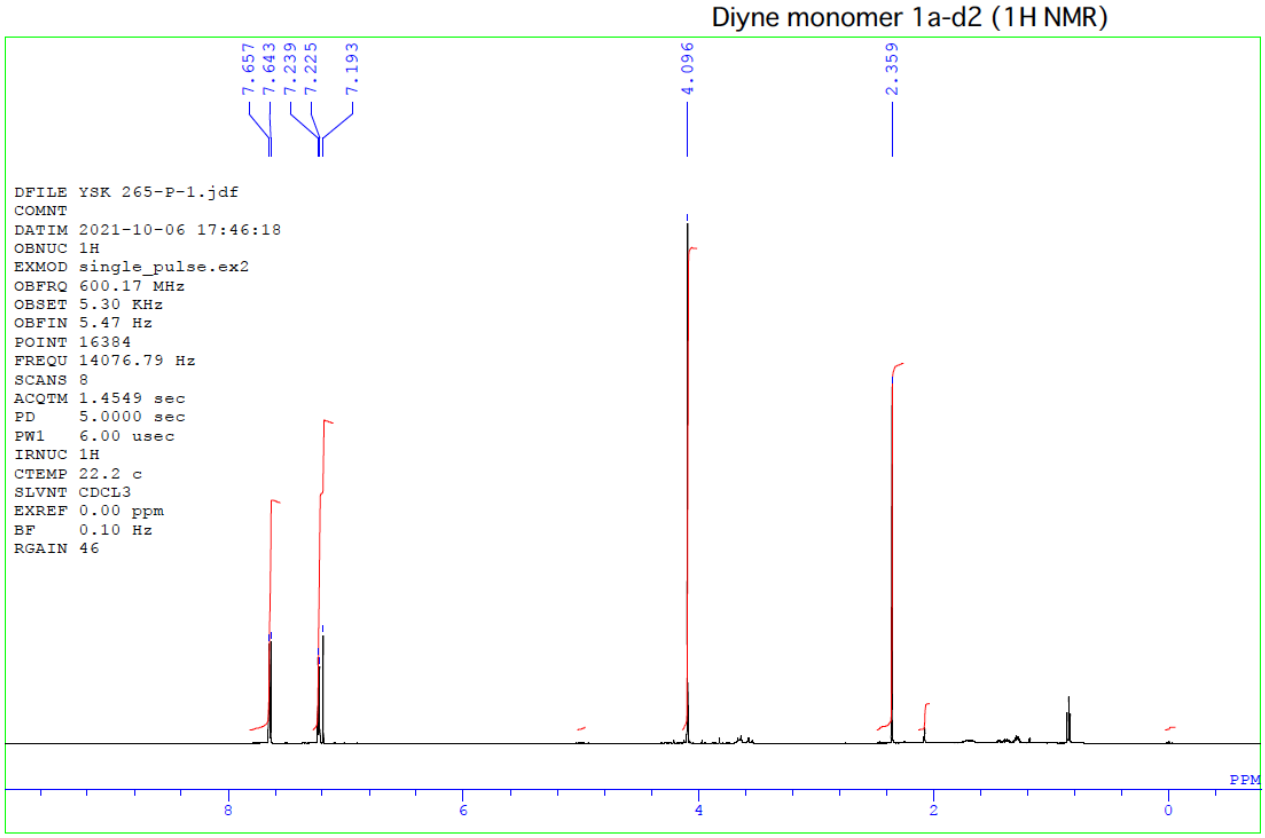


Diyne monomer 1a’

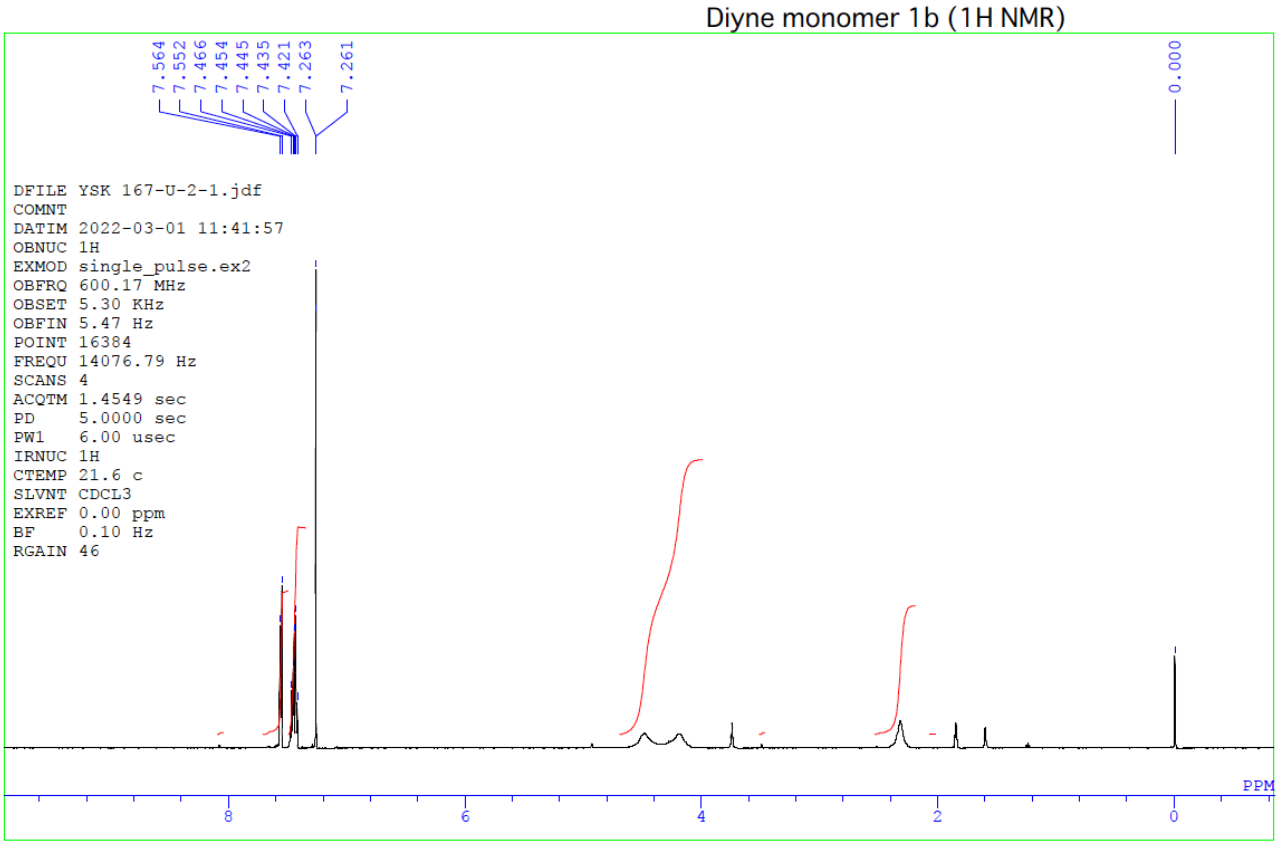




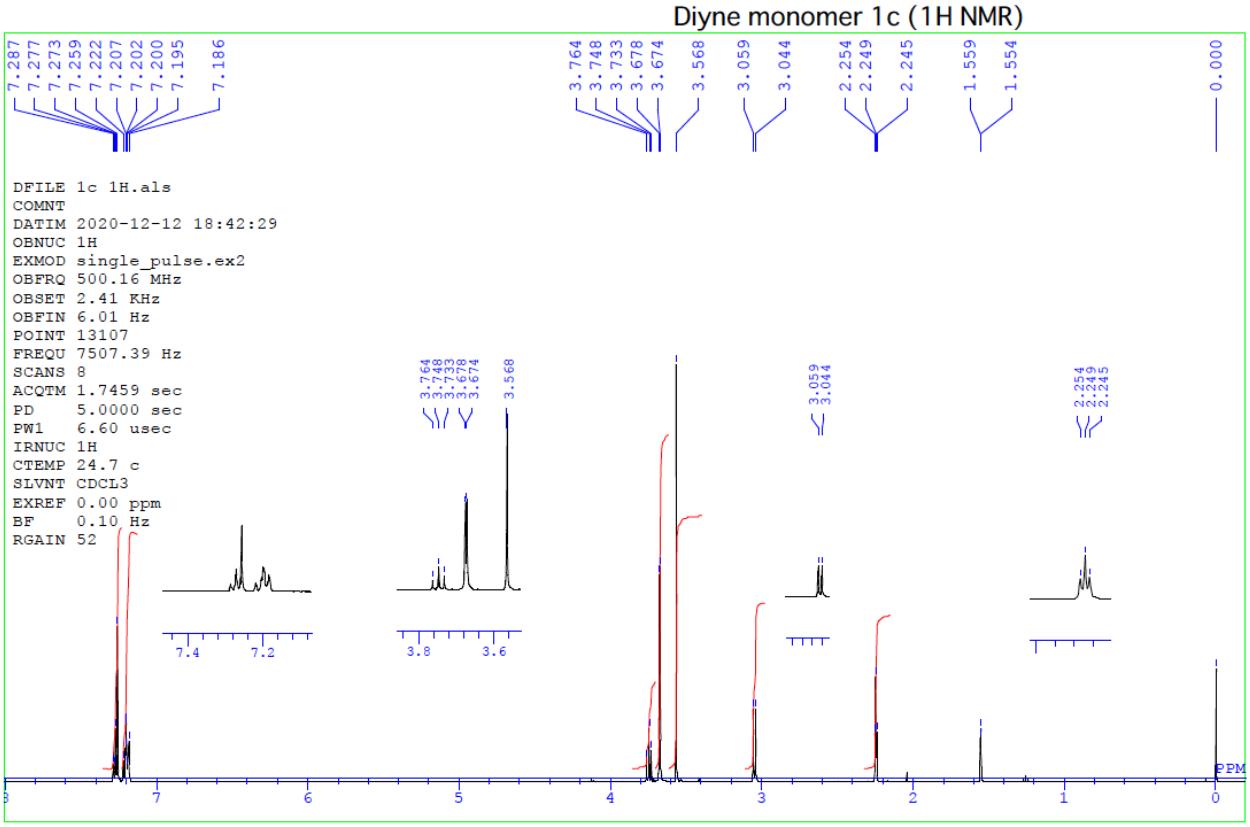
Diyne monomer 1a-d_2



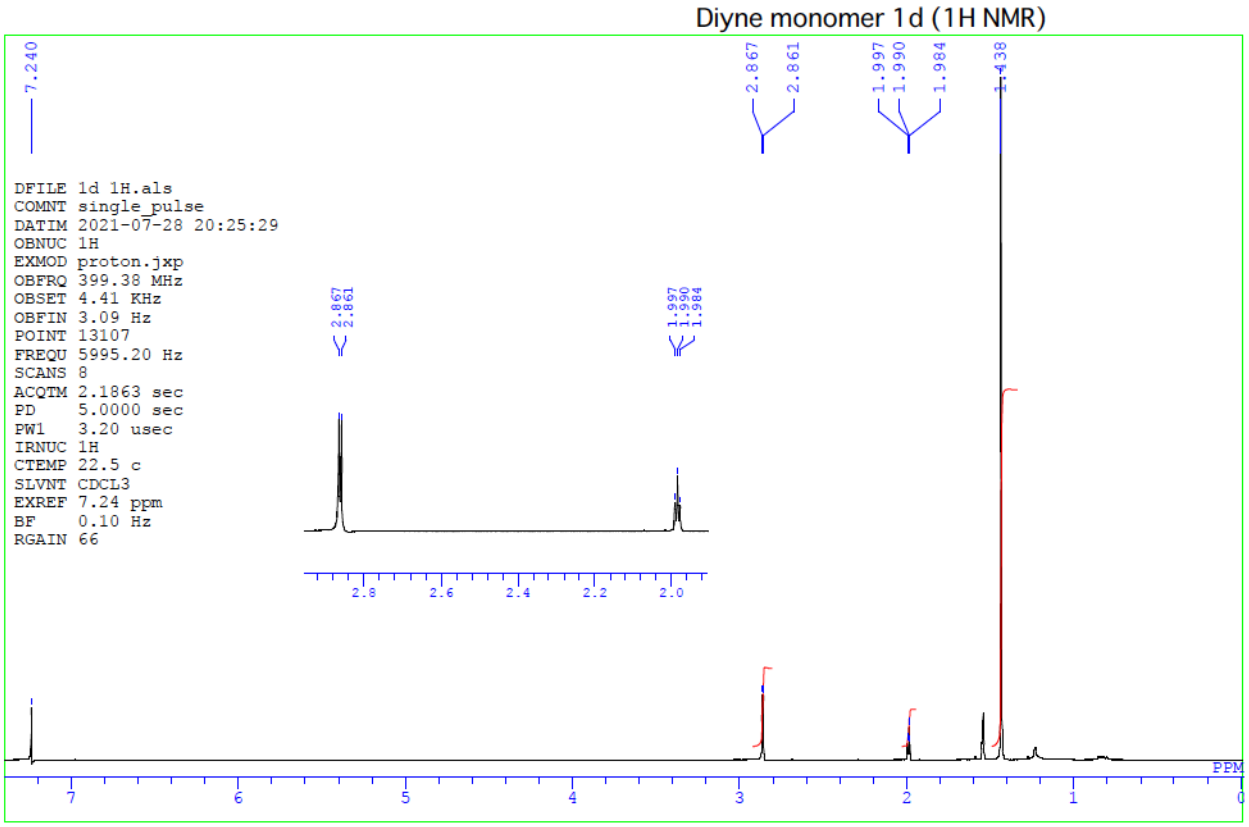
Diyne monomer 1b



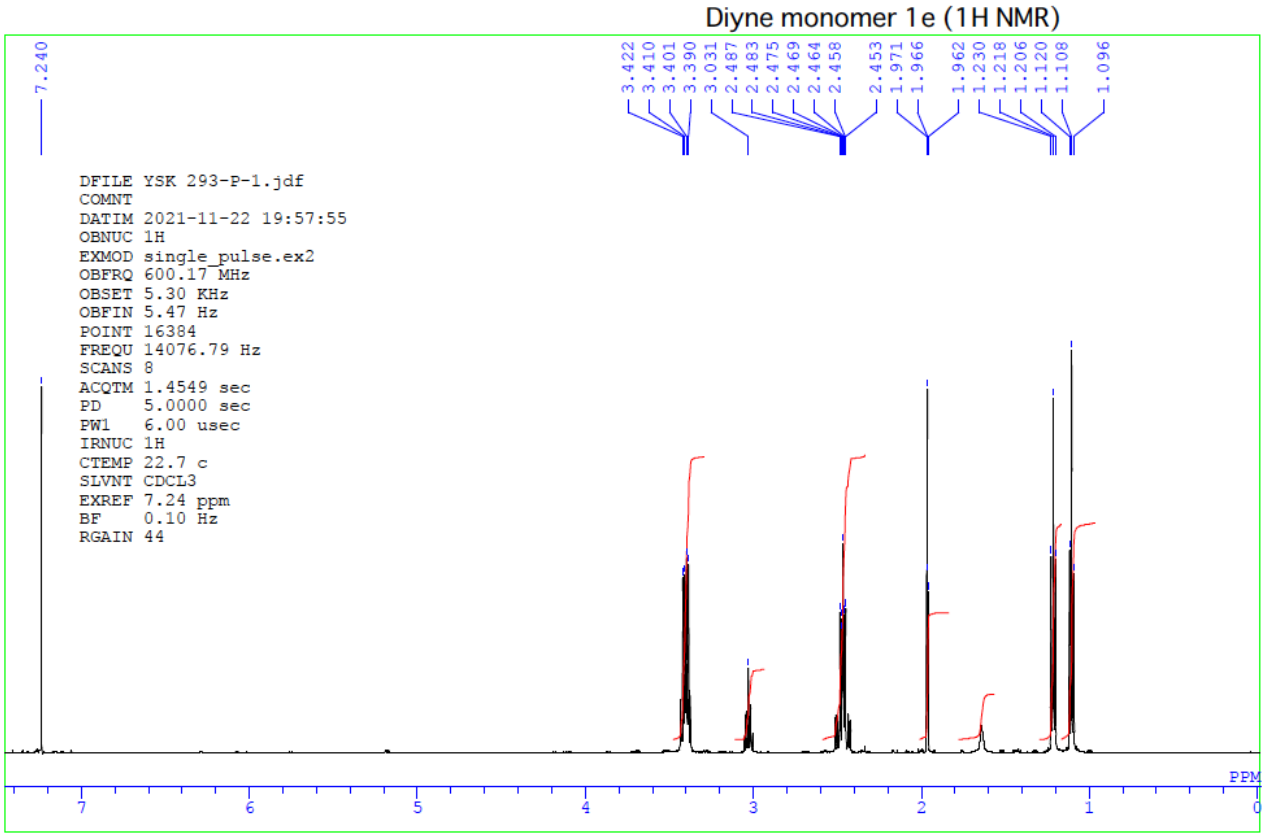
Diyne monomer 1c



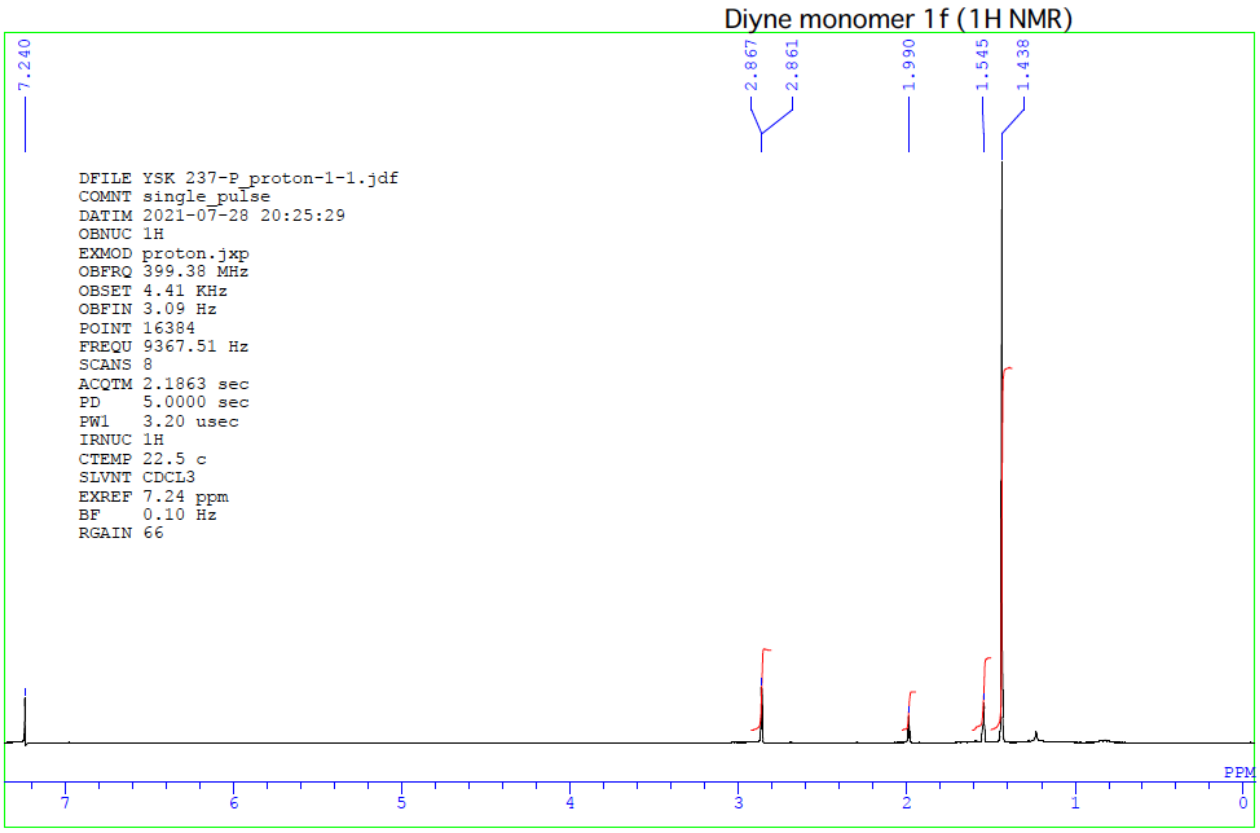
Diyne monomer 1d



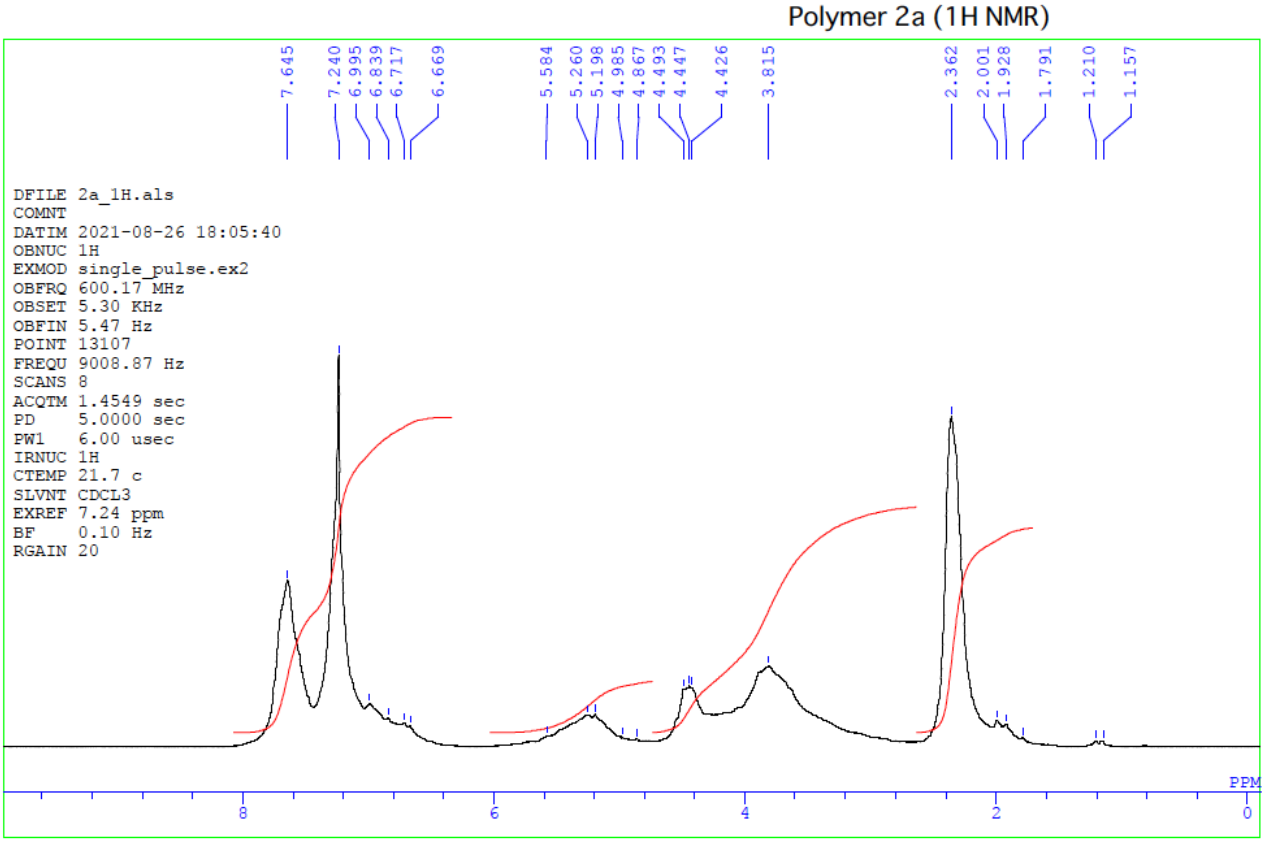
Diyne monomer 1e



Diyne monomer 1f



Polymer 2a


Polymer 2a (1H NMR)
7.645
7.240
6.995
6.839
6.717
6.669
5.584
5.260
5.198
4.985
4.867
4.493
4.447
4.426
3.815
2.362
2.001
1.928
1.791
1.210
1.157
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OBSET 5.30 KHz
OBFIN 5.47 Hz
POINT 13107
FREQU 9008.87 Hz
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PD 5.0000 sec
PW1 6.00 usec
IRNUC 1H
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SLVNT CDCL3
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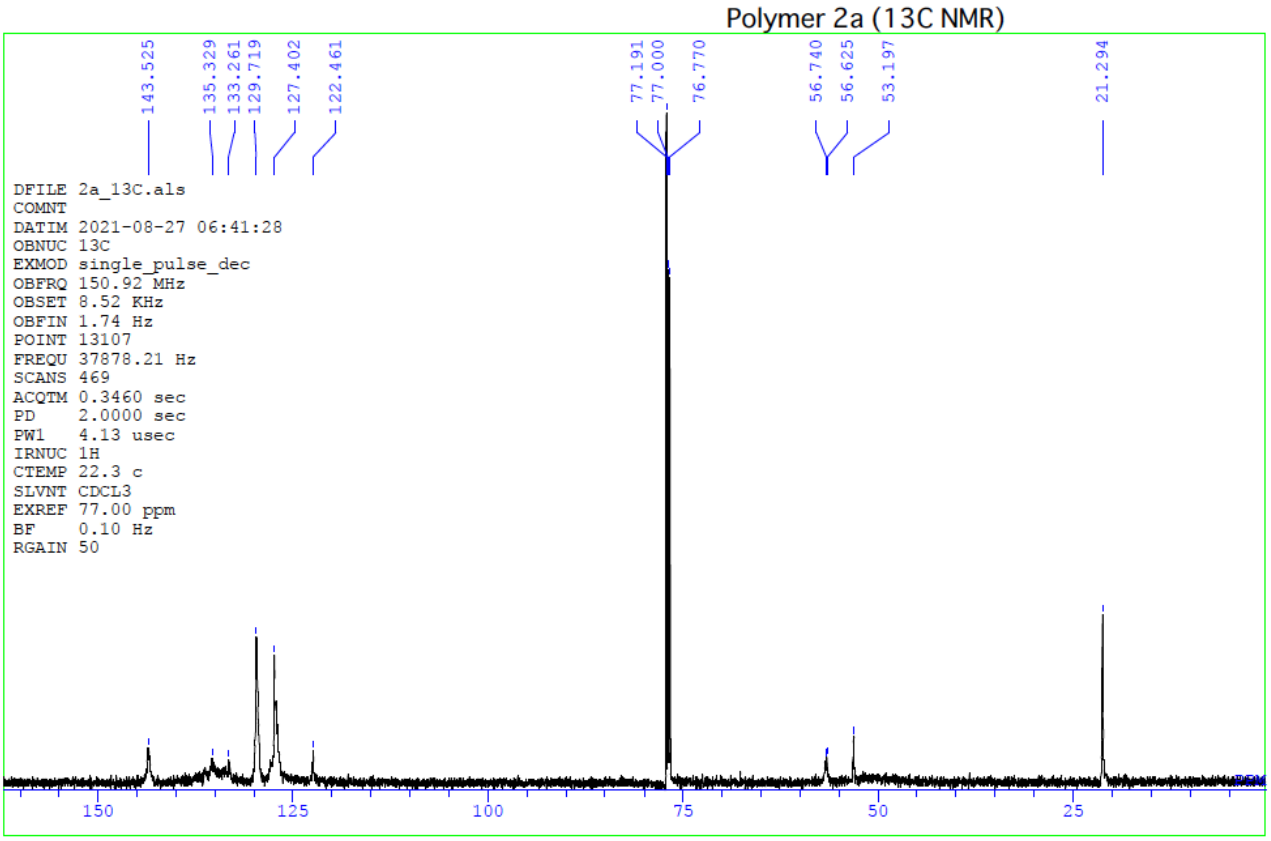
PPM
8
6
4
2
0


Polymer 2a (13C NMR)
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135.329
133.261
129.719
127.402
122.461
77.191
77.000
76.770
56.740
56.625
53.197
21.294
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OBSET 8.52 KHz
OBFIN 1.74 Hz
POINT 13107
FREQU 37878.21 Hz
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PD 2.0000 sec
PW1 4.13 usec
IRNUC 1H
CTEMP 22.3 c
SLVNT CDCL3
EXREF 77.00 ppm
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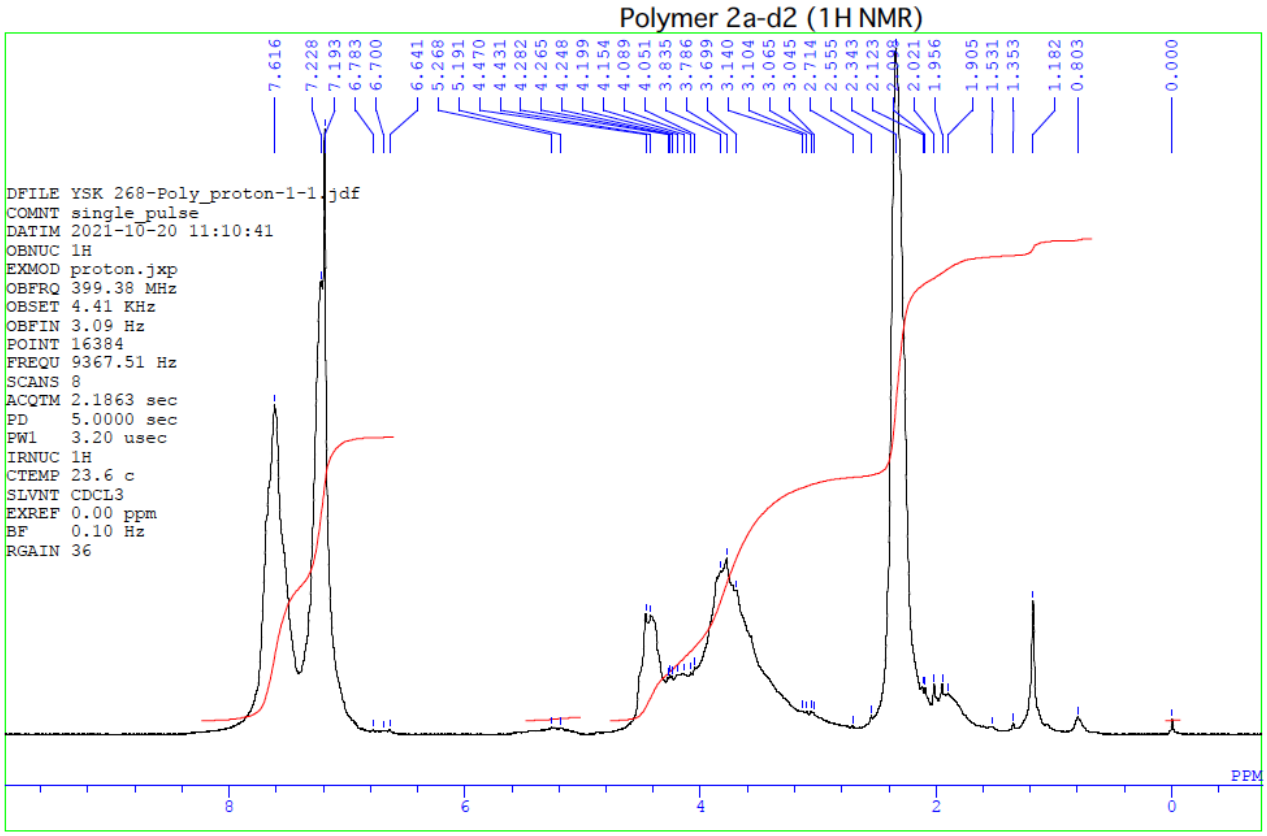
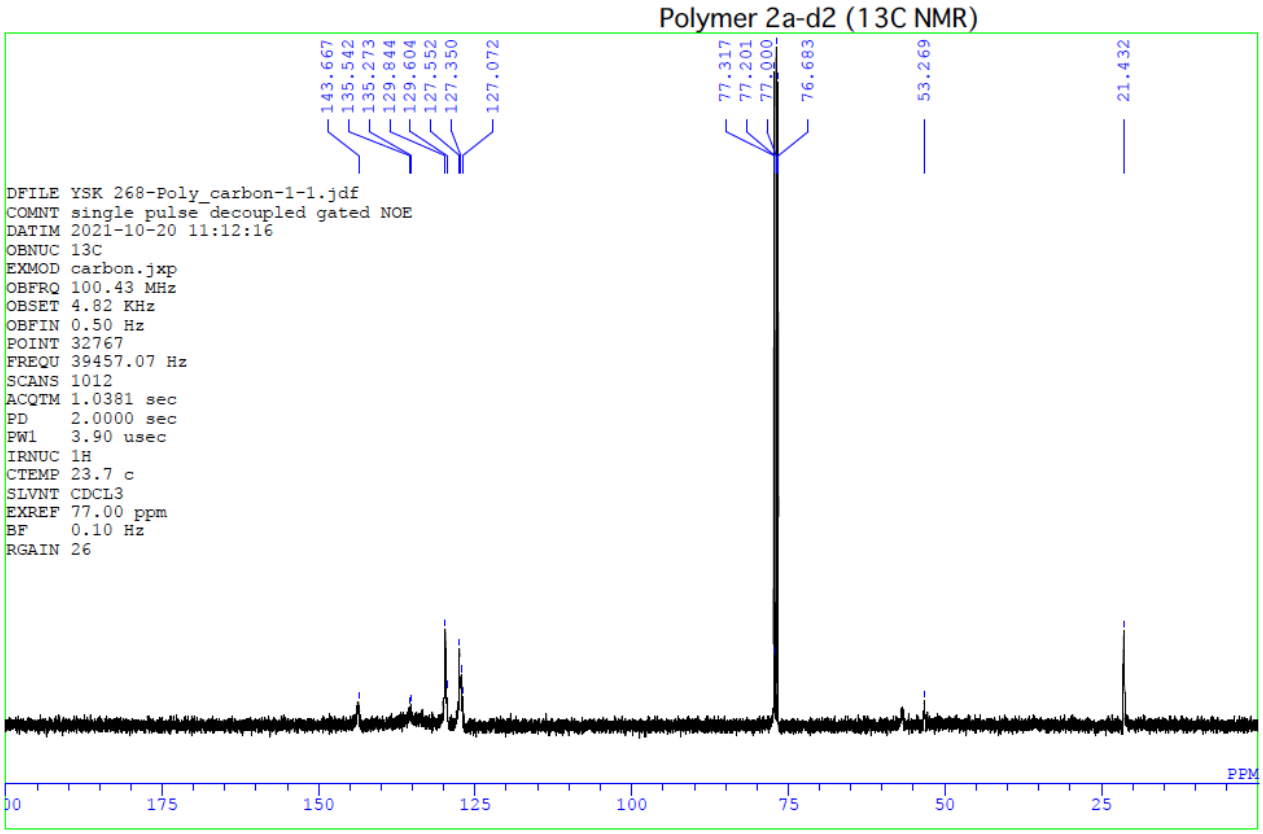
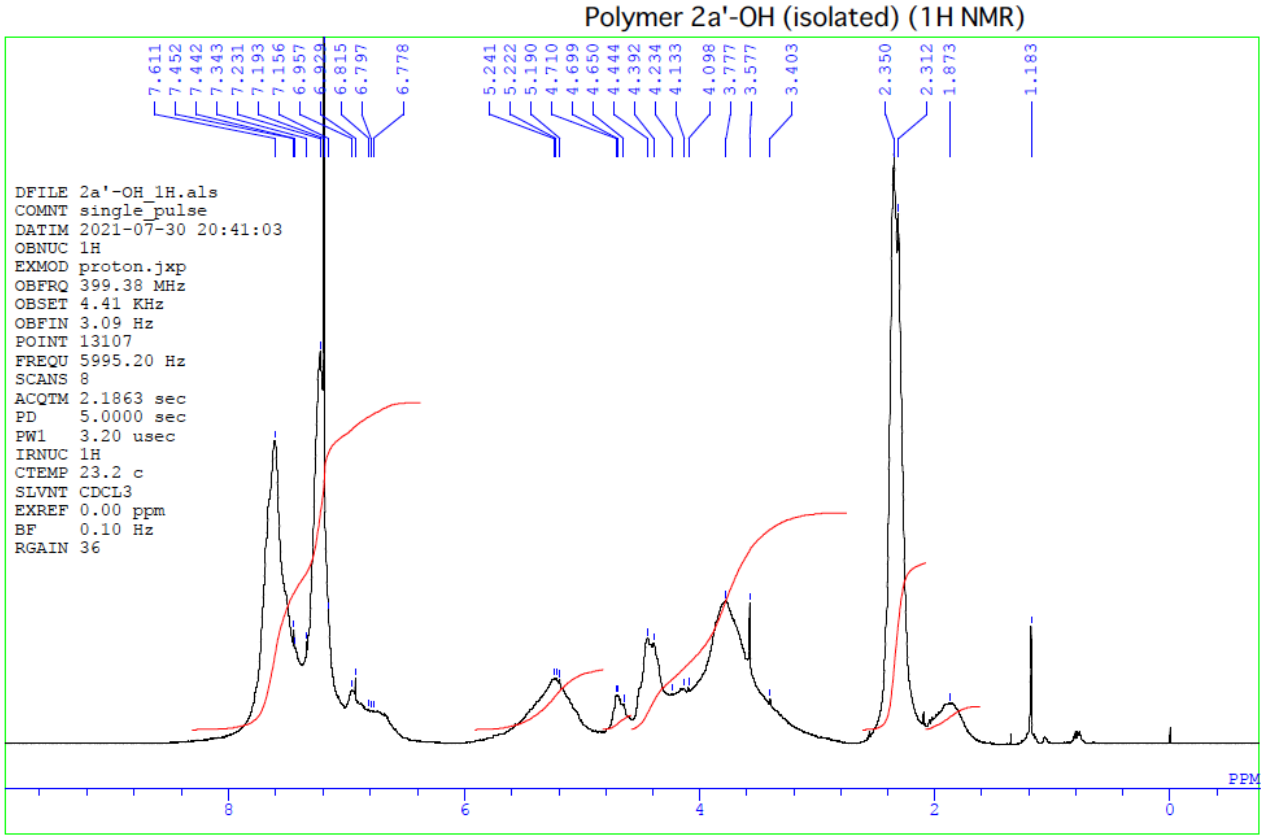
RGAIN 50
150
125
100
75
50
25

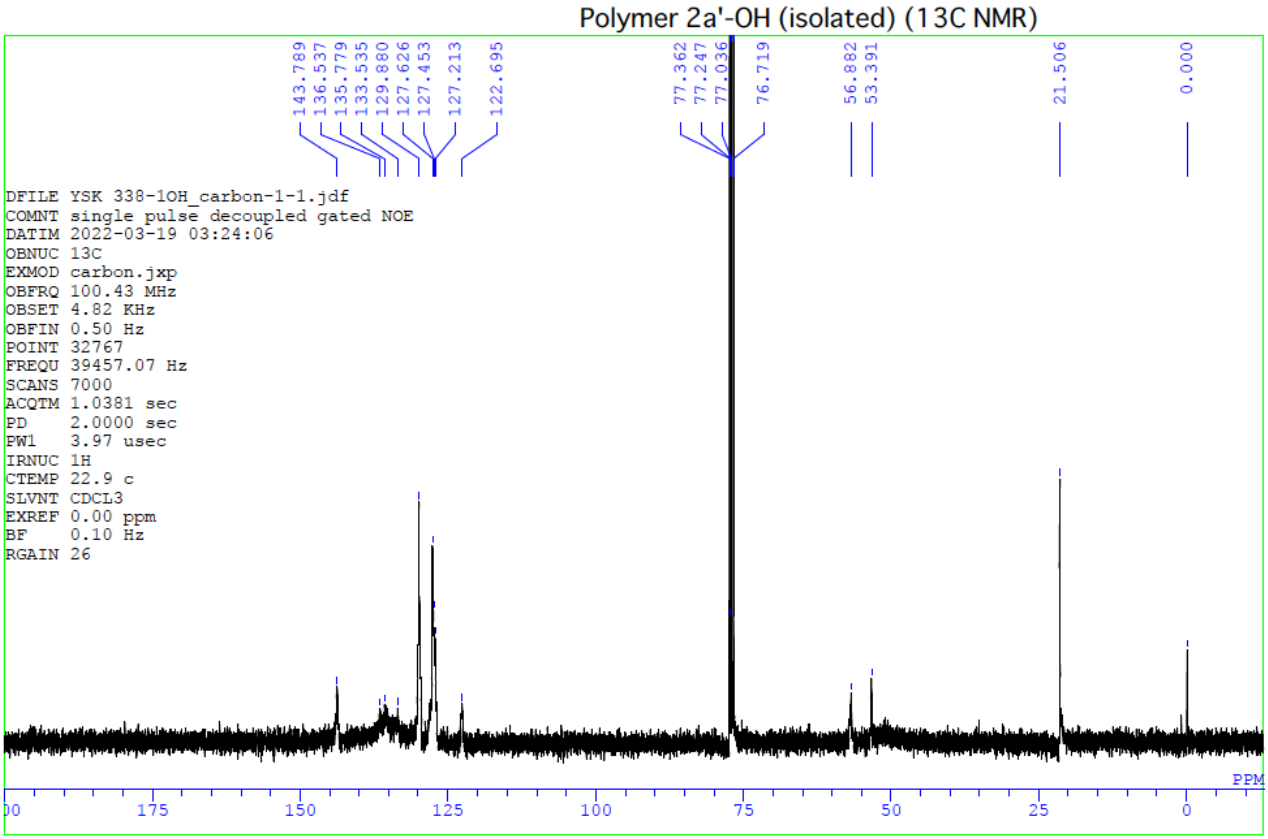
Polymer 2a-d_2



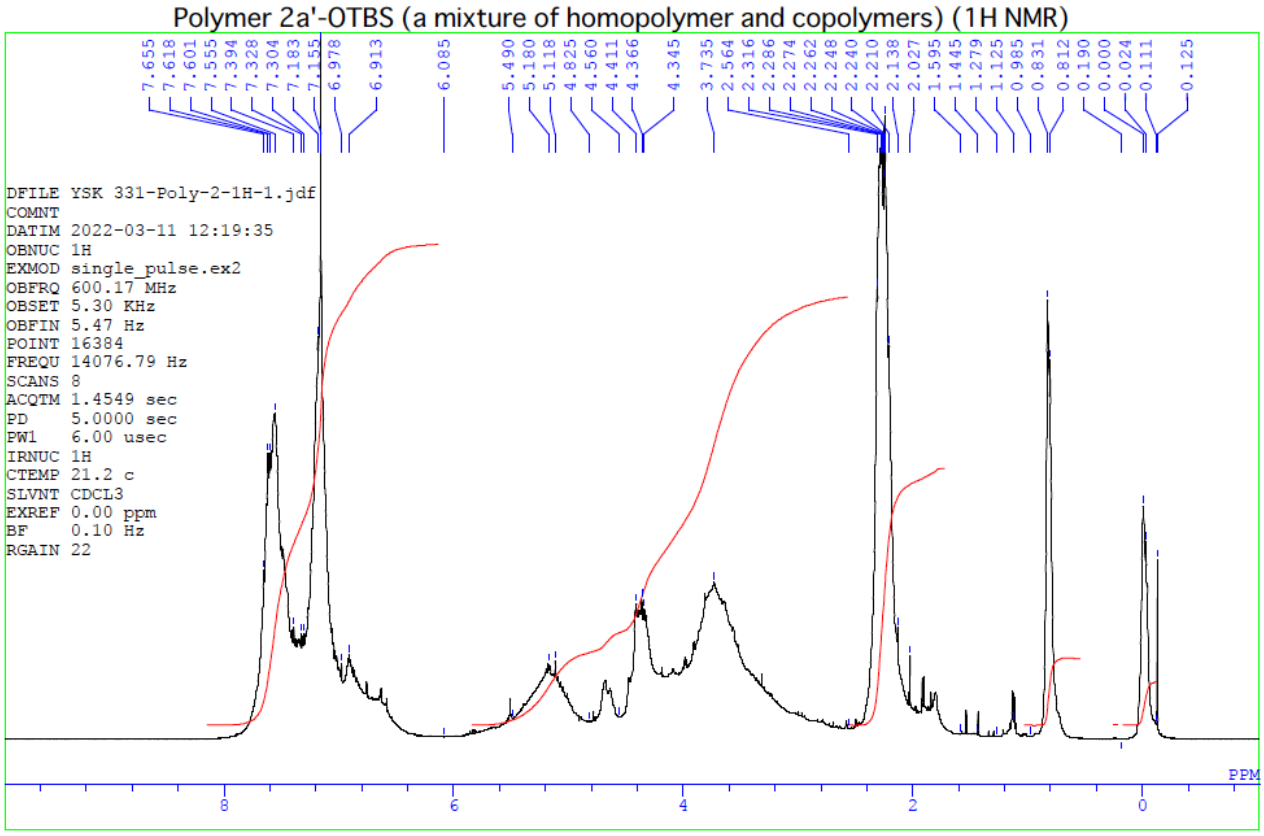


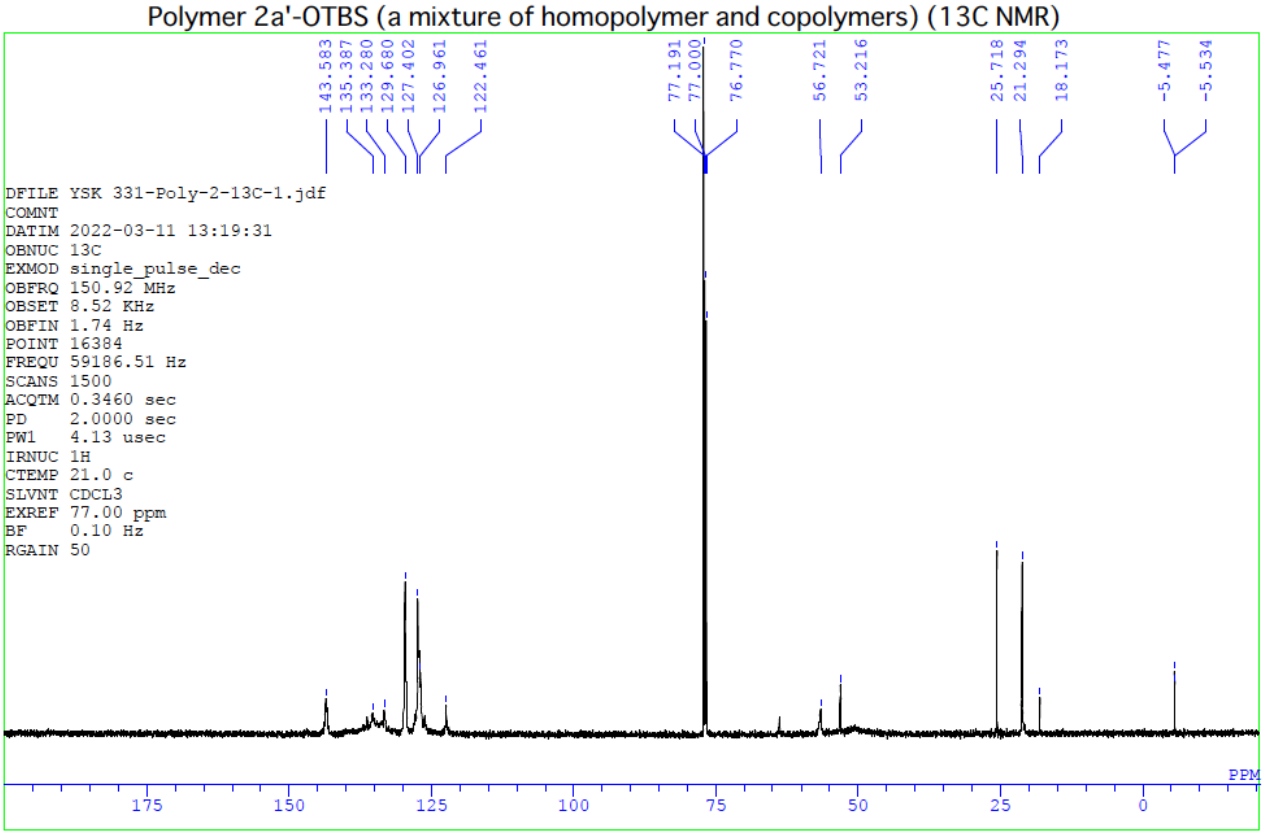
Polymer 2a'-OH



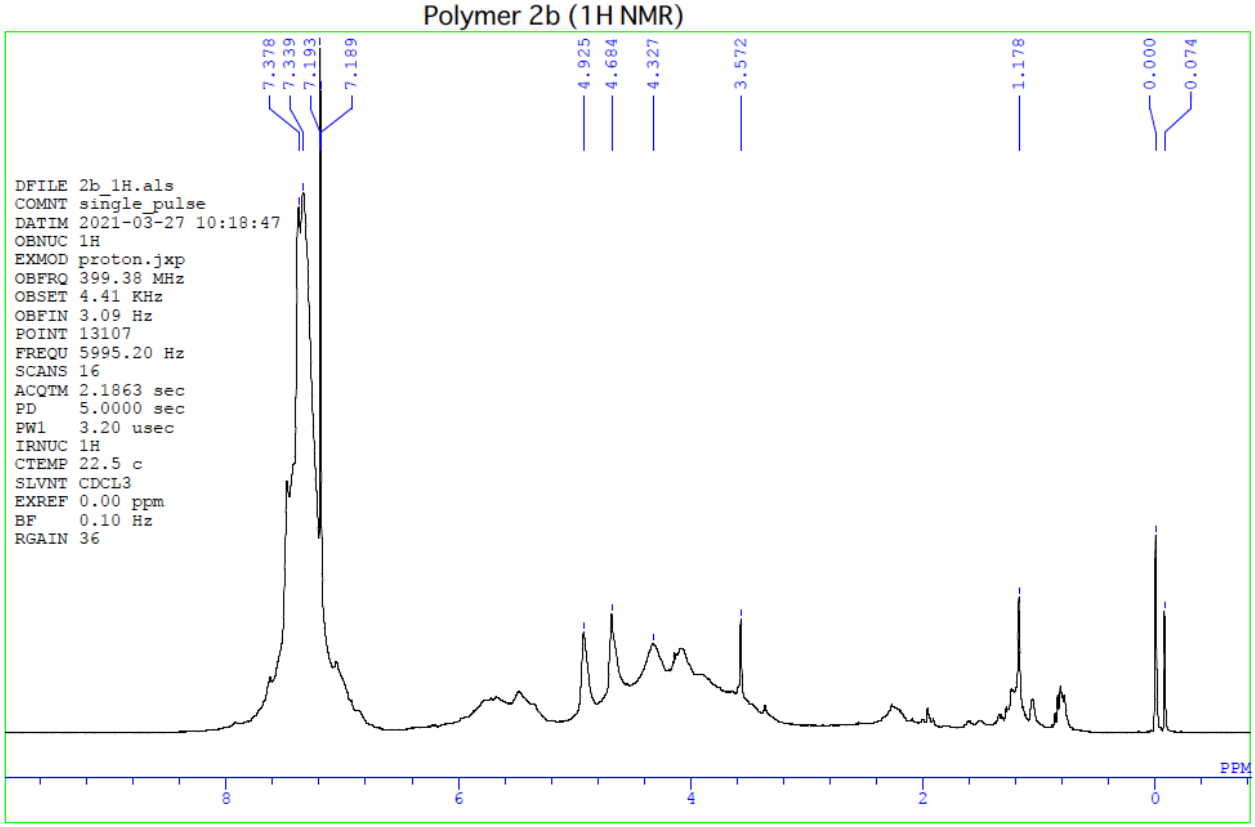


Polymer 2a'-OTBS





Polymer 2b


Polymer 2b (1H NMR)
7.378
7.339
7.193
7.189
4.925
4.684
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3.572
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OBSET 4.41 KHz
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POINT 13107
FREQU 5995.20 Hz
SCANS 16
ACQTM 2.1863 sec
PD 5.0000 sec
PW1 3.20 usec
IRNUC 1H
CTEMP 22.5 c
SLVNT CDCL3
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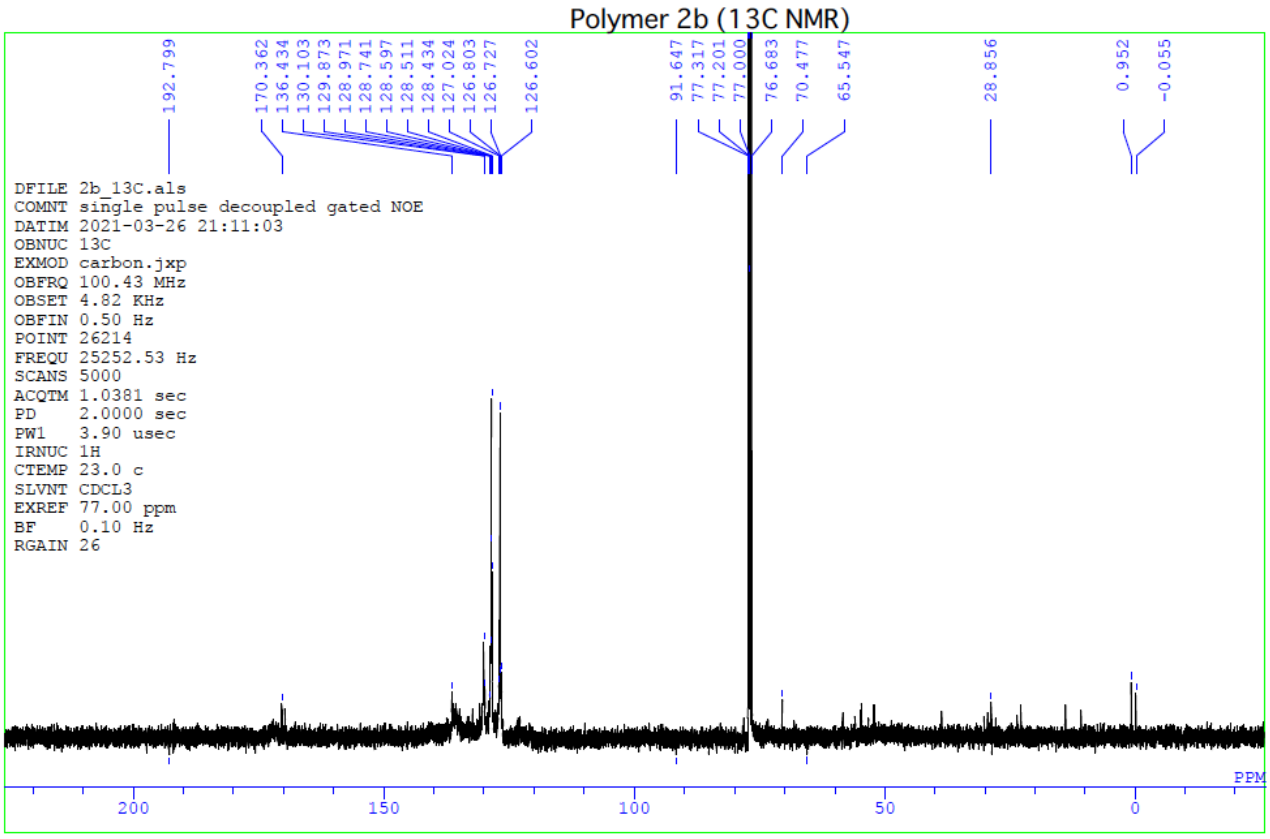
RGAIN 36
PPM
8
6
4
2
0


Polymer 2b (13C NMR)
192.799
170.362
136.434
130.103
129.873
128.971
128.741
128.597
128.511
128.434
127.024
126.803
126.727
126.602
91.647
77.317
77.201
77.000
76.683
70.477
65.547
28.856
0.952
-0.055
DFILE 2b_13C.als
COMNT single pulse decoupled gated NOE
DATIM 2021-03-26 21:11:03
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EXMOD carbon.jxp
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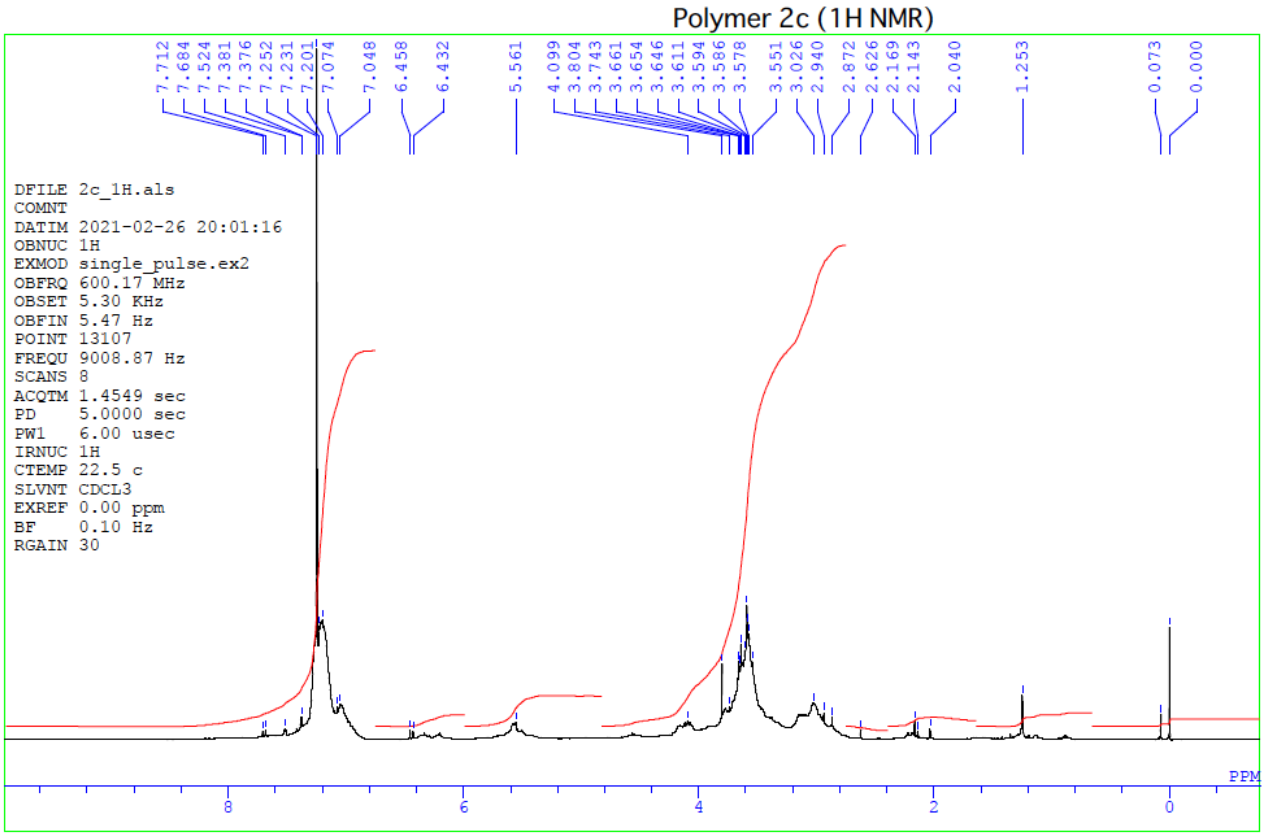
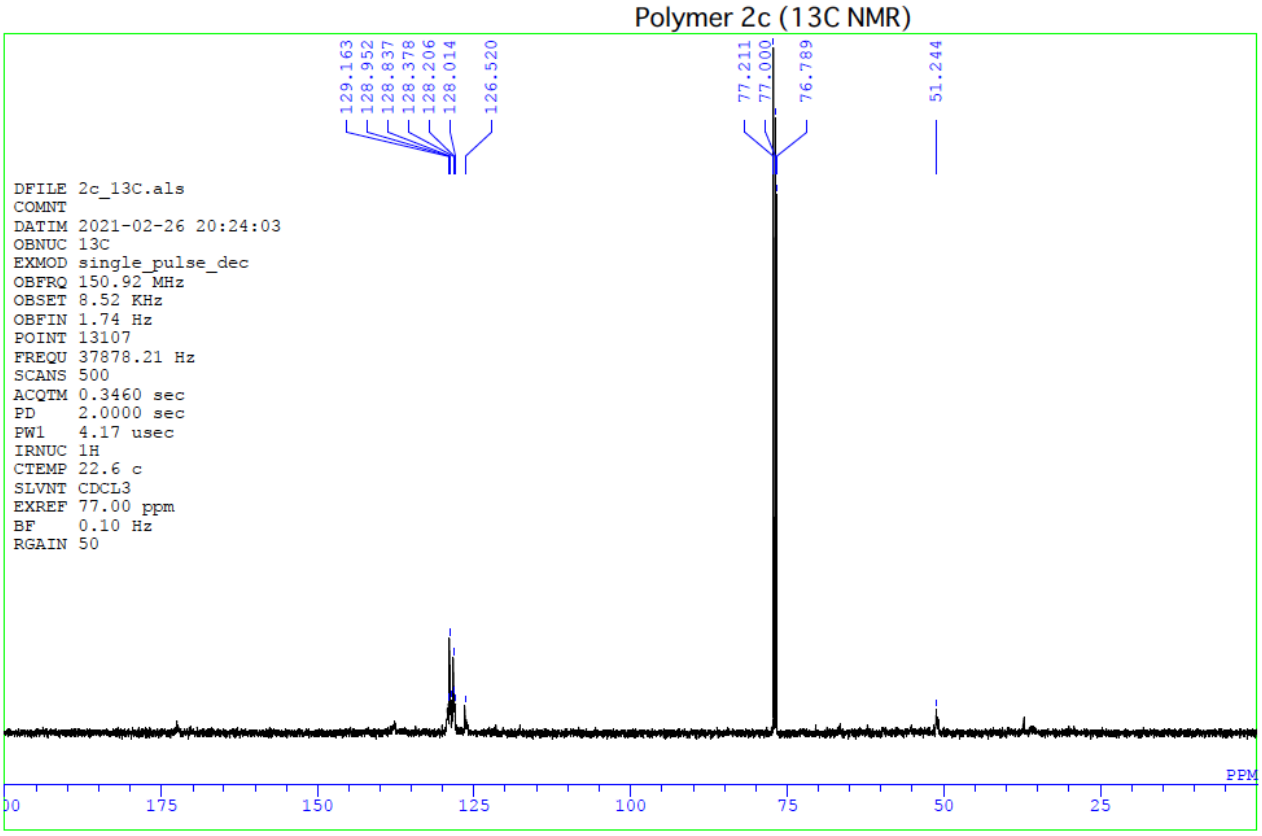
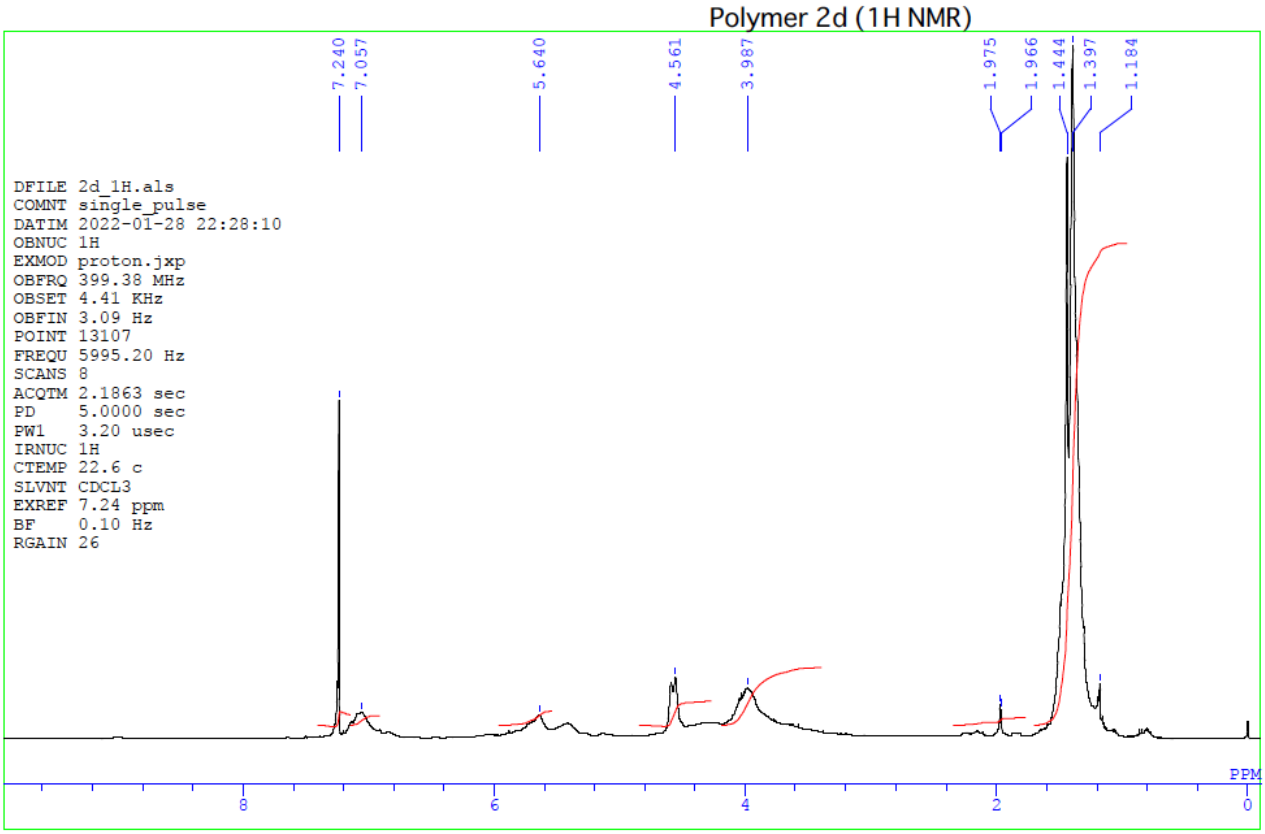
OBSET 4.82 KHz
OBFIN 0.50 Hz
POINT 26214
FREQU 25252.53 Hz
SCANS 5000
ACQTM 1.0381 sec
PD 2.0000 sec
PW1 3.90 usec
IRNUC 1H
CTEMP 23.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.10 Hz
RGAIN 26
PPM
200
150
100
50
0

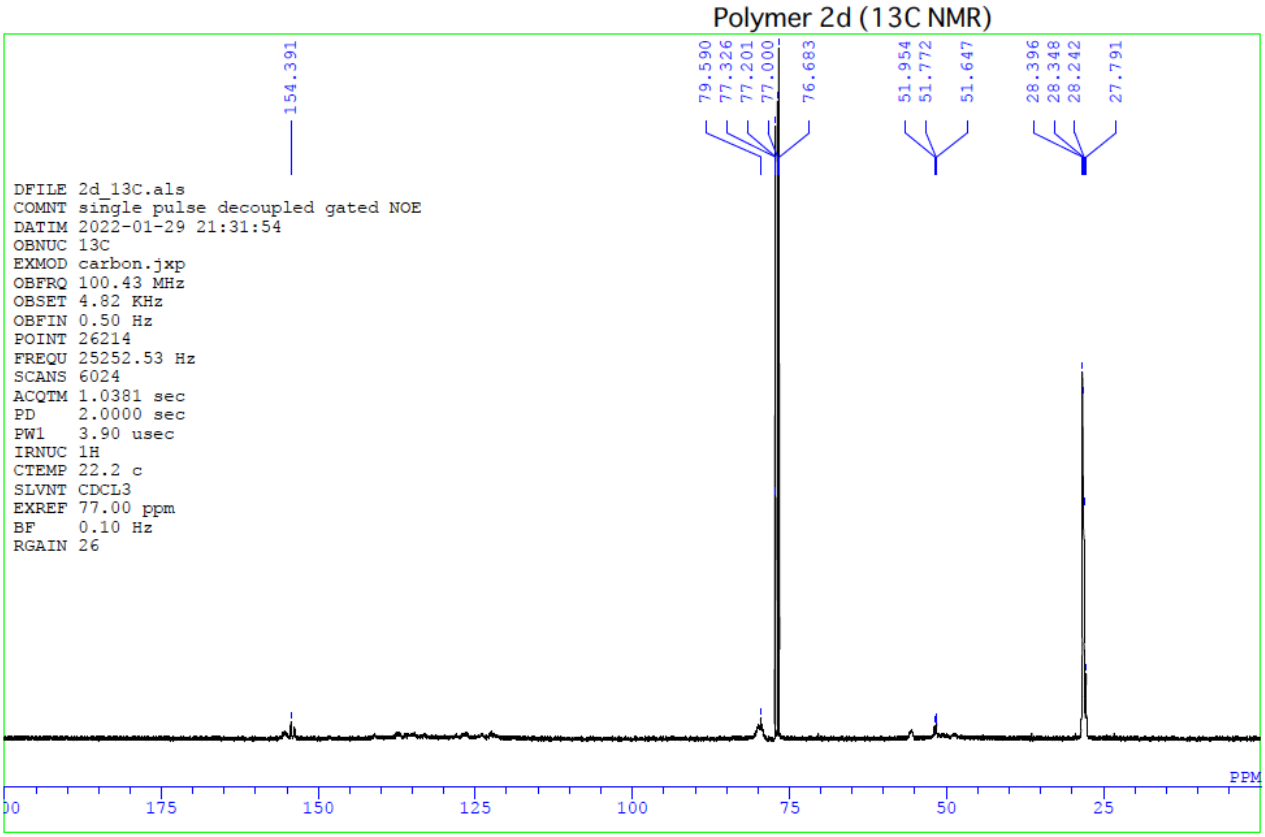
Polymer 2c


Polymer 2c (1H NMR)
7.712
7.684
7.524
7.381
7.376
7.252
7.231
7.201
7.074
7.048
6.458
6.432
5.561
4.099
3.804
3.743
3.661
3.654
3.646
3.611
3.594
3.586
3.578
3.551
3.026
2.940
2.872
2.626
2.169
2.143
2.040
1.253
0.073
0.000
DFILE 2c_1H.als
COMNT
DATIM 2021-02-26 20:01:16
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 600.17 MHz
OBSET 5.30 KHz
OBFIN 5.47 Hz
POINT 13107
FREQU 9008.87 Hz
SCANS 8
ACQTM 1.4549 sec
PD 5.0000 sec
PW1 6.00 usec
IRNUC 1H
CTEMP 22.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.10 Hz
RGAIN 30
PPM
8
6
4
2
0

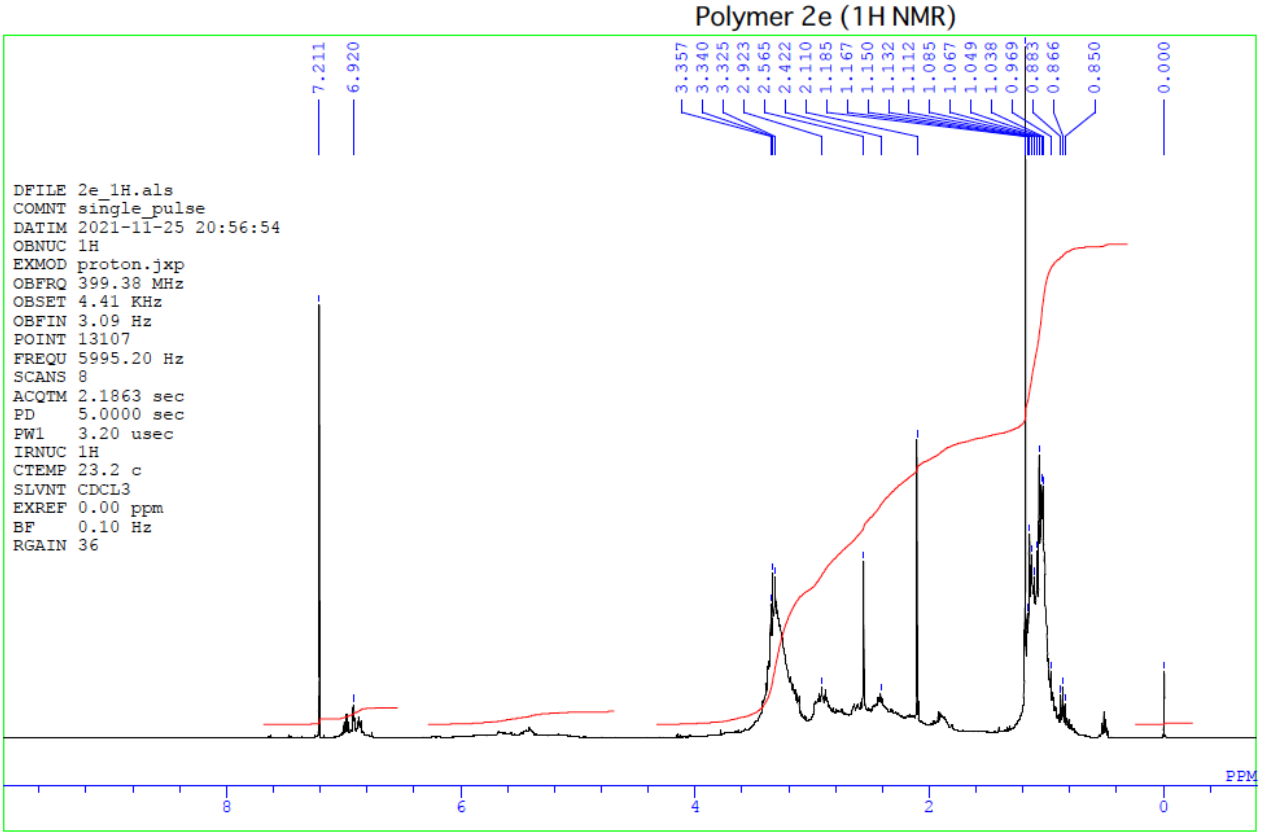

Polymer 2c (13C NMR)
129.163
128.952
128.837
128.378
128.206
128.014
126.520
77.211
77.000
76.789
51.244
DFILE 2c_13C.als
COMNT
DATIM 2021-02-26 20:24:03
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 150.92 MHz
OBSET 8.52 KHz
OBFIN 1.74 Hz
POINT 13107
FREQU 37878.21 Hz
SCANS 500
ACQTM 0.3460 sec
PD 2.0000 sec
PW1 4.17 usec
IRNUC 1H
CTEMP 22.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.10 Hz
RGAIN 50
PPM
00
175
150
125
100
75
50
25

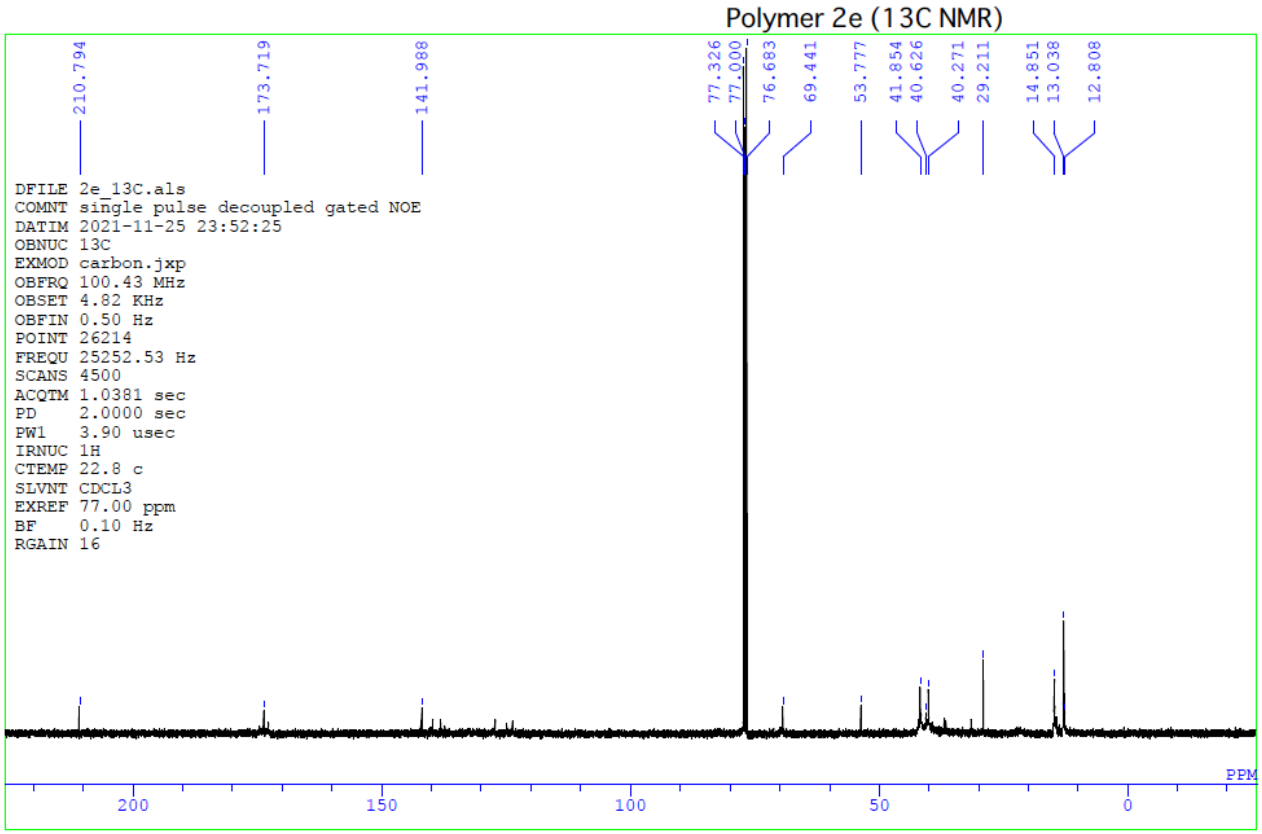
Polymer 2d


Polymer 2d (1H NMR)
7.240
7.057
5.640
4.561
3.987
1.975
1.966
1.444
1.397
1.184
DFILE 2d_1H.als
COMNT single_pulse
DATIM 2022-01-28 22:28:10
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.38 MHz
OBSET 4.41 KHz
OBFIN 3.09 Hz
POINT 13107
FREQU 5995.20 Hz
SCANS 8
ACQTM 2.1863 sec
PD 5.0000 sec
PW1 3.20 usec
IRNUC 1H
CTEMP 22.6 c
SLVNT CDCL3
EXREF 7.24 ppm
BF 0.10 Hz
RGAIN 26
PPM
8
6
4
2
0

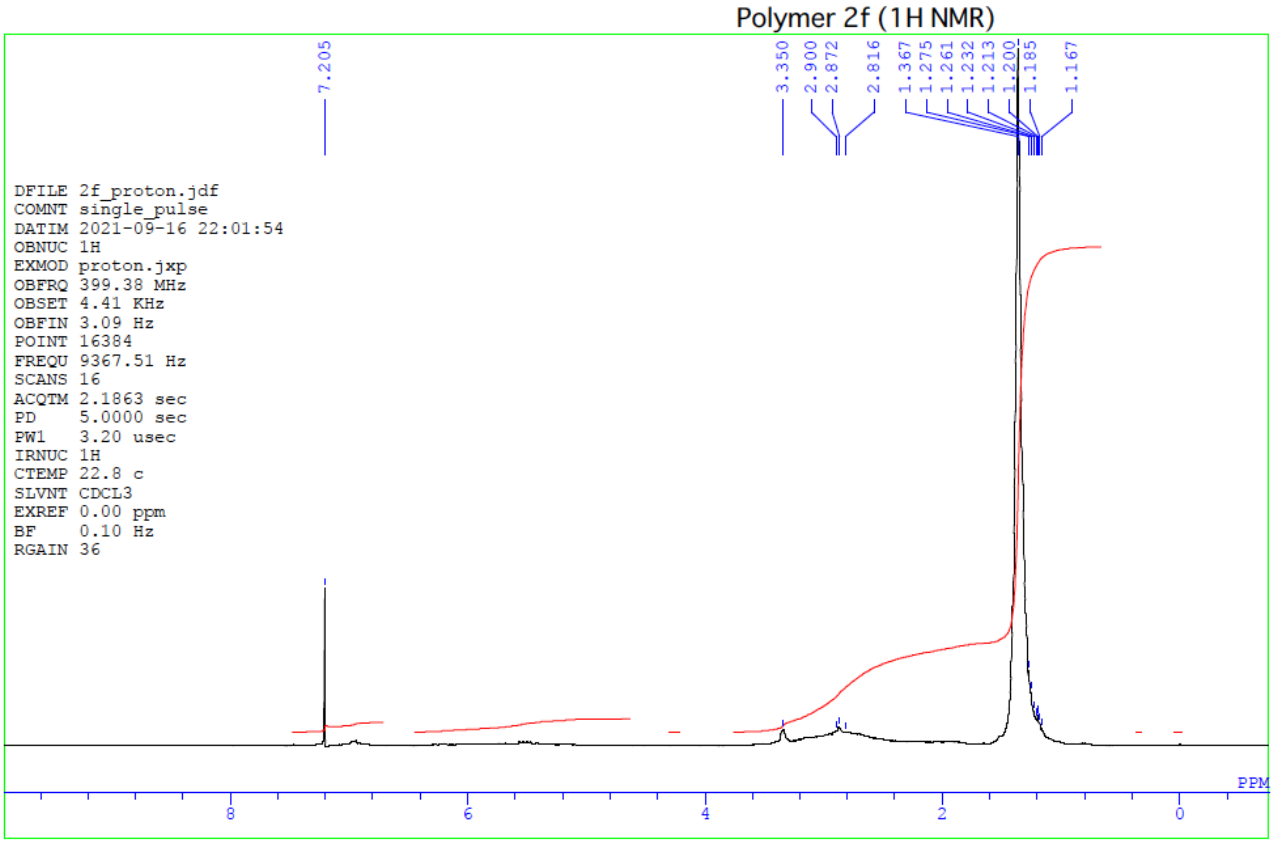

Polymer 2d (13C NMR)
154.391
79.590
77.326
77.201
77.000
76.683
51.954
51.772
51.647
28.396
28.348
28.242
27.791
DFILE 2d_13C.als
COMNT single pulse decoupled gated NOE
DATIM 2022-01-29 21:31:54
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.43 MHz
OBSET 4.82 KHz
OBFIN 0.50 Hz
POINT 26214
FREQU 25252.53 Hz
SCANS 6024
ACQTM 1.0381 sec
PD 2.0000 sec
PW1 3.90 usec
IRNUC 1H
CTEMP 22.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.10 Hz
RGAIN 26
PPM
175
150
125
100
75
50
25

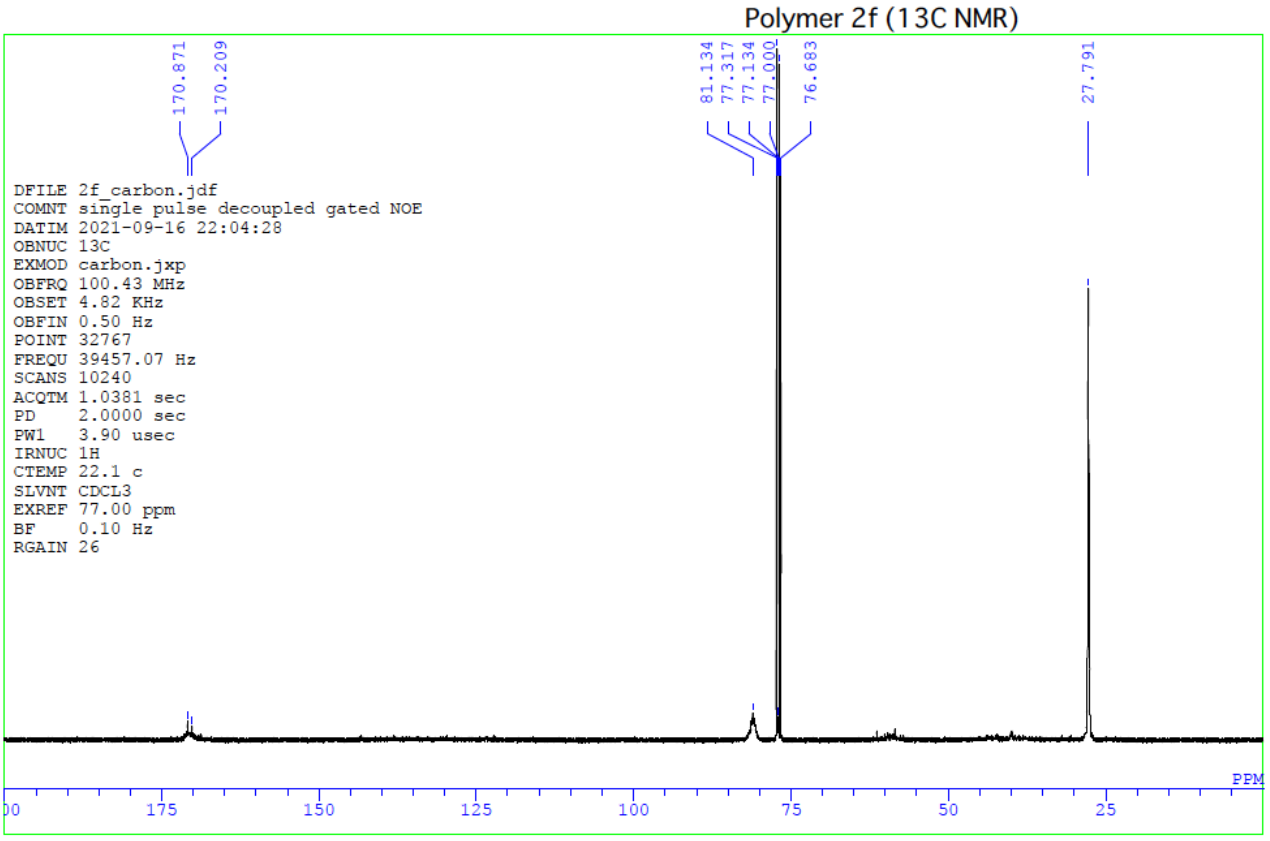
Polymer 2e


Polymer 2e (1H NMR)
7.211
6.920
3.357
3.340
3.325
2.923
2.565
2.422
2.110
1.185
1.167
1.150
1.132
1.112
1.085
1.067
1.049
1.038
0.969
0.883
0.866
0.850
0.000
DFILE 2e_1H.als
COMNT single_pulse
DATIM 2021-11-25 20:56:54
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.38 MHz
OBSET 4.41 KHz
OBFIN 3.09 Hz
POINT 13107
FREQU 5995.20 Hz
SCANS 8
ACQTM 2.1863 sec
PD 5.0000 sec
PW1 3.20 usec
IRNUC 1H
CTEMP 23.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.10 Hz
RGAIN 36
PPM
8
6
4
2
0

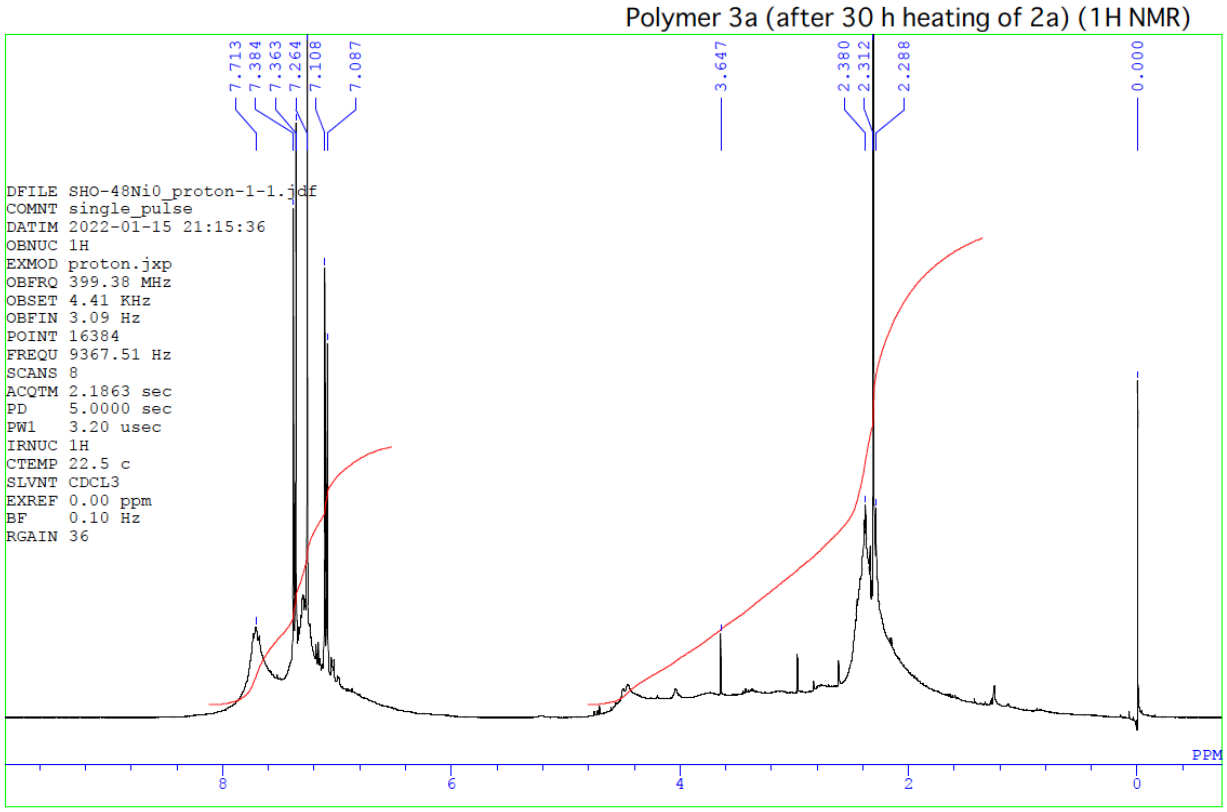

Polymer 2e (13C NMR)
210.794
173.719
141.988
77.326
77.000
76.683
69.441
53.777
41.854
40.626
40.271
29.211
14.851
13.038
12.808
DFILE 2e_13C.als
COMNT single pulse decoupled gated NOE
DATIM 2021-11-25 23:52:25
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.43 MHz
OBSET 4.82 KHz
OBFIN 0.50 Hz
POINT 26214
FREQU 25252.53 Hz
SCANS 4500
ACQTM 1.0381 sec
PD 2.0000 sec
PW1 3.90 usec
IRNUC 1H
CTEMP 22.8 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.10 Hz
RGAIN 16
PPM
200
150
100
50
0

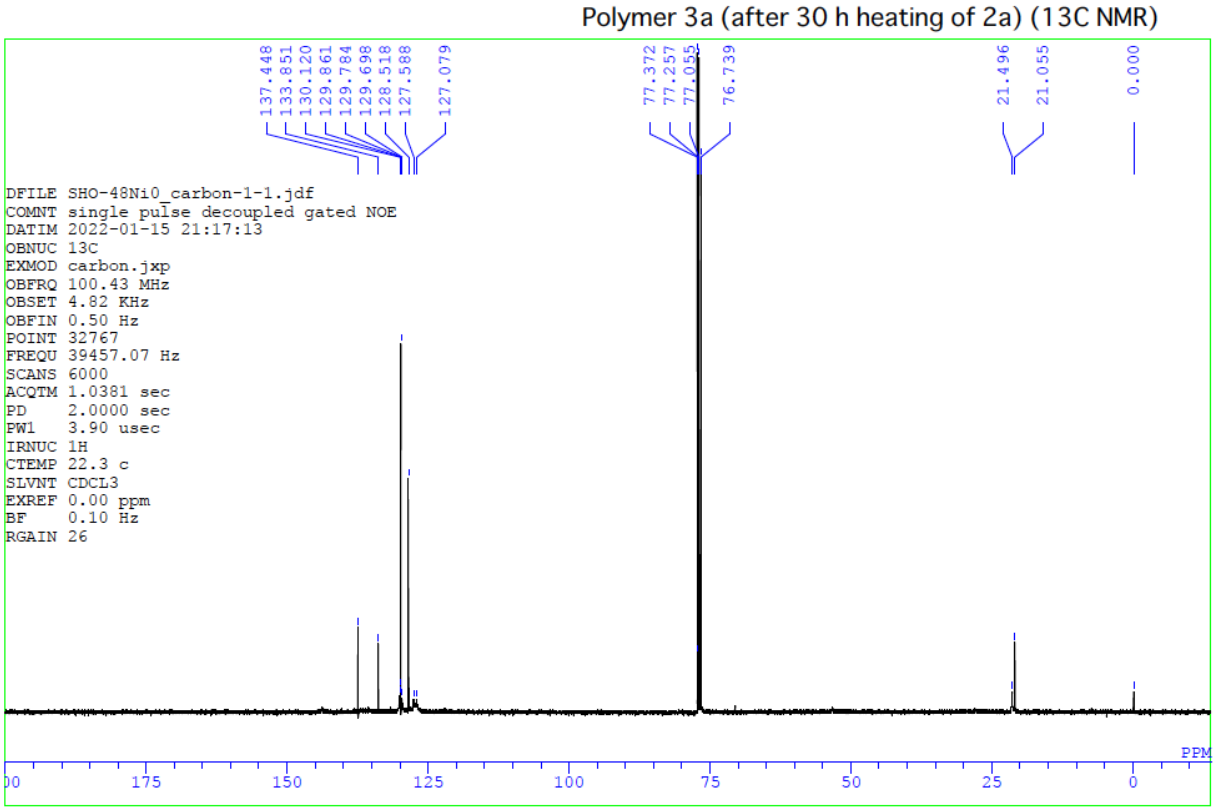
Polymer 2f


Polymer 2f (1H NMR)
7.205
3.350
2.900
2.872
2.816
1.367
1.275
1.261
1.232
1.213
1.200
1.185
1.167
DFILE 2f_proton.jdf
COMNT single_pulse
DATIM 2021-09-16 22:01:54
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.38 MHz
OBSET 4.41 KHz
OBFIN 3.09 Hz
POINT 16384
FREQU 9367.51 Hz
SCANS 16
ACQTM 2.1863 sec
PD 5.0000 sec
PW1 3.20 usec
IRNUC 1H
CTEMP 22.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.10 Hz
RGAIN 36
PPM
8
6
4
2
0

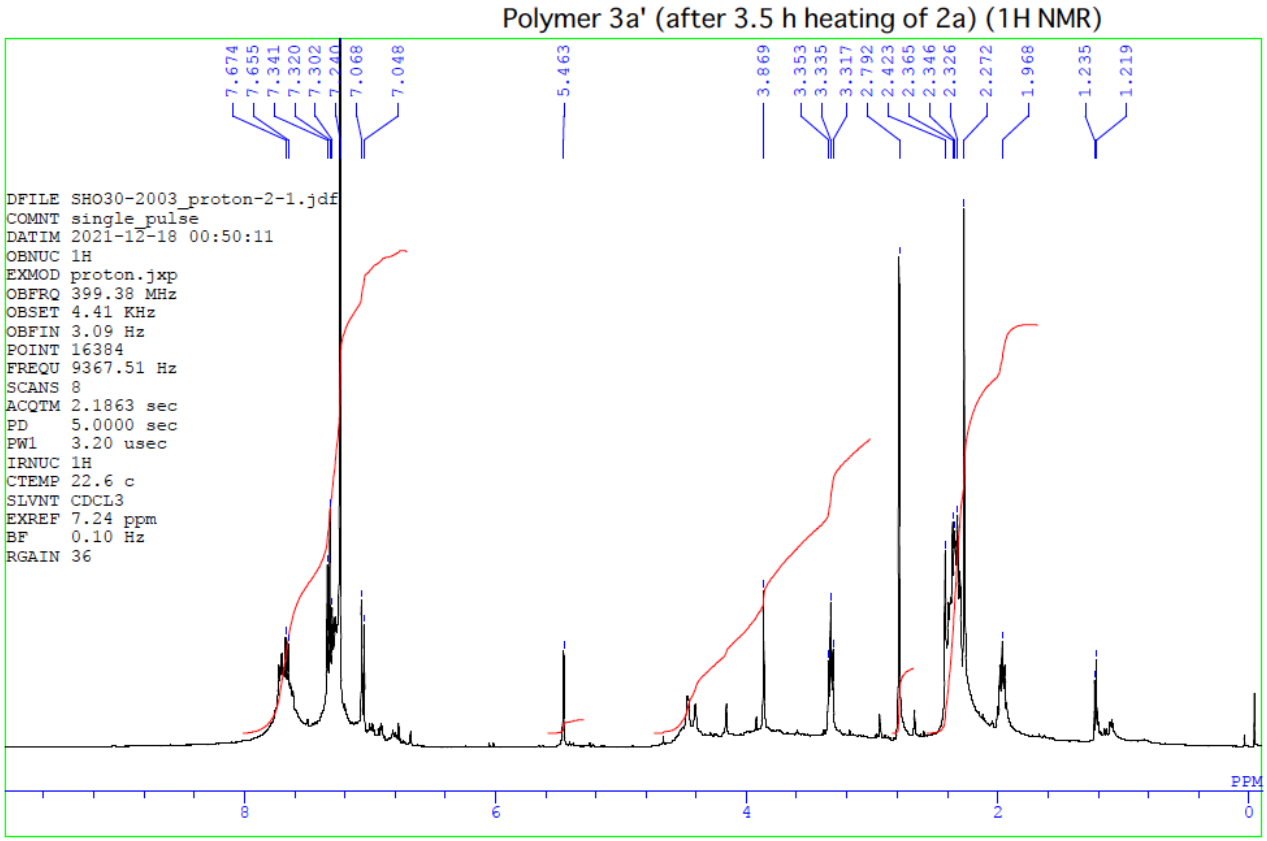

Polymer 2f (13C NMR)
170.871
170.209
81.134
77.317
77.134
77.000
76.683
27.791
DFILE 2f_carbon.jdf
COMNT single pulse decoupled gated NOE
DATIM 2021-09-16 22:04:28
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.43 MHz
OBSET 4.82 KHz
OBFIN 0.50 Hz
POINT 32767
FREQU 39457.07 Hz
SCANS 10240
ACQTM 1.0381 sec
PD 2.0000 sec
PW1 3.90 usec
IRNUC 1H
CTEMP 22.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.10 Hz
RGAIN 26
PPM
175
150
125
100
75
50
25

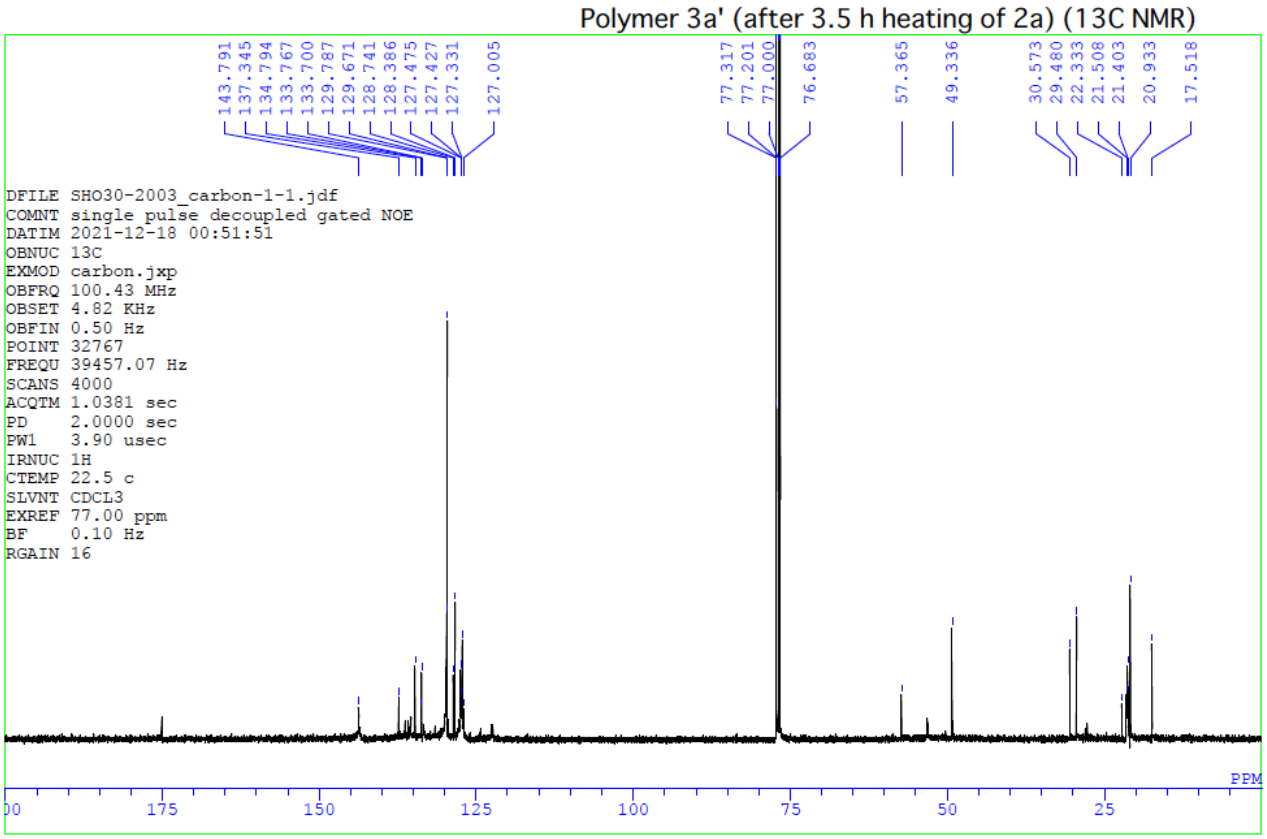
Polymer 3a (after 30h heating of 2a)



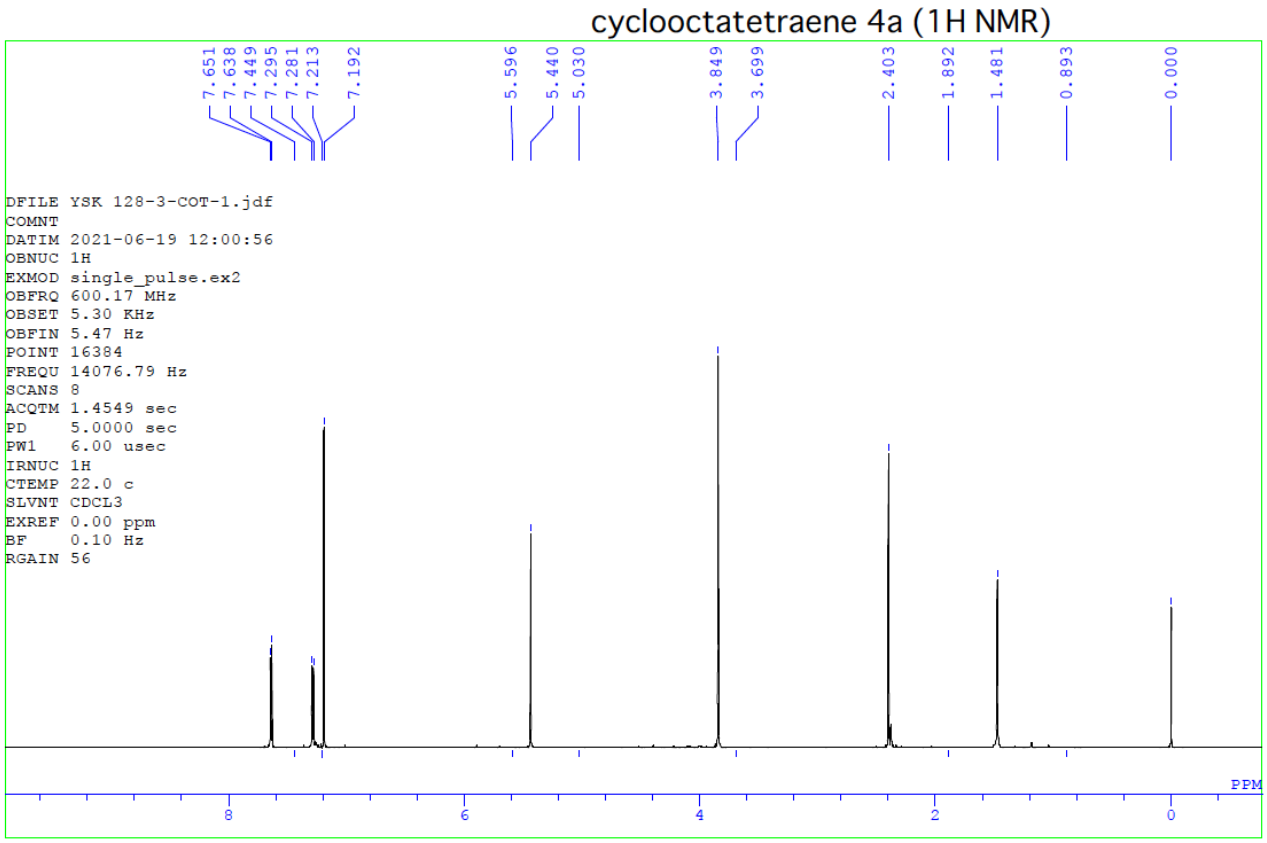


Polymer 3a’ (after 3.5h heating of 2a)



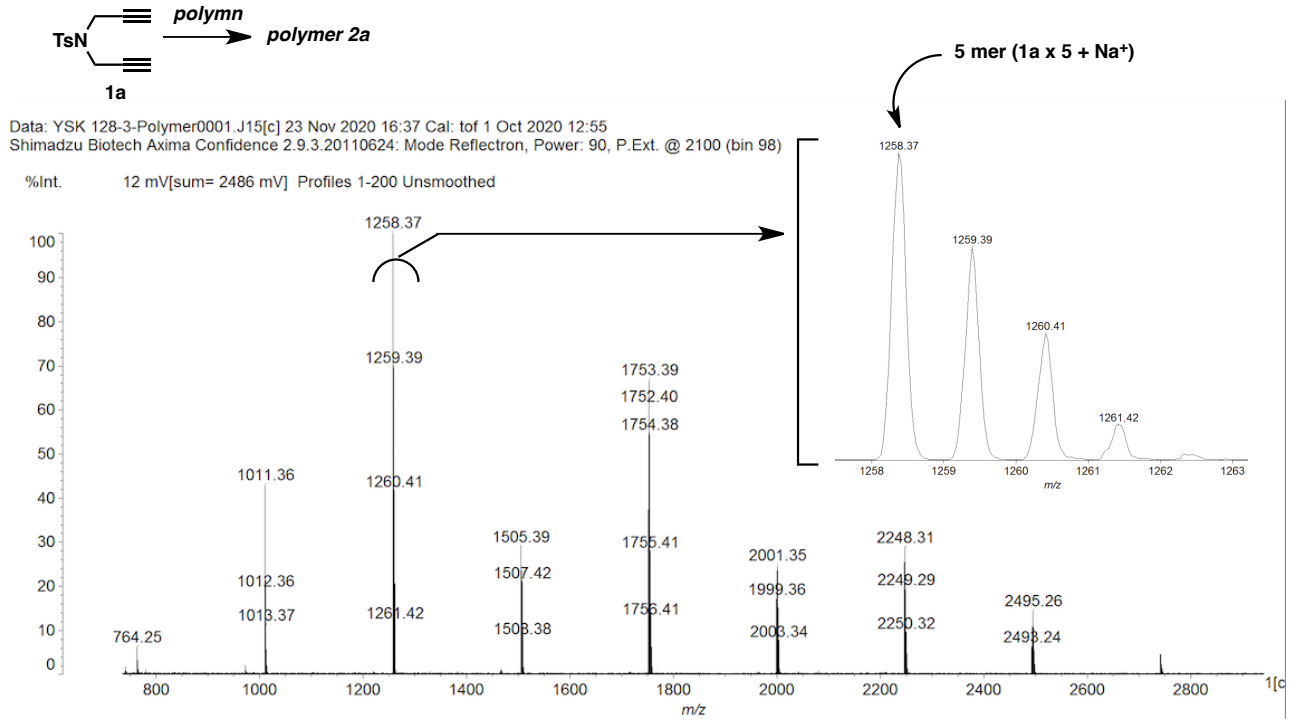


Cyclooctatetraene 4a

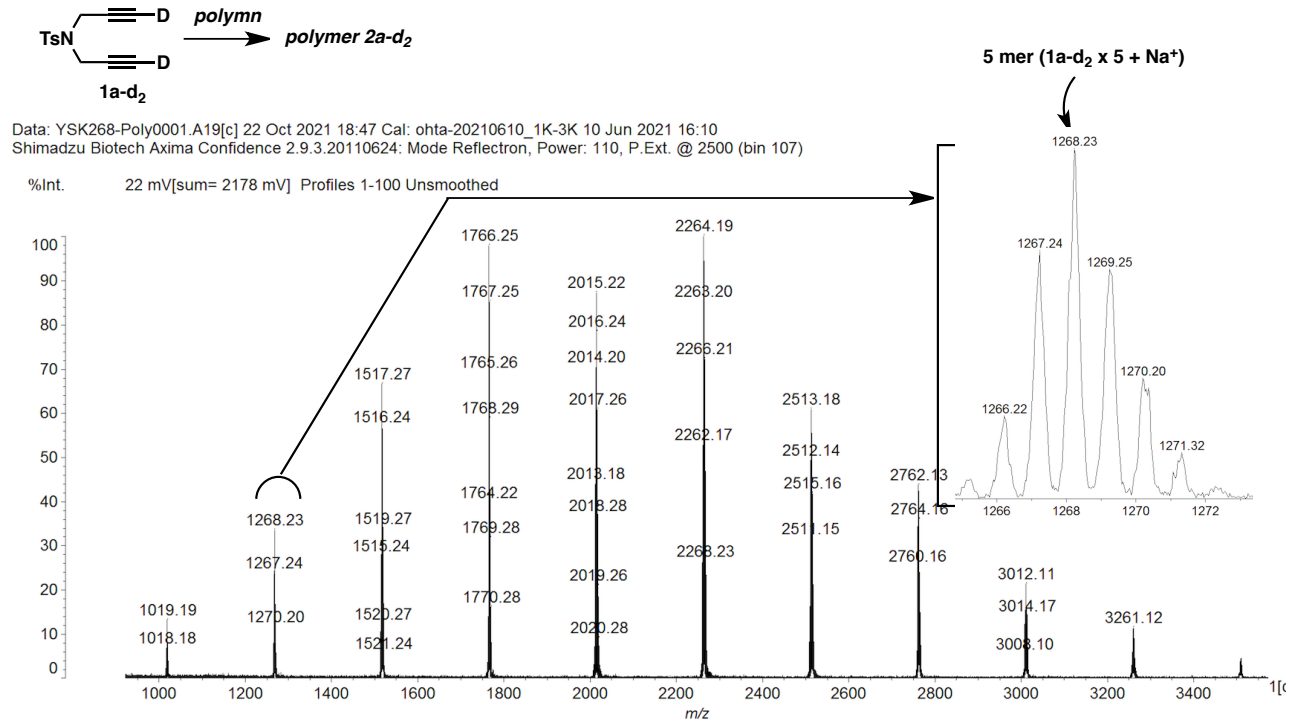


MALDI-TOF-MS Spectra

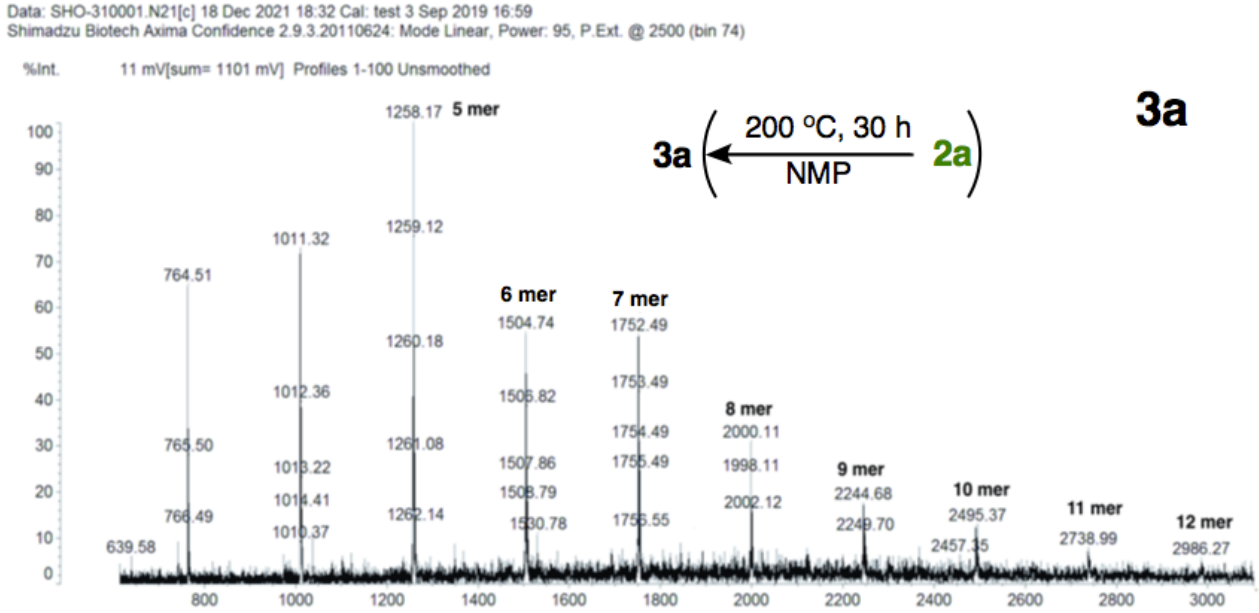
2a



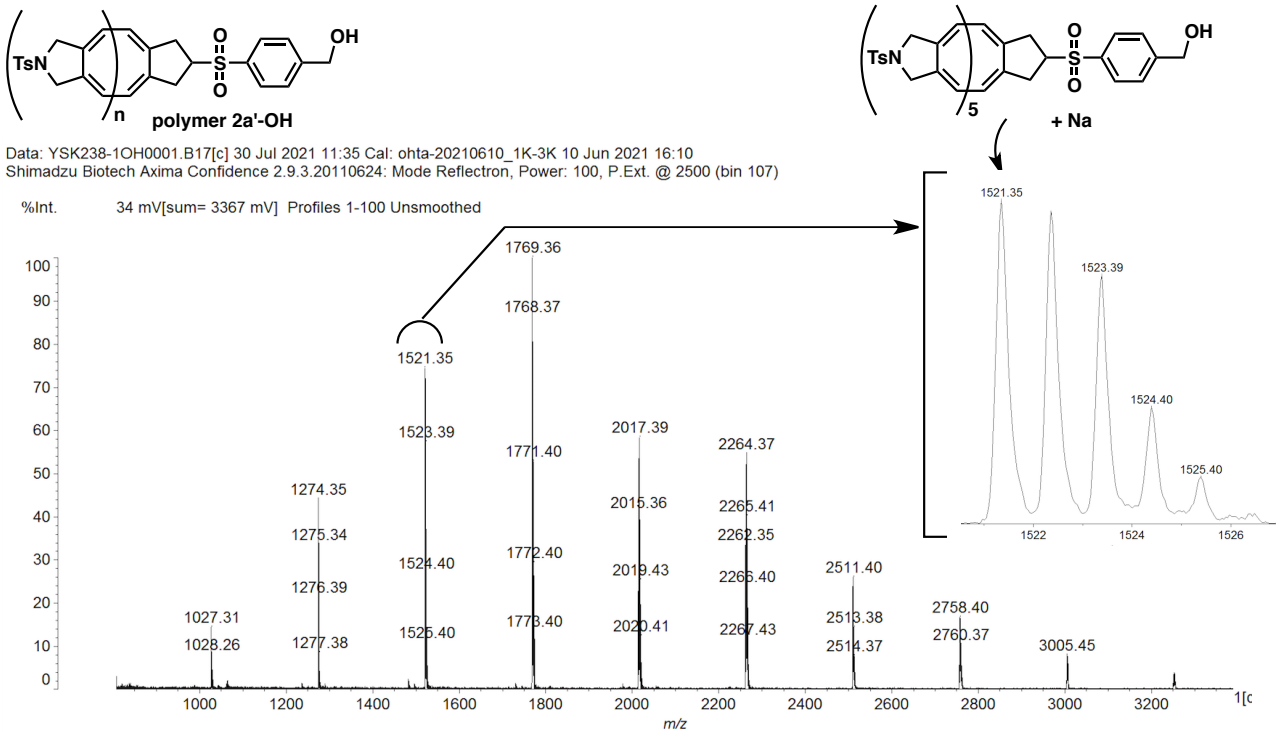
2a-d



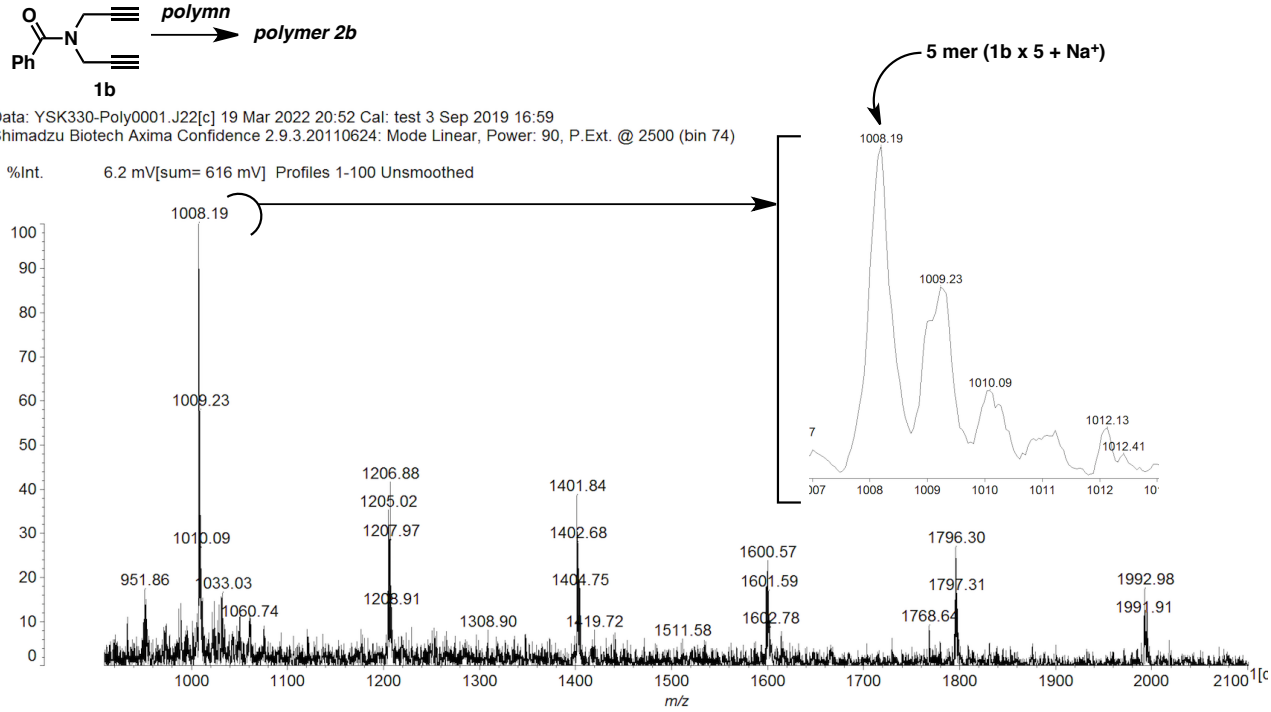
3a


Data: SHO-310001.N21[c] 18 Dec 2021 18:32 Cal: test 3 Sep 2019 16:59
Shimadzu Biotech Axima Confidence 2.9.3.20110624: Mode Linear, Power: 95, P.Ext. @ 2500 (bin 74)
%Int.
11 mV[sum= 1101 mV] Profiles 1-100 Unsmoothed
3a
3a
200 °C, 30 h
NMP
2a
5 mer
6 mer
7 mer
8 mer
9 mer
10 mer
11 mer
12 mer
1258.17
1259.12
1260.18
1261.08
1262.14
1011.32
1012.36
1013.22
1014.41
1010.37
764.51
765.50
766.49
639.58
1504.74
1505.82
1507.86
1508.79
1530.78
1752.49
1753.49
1754.49
1755.49
1755.55
2000.11
1998.11
2002.12
2244.68
2249.70
2495.37
2457.35
2738.99
2986.27

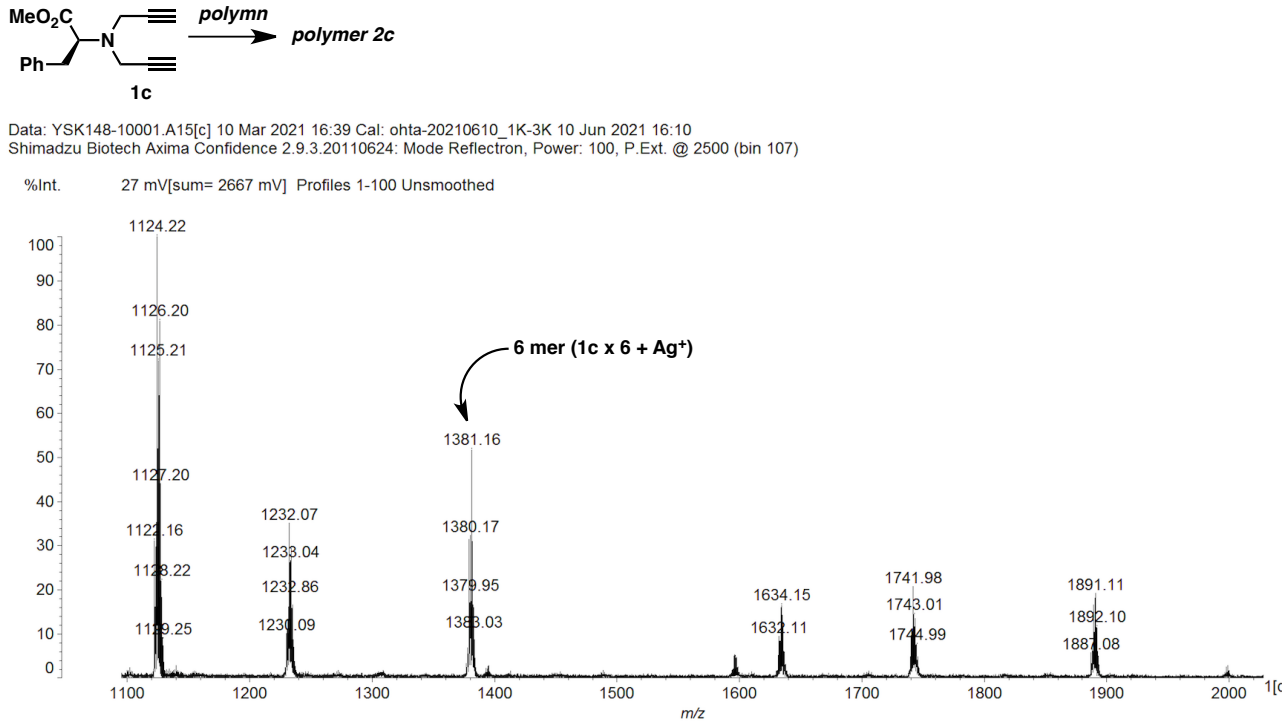
2a'-OH


TsN
OH
n
polymer 2a'-OH
5
+ Na
Data: YSK238-1OH0001.B17[c] 30 Jul 2021 11:35 Cal: ohta-20210610_1K-3K 10 Jun 2021 16:10
Shimadzu Biotech Axima Confidence 2.9.3.20110624: Mode Reflectron, Power: 100, P.Ext. @ 2500 (bin 107)
%Int.
34 mV[sum= 3367 mV] Profiles 1-100 Unsmoothed
m/z
1521.35
1523.39
1524.40
1525.40
1769.36
1768.37
1771.40
1772.40
1773.40
1274.35
1275.34
1276.39
1277.38
1027.31
1028.26
1524.40
1525.40
2017.39
2015.36
2019.43
2020.41
2264.37
2265.41
2262.35
2265.40
2267.43
2511.40
2513.38
2514.37
2758.40
2760.37
3005.45

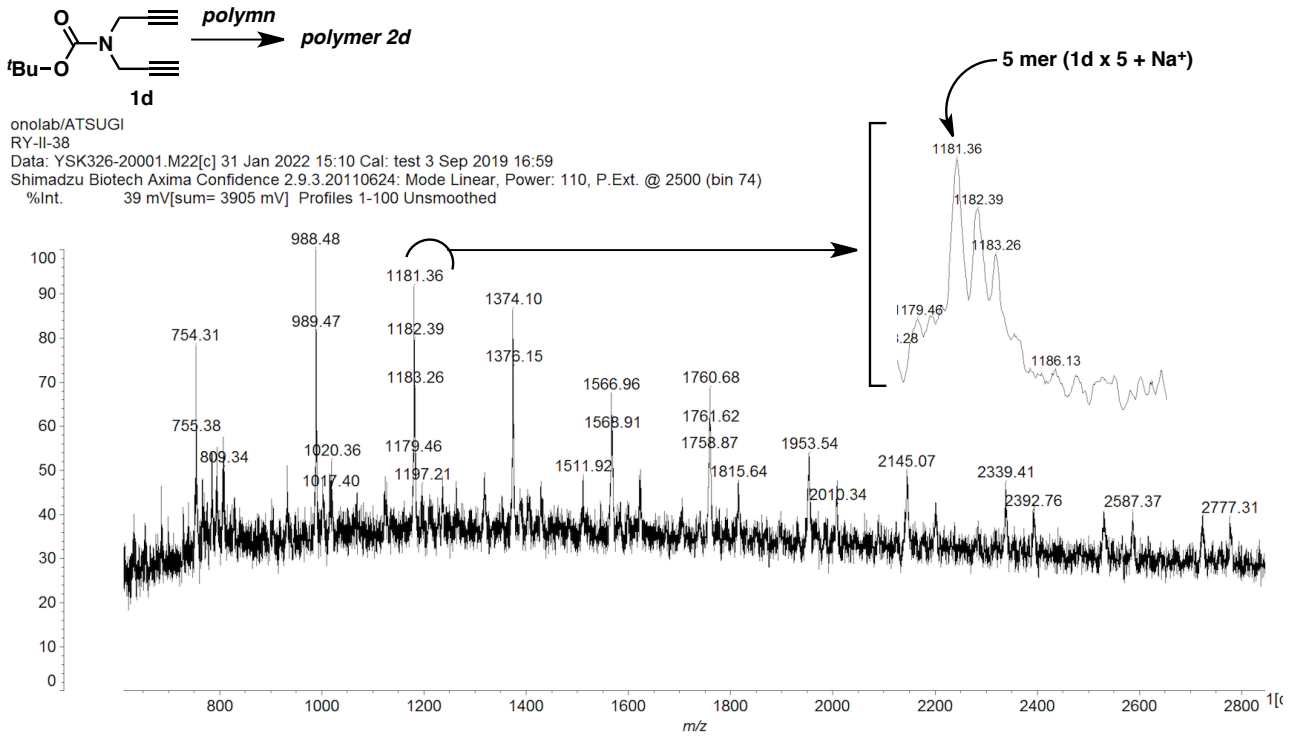
2b


polymn
polymer 2b
1b
5 mer (1b x 5 + Na+)
Data: YSK330-Poly0001.J22[c] 19 Mar 2022 20:52 Cal: test 3 Sep 2019 16:59
Shimadzu Biotech Axima Confidence 2.9.3.20110624: Mode Linear, Power: 90, P.Ext. @ 2500 (bin 74)
%Int.
6.2 mV[sum= 616 mV] Profiles 1-100 Unsmoothed
1008.19
1009.23
1010.09
1206.88
1205.02
1207.97
1401.84
1402.68
1600.57
1601.59
1796.30
1797.31
1992.98
1991.91
951.86
1033.03
1060.74
1208.91
1308.90
1419.72
1404.75
1511.58
1602.78
1768.64
m/z

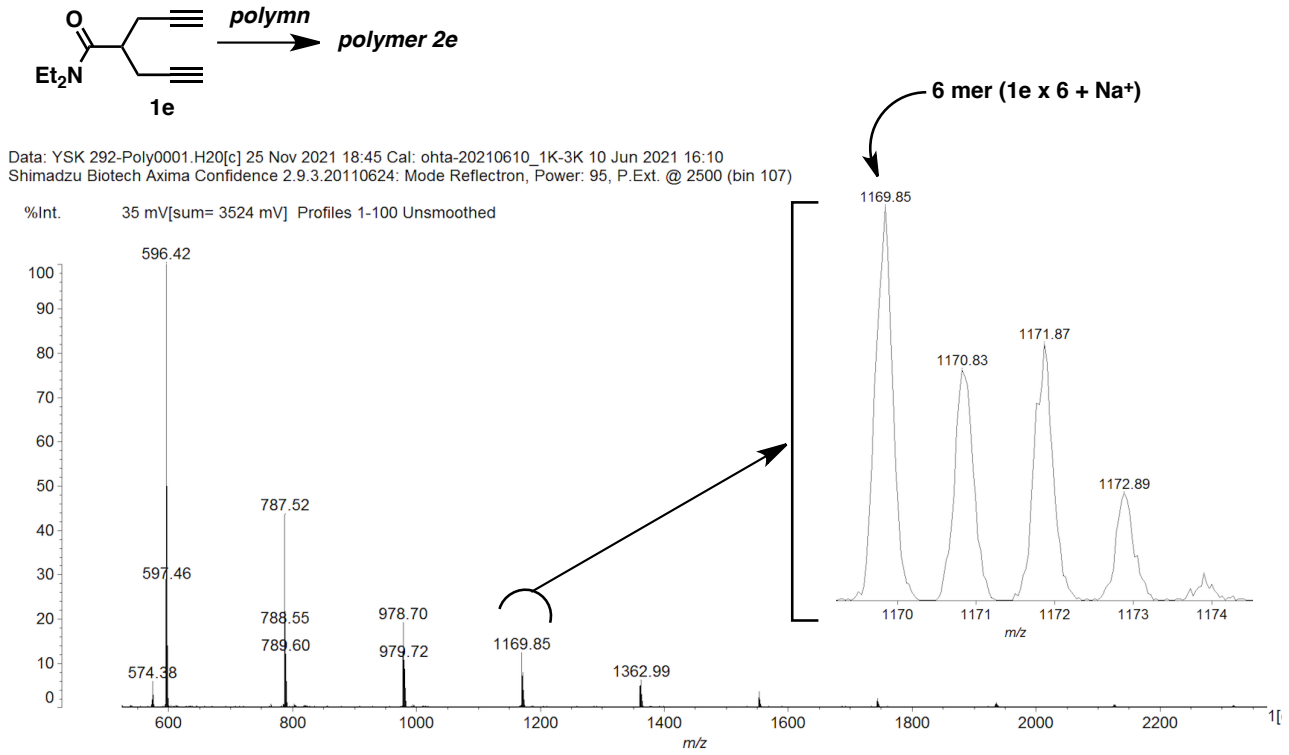
2c


polymn
polymer 2c
1c
6 mer (1c x 6 + Ag+)
Data: YSK148-10001.A15[c] 10 Mar 2021 16:39 Cal: ohta-20210610_1K-3K 10 Jun 2021 16:10
Shimadzu Biotech Axima Confidence 2.9.3.20110624: Mode Reflectron, Power: 100, P.Ext. @ 2500 (bin 107)
%Int.
27 mV[sum= 2667 mV] Profiles 1-100 Unsmoothed
1124.22
1126.20
1125.21
1127.20
1122.16
1128.22
1129.25
1232.07
1233.04
1232.86
1230.09
1381.16
1380.17
1379.95
1383.03
1634.15
1632.11
1741.98
1743.01
1744.99
1891.11
1892.10
1887.08
m/z

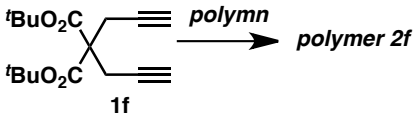
2d


polymn
polymer 2d
1d
5 mer (1d x 5 + Na+)
onolab/ATSUGI
RY-II-38
Data: YSK326-20001.M22[c] 31 Jan 2022 15:10 Cal: test 3 Sep 2019 16:59
Shimadzu Biotech Axima Confidence 2.9.3.20110624: Mode Linear, Power: 110, P.Ext. @ 2500 (bin 74)
%Int. 39 mV[sum= 3905 mV] Profiles 1-100 Unsmoothed
m/z

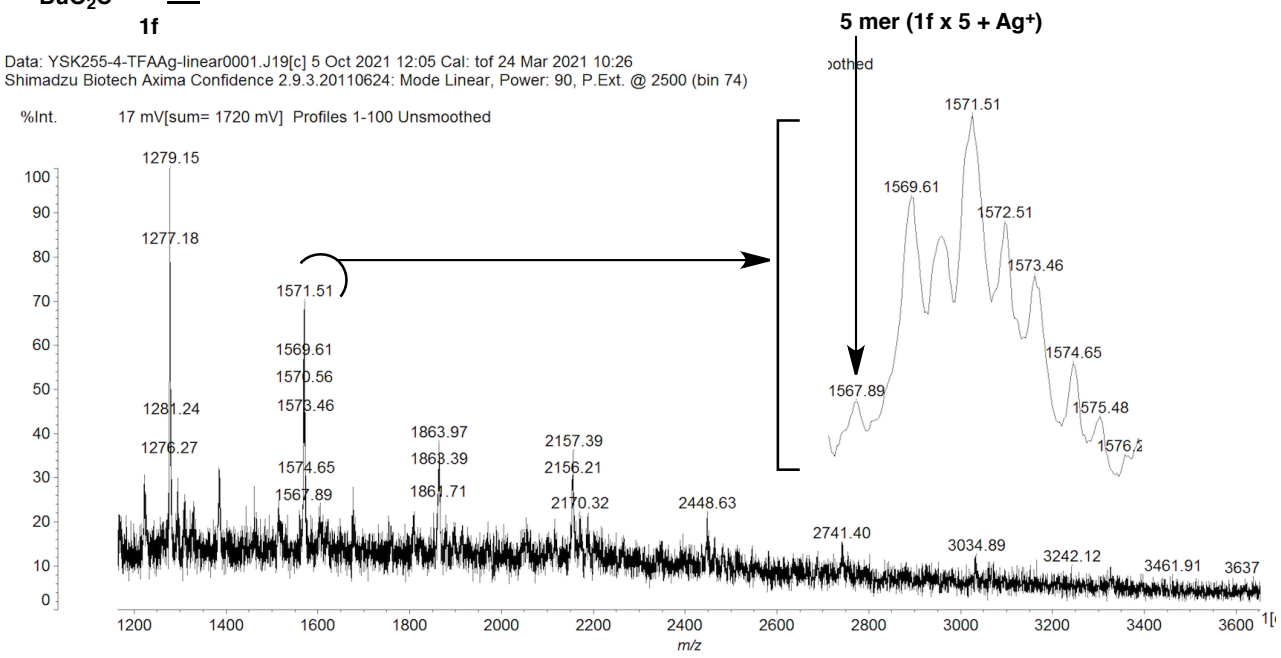
2e


polymn
polymer 2e
1e
6 mer (1e x 6 + Na+)
Data: YSK 292-Poly0001.H20[c] 25 Nov 2021 18:45 Cal: ohta-20210610_1K-3K 10 Jun 2021 16:10
Shimadzu Biotech Axima Confidence 2.9.3.20110624: Mode Reflectron, Power: 95, P.Ext. @ 2500 (bin 107)
%Int. 35 mV[sum= 3524 mV] Profiles 1-100 Unsmoothed
m/z

2f


tBuO2C
tBuO2C
polymn
polymer 2f

1f


5 mer (1f x 5 + Ag+)
Data: YSK255-4-TFAAg-linear0001.J19[c] 5 Oct 2021 12:05 Cal: tof 24 Mar 2021 10:26
Shimadzu Biotech Axima Confidence 2.9.3.20110624: Mode Linear, Power: 90, P.Ext. @ 2500 (bin 74)
%Int.
17 mV[sum= 1720 mV] Profiles 1-100 Unsmoothed
1279.15
1277.18
1571.51
1569.61
1570.56
1573.46
1281.24
1276.27
1574.65
1567.89
1863.97
1863.39
1861.71
2157.39
2156.21
2170.32
2448.63
2741.40
3034.89
3242.12
3461.91
3637
1571.51
1569.61
1572.51
1573.46
1574.65
1567.89
1575.48
m/z