Supplementary Materials

Ionenes as Potential Phase Change Materials with Self-Healing Behavior

Carolina Arriaza-Echanes [1], María V. Velázquez-Tundidor [2], Alejandro Angel-López [1], Ángel Norambuena [1,3], Francisco E. Palay [1], Claudio A. Terraza [2,4], Alain Tundidor-Camba [2,4], Pablo A. Ortiz [1,5,6,*] and Deysma Coll [1,6,7,*]

[1] Vicerrectoría de Investigación, Universidad Mayor, Camino la Pirámide 5750, Santiago 8580745, Chile; angel.norambuena@mayor.cl (Á.N.); francisco.palay@mayor.cl (F.E.P.)
[2] Research Laboratory for Organic Polymers (RLOP), Department of Organic Chemistry, Pontificia Universidad Católica de Chile, Santiago 7820436, Chile
[3] Instituto de Investigaciones y Control del Ejército de Chile (IDIC), Santiago 8370899, Chile
[4] UC Energy Research Center, Pontificia Universidad Católica de Chile, Santiago 7820436, Chile
[5] Escuela de Ingeniería en Medio Ambiente y Sustentabilidad, Facultad de Ciencias, Ingeniería y Tecnología, Universidad Mayor, Camino La Pirámide 5750, Santiago 8580745, Chile
[6] Centro de Nanotecnología Aplicada, Facultad de Ciencias, Ingeniería y Tecnología, Universidad Mayor, Camino La Pirámide 5750, Santiago 8580745, Chile
[7] Núcleo de Química y Bioquímica, Facultad de Ciencias, Ingeniería y Tecnología, Universidad Mayor, Camino La Pirámide 5750, Santiago 8580745, Chile
***** Correspondence: pablo.ortiz@umayor.cl (P.A.O.); deysma.coll@umayor.cl (D.C.)

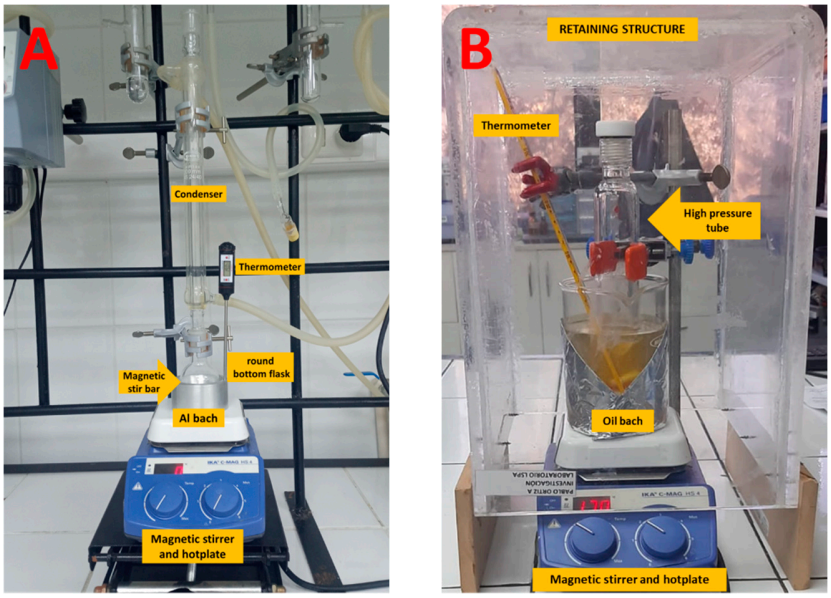
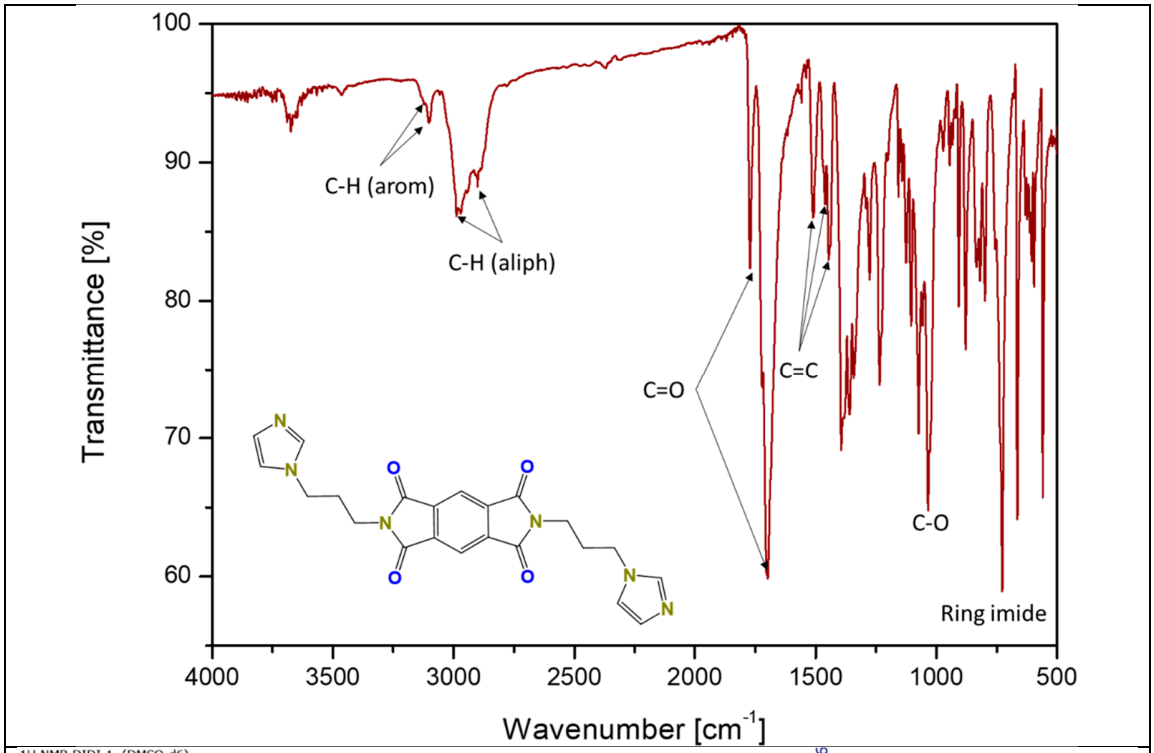
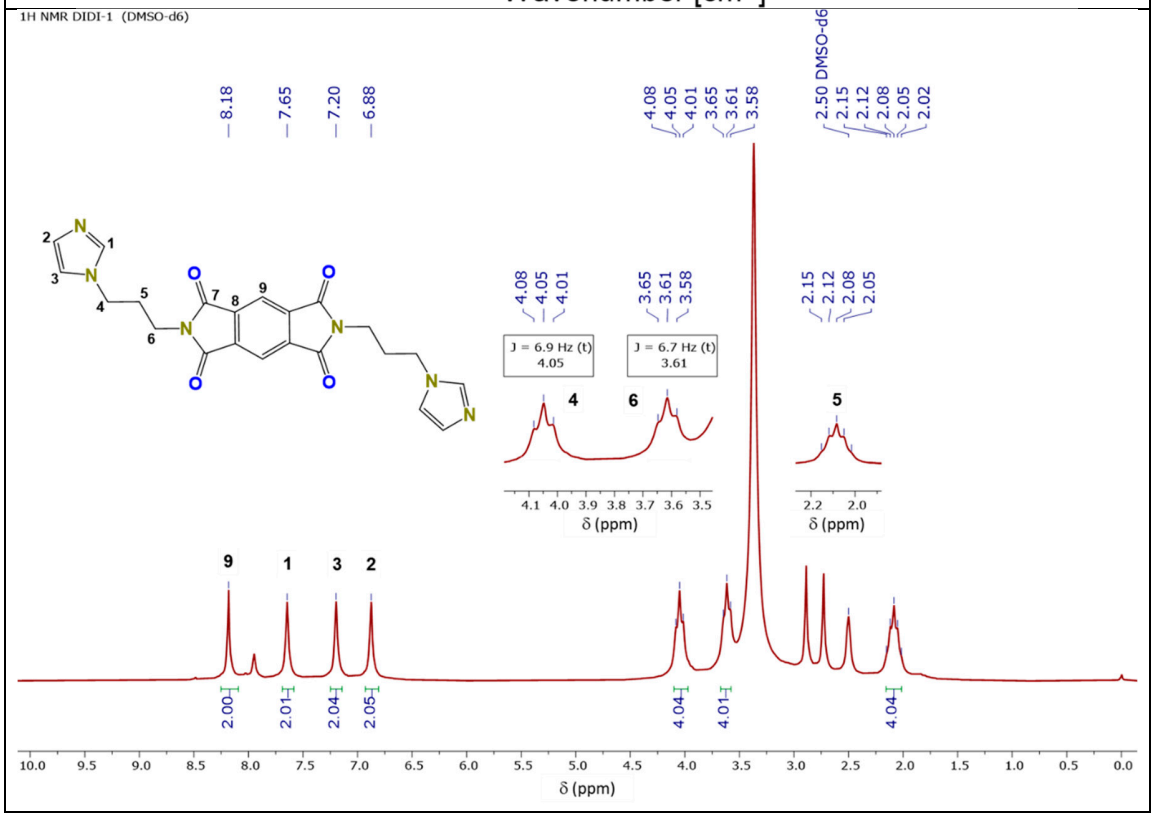


Figure S1. A. Experimental setup for the monomers synthesis. **B.** Experimental setup for the ionenes synthesis.

Spectroscopic data

Below are the infrared and nuclear magnetic resonance spectra of both the synthesized monomers and ionenes.


Transmittance [%]
Wavenumber [cm-1]
C-H (arom)
C-H (aliph)
C=O
C=C
C-O
Ring imide


1H NMR DIDI-1 (DMSO-d6)
δ (ppm)
J = 6.9 Hz (t)
4.05
J = 6.7 Hz (t)
3.61

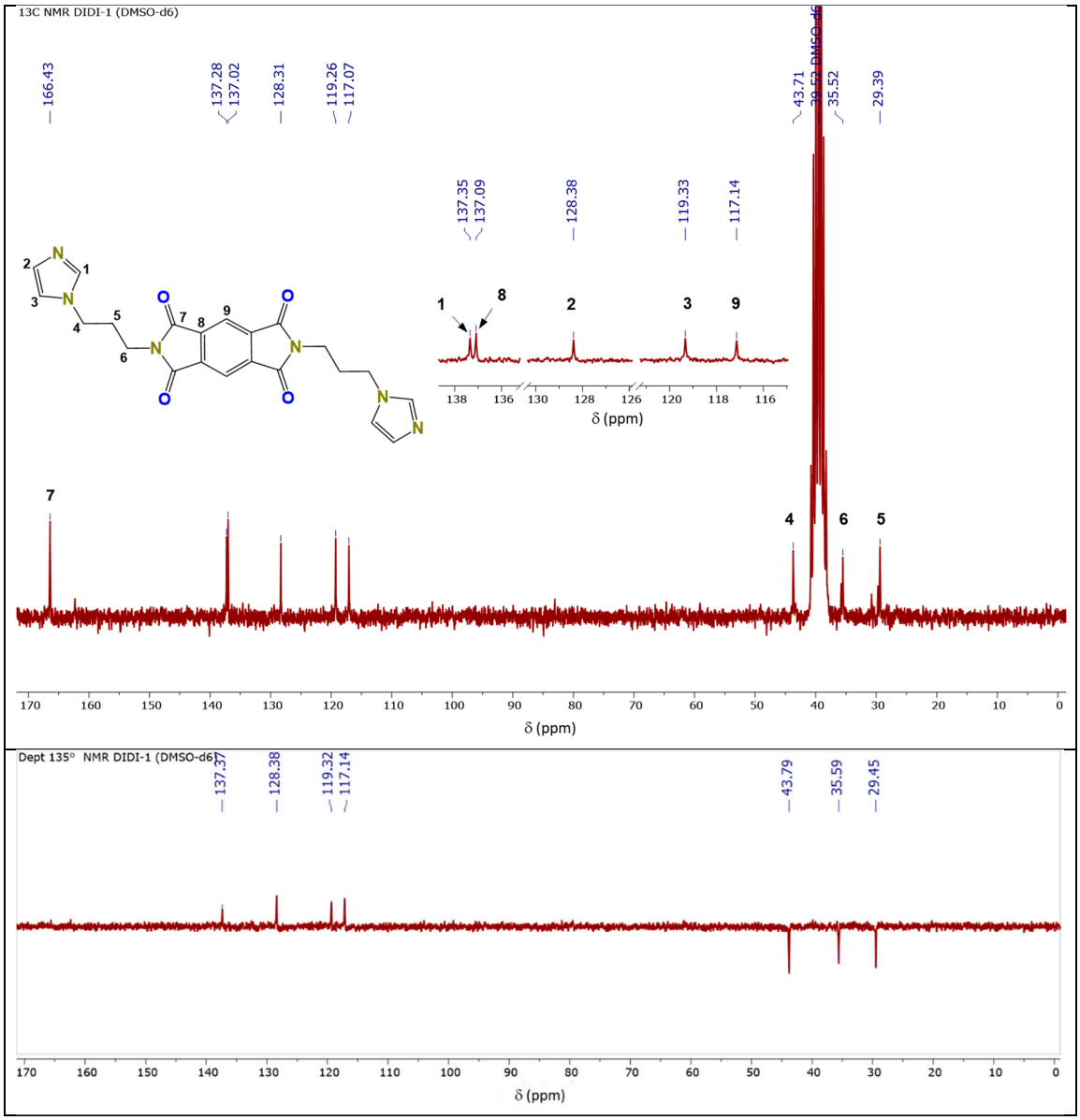
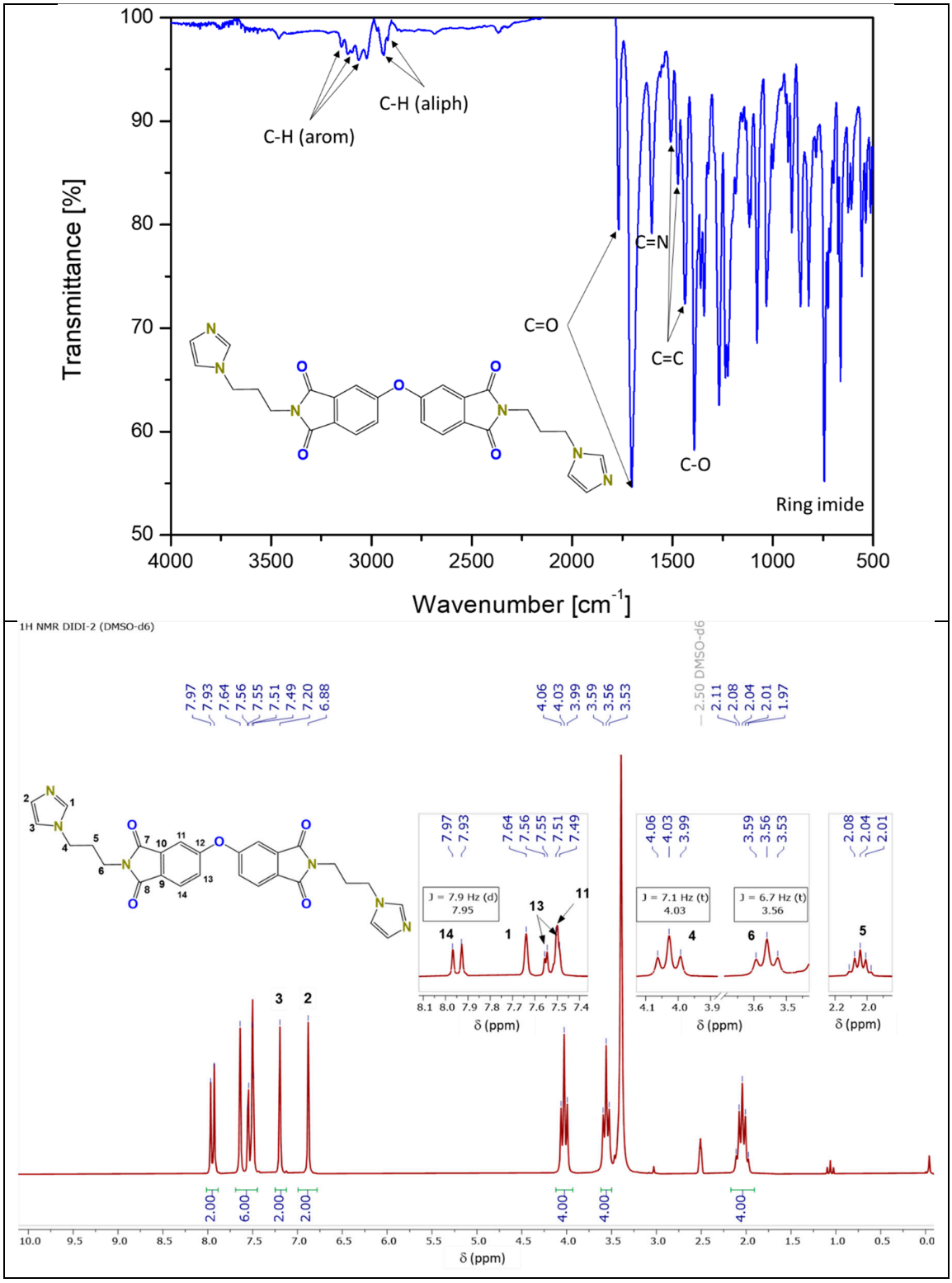


Figure S2. IR and ^{1}H, ^{13}C and Dept 135°NMR spectra of PMDA-API.


Transmittance [%]
Wavenumber [cm-1]
C-H (arom)
C-H (aliph)
C=O
C=N
C=C
C-O
Ring imide
1H NMR DIDI-2 (DMSO-d6)
δ (ppm)

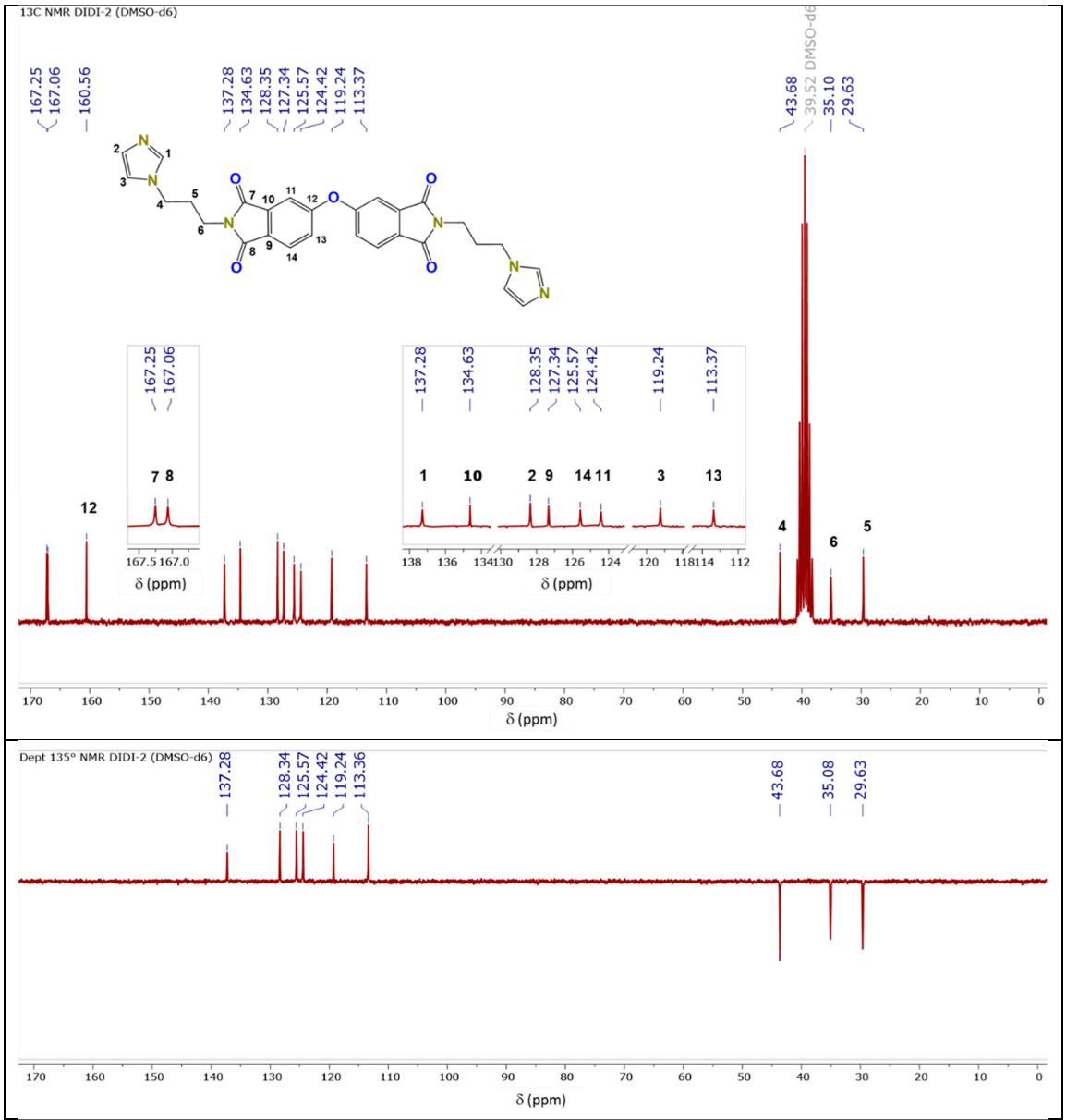
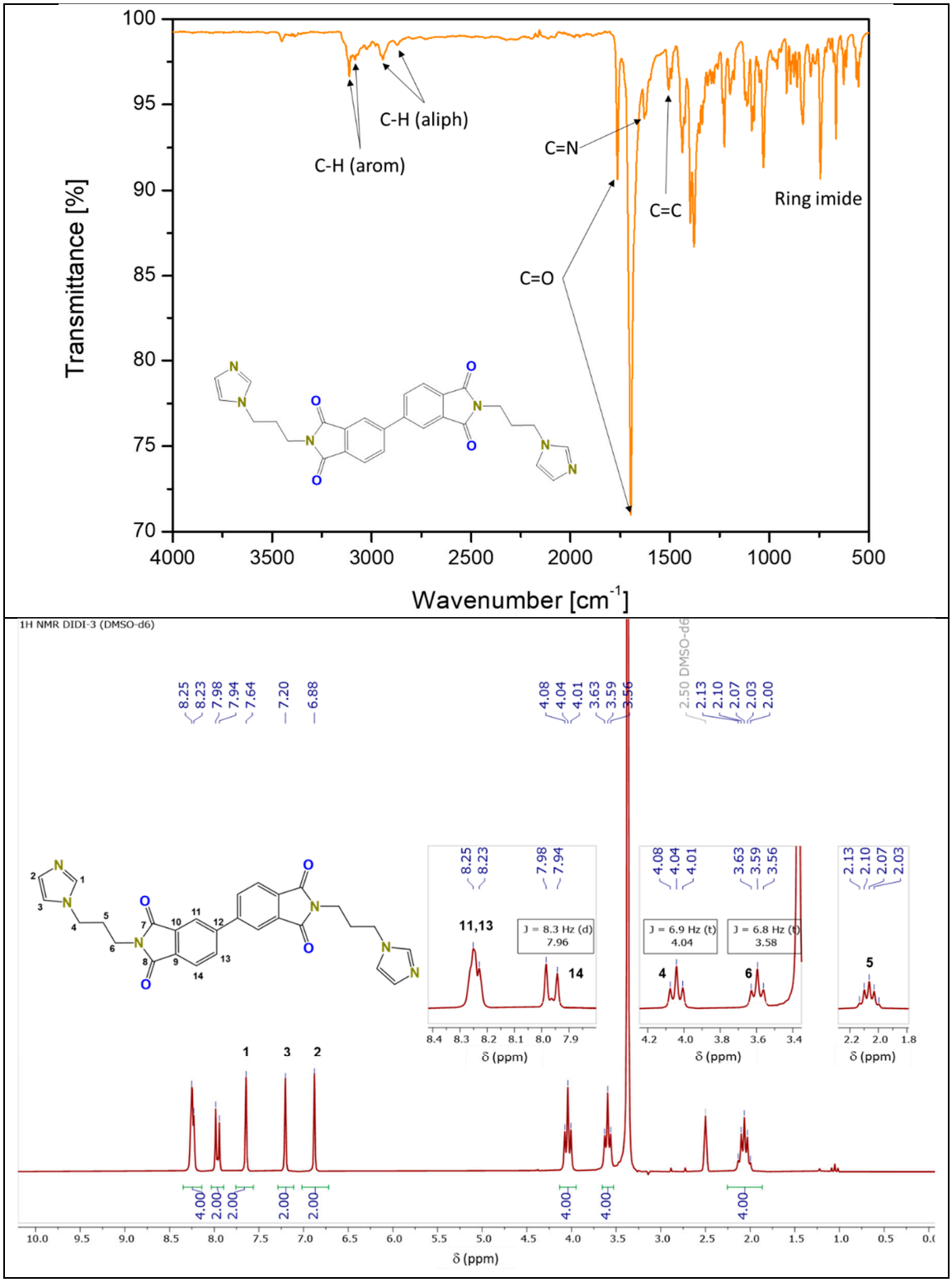


Figure S3. IR and ^{1}H, ^{13}C and Dept 135° NMR spectra of ODPA-API .


Transmittance [%]
Wavenumber [cm-1]
C-H (arom)
C-H (aliph)
C=N
C=C
C=O
Ring imide
1H NMR DIDI-3 (DMSO-d6)
δ (ppm)

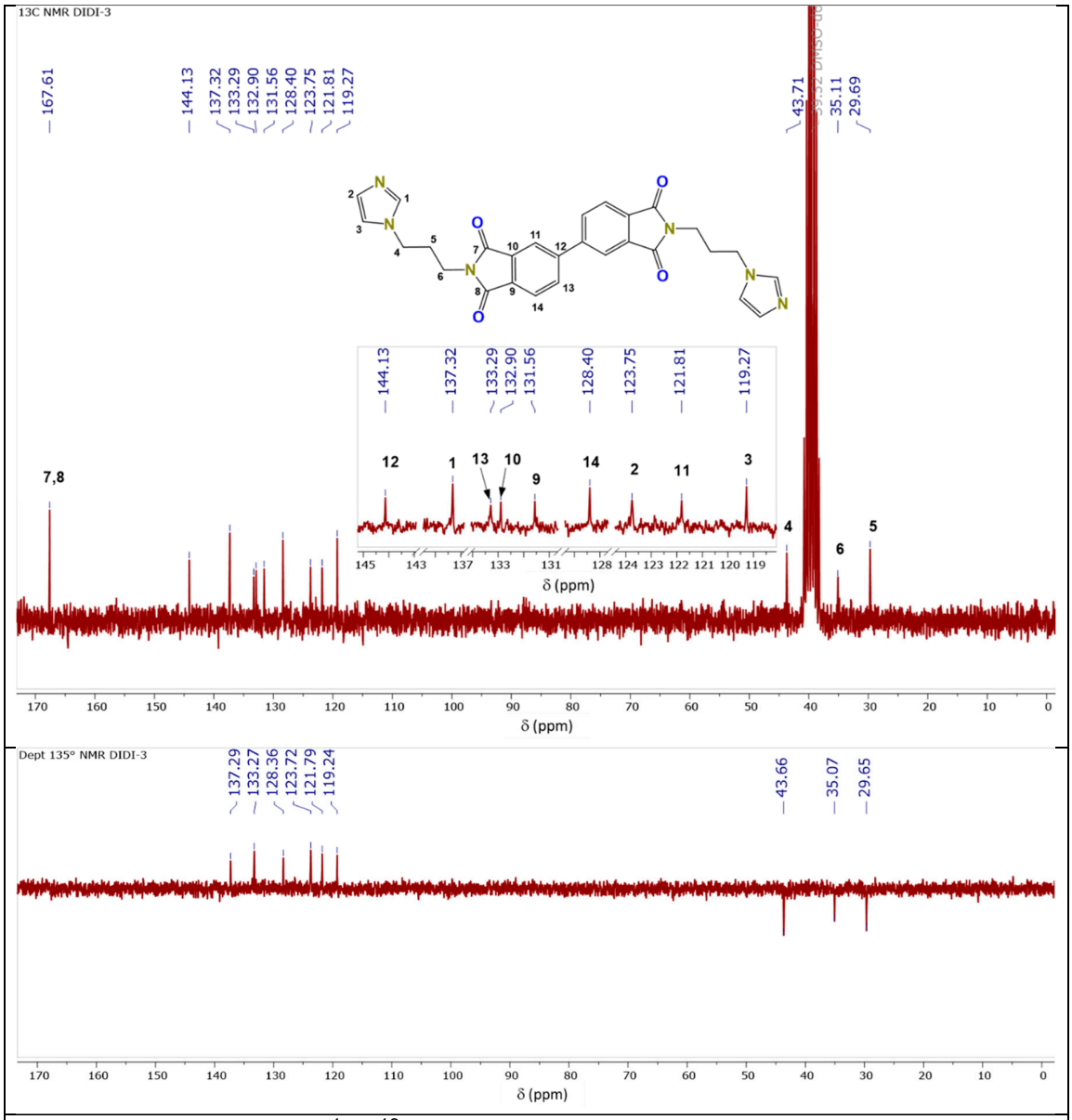
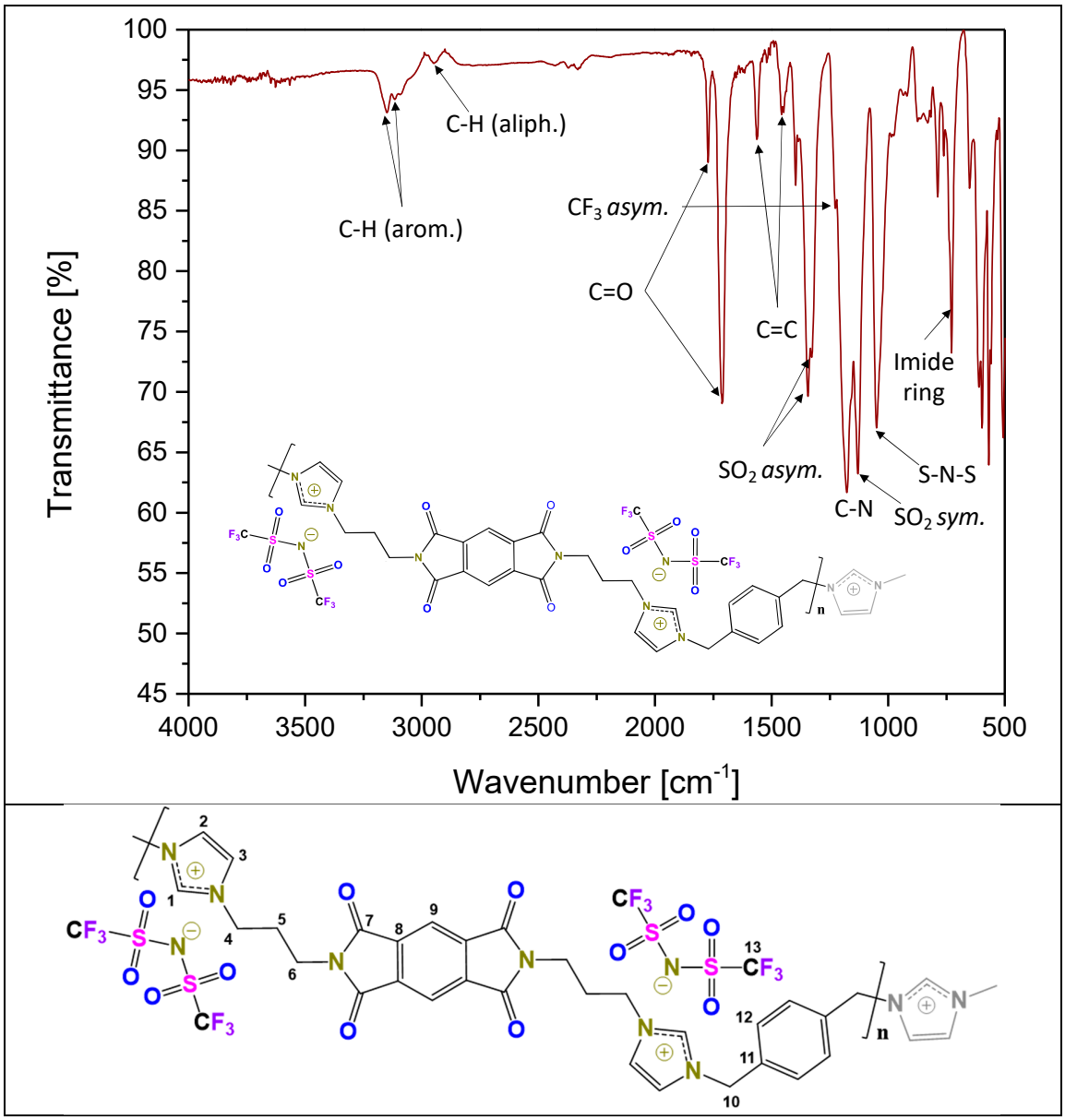
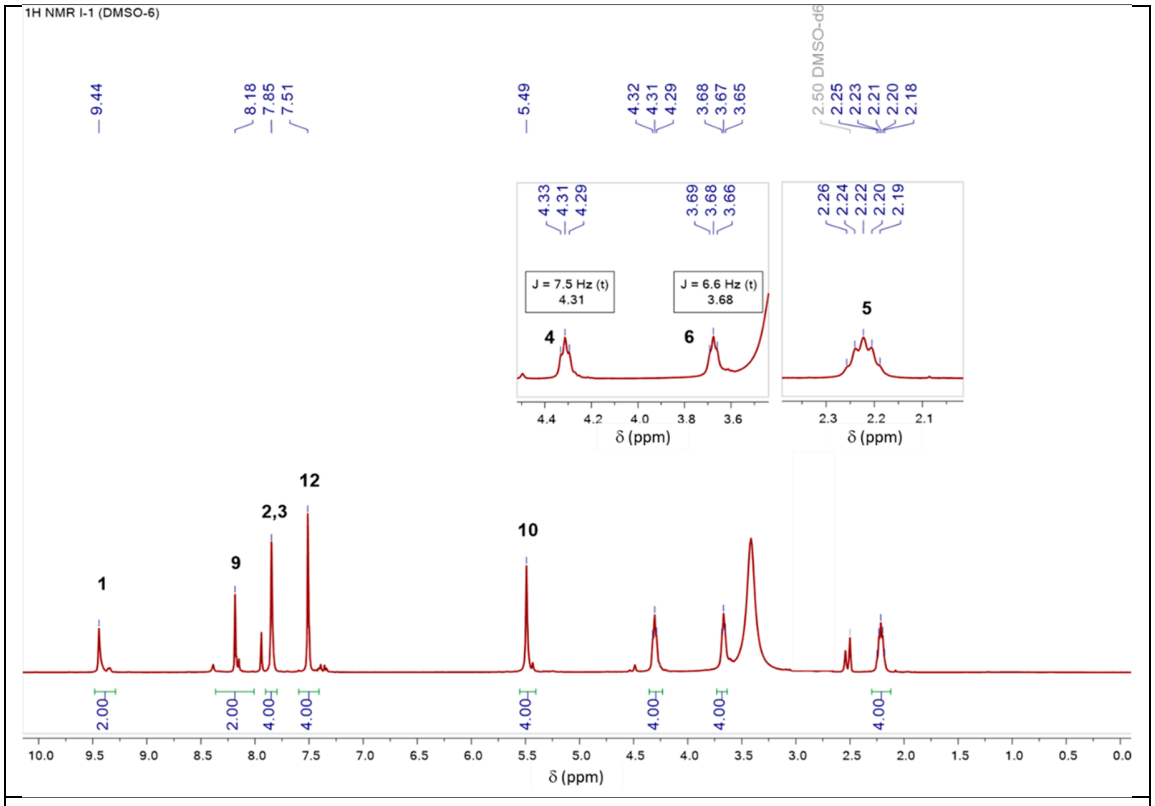
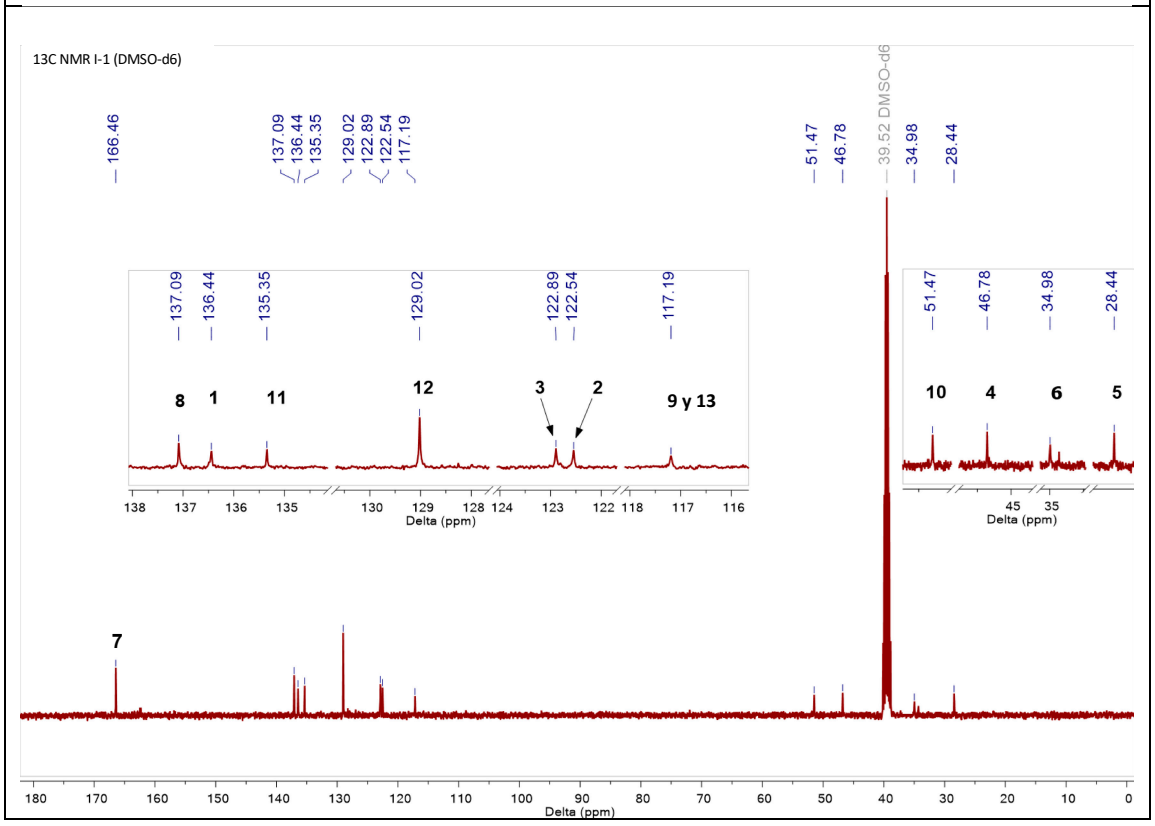


Figure S4. IR and ^{1}H, ^{13}C and Dept 135° NMR spectra of BPDA-API .


Transmittance [%]
Wavenumber [cm-1]
C-H (aliph.)
C-H (arom.)
CF3 asym.
C=O
C=C
Imide ring
SO2 asym.
S-N-S
C-N
SO2 sym.


1H NMR I-1 (DMSO-6)
9.44
8.18
7.85
7.51
5.49
4.32
4.31
4.29
3.68
3.67
3.65
2.50 DMSO-d6
2.25
2.23
2.21
2.20
2.18
4.33
4.31
4.29
3.69
3.68
3.66
J = 7.5 Hz (t)
4.31
J = 6.6 Hz (t)
3.68
4
6
δ (ppm)
2.26
2.24
2.22
2.20
2.19
5
δ (ppm)
12
2,3
10
9
1
2.00
2.00
4.00
4.00
4.00
4.00
4.00
4.00
δ (ppm)


13C NMR I-1 (DMSO-d6)
166.46
137.09
136.44
135.35
129.02
122.89
122.54
117.19
51.47
46.78
39.52 DMSO-d6
34.98
28.44
137.09
136.44
135.35
129.02
122.89
122.54
117.19
8
1
11
12
3
2
9 y 13
Delta (ppm)
51.47
46.78
34.98
28.44
10
4
6
5
Delta (ppm)
7
Delta (ppm)

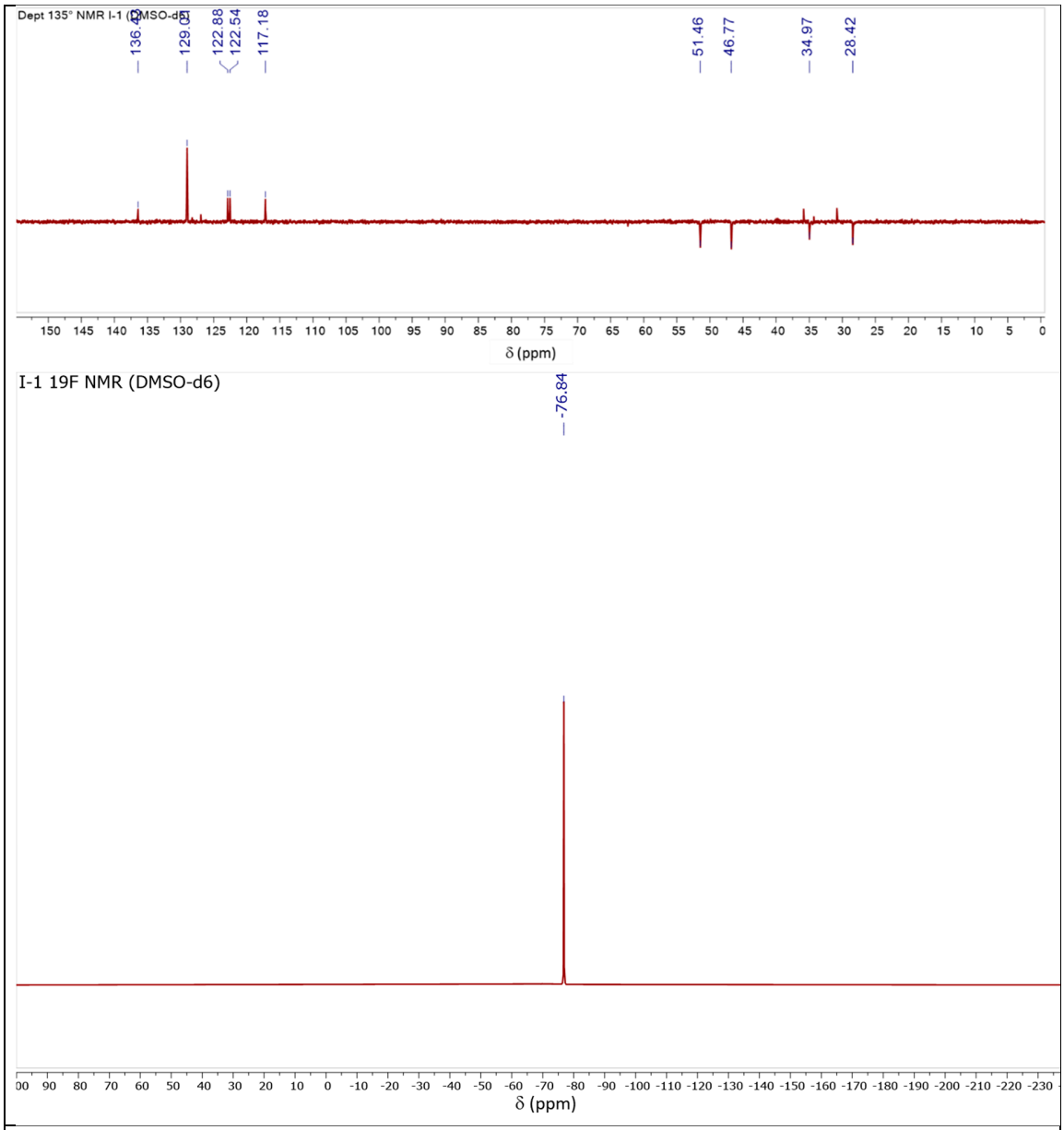
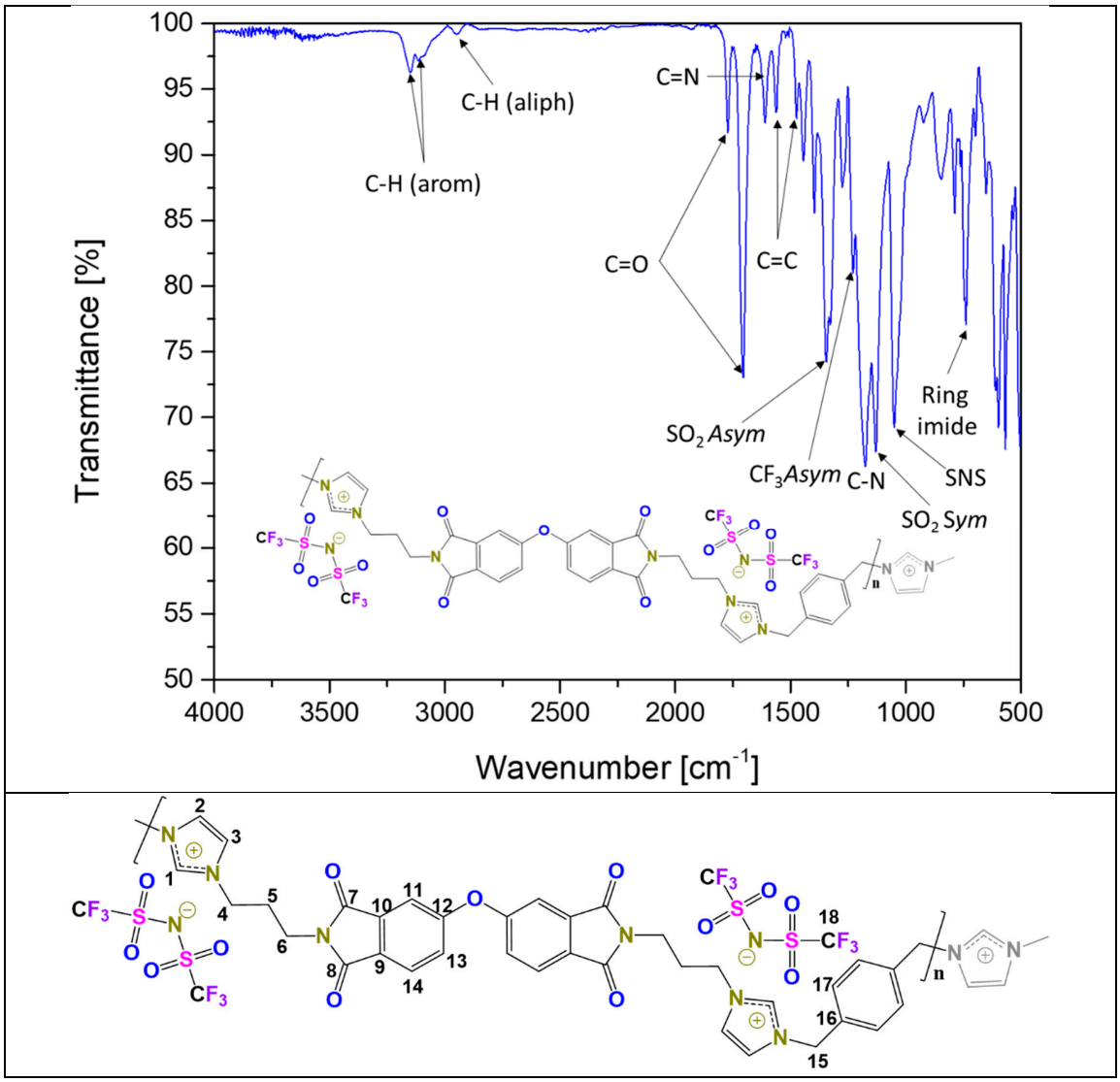
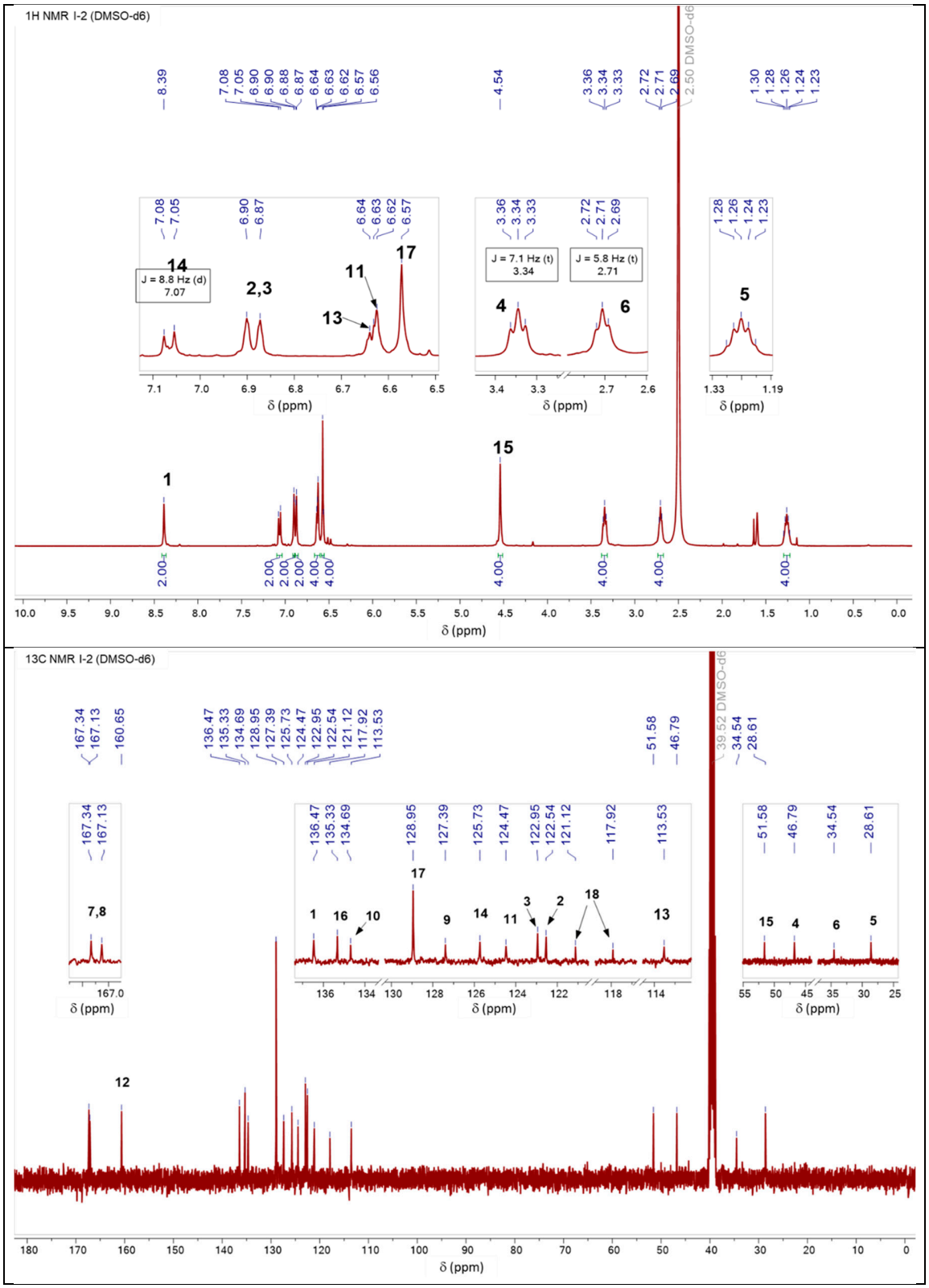


Figure S5. IR and ^{1}H, ^{13}C, Dept 135° and ^{19}F NMR spectra of ionene [PMDA-API-pXy][NTf_2].


Transmittance [%]
Wavenumber [cm-1]
C-H (arom)
C-H (aliph)
C=N
C=O
C=C
SO2 Asym
CF3 Asym
C-N
SNS
SO2 Sym
Ring imide


1H NMR I-2 (DMSO-d6)
8.39
7.08
7.05
6.90
6.90
6.88
6.87
6.64
6.63
6.62
6.57
6.56
4.54
3.36
3.34
3.33
2.72
2.71
2.69
2.50 DMSO-d6
1.30
1.28
1.26
1.24
1.23
J = 8.8 Hz (d)
7.07
J = 7.1 Hz (t)
3.34
J = 5.8 Hz (t)
2.71
δ (ppm)
13C NMR I-2 (DMSO-d6)
167.34
167.13
160.65
136.47
135.33
134.69
128.95
127.39
125.73
124.47
122.95
122.54
121.12
117.92
113.53
51.58
46.79
39.52 DMSO-d6
34.54
28.61
δ (ppm)

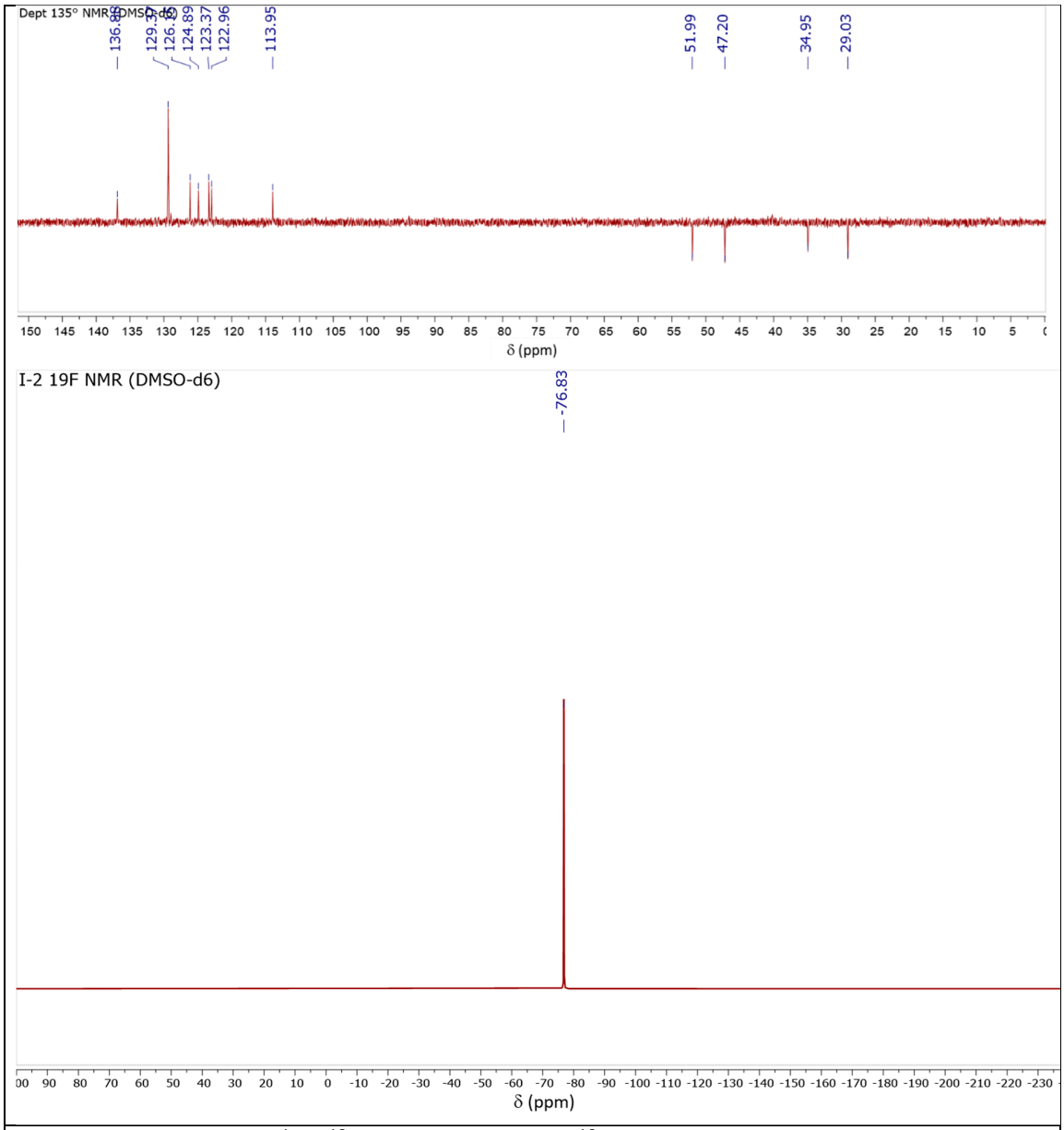
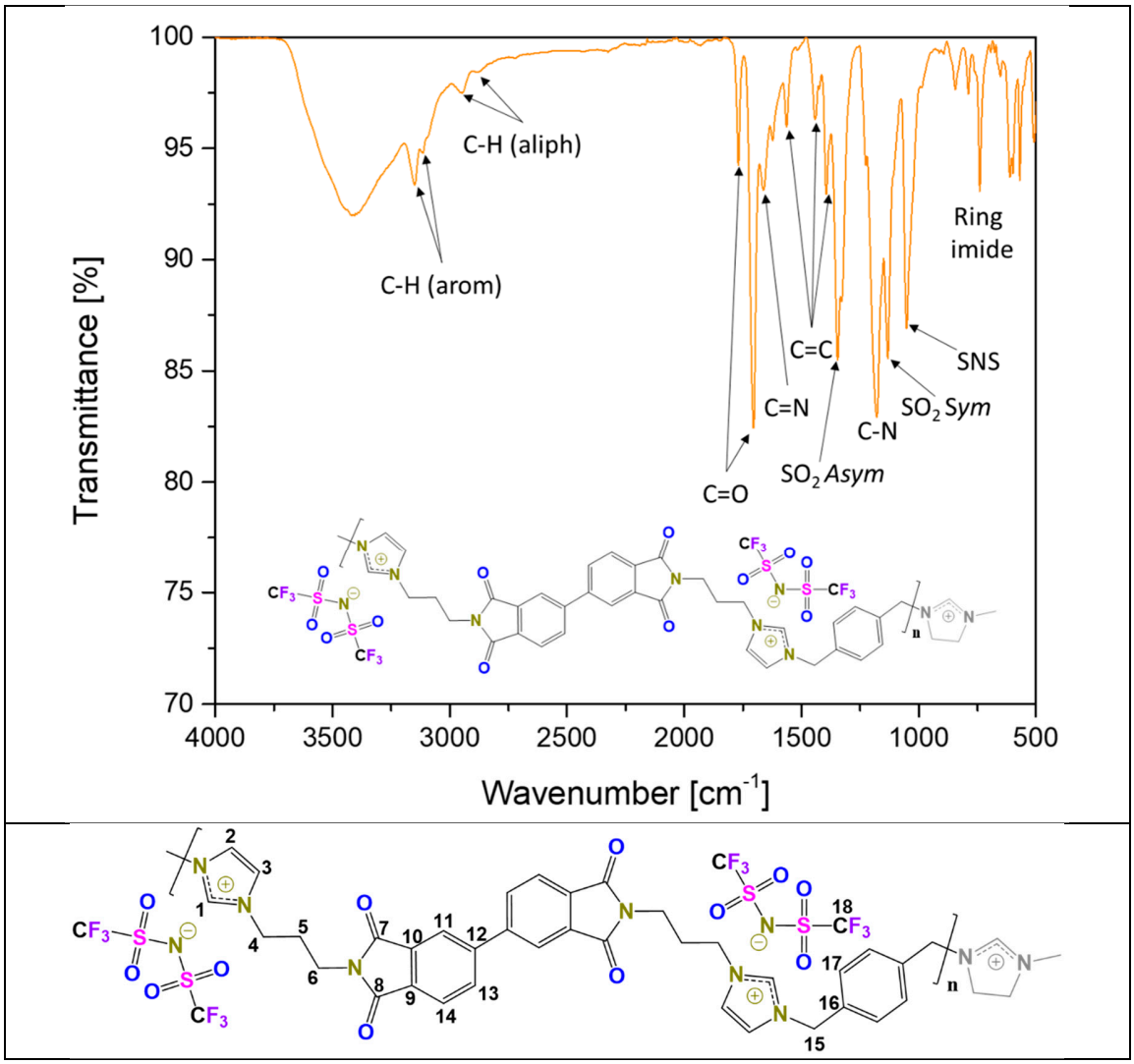
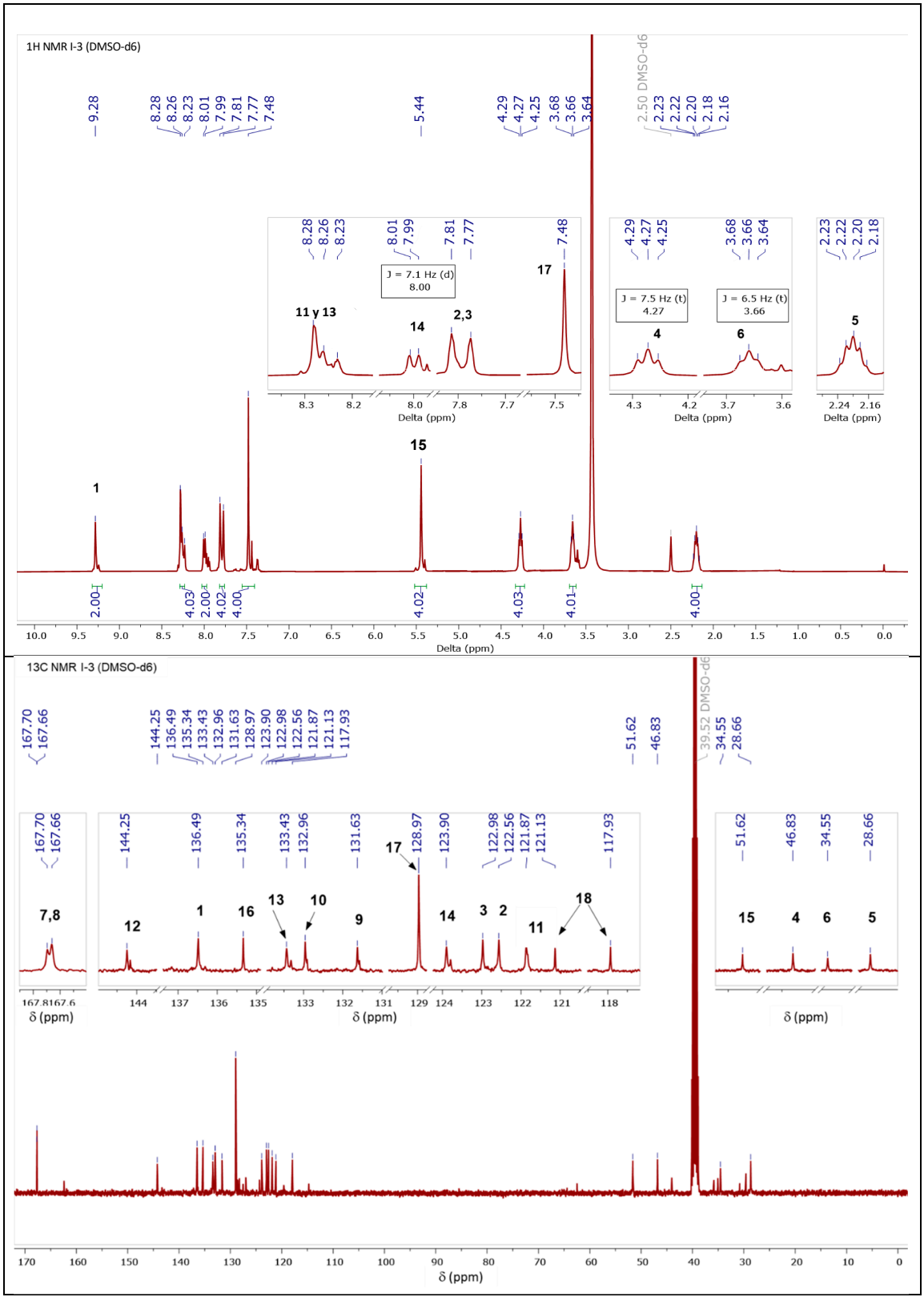


Figure S6. IR and ^{1}H, ^{13}C, Dept 135° and ^{19}F NMR spectra of ionene [ODPA-API-pXy][NTf_2].


Transmittance [%]
Wavenumber [cm-1]
C-H (aliph)
C-H (arom)
C=O
C=N
C=C
C-N
SO2 Asym
SO2 Sym
SNS
Ring imide


1H NMR I-3 (DMSO-d6)
9.28
8.28
8.26
8.23
8.01
7.99
7.81
7.77
7.48
5.44
4.29
4.27
4.25
3.68
3.66
3.64
2.50 DMSO-d6
2.23
2.22
2.20
2.18
2.16
J = 7.1 Hz (d)
8.00
J = 7.5 Hz (t)
4.27
J = 6.5 Hz (t)
3.66
11 y 13
14
2,3
17
4
6
5
15
1
2.00
4.03
2.00
4.02
4.00
4.02
4.03
4.01
4.00
Delta (ppm)
13C NMR I-3 (DMSO-d6)
167.70
167.66
144.25
136.49
135.34
133.43
132.96
131.63
128.97
123.90
122.98
122.56
121.87
121.13
117.93
51.62
46.83
39.52 DMSO-d6
34.55
28.66
7,8
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13
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9
17
14
3
2
11
18
15
4
6
5
δ (ppm)

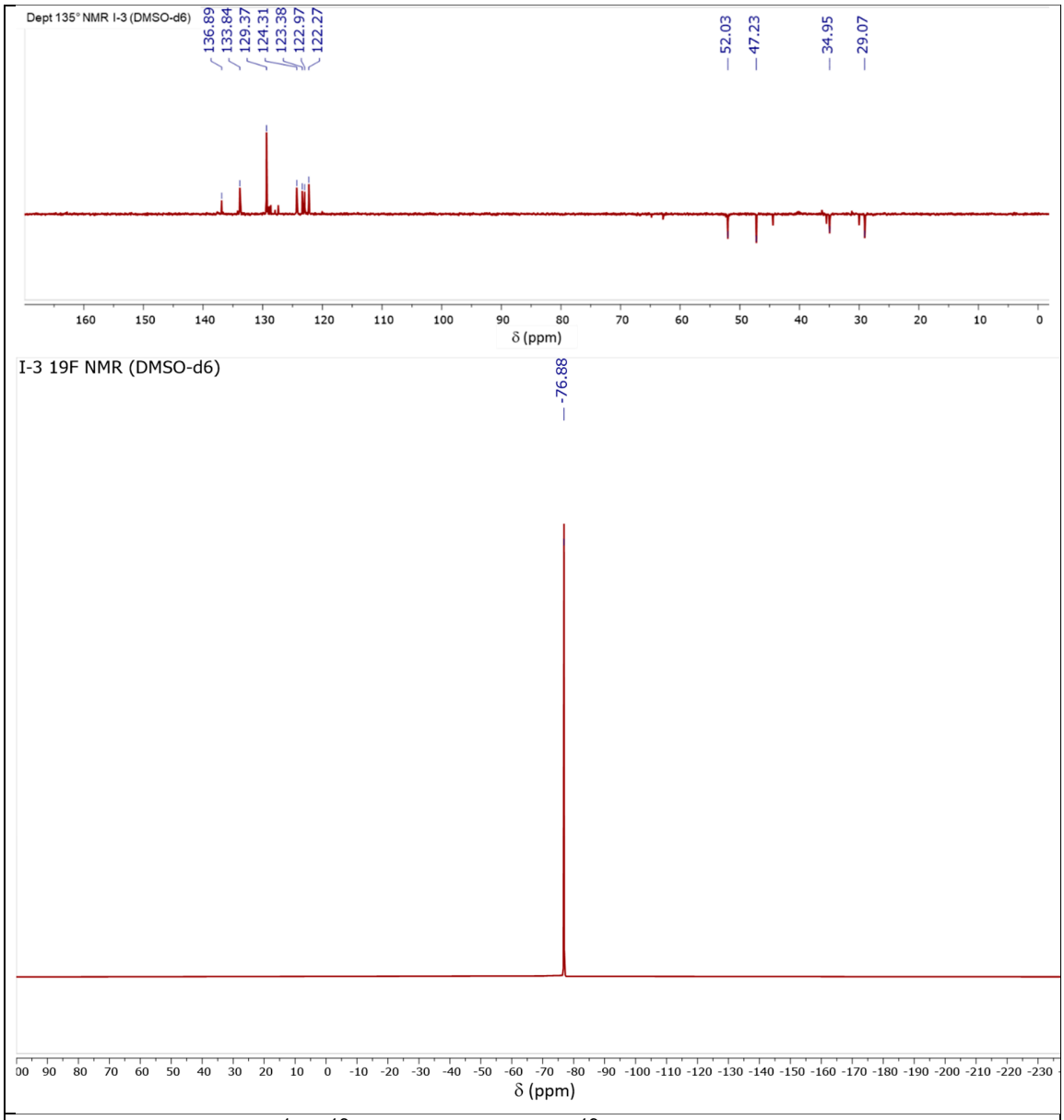
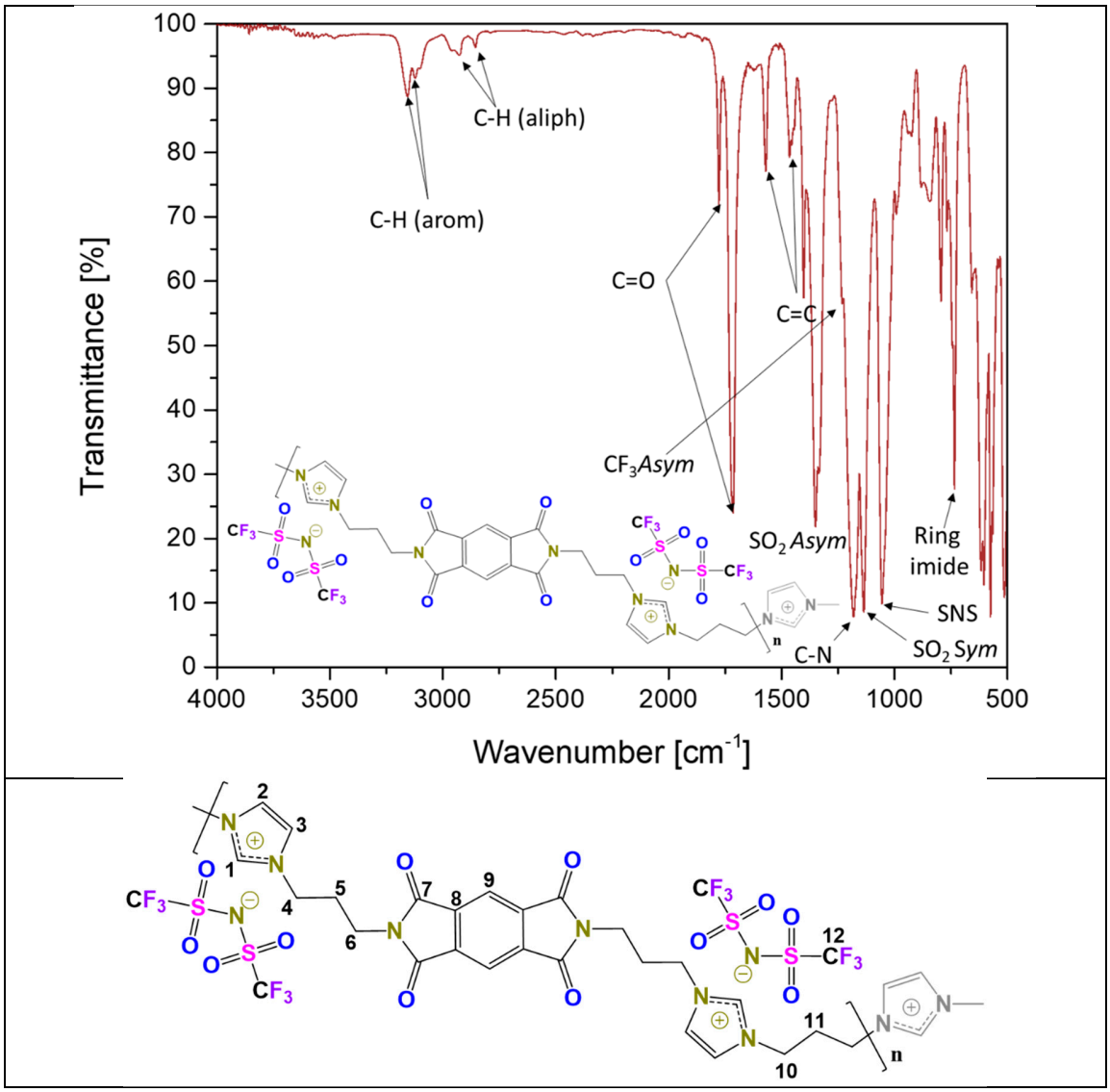
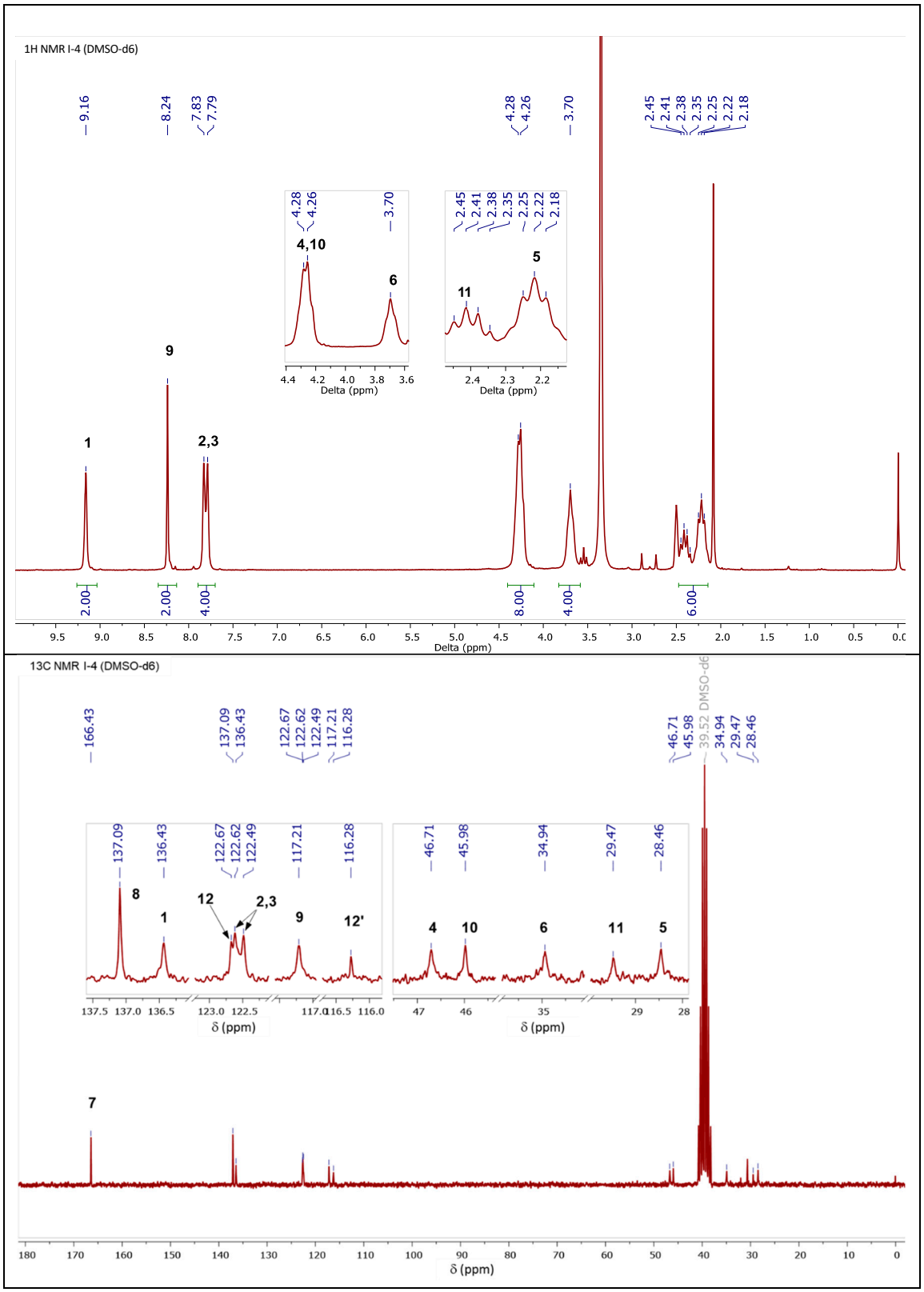


Figure S7. IR and ^{1}H, ^{13}C, Dept 135° and ^{19}F NMR spectra of ionene [BPDA-API-pXy] [NTf_2].


Transmittance [%]
Wavenumber [cm-1]
C-H (arom)
C-H (aliph)
C=O
C=C
CF3Asym
SO2 Asym
Ring imide
SNS
C-N
SO2 Sym


1H NMR I-4 (DMSO-d6)
9.16
8.24
7.83
7.79
4.28
4.26
3.70
2.45
2.41
2.38
2.35
2.25
2.22
2.18
4,10
6
5
11
9
1
2,3
2.00
2.00
4.00
8.00
4.00
6.00
Delta (ppm)
13C NMR I-4 (DMSO-d6)
166.43
137.09
136.43
122.67
122.62
122.49
117.21
116.28
46.71
45.98
39.52 DMSO-d6
34.94
29.47
28.46
8
1
12
2,3
9
12'
4
10
6
11
5
7
δ (ppm)

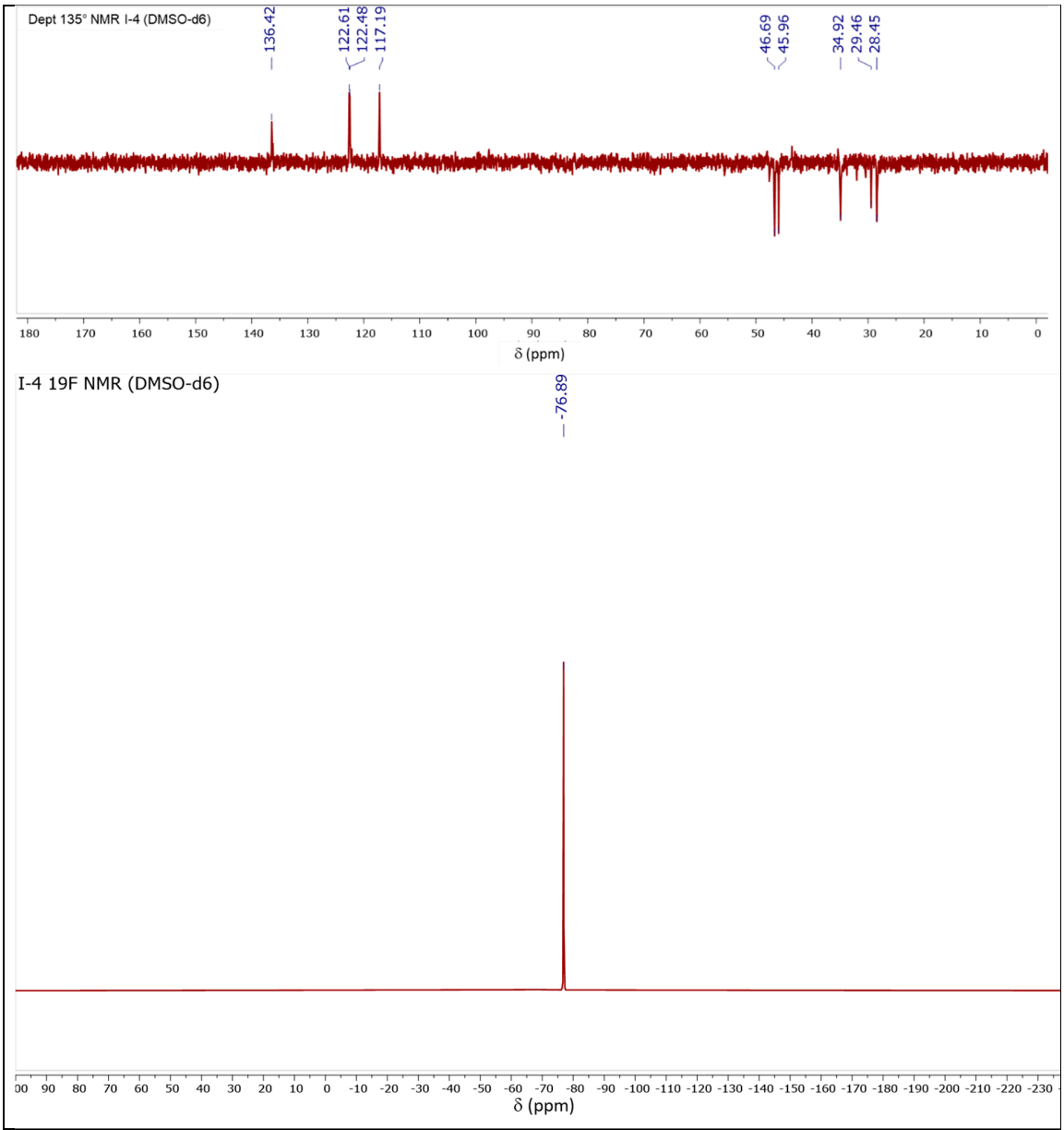
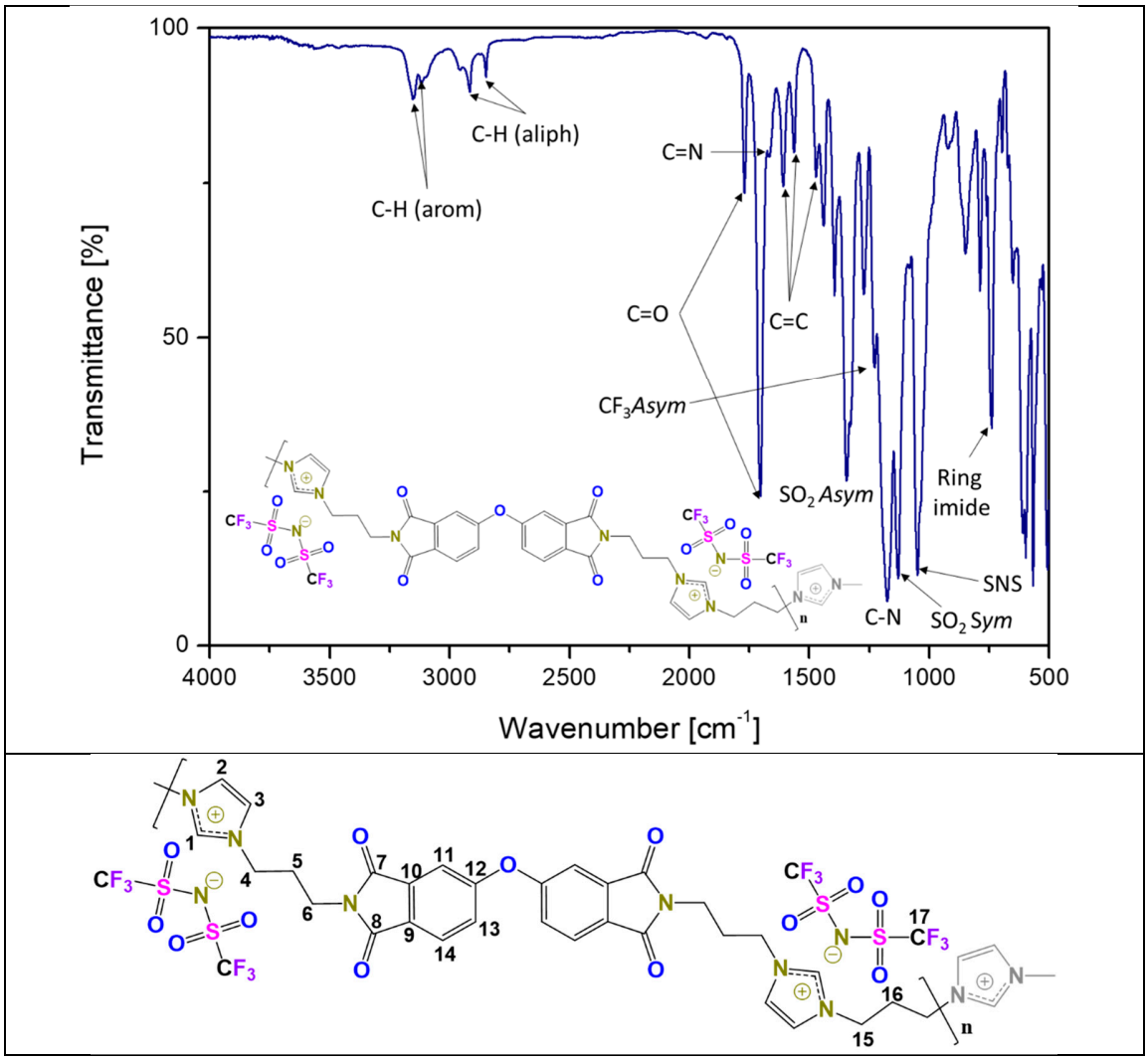
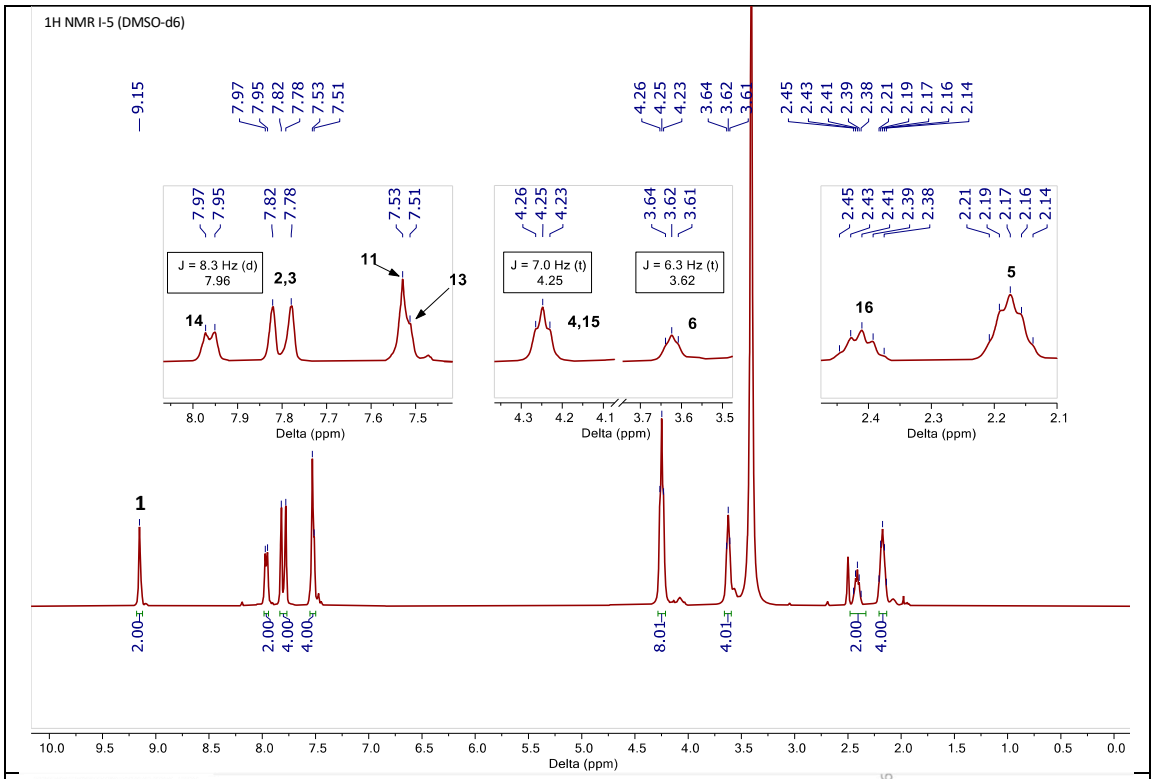
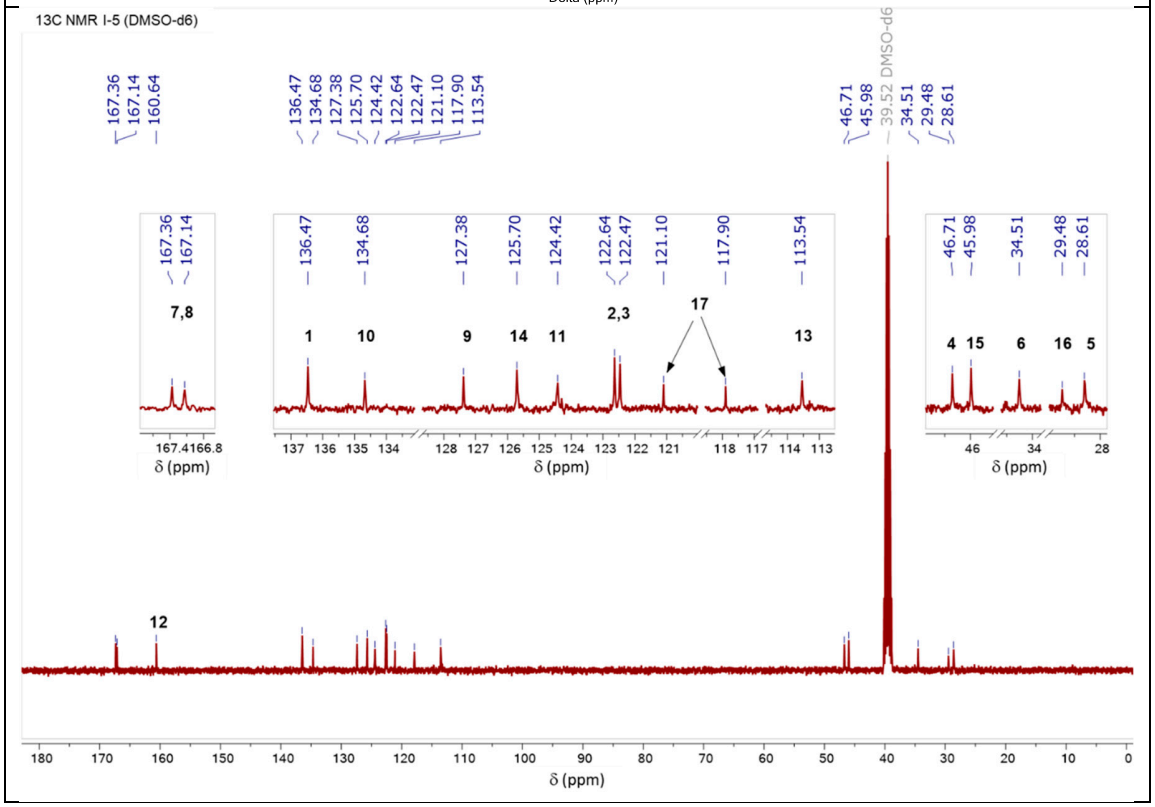


Figure S8. IR and ^{1}H, ^{13}C, Dept 135° and ^{19}F NMR spectra of ionene [PMDA-API-C_3] [NTf_2].


Transmittance [%]
100
50
0
4000
3500
3000
2500
2000
1500
1000
500
Wavenumber [cm-1]
C-H (arom)
C-H (aliph)
C=N
C=O
C=C
CF3Asym
SO2 Asym
Ring imide
SNS
C-N
SO2 Sym


1H NMR I-5 (DMSO-d6)
9.15
7.97
7.95
7.82
7.78
7.53
7.51
4.26
4.25
4.23
3.64
3.62
3.61
2.45
2.43
2.41
2.39
2.38
2.21
2.19
2.17
2.16
2.14
J = 8.3 Hz (d)
7.96
J = 7.0 Hz (t)
4.25
J = 6.3 Hz (t)
3.62
1
2,3
4,15
5
6
11
13
14
16
2.00
2.00
4.00
4.00
8.01
4.01
2.00
4.00
Delta (ppm)


13C NMR I-5 (DMSO-d6)
167.36
167.14
160.64
136.47
134.68
127.38
125.70
124.42
122.64
122.47
121.10
117.90
113.54
46.71
45.98
39.52 DMSO-d6
34.51
29.48
28.61
7,8
1
10
9
14
11
2,3
17
13
4
15
6
16
5
12
δ (ppm)

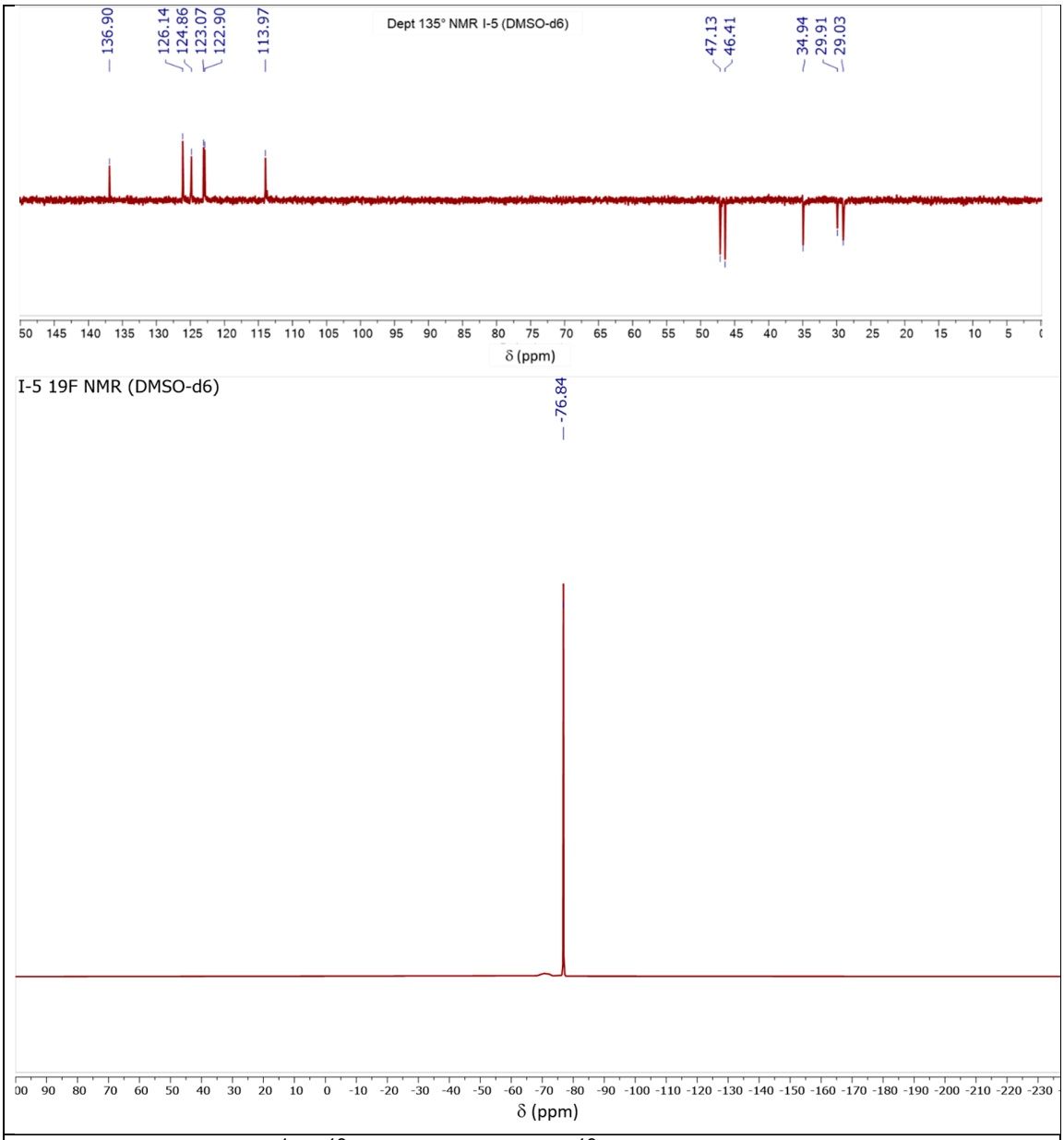
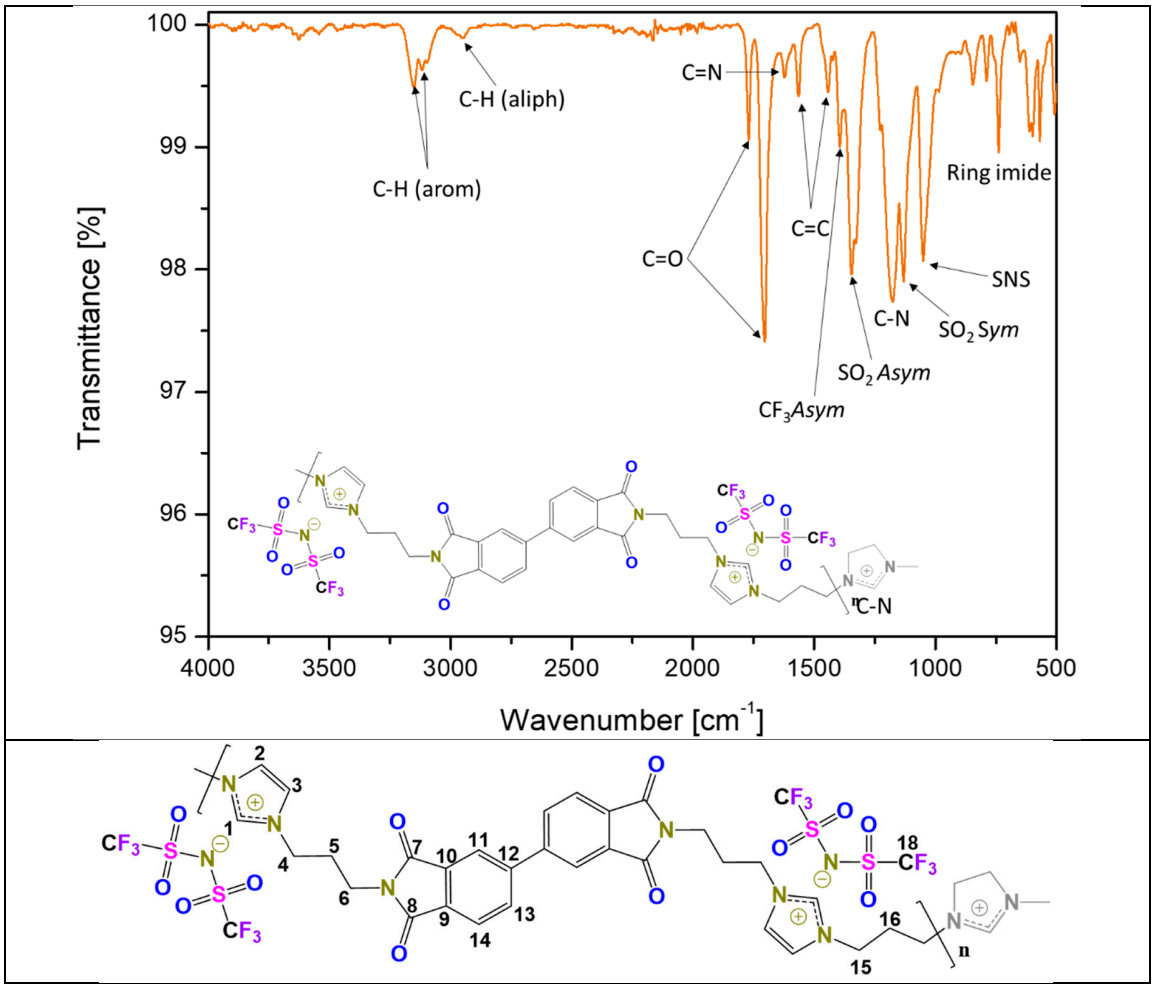
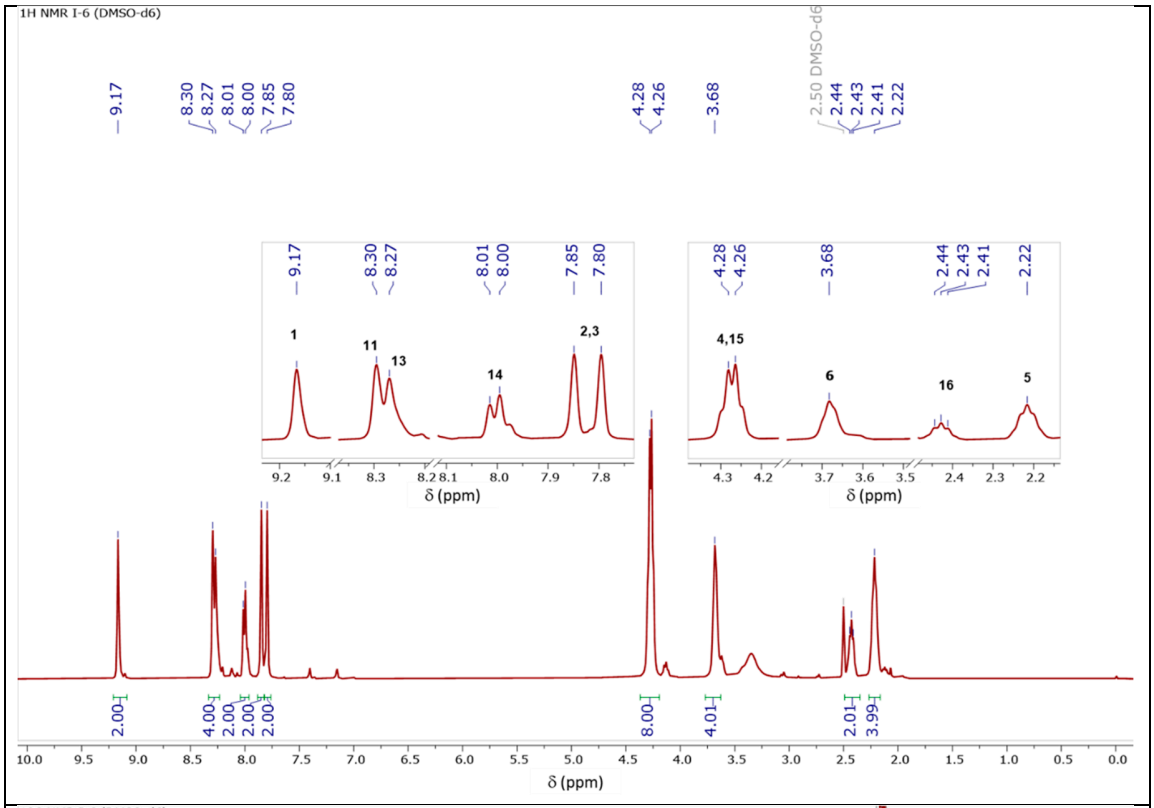
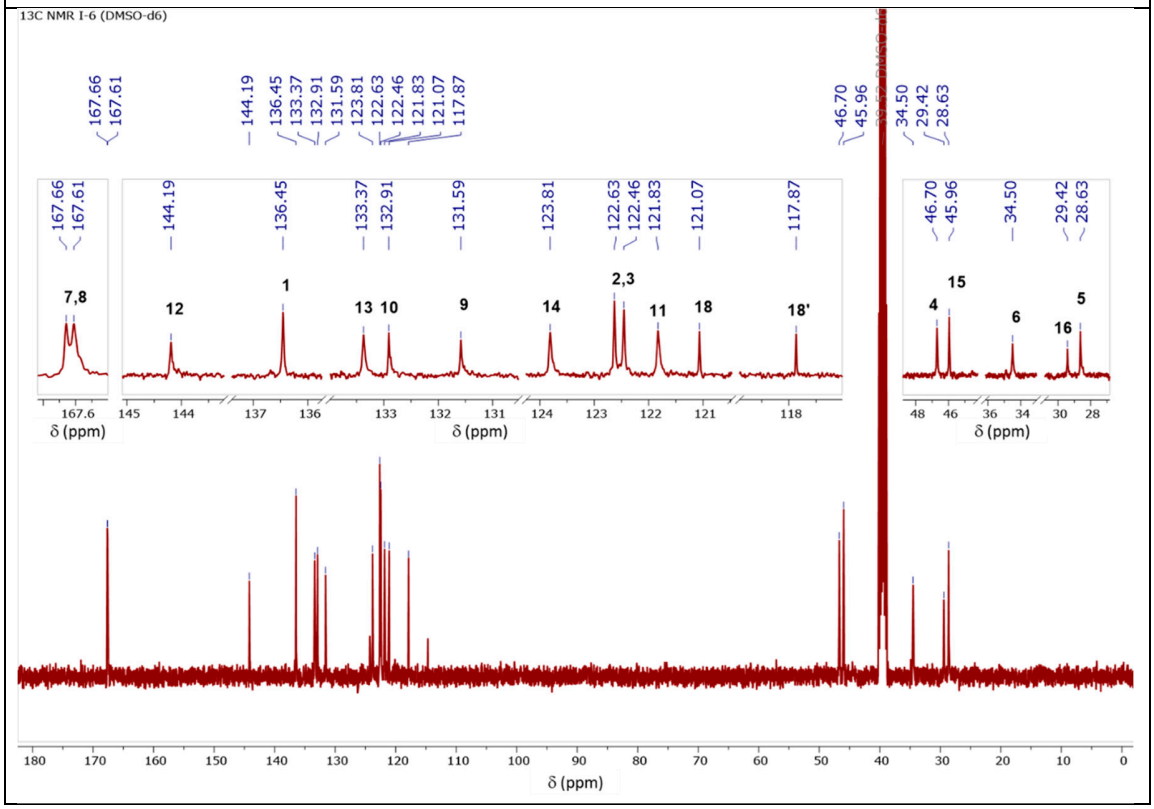


Figure S9. IR and ^{1}H, ^{13}C, Dept 135° and ^{19}F NMR spectra of ionene [ODPA-API-C_3] [NTf_2].


Transmittance [%]
Wavenumber [cm-1]
C-H (arom)
C-H (aliph)
C=N
C=O
C=C
Ring imide
SNS
C-N
SO2 Sym
SO2 Asym
CF3Asym
nC-N


1H NMR I-6 (DMSO-d6)
9.17
8.30
8.27
8.01
8.00
7.85
7.80
4.28
4.26
3.68
2.50 DMSO-d6
2.44
2.43
2.41
2.22
1
11
13
14
2,3
4,15
6
16
5
δ (ppm)


13C NMR I-6 (DMSO-d6)
167.66
167.61
144.19
136.45
133.37
132.91
131.59
123.81
122.63
122.46
121.83
121.07
117.87
46.70
45.96
39.52 DMSO-d6
34.50
29.42
28.63
7,8
12
1
13
10
9
14
2,3
11
18
18'
15
4
6
16
5
δ (ppm)

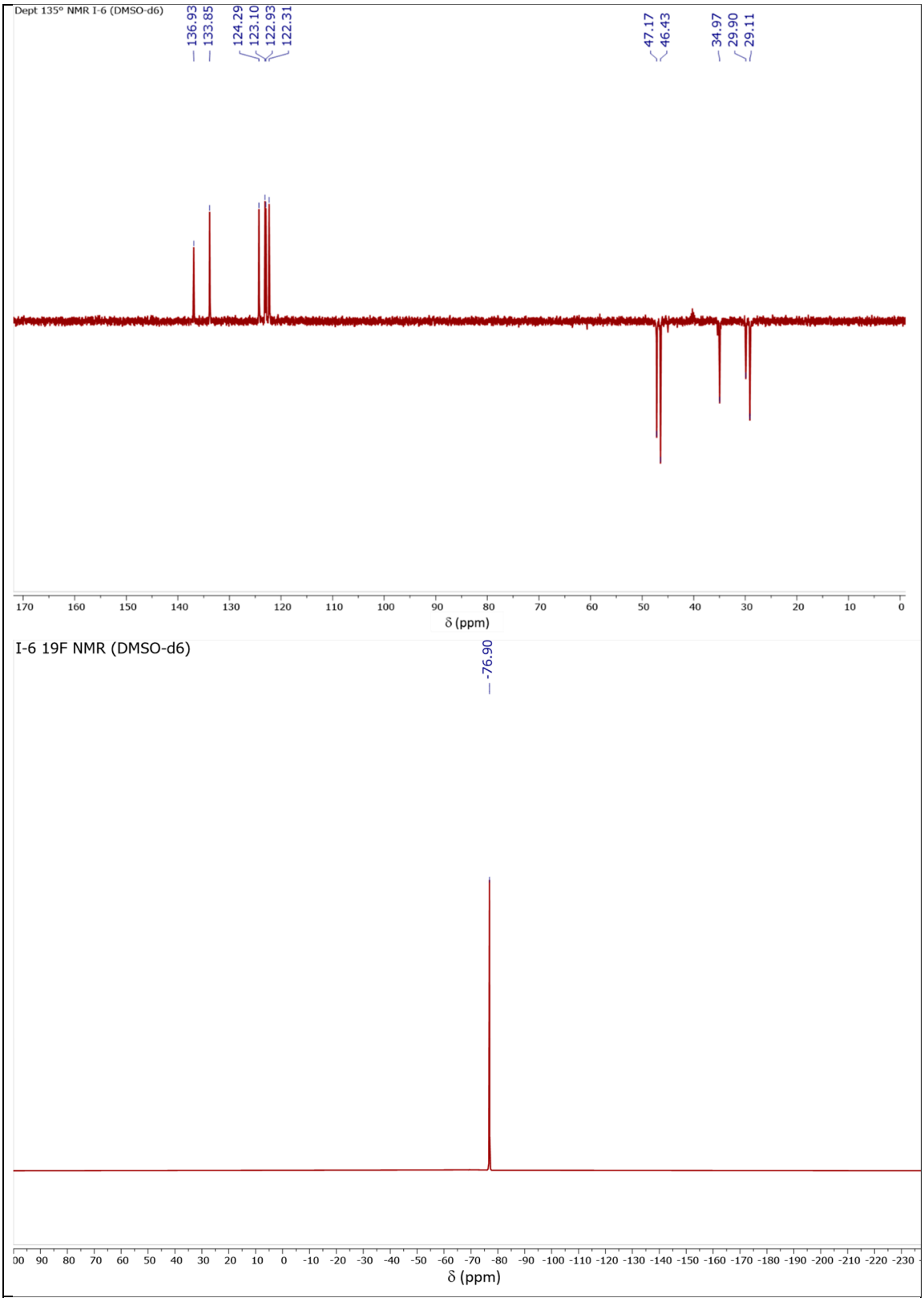
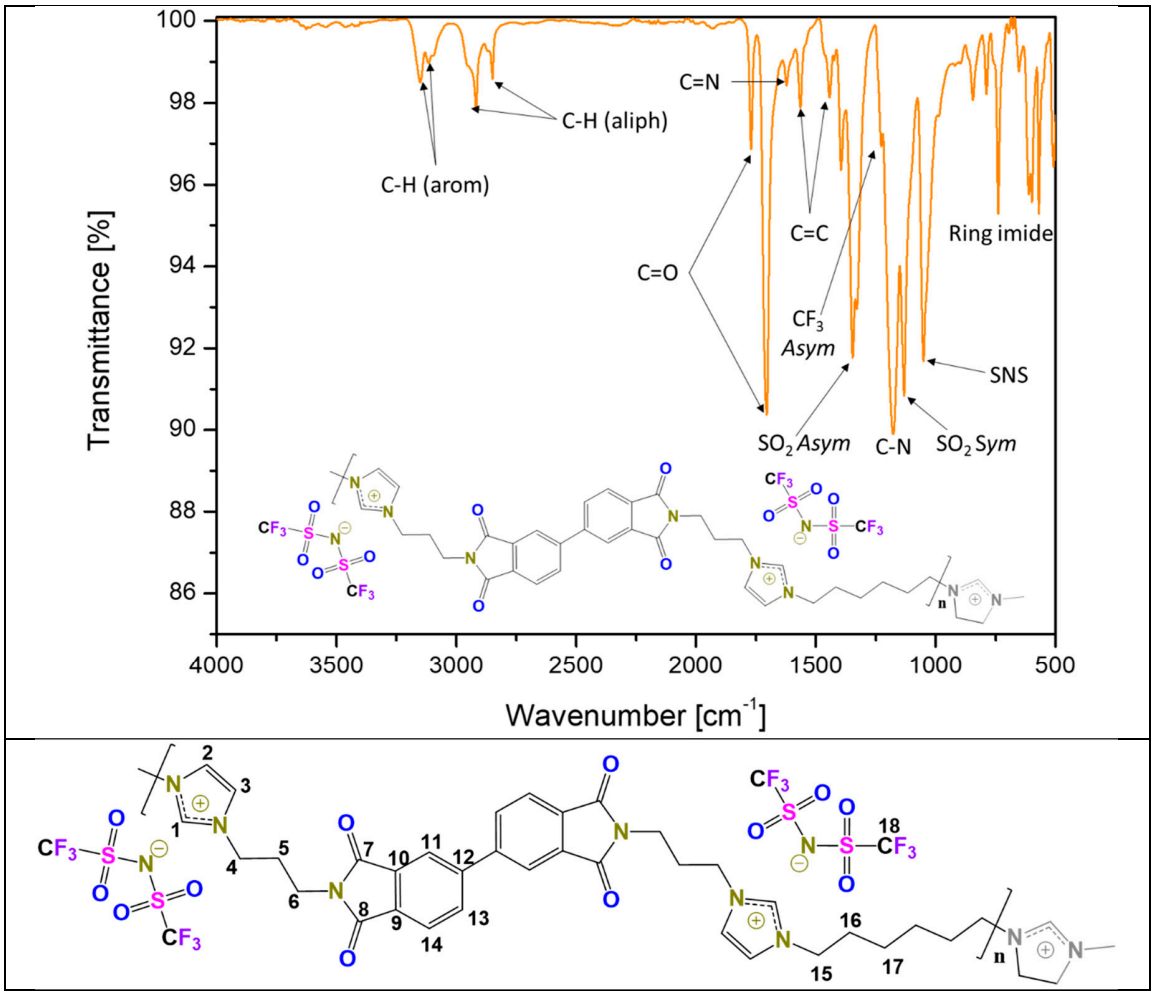
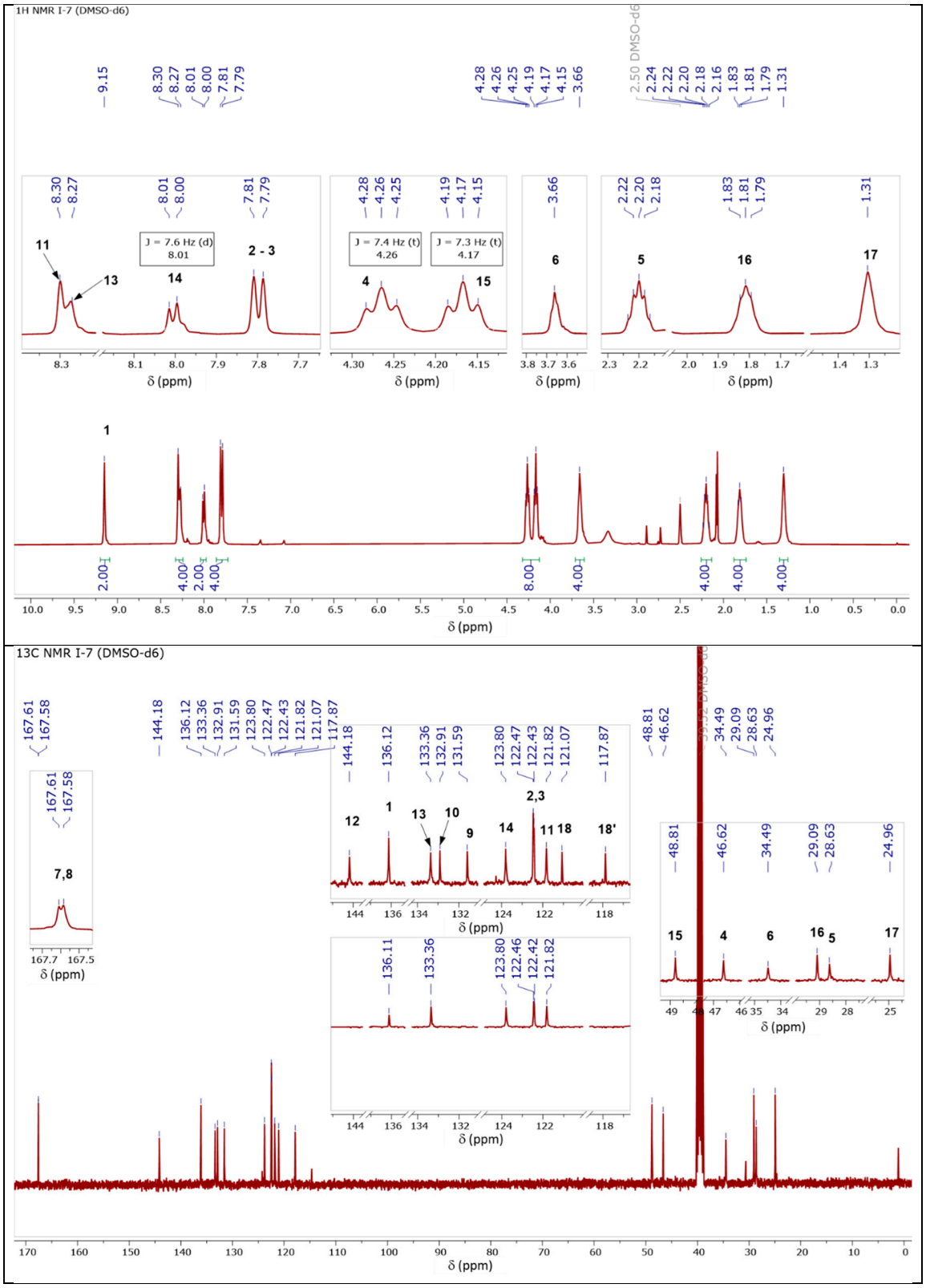


Figure S10. IR and ^{1}H, ^{13}C, Dept 135° and ^{19}F NMR spectra of ionene [BPDA-API-C_3] [NTf_2].


Transmittance [%]
Wavenumber [cm-1]
C-H (arom)
C-H (aliph)
C=N
C=C
C=O
CF3 Asym
SO2 Asym
C-N
SO2 Sym
SNS
Ring imide


1H NMR I-7 (DMSO-d6)
9.15
8.30
8.27
8.01
8.00
7.81
7.79
4.28
4.26
4.25
4.19
4.17
4.15
3.66
2.50 DMSO-d6
2.24
2.22
2.20
2.18
2.16
1.83
1.81
1.79
1.31
J = 7.6 Hz (d)
8.01
J = 7.4 Hz (t)
4.26
J = 7.3 Hz (t)
4.17
11
13
14
2 - 3
4
15
6
5
16
17
1
2.00
4.00
2.00
4.00
8.00
4.00
4.00
4.00
4.00
δ (ppm)
13C NMR I-7 (DMSO-d6)
167.61
167.58
144.18
136.12
133.36
132.91
131.59
123.80
122.47
122.43
121.82
121.07
117.87
48.81
46.62
39.52 DMSO-d6
34.49
29.09
28.63
24.96
7,8
12
1
13
10
9
14
2,3
11
18
18'
15
4
6
16
5
17
δ (ppm)

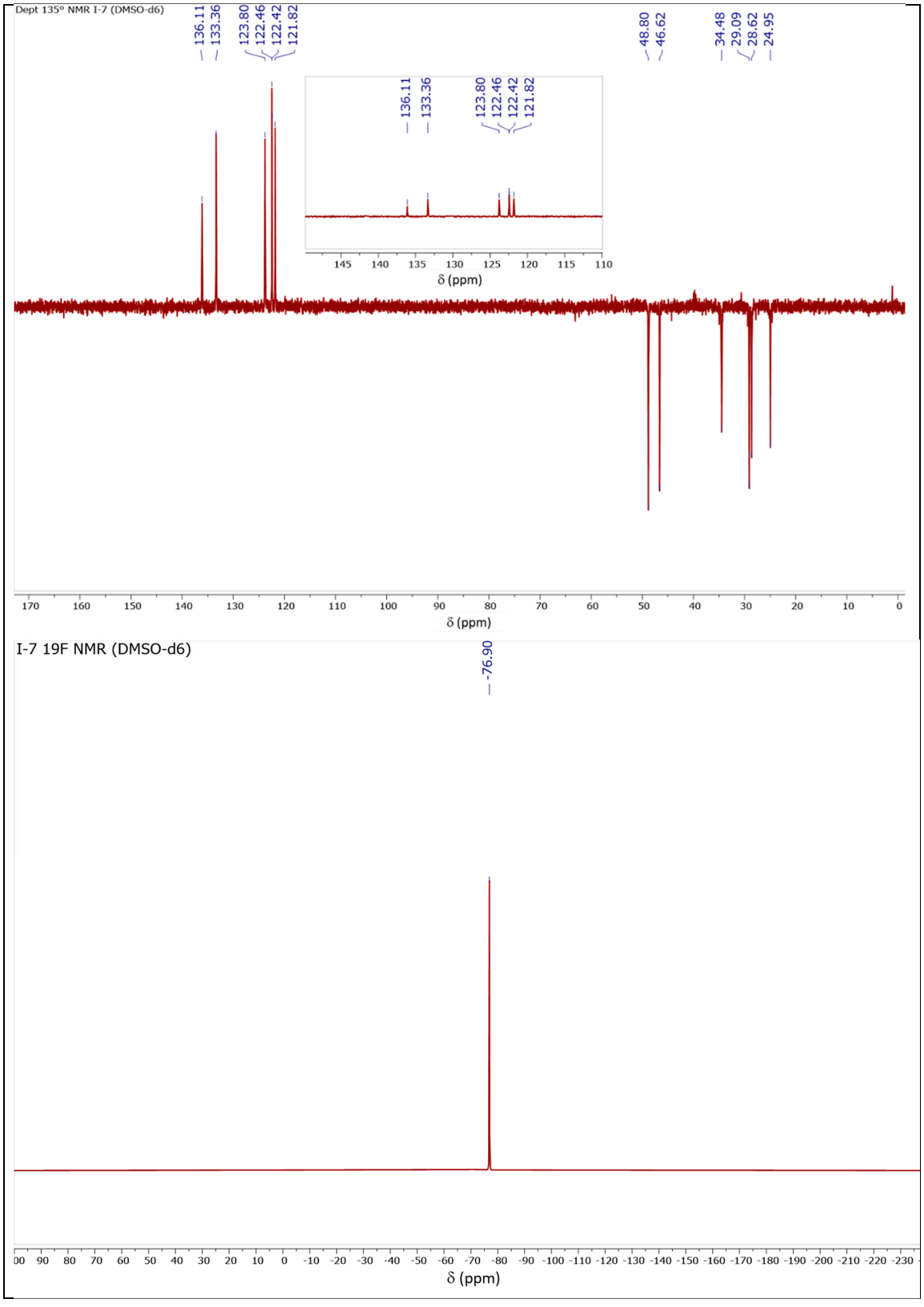
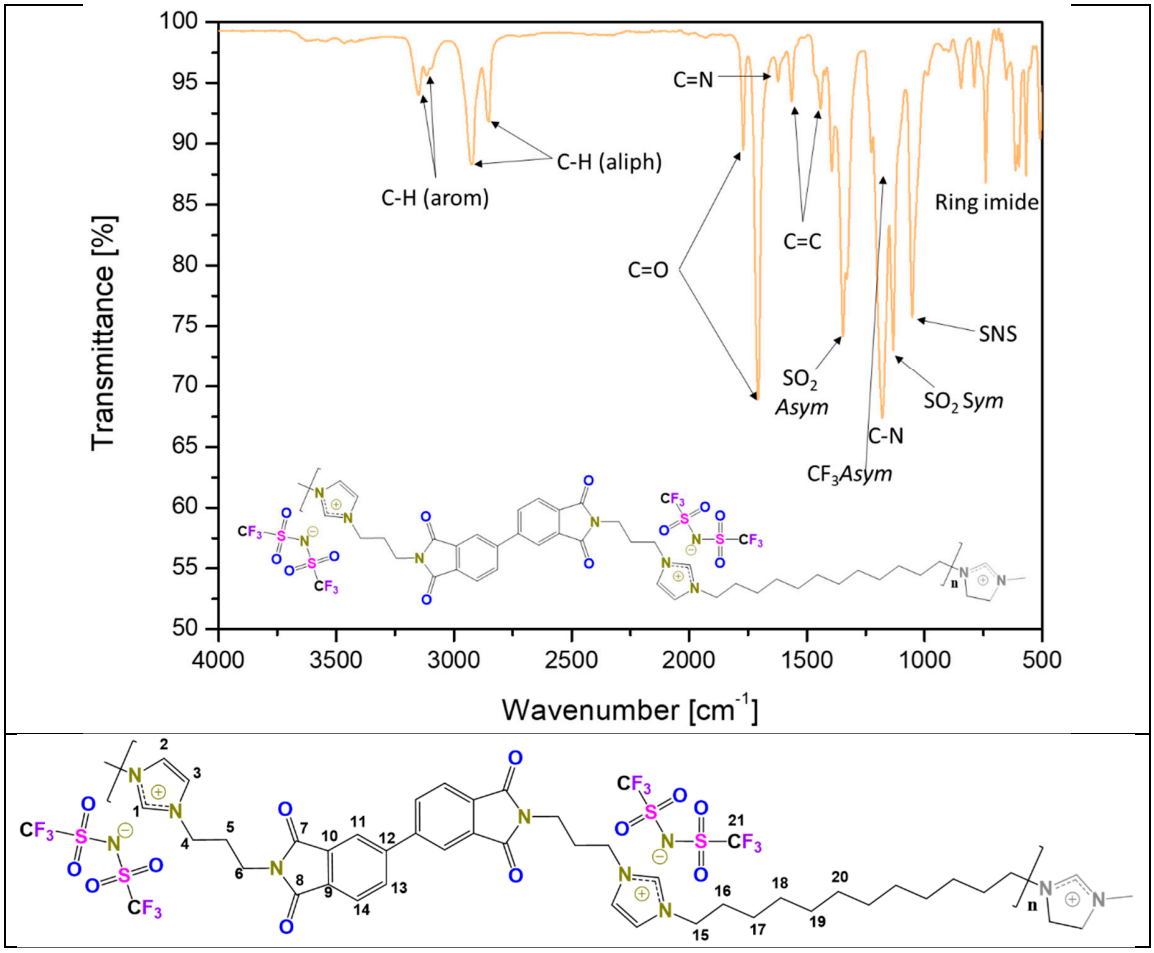
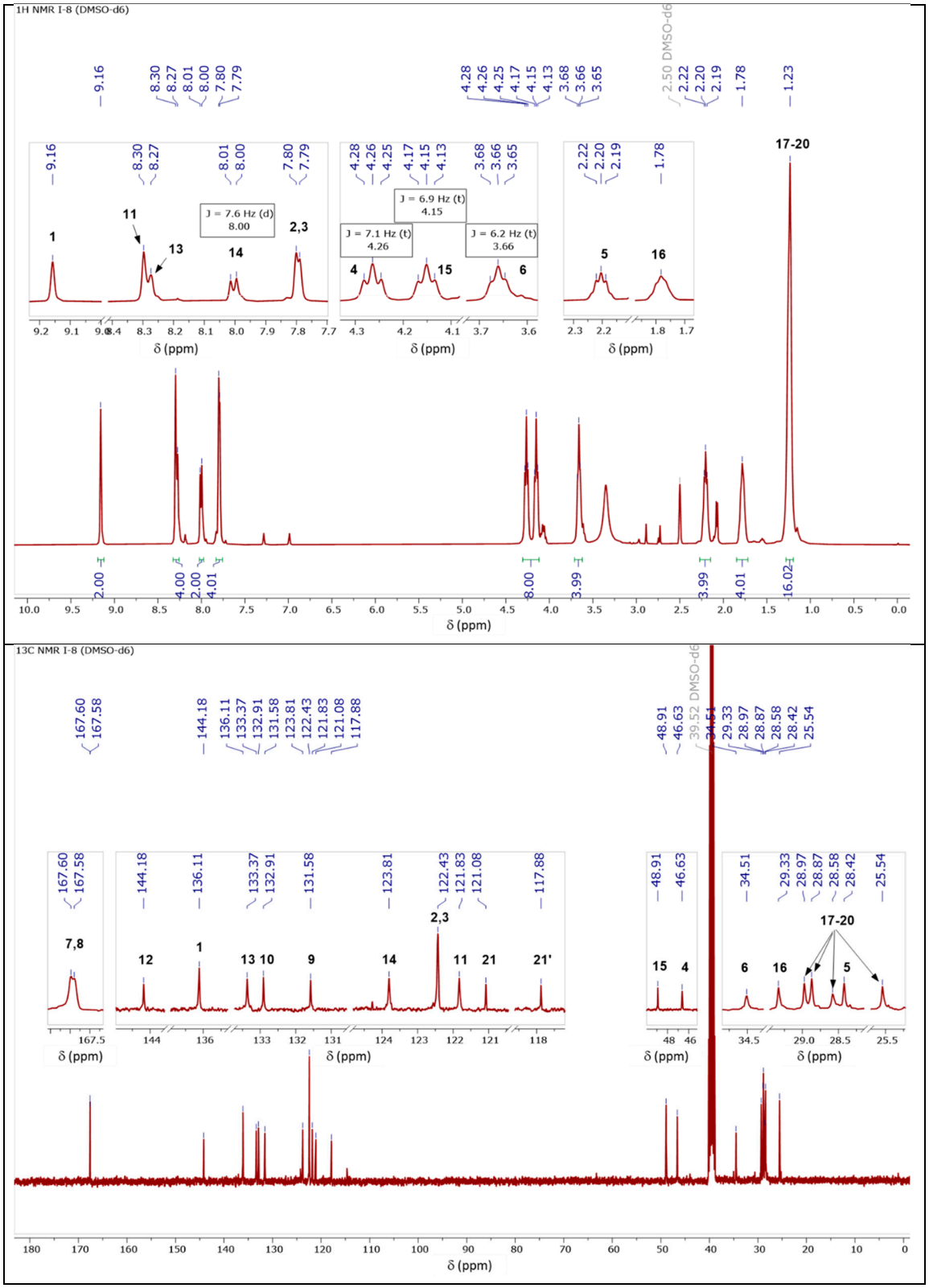

Dept 135° NMR I-7 (DMSO-d6)
136.11
133.36
123.80
122.46
122.42
121.82
48.80
46.62
34.48
29.09
28.62
24.95
δ (ppm)
I-7 19F NMR (DMSO-d6)
-76.90
δ (ppm)

Figure S11. IR and ^{1}H, ^{13}C, Dept 135° and ^{19}F NMR spectra of ionene [BPDA-API-C_6] [NTf_2].




1H NMR I-8 (DMSO-d6)
9.16
8.30
8.27
8.01
8.00
7.80
7.79
4.28
4.26
4.25
4.17
4.15
4.13
3.68
3.66
3.65
2.50 DMSO-d6
2.22
2.20
2.19
1.78
1.23
17-20
J = 7.6 Hz (d)
8.00
J = 6.9 Hz (t)
4.15
J = 7.1 Hz (t)
4.26
J = 6.2 Hz (t)
3.66
1
11
13
14
2,3
4
15
6
5
16
δ (ppm)
2.00
4.00
2.00
4.01
8.00
3.99
3.99
4.01
16.02
13C NMR I-8 (DMSO-d6)
167.60
167.58
144.18
136.11
133.37
132.91
131.58
123.81
122.43
121.83
121.08
117.88
48.91
46.63
39.52 DMSO-d6
34.51
29.33
28.97
28.87
28.58
28.42
25.54
7,8
12
1
13
10
9
14
2,3
11
21
21'
15
4
6
16
17-20
5
δ (ppm)

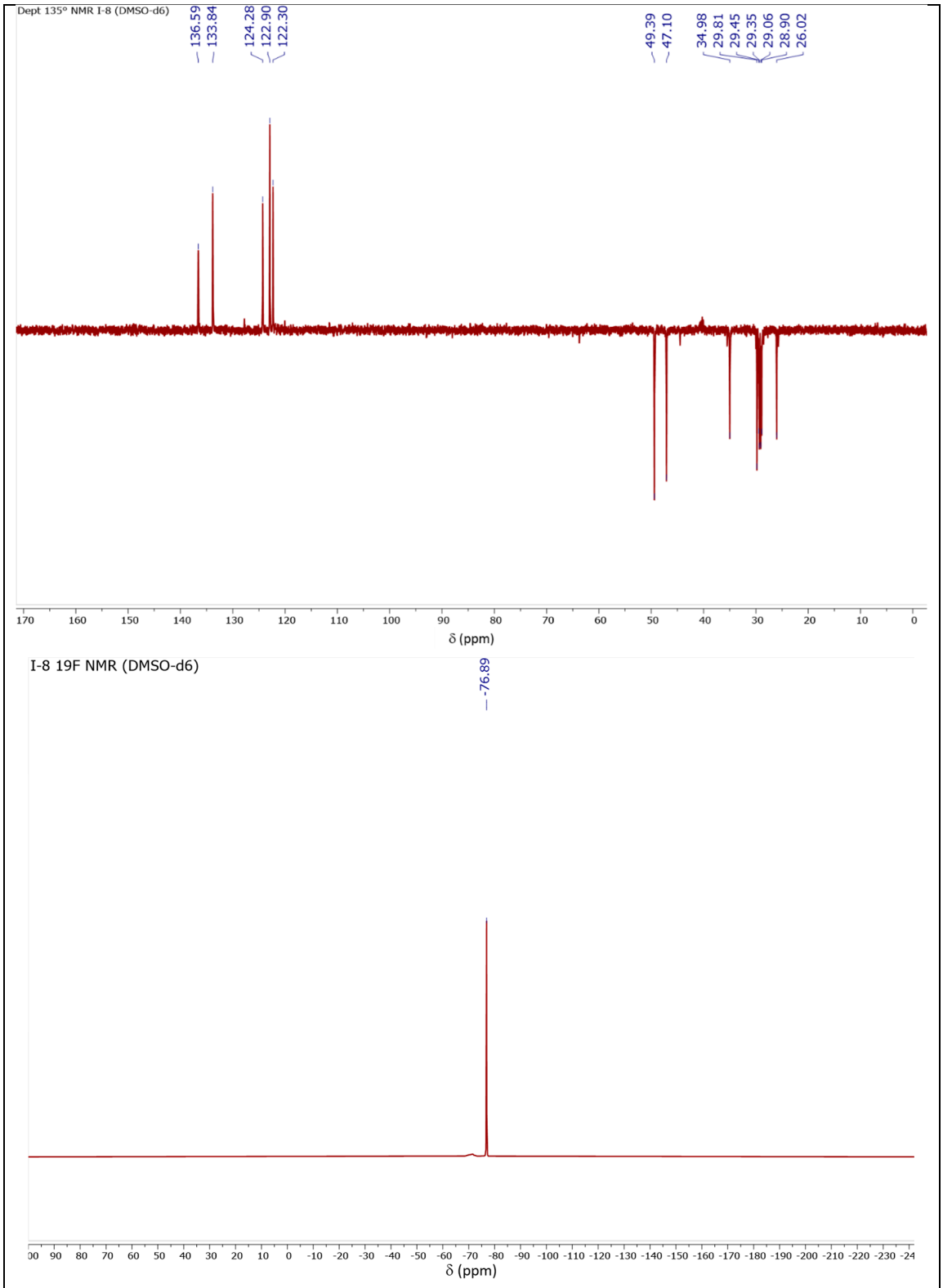


Figure S12. IR and ^{1}H, ^{13}C, Dept 135° and ^{19}F NMR spectra of ionene [BPDA-API-C_{12}] [NTf_2].

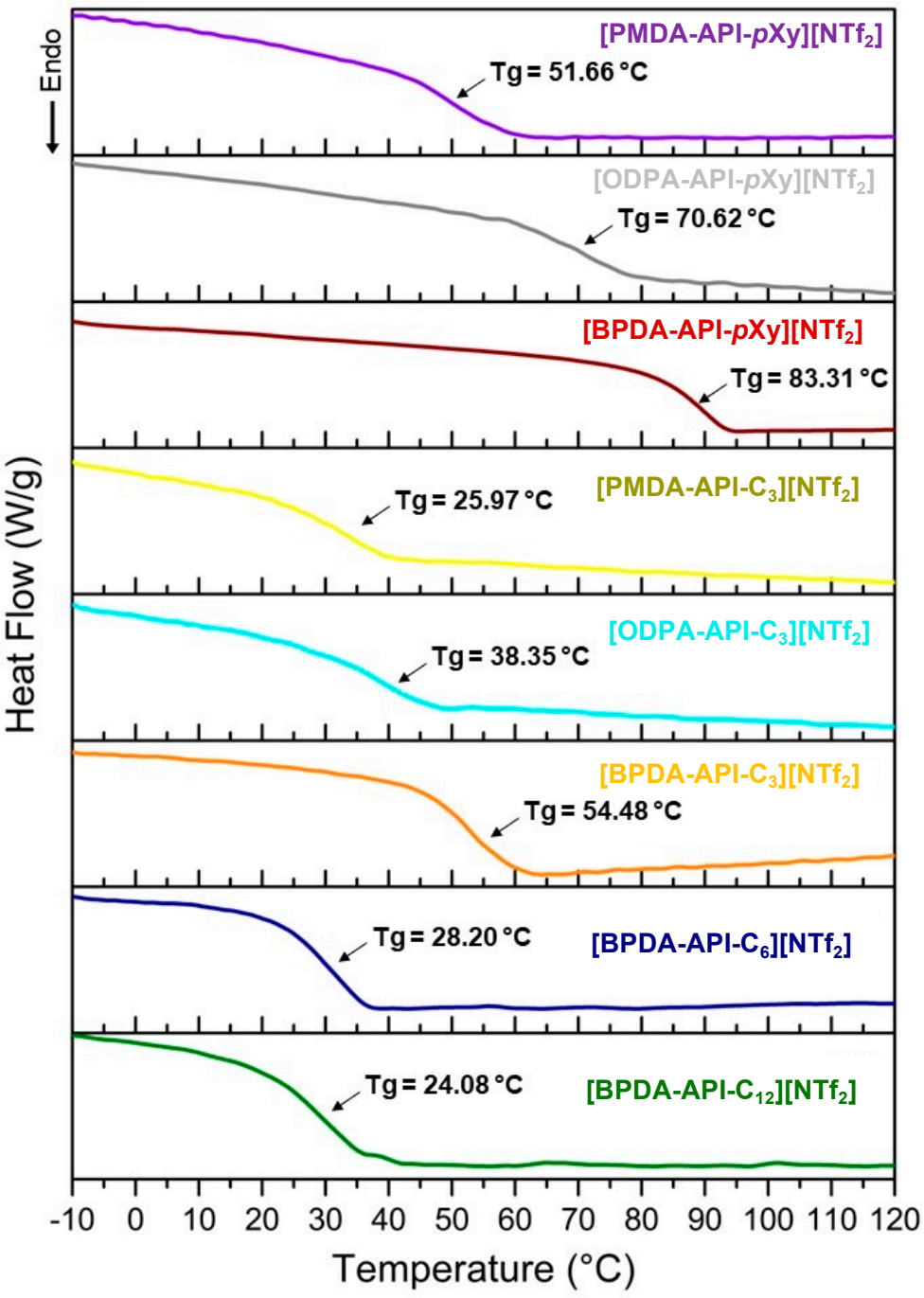
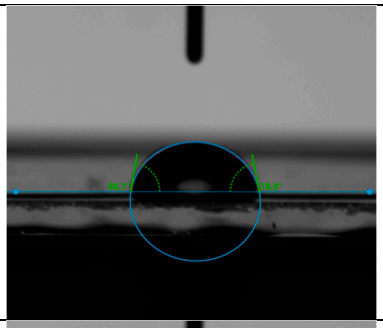
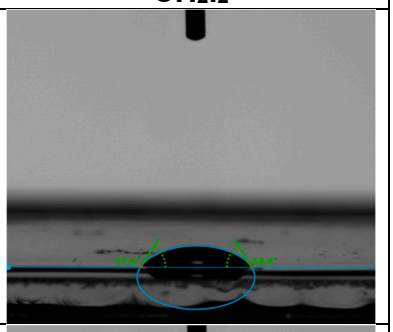
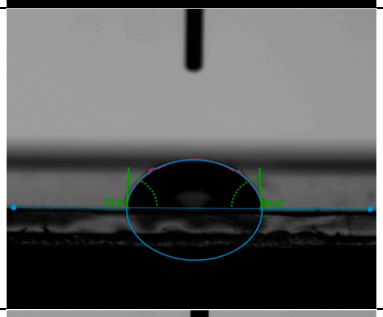
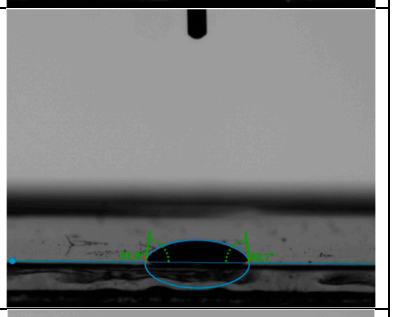
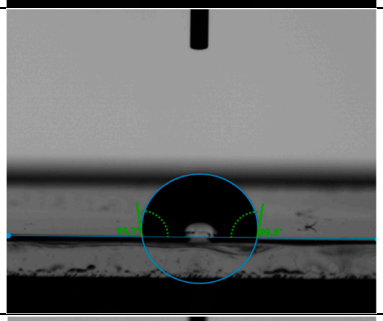
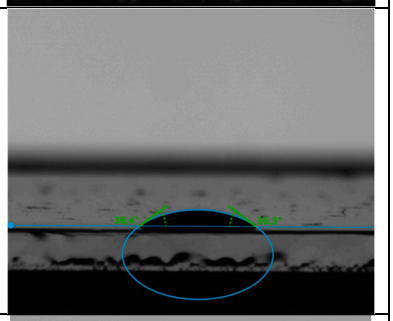
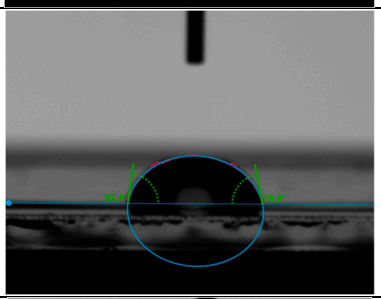
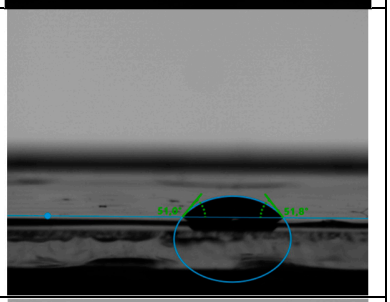
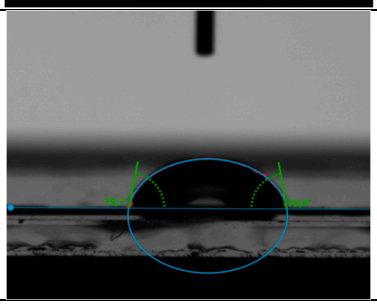
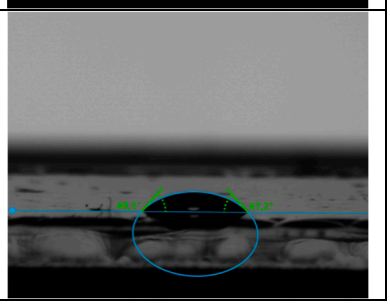


Figure S13. Heating curves of the DSC assays of the synthesized ionenes.

	Water	CH_2I_2
[PMDA-API-*p*Xy][NTf_2]		
[ODPA-API-*p*Xy][NTf_2]		
[BPDA-API-*p*Xy] [NTf_2]		
[PMDA-API-C_3] [NTf_2]		
[ODPA-API-C_3] [NTf_2]		

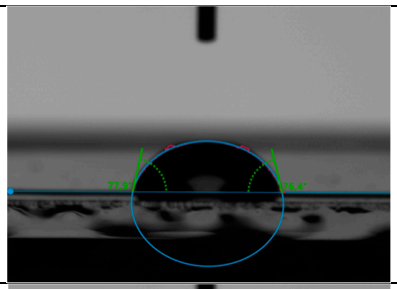
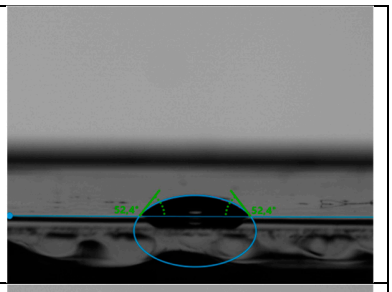
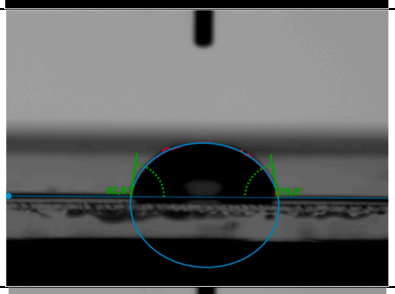
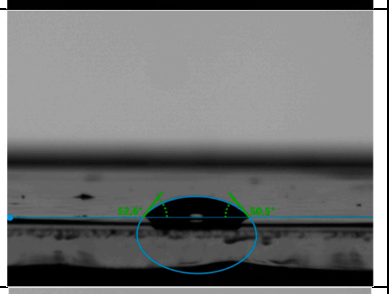
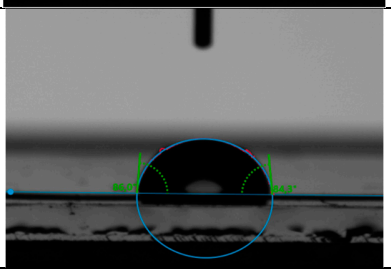
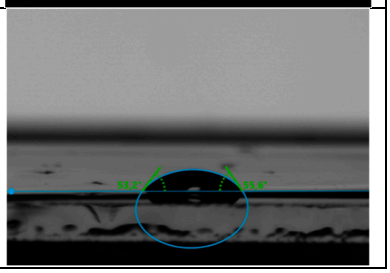
[BPDA-API-C_3] [NTf_2]		
[BPDA-API-C_6] [NTf_2]		
[BPDA-API-C_{12}] [NTf_2]		

Figure S14. Some contact angle measurements for the synthesized ionenes.